

UNITED STATES PATENT AND TRADEMARK OFFICE

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BEFORE THE PATENT TRIAL AND APPEAL BOARD

PREFERRED PROPPANTS, LLC, PREFERRED PIPELINE, LLC, PREFERRED  
RESIN SALES, LLC, PREFERRED RESIN OF GENOA, LLC, AND  
PREFERRED SANDS OF WISCONSIN, LLC,  
PETITIONERS

V.

TRICAN WELL SERVICE LTD.,  
PATENT OWNER

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U.S. PATENT NO. 8,236,738 TO ZHANG

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*INTER PARTES* REVIEW IPR2017-02099, IPR2017-02100

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DECLARATION OF GARY R. WOOLEY IN SUPPORT OF THE  
PETITIONS FOR *INTER PARTES* REVIEW OF U.S. PATENT NO. 8,236,738

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## EXHIBITS CONSIDERED<sup>1</sup>

| Ex. # | Description                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |
|-------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1001  | U.S. Patent No. 8,236,738, Zhang, “Control of Particulate Entrainment by Fluids” (“the ’738 patent”)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |
| 1002  | <p>Declaration of Dr. Gary Wooley, Ph.D.</p> <ul style="list-style-type: none"> <li>• Ex. A: Resume of Gary Wooley</li> <li>• Ex. B: Recent Case Listing of Gary Wooley</li> <li>• Ex. C: ’738 Patent v. Nguyen Claim Chart</li> <li>• Ex. D: ’738 Patent v. Dawson Claim Chart</li> <li>• Ex. E: ’738 Patent v. Vincent Claim Chart</li> <li>• Ex. F: ’738 Patent v. Weaver Claim Chart</li> <li>• Ex. G: ’738 Patent v. Needham Claim Chart</li> <li>• Ex. H: ’738 Patent v. Nguyen in view of Weaver or Plummer Claim Chart</li> <li>• Ex. I: ’738 Patent v. Dawson in view of Weaver or Plummer Claim Chart</li> <li>• Ex. J: ’738 Patent v. Vincent in view of Weaver or Plummer Claim Chart</li> <li>• Ex. K: ’738 Patent v. Needham in view of Weaver or Plummer Claim Chart</li> </ul> |
| 1003  | Prosecution History of the ’738 Patent                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |
| 1004  | Prosecution History of U.S. Patent App. No. 12/759,469, Zhang, “Method for Making Particulate Slurries and Particulate Slurry Compositions” (“the ’469 application”)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |
| 1005  | U.S. Patent App. Publication No. US 2004/0023818, Nguyen, “Method and Product for Enhancing the Clean-Up of Hydrocarbon-Producing Well” (“Nguyen”)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |
| 1006  | Canadian Patent No. 2,423,031, Dawson, “Lightweight Particulate and Uses Therefor” (“Dawson”)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |
| 1007  | U.S. Patent App. Publication No. US 2005/0244641, Vincent, “Coating and/or Treating Hydraulic Fracturing Proppants to Improve Wettability, Proppant Lubrication, and/or to Reduce Damage by Fracturing Fluids and Reservoir Fluids” (“Vincent”)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                |
| 1008  | U.S. Patent App. Publication No. US 2006/0260808, Weaver, “Methods of Treating Particulates and Use in Subterranean Formations” (“Weaver”)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |

<sup>1</sup>This Exhibits Considered list is consistent with the Joint Listing of Exhibits for IPR2017-02099 and IPR2017-02100.

|      |                                                                                                                                                           |
|------|-----------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1009 | U.S. Patent No. 4,231,428, Needham, “Well Treatment Method” (“Needham”)                                                                                   |
| 1010 | U.S. Patent No. 3,980,136, Plummer, “Fracturing Well Formations Using Foam” (“Plummer”)                                                                   |
| 1011 | U.S. Patent No. 6,330,916, Rickards, “Formation Treatment Method Using Deformable Particles” (“Rickards”)                                                 |
| 1012 | U.S. Patent No. 3,659,651, Graham, “Hydraulic Fracturing Using Reinforced Resin Pellets” (“Graham”)                                                       |
| 1013 | U.S. Patent No. 3,378,074, Kiel, “Method for Fracturing Subterranean Formations” (“Kiel”)                                                                 |
| 1014 | Prosecution History of U.S. Patent No. 7,723,274, Zhang, “Method for Making Particulate Slurries and Particulate Slurry Compositions” (“the ’274 patent”) |
| 1015 | File History of Vincent                                                                                                                                   |
| 1016 | File History of Nguyen                                                                                                                                    |
| 1017 | U.S. Patent No. 3,977,472, Graham, “Method of Fracturing Subterranean Formations using Oil-in-Water Emulsions”                                            |
| 1018 | U.S. Patent No. 5,633,220, Cawiezel, “High Internal Phase Ratio Water-in-Oil Emulsion Fracturing Fluid”                                                   |
| 1019 | U.S. Patent No. 2,999,063, Hoeppel, “Water-in-Oil Emulsion Drilling and Fracturing Fluid”                                                                 |



## **U.S. PATENT NO. 8,236,738 CLAIM LISTING**

| <b>No.</b>  | <b>Limitation</b>                                                                                                                                                                                                  |
|-------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1 preamble  | 1. An aqueous slurry composition comprising:                                                                                                                                                                       |
| 1A          | water;                                                                                                                                                                                                             |
| 1B          | particulates;                                                                                                                                                                                                      |
| 1C          | a chemical compound for rendering the surface of the particulates hydrophobic; and                                                                                                                                 |
| 1D          | an oil.                                                                                                                                                                                                            |
| 21A         | 21. The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates. |
| 22A         | 22. The composition of claim 1, wherein the particulates are sands.                                                                                                                                                |
| 32 preamble | 32. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:                                                                                                        |
| 32A         | mixing water,                                                                                                                                                                                                      |
| 32B         | particulates,                                                                                                                                                                                                      |
| 32C         | a chemical compound for rendering the surface of the particulates hydrophobic and                                                                                                                                  |
| 32D         | an oil; and                                                                                                                                                                                                        |
| 32E         | pumping the mixture into the formation                                                                                                                                                                             |
| 40A         | 40. The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates.                               |
| 41A         | 41. The method of claim 32, wherein the particulates are sands.                                                                                                                                                    |
| 51 preamble | 51. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:                                                                                                        |
| 51A         | contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic,                                                                                        |
| 51B         | separating the particulate from the medium;                                                                                                                                                                        |
| 51C         | and blending the hydrophobic particulate with water and an oil to form a mixture;                                                                                                                                  |
| 51D         | and pumping the mixture into the formation.                                                                                                                                                                        |
| 57          | 57. The composition of claim 51, wherein the particulates are sands.                                                                                                                                               |

## DECLARATION OF DR. GARY WOOLEY

I, Dr. Gary Wooley, declare as follows:

1. I am over the age of 18 years and am fully competent to make this declaration. I make the following statements based on personal knowledge and, if called to testify to them, could and would do so.

### **I. INTRODUCTION**

2. I have been retained on behalf of Petitioners, Preferred Proppants, LLC; Preferred Pipeline, LLC; Preferred Resin Sales, LLC; Preferred Resin of Genoa, LLC; and Preferred Sands of Wisconsin, LLC (collectively, “Petitioners”) to, among other things, provide this Declaration as an expert in wellbore fluids including drilling, completion, hydraulic fracturing, and gravel packing fluids, and pipeline fluids and render an opinion regarding the validity of claims 1, 21, 22, 32, 40, 41, 51, and 57 of U.S. Patent No. 8,236,738 (the “’738 patent”) (Ex. 1001).

### **II. BACKGROUND & QUALIFICATIONS**

3. I am President of Wooley & Associates, Inc., a petroleum and mechanical engineering consulting firm with offices at 3100 South Gessner, Suite 325, Houston, Texas 77063. I am a registered Professional Engineer in the State of Texas (#40049). I have a B.S. degree in Mechanical Engineering (1969), an M.S. degree in Engineering Mechanics (1970), and a Ph.D. degree in Engineering Science

with minors in Applied Mathematics and Mechanical Engineering (1972), from Louisiana State University.

4. My relevant experience began during my undergraduate studies. I did much educational work with fluid mechanics, including a number of undergraduate and graduate level classes, as well as a Ph.D. dissertation on numerical modeling of 2D viscous flow.

5. I have over fifty years of experience in the petroleum industry, and have worked with wellbore and pipeline fluids since the 1960s. Much of my work in the petroleum industry has focused on planning, designing and executing drilling, completion, and production of oil and gas wells from conventional reservoirs (onshore and offshore including unconsolidated sands requiring gravel packs) and shale reservoirs (requiring horizontal wells and fracture stimulation). In addition, I have worked on design, installation and analysis of flow lines, gathering lines and large pipelines, including new lines and failures and repairs of old lines.

6. During my career in the petroleum industry, I have worked for Shell, Chevron, Humble Oil (Exxon), and Atlantic Richfield Company (Arco) in various engineering positions. While working as an operator employee at Chevron, Exxon, and Arco, I learned hydraulic fracturing operations.

7. In the 1960s and 1970s as an employee of a major oil company, I helped write specifications for fracture stimulations in conventional wells, solicited bids,

selected contractors, and supervised field operations in south Louisiana and the Gulf of Mexico. I continued my work in hydraulic fracturing in the 1970s, when I worked with Tom Perkins and Lloyd Kern, two of the pioneers in hydraulic fracturing, studying rock and fluid mechanics, following literature, conducting laboratory testing and computer modeling. I worked on drilling, completions and production including gravel packs to limit the quantity of sand from unconsolidated sand reservoirs that is suspended in oil, gas and water production fluids. I worked on installation and repairs of tubings, casings, flow lines, gathering lines and pipelines for produced oil, gas and water, including chemicals injected in fluids to reduce steel corrosion, to reduce the buildup of paraffin and scale, and for other benefits.

8. Since 1978, I have served as a petroleum and mechanical engineering consultant to the oil and gas industry. My clients include all the major oil companies and many independents and smaller oil companies, all the major and many smaller oilfield service contractors, manufacturers and suppliers of oilfield equipment. The majority of this consulting work has been on upstream activities such as drilling, completions, and production, including hydraulic fracturing. I have helped clients with designing, selecting proposals, and supervision of fracture stimulations in horizontal wells since the late 1980s. I have also helped clients with evaluating the suitability of fracture stimulations for specific wells, and with evaluating the causes of problems encountered during fracture stimulations, including failure analyses.

9. I have served as an expert witness for operators, contractors, and suppliers in various litigations involving fracture stimulation equipment, fluids, and procedures. As one representative example, in 2001 and 2002, I worked with BJ Services on a trade secrets and patent infringement dispute with Halliburton over BJ's Vistar fracturing fluid and Halliburton's Phoenix fracturing fluid. The case went to trial and BJ won a one hundred million dollar jury verdict, and the case was upheld on appeal.

10. I have consulted with clients in drilling matters which involved the flow of oil, water, and polymer based drilling muds, chemical inhibitors, viscosifiers and other mud additives, oil, gas, and water, and other fluids as they flow through drill pipe, bottom hole assemblies, drill bits, the annular space between drill pipe and formations, gathering tanks, separators, pumps of a drilling circulating system, and solids separation equipment—such as shale shakers, sand traps, and hydrocyclone, desanders and desilters. One example involved the 20 April 2010 blowout of the Macondo well in the Gulf of Mexico. Another matter was a 2010-2011 Canadian litigation involving an automatic driller patent owned by NOV that alleged infringement by an automatic drilling system AutoDriller offered by Pason Systems.

11. I have represented clients in completions and production operations, procedures, and equipment. Included in this work are flows of, oil, gas, and water through reservoirs, gravel packs, perforations, gas lift valves, production tubing,

wellheads, flow lines, separators, heater treaters, and other components. Besides fracture stimulation, chemicals such as acids are injected down wellbores, through casing perforations and into formations to improve production. Corrosion inhibitors, paraffin inhibitors and other chemicals may be injected into oil, gas and water flowing in production tubing, flow lines, gathering lines, pipelines, or other equipment. Cyclones and other devices are used to separate solids such as sand from the produced oil, gas, and water. One example would be the 2010-2014 litigation between Sepco Tubulars and United Resources involving a production casing failure and obstruction and the expected oil and gas production from three reservoirs. Two examples of litigation involving pipelines were the investigation of the 27 June 1996 leak of the 36" Colonial pipeline in South Carolina carrying gasoline from Texas to New York, and the investigation of the 21 October 1994 leak of the 40" pipeline into the San Jacinto River near Houston.

12. A copy of my resumé is attached to this Declaration as Ex. 1002A. I have testified as an expert witness in the oil and gas industry in a number of cases over the last five years, as shown by the list of cases attached to this Declaration as Ex. 1002B.

13. I am being compensated for my services at my normal billing rate of \$520/hour. My compensation is not in any way affected by the outcome of this matter, and I do not have any other financial interests in this matter or in the pending

litigation between Petitioners and Trican Well Service Ltd. (“Trican” or “Patent Owner”) in federal court.

### **III. BASES FOR OPINIONS EXPRESSED**

14. The opinions set forth in this declaration are my own. My opinions are based on my review of materials provided in this matter and cited throughout this Declaration and itemized in “Exhibits Considered” preceding this declaration, my educational background, my experience working in the oil and gas industry, including working with hydraulic fracturing, drilling, completions, production, and oil and gas technology, and my experience consulting and providing expert testimony in many cases involving similar and/or related technology.

15. My analysis of the materials relating to this matter is ongoing and I will continue to review any new material that is provided to me. This Declaration represents certain opinions that I have formed to date and the bases for those opinions. I reserve the right to revise, supplement, and/or amend my opinions based on new information presented to me and on my continuing analysis of the materials cited throughout this Declaration and those identified in the “Exhibits Considered.”

### **IV. UNDERSTANDING OF LEGAL STANDARDS**

16. I am not an attorney. For purposes of this declaration, I have been informed about certain aspects of the law that are relevant to my analysis and opinions, as set forth below.

**A. Claim Construction**

17. In an *inter partes* review, it is my understanding the pending claims are given their broadest reasonable construction in light of the specification and prosecution history as it would be interpreted by one of ordinary skill in the art at the time of the claimed invention.

18. Under the broadest reasonable interpretation standard, it is my understanding that the words of the claims must be given their ordinary and customary meaning as understood by those of ordinary skill in the art at the time of the invention.

19. It is my understanding that the ordinary and customary meaning of a term may be evidenced by a variety of sources, including the words of the claims themselves, the specification, the prosecution history, and extrinsic evidence concerning relevant scientific principles, the meaning of technical terms, and the state of the art.

20. I also understand that some claims are independent, and that those independent claims are complete by themselves. Other claims refer to these independent claims and are “dependent” on those independent claims. The dependent claims include all of the limitations of the claims on which they depend.



**B. Prior Art**

21. Among other things, I understand prior art could include printed publications in or outside the United States by the applicant before the claimed invention was discovered, patents or printed publications more than one year before the date of the application, and patent applications filed or patents granted on an application filed in the United States before the invention was discovered.

22. I have been instructed to assume for purposes of my analysis that April 26, 2007 is the relevant date for determining what is “prior art.” In other words, I should consider as “prior art” any patents or publications publicly available prior to April 26, 2007 or a patent application filed in the United States before the invention was discovered. I further understand that for purposes of this proceeding in the United States Patent Trial and Appeal Board, only patents and other documents that have the legal status of a “printed publication” may be relied on as prior art.

**C. Anticipation**

23. It is my understanding that anticipation is a question of fact that is determined from the perspective of one of ordinary skill in the art at the time of the claimed invention. I also understand that the standard for anticipation in an *inter partes* review proceeding is by a preponderance of the evidence.

24. It is my understanding that a claim is anticipated only if each and every element as set forth in the claim is found, either expressly or inherently described, in a single prior art reference.

25. I understand that if the prior art reference does not expressly set forth a particular claim element, the prior art may still anticipate if that element is “inherent” in its disclosure. To establish inherency, it is my understanding that evidence must make clear that the missing descriptive matter is necessarily present in the thing described in the reference. It is my understanding that inherency may not be established by probabilities or possibilities. In other words, it is my understanding that the mere fact that a certain thing may result from a given set of circumstances is not sufficient to establish inherency.

26. I understand that if a claim recites alternatives, for example in the format “selected from the group consisting of A, B, and C,” anticipation of the entire claim limitation can be achieved through prior art that discloses one alternative, for example, a prior art reference that simply discloses B.

27. I also have been advised that a prior art document can disclose a claim feature, and anticipate a claimed invention, if that feature is described in another document that has been incorporated by reference.

**D. Obviousness**

28. It is my understanding that obviousness is a question of law with underlying factual inquiries that are determined from the perspective of one of ordinary skill in the art at the time of the claimed invention. I also understand that the standard for obviousness in an *inter partes* review proceeding is by a preponderance of the evidence.

29. It is my understanding that a claimed invention is obvious if the differences between the claimed invention and the prior art are such that the claimed invention as a whole would have been obvious before the effective filing date of the claimed invention to a person having ordinary skill in the art to which the claimed invention pertains. I understand that a person of ordinary skill in the art is presumed to have knowledge of all relevant prior art.

30. It is my understanding is that obviousness requires consideration of the following underlying factual inquiries:

- determining the scope and content of the prior art;
- ascertaining the differences between the claimed invention and the prior art;
- resolving the level of ordinary skill in the art; and

- evaluating objective evidence or “secondary considerations” of non-obviousness, such as commercial success, long-felt but unresolved needs, failure of others, industry praise, and unexpected results.

31. I understand, however, that for such objective evidence to be relevant to the obviousness of a claim, there must be a causal relationship (called a “nexus”) between the claim and the evidence. I also understand that this nexus must be based on a novel element of the claim rather than something in the prior art. I also understand that there are instances when secondary considerations are unable to overcome primary evidence of obviousness (e.g., motivation to combine with predictable results) that is sufficiently strong.

32. It is my understanding that a reference must be analogous art to the claimed invention in order to be properly considered for use in an obviousness inquiry. It is my understanding that a reference is analogous art to the claimed invention if:

- the reference is from the same field of endeavor as the claimed invention (even if it addresses a different problem); or
- the reference is reasonably pertinent to the problem faced by the inventor (even if it is not in the same field of endeavor as the claimed invention).

33. It is my understanding that whether a reference is reasonably pertinent turns on how the problem faced by the inventor is reflected, either explicitly or implicitly, in the specification.

34. It is my understanding that the following are exemplary rationales that may support a conclusion of obviousness:

- 1) Combining prior art elements according to known methods to yield predictable results;
- 2) Simple substitution of one known element for another to obtain predictable results;
- 3) Use of known technique to improve a similar method or product in the same way;
- 4) Applying a known technique to a known method or product ready for improvement to yield predictable results;
- 5) Obvious to try – choosing from a finite number of identified, predictable solutions, with a reasonable expectation of success;
- 6) Known work in one field of endeavor may prompt variations of it for use in either the same field or a different one based on design incentives or other market forces if the variations are predictable to one of ordinary skill in the art; and

- 7) Some teaching, suggestion, or motivation in the prior art that would have led one of ordinary skill to modify the prior art reference or to combine prior art reference teachings to arrive at the claimed invention.

## V. SUMMARY OF OPINIONS

35. For reasons described in more detail in this Declaration, my opinions are as follows:

- Claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent (the “challenged claims”) are merely a reflection of the state of the art in hydraulic fracture fluids at the time the '738 patent was filed. Not only was each and every element of the challenged claims known in the art, but they were understood by those skilled in the art to be useful together.
- Claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent are anticipated based on U.S. Patent Application Publication No. 2004/0023818 (“Nguyen”) (Ex. 1005).
- Claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent are anticipated based on Canadian Patent Application No. 2,423,031 (“Dawson”) (Ex. 1006).

- Claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent are anticipated based on U.S. Patent Application Publication No. 2005/0244641 ("Vincent") (Ex. 1007).
- Claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent are anticipated based on U.S. Patent Application Publication No. 2006/0260808 ("Weaver") (Ex. 1008).
- Claims 1, 21 and 22 of the '738 patent are anticipated based on U.S. Patent No. 4,231,428 ("Needham") (Ex. 1009).
- Claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent would have been obvious over Nguyen in view of Weaver or U.S. Patent No. 3,980,136 ("Plummer") (Ex. 1010).
- Claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent would have been obvious over Dawson in view of Weaver or Plummer.
- Claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent would have been obvious over Vincent in view of Weaver or Plummer.
- Claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent would have been obvious over Needham in view of Weaver or Plummer.

## **VI. LEVEL OF ORDINARY SKILL IN THE ART AND TECHNICAL DISCUSSION**

### **A. Level of Ordinary Skill in the Art**

36. I have been asked to provide my opinions regarding the knowledge and understanding of a person of ordinary skill in the art as of the earliest effective filing date of the '738 patent.

37. I understand that the factors considered in determining the ordinary level of skill in the art include: (i) the levels of education and experience of persons working in the field; (ii) the types of problems encountered in the field; and (iii) the sophistication of the technology.

38. I understand that a person of ordinary skill in the art is not a specific real individual, but rather a hypothetical individual having the qualities reflected by the factors above. This hypothetical person has knowledge of all prior art in the relevant field as if it were arranged on a workshop wall and takes from each reference what it would teach to a person having the skills of a person of ordinary skill in the art.

39. I also understand that a person of ordinary skill in the art is also a person of ordinary creativity, not an automaton. Therefore, a person of ordinary skill in the art has and applies common sense to modify or combine references in the prior art, and can also find any teaching, suggestion, or motivation, whether to modify or combine, whether explicit or implicit.



40. It is my opinion that a person of ordinary skill in the art (as of April 2007) as to the '738 patent would have either (i) at least a bachelor's degree in engineering or chemistry, or equivalent experience and education, and (ii) 3-5 years of experience in the petroleum or other industry working with wellbore and/or pipeline fluids. Wellbore fluids would include drilling, completion, hydraulic fracturing, and gravel packing fluids.

41. As discussed below, it is my understanding that claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent are entitled to an effective filing date no earlier than April 26, 2007. Accordingly, the analysis set forth herein is based on the timeframe of the alleged invention being April 26, 2007. As of April 2007, I qualified for or exceeded the level of ordinary skill required by the above definition, and am in a position to opine on the understanding of a person of ordinary skill in the art as of that date. When I address the understanding of a person of ordinary skill in the art, I am referring to such a person's understanding as of that date, which I refer to as the "effective filing date."

#### **B. Technical Discussion**

42. To produce oil and gas from a subterranean hydrocarbon bearing formation, a borehole must be drilled down to the formation. In general, a surface borehole is drilled using a drill bit and a drillstring to a predetermined depth, after which a steel pipe known as casing is run and cemented into place to prevent the

borehole from collapsing and to seal the annular space between the borehole and the outer diameter of the casing. Thereafter, a smaller borehole is drilled beyond the end of the previous casing string (using a smaller drill bit) to another deeper, predetermined depth. A second, smaller casing string is lowered into the wellbore and cemented to protect the new section of the borehole. This process is repeated as necessary until the hydrocarbon bearing formation is penetrated by the well. During the drilling process, a drilling fluid (a/k/a drilling mud) is pumped down the drillstring and back to the surface. The drilling fluid serves several purposes, including lifting the drill cuttings out of the borehole, lubricating and cooling the drill bit, keeping the open borehole from collapsing and helping control the pressure exerted by downhole formation fluids (i.e., preventing the well from blowing out). Once the drilling fluid returns to the surface, the fluid is treated by special surface equipment, such as shale shakers, desilters, mud cleaners and centrifuges, to remove the drill cuttings and other solids before the drilling mud is again pumped back down the drillstring. Thus, the drilling fluid is circulated and constantly reused during the drilling of the borehole. This process for constructing the wellbore has been used for decades (i.e., well before the effective filing date of the patent).

43. Once the wellbore reaches the hydrocarbon bearing formation, it is tested to see if the well contains commercial quantities of oil and/or gas. If so, the well is then completed. Depending on the well, the completion process may include

further well testing, running wireline logs to measure various properties of the formation and taking core samples of the formation rock, running and cementing a production casing string or a slotted liner across the hydrocarbon bearing formation, perforating the production casing to establish communication between the formation and the wellbore, running production tubing and downhole production equipment such as packers and safety valves, and attaching a surface wellhead and valves (Christmas tree) and related surface flow lines and/or surface production equipment. In essence, completing the well includes the various steps needed to begin producing oil and/or gas from the well. The main constituent of the natural gas found in hydrocarbon bearing formations is methane gas. Oil found in a hydrocarbon bearing formation is an example of a liquid hydrocarbon.

44. Hydraulic fracturing (a/k/a fracking) is a common treatment for stimulating hydrocarbon bearing formations, particularly low permeability formations such as shales. In hydraulic fracturing, a fracturing fluid is pumped from the surface of the well, down the wellbore, to the hydrocarbon bearing formation. The pump pressure is steadily increased until the pressure of the fracturing fluid exceeds the shear strength of the rock formation, causing the formation to crack or fracture. Once the formation fractures, pumping is continued to lengthen the extent or reach of the fracture. In addition to initiating and propagating the fracture, the fracturing fluid also transports proppant into the

fracture. Once pumping ceases, the proppant that remains in the fracture prevents the fracture from fully closing. The propped fracture provides a flow path to the wellbore with improved permeability, allowing more oil and gas to flow into the wellbore, thereby increasing the production of oil and gas from the well. The most common proppant used for hydraulic fracturing is sand. After fracture stimulation pumping is stopped, the fracturing fluid (without the portion of the proppant that is trapped in the fractures) flows back to the surface where it is collected in surface storage containers. The recovered fracturing fluid may then be re-used and/or disposed of.

45. Another common treatment for oil and gas wells is gravel packing (a/k/a sand packing). Gravel packing is a treatment used to reduce the amount of formation sand that is produced with the oil and gas from a reservoir. For example, in an unconsolidated sandstone formation it is not uncommon for sand grains from the formation rock to break loose and be produced with the flowing gas. The sand grains produced with the formation gas cause excessive wear (comparable to sandblasting) on the production tubing string and surface production equipment such as the Christmas tree, flow lines and related valving, shortening the useful life of such equipment. In a typical gravel pack, a slotted or perforated, wire wrapped screen is placed on the end of the production tubing adjacent the gas producing formation. The spacing between the wrapped wire forming the outer surface of the

screen is sized to prevent individual formation sand grains from passing through the screen. In addition, a sand or gravel-laden slurry is circulated down the production string and out ports above the screen so that the sand from the slurry is deposited in the annular space between the screen and the formation (e.g., between the screen and the perforations that extend from the production casing into the formation) while the slurry fluid passes through the screen and slotted pipe. The slurry fluid without the sand is circulated back to the surface where it is collected in surface containers for either re-use or disposal. The deposited gravel (i.e., sand) packed in the annular space between the formation and screen acts as a filter to keep formation sand grains from being produced the gas, thereby protecting the production tubing and surface equipment. See Fundamentals of Petroleum 184 (Mildred Gerding ed., Petroleum Extension Service 3d ed. 1986).

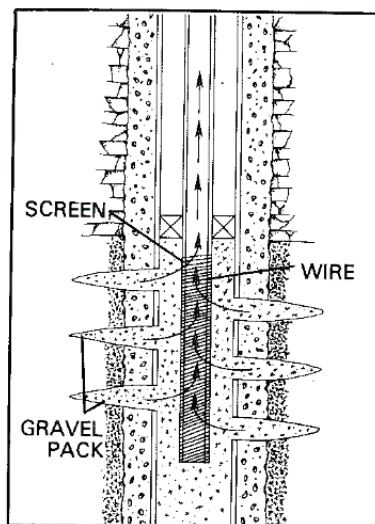


Fig. 5.7. A wire-wrapped screen completion is often run with a gravel pack inside perforated casing.

46. The word shear is used to describe the relative movement of layers and to describe the force required to cause that movement.<sup>2</sup> Generally, shear is described by shear strain or shear stress, which are related to each other by the properties of the material. Shear strain is a measure of the movement of one layer relative to another, while shear stress is a measure of the force required to cause that movement. For a more in-depth description of shear, *see* Victor Streeter, *FLUID MECHANICS* 4-7 (McGraw Hill Inc. 4th ed. 1966); *see also* Adam T. Bourgoyne Jr. et al, *APPLIED DRILLING ENGINEERING SPE TEXTBOOK SERIES*, VOL. 2 143-44 (Jack F. Evers and David S. Pye eds. 1991).

47. Fluids in oil and gas services experience shear during many operations. In drilling, drilling fluids are mixed at the surface, pumped down the inside of the drill pipe, travel through drill bits, and return to the surface on the outside of the drill pipe where solids are filtered and the drilling fluid reconditioned, then pumped back into the well. At each of these stages, the drilling fluid is subjected to shear. For example, when drilling fluid are forced through small jets in a drill bit, they are subjected to dramatic shear.

48. In hydraulic fracturing, fracture fluids are mixed at the surface and combined with proppants, then pumped down the well to the reservoir to be

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<sup>2</sup>One of ordinary skill in the art would understand that the '738 patent uses the term "shear" to mean the relative displacement or movement of layers of fluid. *See* Section IX.B, *infra*.

stimulated. The fracture fluids and proppants create fractures in the reservoir through which the fluids and proppant flow. After fracture stimulation, the pressure on the well is removed, and the fracture fluids flow back to the surface. At each of these stages the fracture fluid experiences shear. For example, when fracture fluids and proppants are injected into the well and travel downhole, the fluid is subjected to shear, and when fracture fluids and proppants create a fracture in the reservoir or formation, the shear is more dramatic when the fracture fluid and proppant enter the fracture.

49. During production, oil, gas, and water flow through pore spaces and/or fractures in the reservoir to the wellbore, up the wellbore inside production tubing, through the well head to the surface for separation and treating. The fluid is then pumped through flowlines to a pipeline or tank. In some wells, sand from a producing reservoir is carried with oil, gas, and water to the surface. As described above, to reduce the amount of sand in the production fluid, a gravel pack with sand or particles may be installed during completion. During installation of the gravel pack, the particles are mixed with a carrier fluid and injected to the bottom of the well, the sand is placed in the annulus between the gravel pack screen and the perforations, and the carrier fluid returns to the surface in the annulus. During mixing, injecting, and placing of the particles, the carrier fluid is subjected to shear.

50. In pipeline flow, fluids and slurries with particulates are pumped into a line to apply pressure to cause flow to the other end. During the pumping and flowing operations, the fluid or slurry is subjected to shear.

51. Chemical compounds are named according to a common system created by the International Union of Pure and Applied Chemistry (IUPAC). JOHN McMURRAY, ORGANIC CHEMISTRY 81 (3rd ed. 1992). A chemical name has three parts in IUPAC: prefix, parent, and suffix. The prefix specifies the location of the functional groups and other substituents on the chain; the parent specifies the number of carbon atoms (if any) in the main chain and the length of that chain; and the suffix identifies the functional groups present in the molecule. *Id.* A functional group is part of a larger molecule and is composed of an atom or group of atoms that have a characteristic chemical behavior. *Id.* at 67. The chemistry and way a molecule acts, regardless of the size and complexity of the molecule, is determined by the functional groups the molecule contains. *Id.* at 68. To name a molecule, begin with identifying the parent in terms of number of carbons or other base elements. *Id.* at 81-82. After identifying the parent, identify and name the substituents (suffix). *Id.* at 82. If two or more identical substituents are present, use one of the prefixes di-, tri-, tetra-, and so forth. *Id.* at 83.

52. The simplest silicon-based compound is known as silane and consists of a silicon atom bonded to four hydrogens (SiH<sub>4</sub>). Eugene G. Rochow, AN



INTRODUCTION TO THE CHEMISTRY OF THE SILICONES 4 (2nd ed. 1951). Silane is a shorter name for the more formal term silicane. *Id.* A silane that contains at least one carbon-silicon bond ( $\text{CH}_3\text{-Si-}$ ) structure is called an organosilane. Dow Corning, “A Guide to Silane Solutions: The Basics of Silane Chemistry” (2009). Silanes can act as water repellents or surface protection. *Id.* A siloxane is created when one of the four hydrogen atoms of a silane is replaced with an oxygen. *See* VANNOSTRAND’S SCIENTIFIC ENCYCLOPEDIA 2583 (Douglas M. Considine and Glenn Considine eds. 7th ed. 1989). Siloxanes can similarly act as strong water repellents. Eugene G. Rochow, AN INTRODUCTION TO THE CHEMISTRY OF THE SILICONES 132-33 (2nd ed. 1951). As disclosed in U.S. Patent No. 8,236,738, “[o]rganosilanes are compounds containing silicon to carbon bonds. Organosiloxanes are compounds containing  $\text{Si—O—Si}$  bonds. Polysiloxanes are compounds in which the elements silicon and oxygen alternate in the molecular skeleton, i.e.,  $\text{Si—O—Si}$  bond are repeated. The simplest polysiloxanes are polydimethylsiloxanes.” Ex. 1001, ’738 patent at 5:4-9.

53. The attachment of a chlorine directly to a silicon yields a chlorosilane. Dow Corning, “A Guide to Silane Solutions: The Basics of Silane Chemistry” (2009). Examples of chlorosilanes include dimethyldichlorosilane ( $(\text{CH}_3)_2\text{SiCl}_2$ ), trimethylchlorosilane ( $(\text{CH}_3)_3\text{SiCl}$ ), and methyltrichlorosilane ( $\text{CH}_3\text{SiCl}_3$ ). Chlorosilanes are very reactive and exhibit unique inorganic reactivity with water.

*Id.* This is the key chemistry that makes chlorosilanes hydrophobic and function as valuable surface-treating and coupling agents. *Id.*

## **VII. U.S. PATENT NO. 8,236,738**

54. The '738 patent titled "Control of Particulate Entrainment by Fluids" was filed as a PCT application on April 25, 2008 and issued on August 7, 2012. Ex. 1001 (cover). The '738 patent claims priority to U.S. Provisional Application Ser. No. 60/924,006, filed on April 26, 2007. *Id.*

### **A. The Specification of the '738 Patent**

55. The '738 patent states that its alleged invention "relates to [a]n aqueous slurry composition having water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic and an oil" (*id.* at 3:6-9) and to "a method of controlling sand in a hydrocarbon producing formation comprising the steps of mixing water, particulates and a chemical compound for rendering the surface of the particulates hydrophobic, [and] pumping the mixture into the formation" (*id.* at 3:10-15). The abstract states that the slurry composition is used in the petroleum and pipeline industries, and that the composition includes an aqueous carrier fluid and "a small amount of an oil." According to the specification, the purported invention is useful in controlling proppant flowback in hydraulic fracturing treatments and in reducing formation sand production during well production. *Id.* at 4:33-35.

56. The '738 patent recites that there are “various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” *Id.* at 4:62-67.

57. The '738 patent specifies that “[t]he organosiloxane compounds also include alkylsiloxanes including hexamethylcyclotrisiloxane, octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, hexamethyldisiloxane, hexaethyldisiloxane, 1,3-divinyl-1,3,3-tetramethyldisiloxane, octamethyltrisiloxane, decamethyltetrasiloxane. The organosilane compounds include alkylchlorosilane, for example methyltrichlorosilane, dimethyldichlorosilane, trimethylchlorosilane, octadecyltrichlorosilane; alkyl-alkoxysilane compounds, for example methyl-, propyl-, isobutyl- and octyltrialkoxysilanes, and fluoro-organosilane compounds.” *Id.* at 5:34-45.

58. The '738 patent provides several examples of organosilanes including:

$\text{CH}_3\text{SiCl}_3$ ,  $\text{CH}_3\text{CH}_2\text{SiCl}_3$ ,  $(\text{CH}_3)_2\text{SiCl}_2$ ,  $(\text{CH}_3\text{CH}_2)_2\text{SiCl}_2$ ,  
 $(\text{C}_6\text{H}_5)_2\text{SiCl}_2$ ,  $(\text{C}_6\text{H}_5)\text{SiCl}_3$ ,  $(\text{CH}_3)_3\text{SiCl}$ ,  $\text{CH}_3\text{HSiCl}_2$ ,  $(\text{CH}_3)_2\text{HSiCl}$ ,  
 $\text{CH}_3\text{SiBr}_3$ ,  $(\text{C}_6\text{H}_5)\text{SiBr}_3$ ,  $(\text{CH}_3)_2\text{SiBr}_2$ ,  $(\text{CH}_3\text{CH}_2)_2\text{SiBr}_2$ ,  
 $(\text{C}_6\text{H}_5)_2\text{SiBr}_2$ ,  $(\text{CH}_3)_3\text{SiBr}$ ,  $\text{CH}_3\text{HSiBr}_2$ ,  $(\text{CH}_3)_2\text{HSiBr}$ ,  $\text{Si}(\text{OCH}_3)_4$ ,  
 $\text{CH}_3\text{Si}(\text{OCH}_3)_3$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_3)_3$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_3$ ,

$\text{CH}_3\text{Si}[\text{O}(\text{CH}_2)_3\text{CH}_3]_3$ ,  $\text{CH}_3\text{CH}_2\text{Si}(\text{OCH}_2\text{CH}_3)_3$ ,  $\text{C}_6\text{H}_5\text{Si}(\text{OCH}_3)_2$ ,  
 $\text{C}_6\text{H}_5\text{CH}_2\text{Si}(\text{OCH}_3)_3$ ,  $\text{C}_6\text{H}_5\text{Si}(\text{OCH}_2\text{CH}_3)_3$ ,  $\text{CH}_2=\text{CHCH}_2\text{Si}(\text{OCH}_3)_3$ ,  
 $(\text{CH}_3)_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{CH}_2=\text{CH})\text{Si}(\text{CH}_3)_2\text{Cl}$ ,  $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$ ,  
 $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_2$ ,  $(\text{CH}_3)_2\text{Si}[\text{O}(\text{CH}_2)_3\text{CH}_3]_2$ ,  
 $(\text{CH}_3\text{CH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_3)_2$ ,  
 $(\text{C}_6\text{H}_5\text{CH}_2)_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$ ,  
 $(\text{CH}_2=\text{CH})_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{CH}_3)_3\text{SiOCH}_3$ ,  
 $\text{CH}_3\text{HSi}(\text{OCH}_3)_2$ ,  $(\text{CH}_3)_2\text{HSi}(\text{OCH}_3)$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_3$ ,  
 $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $(\text{CH}_2=\text{CH})_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $\text{CH}_3\text{Si}(\text{CH}_3\text{COO})_3$ , 3-  
 aminotriethoxysilane, methyldiethylchlorosilane, butyltrichlorosilane,  
 diphenyldichlorosilane, vinyltrichlorosilane, methyltrimethoxysilane,  
 vinyltriethoxysilane, vinyltris(methoxyethoxy)silane,  
 methacryloxypropyltrimethoxysilane,  
 glycidoxypropyltrimethoxysilane, aminopropyltriethoxysilane,  
 divinyl-di-2-methoxysilane, ethyltributoxysilane,  
 isobutyltrimethoxysilane, hexyltrimethoxysilane, n-  
 octyltriethoxysilane, dihexyldimethoxysilane, octadecyltrichlorosilane,  
 octadecyltrimethoxysilane, octadecyldimethylchlorosilane,  
 octadecyldimethylmethoxysilane and quaternary ammonium silanes  
 including 3-(trimethoxysilyl) propyldimethyloctadecyl ammonium  
 chloride, 3-(trimethoxysilyl)propyldimethyloctadecyl ammonium  
 bromide, 3-(trimethylethoxysilylpropyl)didecylmethyl ammonium  
 chloride, triethoxysilyl soyapropyl dimoniumchloride, 3-  
 (trimethylethoxysilylpropyl)didecylmethyl ammonium bromide, 3-

(trimethylethoxysilylpropyl)didecylmethyl ammonium bromide,  
triethoxysilyl soyapropyl dimonium bromide,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_5)_3\text{Cl}^-$ ,  $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_5)_3\text{Br}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{CH}_3)_3\text{Cl}^-$ ,  $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_{13})_3\text{Cl}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{C}_4\text{H}_9\text{Cl}$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{C}_6\text{H}_5\text{Cl}$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{CH}_2\text{OHCl}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{C}_2\text{H}_5)_3\text{Cl}^-$ ,  
 $(\text{C}_2\text{H}_5\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{C}_{18}\text{H}_{37}\text{Cl}^-$ .

*Id.* at 6:1-43.

59. The '738 patent further discloses that the hydrophobic-coated particulate can be prepared in numerous ways, including:

- “[t]he hydrophobizing agent can be simply added into a sand slurry comprising sand and fracturing fluid which is pumped down the well” (*id.* at 3:51-53); or
- “[t]he hydrophobizing agent can be applied throughout the proppant stage or during a portion of the proppant stage such as the last portion of the proppant stage, i.e. tail-in” (*id.* at 3:60-62); or
- “sand can be hydrophobized first and dried and then used to make a slurry and pumped into fracture” (*id.* at 3:63-64).

60. Moreover, the '738 patent discloses that “when a small amount of an oil, including hydrocarbon oil and silicone oil, is mixed into the aqueous slurry

containing the hydrophobized sands, the hydrophobic aggregation is enhanced significantly.” *Id.* at 3:65-4:1. The oil can be added to the slurry in a number of ways including:

- “proppant such as sand is mixed with a hydrophobizing agent in water based slurry and pumped into the fractures, and then followed by over flush with oil or water containing a small amount of oil to strengthen the bridge between the sand grains” (*id.* at 4:6-10); or
- “the slurry containing a hydrophobizing agent can be pumped into the fracture forming the proppant pack, which can be further consolidated by oil or condensate contained in the formation” (*id.* at 4:12-15); or
- “the pre-hydrophobized sand is used as proppant and then followed by flushing with water, containing small amount of oil” (*id.* at 4:15-17); or
- “the pre-hydrophobized sand is used as a proppant which can be further consolidated by oil or condensate contained in the formation” (*id.* 4:17-19); or
- “the pre-hydrophobizing sand is tailed in and followed by flushing with water containing small amount of oil” (*id.* at 4:19-21).

61. The ’738 specification provides no meaningful disclosure of or guidance regarding the amount of oil to add to the slurry composition, how to determine the appropriate amount of oil, how to determine the type of oil to be used

for a given composition, or how to determine the effectiveness of a given oil, especially for downhole conditions. Due to the absence of such disclosure, a person of ordinary skill in the art would have to conduct his or her own experiments to determine the same.

**B. The Challenged Claims of the '738 Patent**

62. I have considered claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent, which are recited below. I have also reviewed and considered the '738 patent prosecution history.

63. Claims 1, 32, and 51 are independent claims. Claims 21-22, 40-41, and 57 are dependent claims, and include all of the features of claims 1, 32, and 51 respectively.

64. Claims 1 and 21-22 are composition claims. Claims 32, 40-41, 41 and 57<sup>3</sup> are method claims that recite a method of producing the composition recited in claims 1 and 21-22.

65. Claim 1 recites:

An aqueous slurry composition comprising:

water;

particulates;

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<sup>3</sup> I note that claim 57 recites “[t]he **composition** of claim 51, wherein the particulates are sands,” yet claim 51 recites “[a] **method** of controlling proppant flowback . . .,” without ever mentioning a composition. Nevertheless, the prior art recites each and every limitation of claims 51 and 57.

a chemical compound for rendering the surface of the particulates hydrophobic; and  
an oil.

66. Claim 21 recites:

The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates.

67. Claim 22 recites:

The composition of claim 1, wherein the particulates are sands.

68. Claim 32 recites:

A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:  
mixing water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic and an oil; and pumping the mixture into the formation.

69. Claim 40 recites:

The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates.

70. Claim 41 recites:

The method of claim 32, wherein the particulates are sands.

71. Claim 51 recites:



A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic, separating the particulate from the medium; and blending the hydrophobic particulate with water and an oil to form a mixture; and pumping the mixture into the formation.

72. Claim 57 recites:

The composition of claim 51, wherein the particulates are sands.

**C. The Prosecution History of the '738 Patent**

73. The '738 patent was filed as U.S. Patent Application No. 12/597,370 (the "'370 application") on October 23, 2009. The '370 application is the U.S. division of PCT application PCT/CA2008/000786 filed April 25, 2008. The '370 application claims priority to Provisional Application Serial No. 60/924,006 filed on April 26, 2007.

74. When filed, the '370 application had 45 total claims, made up of 2 independent claims and 43 dependent claims. Ex. 1003 at 21-28, Prosecution History of the '738 Patent, Claims.

75. Claims 1 and 26 of the '370 application are analogous to challenged claims 1 and 32 of the '738 patent.

76. Claim 1 of the '370 application as filed recited:

1. An aqueous slurry composition comprising:  
water;

particulates;  
a chemical compound for rendering the surface of the particulates hydrophobic; and  
an oil.

77. Claim 26 of the '370 application as filed recited:

26. A method of controlling sand in a hydrocarbon producing formation comprising the steps of mixing water, particulates and a chemical compound for rendering the surface of the particulates hydrophobic, pumping the mixture into the formation.

78. Claim 26 as filed is noteworthy in that the mixture did not require oil.

*Id.* at 25, File History of the '738 Patent, Original Claim 26.

79. On April 18, 2011, the Patent Office issued a restriction requirement for claims 1-46. *See generally*, File History of the '738 Patent at 865-68, Restriction Requirement, Apr. 18, 2011. The Examiner found that the application “contains claims directed to more than one species of the generic invention. *Id.* at 867. These species are deemed to lack unity of invention because they are not so linked as to form a single general inventive concept under PCT Rule 13.1. *Id.* The species is as follows: [t]he chemical compound.” *Id.* Thus, the Examiner required Trican to elect a single species. *Id.*

80. On May 18, 2011, the Applicant responded to the restriction requirement by “elect[ing] without traverse organosilane as the chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1003 at 873, File

History of the '738 Patent, Response to Restriction Requirement, May 18, 2011. The Applicant also submitted that claims 1-4, 7-28, and 30-46 read on the elected species. *Id.*

81. On July 8, 2011, the Examiner issued the first, Non-Final Office Action rejecting claims 1-23, 25-42, 45, and 46, and objecting to claims 24, 43, and 44. *See generally*, Ex. 1003 at 881-87, File History of the '738 Patent, Non-Final Office Action, July 8, 2011. The Examiner rejected claims 7-20, 25, 30, 31, 39-42, and 46 under 35 U.S.C. § 112 as indefinite. *Id.* at 884. With respect to claims 7, 14, 30, 39, and 41, the Examiner stated that “the phrase ‘typically’ renders the claim indefinite because it is unclear whether the limitation(s) follow the phrase are part of the claimed invention.” *Id.* Regarding claims 25 and 46, the Examiner stated that “each recites the limitation ‘functionally effective amount,’ however, this term is not sufficiently defined in the specification or claims such that a person of ordinary skill in the art could interpret the metes and bounds of the claim so as to understand how to avoid infringement.” *Id.* Moreover, the Examiner stated that “[c]laims 8-13 depend on claim 7, claims 15-20 depend on claim 14, claim 31 depends on claim 30, claim 40 depends on claim 39 and claim 42 depends on claim 41.” *Id.*

82. The Examiner also rejected claims 1-23, 26-42 and 45 under 35 U.S.C. § 102(b) as anticipated by U.S. Application Publication 2007/0015669 to Zhang, which were cited as X-references on the International Search Report for

PCT/CA2008/000786, from which the '370 application claims priority. *Id.* at 885.

The Examiner found that

Zhang teaches a [sic] methods of treating a wellbore and subterranean formation—specifically fracturing operations (0007)—using an aqueous slurry comprising water (0003), particulates—including those instantly exemplified and claimed (0005)—and a chemical compound—including betaine, dibetaine, quaternary, di-quaternary and other siloxanes and polysiloxanes and instantly claimed (0023-0025), and further comprising a gas—such as air, nitrogen, carbon dioxide and methane (0020)—mixed into the composition via shear through pumping (0020), and further teaches that such composition is produced as claimed (0028-0029). ***Since Zhang teaches the composition being placed into the hydrocarbon oil bearing formation, it is taken by the examiner that such hydrocarbon oil is included in the composition itself.***

*Id.* (emphasis added).

83. The Examiner stated that claims 23, 43, and 44 are objected to as being dependent upon rejected claims, but would be allowable if rewritten in independent form, and stated that claims 25 and 46 would be allowable if rewritten to overcome the § 112 rejections because “Zhang, the closest prior art made of record, fails to teach or fairly suggest the oil being kerosene or frac oil or the amount of oil present in the composition and further fails to teach or fairly suggest introducing the oil into the formation.” *Id.* at 886.

84. The Applicant filed amendments to the specification and the claims and remarks in response to the Office Action on November 8, 2011. *See generally, id.* at 905, File History of the '738 Patent, Amendment and Remarks, Nov. 8, 2011.

85. The Applicant amended the claims to: (1) add 13 additional dependent claims; and (2) amend claims 1-4, 7-16, 18, 19, 23-27, 30, 39, 41, 45, and 46. *See id.* at 907-12. Independent claim 1 was amended to include the limitation of “[a]n aqueous slurry composition on the ground surface suitable for use as a well service slurry composition.” *Id.* at 914. Independent claim 26 was amended to “incorporate the limitations previously claimed in claim 43, i.e. the oil is added to the composition prior to it being pumped into the formation. Claim 26 [was also amended] to specify that it is a method of controlling proppant flowback.” *Id.* at 915. Claims 7, 14, 30, 39, and 41 were amended to remove the indefinite phrase “typically a methyl group.” *Id.* Claims 25 and 46 were amended to remove the indefinite phrase “functionally effective amount.” *Id.*

86. The Applicant argued that “Zhang is not properly citable against the present application. The present application claims priority from the United States Provisional Application 60/924,006, which was filed April 26, 2007. Zhang was published in January 2007, i.e. within one year of the filing date of the U.S. provisional application. Accordingly, Zhang is not citable pursuant to Chapter 35 U.S. Code section 102(b).” *Id.* at 916. Moreover, the Applicant stated that “[t]he

Applicant has amended independent claim 1 to incorporate the limitation of ‘An aqueous slurry composition on the ground surface suitable for use as a well service slurry composition.’ As the Office Action notes, Zhang does not teach or fairly suggest introducing the oil into the formation. Accordingly, Zhang does not disclose, teach or fairly suggest an aqueous slurry composition on the ground surface for use as a well service fluid wherein the slurry composition comprises an oil. . . . The Applicant respectfully submits that amended claims 1 and 26, and the claims dependent directly or indirectly upon them, are not anticipated by Zhang.” *Id.* at 916-17. By amending claim 26 to add oil to the mixture to overcome the Examiner’s anticipation rejection, Trican acquiesced that the method of original claim 26 was known in the prior art. Thus, Trican acknowledged that a method comprising the steps of mixing water, particulates, and a chemical compound for rendering the surface of the particulates hydrophobic, and pumping the mixture into the formation was not patentable.

87. On November 29, 2011, the Examiner issued a final Office Action rejecting claims 1-25 and 46-52, and allowing claims 26-42, 45, and 53-59. *See generally*, Ex. 1003 at 977, Prosecution History of the ’738 Patent, Final Office Action, Nov. 29, 2011. The Examiner stated that the Applicant’s amendments to the claims overcame the § 112 rejection of claims 7-20, 25, 30, 31, 39-42, and 46. *Id.* at 978. The Examiner also withdrew the § 102 rejection over Zhang and found that

the Applicant's argument that Zhang was not prior art under 35 U.S.C. § 102(b) was persuasive. *Id.* at 979. The Examiner also rejected claims 1-25 and 46-52 under 35 U.S.C. § 112 as failing to comply with the written description requirement and as indefinite because the phrase "on the ground surface" finds no basis in the original disclosure or claims, and it is not sufficiently defined in the specification or claims such that a person of ordinary skill in the art could interpret the metes and bounds of the claim so as to understand how to avoid infringement. *Id.* at 979-80.

88. On March 29, 2012, the Applicant filed a Request for Continued Examination, amendment to the claims, and remarks in response to the November 29, 2011 Office Action. *See generally*, Ex. 1003 at 994-1007, File History of the '738 Patent, Request for Continued Examination, March 29, 2012. Applicant amended claim 1 to remove the limitation "on the ground surface suitable for use as a well service operation." *Id.* at 1006. The Applicant therefore removed the limitation intended to exclude formation oil from the composition of claim 1. The Applicant also cancelled claims 24, 25, 43, and 44. *Id.*

89. The Patent Office issued a Notice of Allowance for the '370 application on April 10, 2012 and issued as the '738 patent thereafter.

**D. The Prosecution History of the '274 Patent**

90. The named inventor for U.S. Patent 7,723,274 ("the '274 patent") is Kewei Zhang, the same named inventor for the '738 patent. The '274 patent is the

subject to pending IPR Petitions IPR2017-01807 and IPR2017-01815. Like the '738 patent, the '274 patent is directed to slurry compositions that include water, particulates, and a chemical compound for rendering the surface of the particulates hydrophobic. As-filed claim 1 of the '274 patent is representative:

1. An aqueous slurry composition comprising:
  - (a) water;
  - (b) particulates; and
  - (c) a chemical compound for rendering the surface of the particulates extremely hydrophobic.

Ex. 1014 at 25, File History of the '274 Patent, Claims.

91. The Examiner rejected various versions of this claim as unpatentable, including after amendments were made to specify that the particulates “ranged in size from 10 to 100 US mesh.” *See id.* at 267-75, File History of the '274 Patent, Non-Final Office Action, Sept. 5, 2008; *id.* at 321-29, File History of the '274 Patent, Final Office Action, July 14, 2009. However, in one of the rejections, the Examiner allowed certain claims because the prior art “failed to teach or fairly suggest . . . the slurry composition is subjected to shear in the presence of a gas.” *Id.* at 327, File History of the '274 Patent, Final Office Action, July 14, 2009.

92. On this basis, the Applicant filed a request for continued examination and amended the independent claims to include “subjecting the composition to shear in the presence of a gas” or to include that “the composition further includes a gas.”



*Id.* at 347, 349, 352, File History of the '274 Patent, Amendment and Request for Continued Examination. Thereafter, the Patent Office issued a Notice of Allowance. Thus, the purportedly novel feature of the challenged claim was the addition of gas to the composition.

**E. The Prosecution History of the '469 Application**

93. Prior to the issuance of the '738 patent, Trican filed U.S. Patent Application No. 12/759,469 (the "'469 application") on April 13, 2010. The '469 application is a continuation of the '274 patent, and thus claims priority solely to the '274 patent. Accordingly, the '469 application shares the exact same specification with the '274 patent. The named inventor for the '469 application is Kewei Zhang, the same named inventor for the '738 patent. Like the '738 patent, the '469 application is directed to slurry compositions that include water, particulates, and a chemical compound for rendering the surface of the particulates hydrophobic. *Compare, e.g.,* Ex. 1003 at 25, File History of the '738 Patent, Original Claim 26 *and* Ex. 1001, '738 patent at 3:10-15 *with* Ex. 1004 at 25, File History of the '469 Application, Original Claim 1 (illustrating that both applications originally claimed compositions without gas or oil). Consequently, as discussed below, the prosecution of the '469 application is highly relevant to the patentability of the challenged claims, particularly in light of the Examiner's observation, along with Trican's

acknowledgement of the same, that formation oil can satisfy the oil limitation in the challenge composition claims of the '738 patent.

94. In the first Office Action of May 25, 2010, the Examiner rejected claim 1 of the '469 application as anticipated by and/or as obvious over Dawson. Ex. 1004 at 40, File History of the '469 Application, Office Action, May 25, 2010. According to the Examiner, Dawson

[T]eaches methods and compositions useful for sand control and/or hydraulic fracturing of subterranean formations that utilize relatively lightweight and or substantially neutrally buoyant particles as particulate sand control or proppant material. Particles that may be employed include particulates of naturally-occurring materials that may be optionally strengthened or hardened by exposure to a modifying agent. . . . Dawson further teaches the modifying process may include exposing particles of the naturally-occurring material to a modifying agent (e.g., that includes an aqueous mixture of alkyl silanes, such as aminopropyltriethoxy silane) to strengthen the naturally-occurring material against deformation under closure stress. . . . Dawson further teaches the particle size of the particulates range from 4 mesh to about 100 mesh as instantly claimed. Examples of suitable modifying agents include compounds containing silicon-oxygen linkages. Specific examples include silane-based compounds and siloxane-based compounds as claimed instantly. . . . Since Dawson teaches the same composition as claimed, the particulates surface rendering hydrophobic upon treatment with silane and siloxanes would inherently be the same as claimed. If there is any difference between the product of Dawson

and the product of the instant claims, the difference would have been minor and obvious. ‘Products of identical chemical composition can not have mutually exclusive properties.’ A chemical compound and its properties are inseparable. Therefore, if the prior art teaches the identical chemical structure, the properties applicant discloses and/or claims are necessarily present.

Ex. 1004 at 43-45, File History of the ’469 Application, Non-Final Office Action, May 25, 2010. The Applicant attempted to distinguish Dawson by adding the limitation that the particulates were “inorganic.”

95. In response to the initial Office Action of May 25, 2010, Trican added new claim 13. Ex. 1004 at 62, File History of the ’469 Application, Amendment and Request for Consideration, Nov. 24, 2010. As seen below, claim 13 of the ’469 applications contains all of the limitations of challenged claim 17 of the ’274 patent (as illustrated by the highlighted text that points out commonality). Claim 17 of the ’274 patent includes all of the limitations of challenged claim 18 of the ’738 patent, with the exception of the oil limitation. However, both Trican and the examiner acknowledged that formation oil can satisfy the oil limitation of the challenged composition claims. Ex. 1003 at 885, File History of the ’738 Patent, Non-Final Office Action, July 8, 2011 (“Since Zhang teaches the composition being placed into the hydrocarbon oil bearing formation, it is taken by the examiner that such hydrocarbon oil is included in the composition itself”); Ex. 1001, ’738 patent at 4:11-

15 (“which can further be consolidated by oil or condensate *contained in the formation*”).

| Claim 13 of the '469 Application                                                                                                                                                                                                                                                                            | Issued Claim 17 of the '274 Patent                                                                                                                                                                                                                                             |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 13. (New) An aqueous slurry composition comprising:<br>(a) water;<br>(b) inorganic particulates ranging in size from about 10 to about 100 US mesh; and<br>(c) a chemical compound for rendering the surface of the particulates extremely hydrophobic,<br>wherein the composition further comprises a gas. | 17. An aqueous slurry composition comprising:<br>(a) water;<br>(b) particulates ranging in size from 10 to 100 US mesh; and<br>(c) a chemical compound for rendering the surface of the particulates extremely hydrophobic,<br>wherein the composition further includes a gas. |

96. Trican subsequently amended claim 13 of the '469 application several times by adding additional limitations to the claim. The amended claim included the elements shared with issued claim 17 of the '274 patent (as shown by highlighted text) until the last amendment when Trican withdrew the claim from consideration without explanation.

97. In the same response of November 24, 2010, Trican also added dependent claim 29, which depended from independent claim 13 and further specified a gas selected from a group of air, nitrogen, carbon dioxide, methane and mixtures thereof. Ex. 1004 at 67, File History of the '469 Application, Amendment and Request for Consideration, Nov. 24, 2010. Dependent claim 29 includes the

identical limitations found in challenged dependent claims 19 and 39 of the '738 patent. *Id.* Trican never relied upon the limitations of claim 29 to distinguish the prior art, nor amended the claim, relying instead on the limitations found in independent claim 13 to argue patentability.

98. On February 11, 2011, the Examiner issued an Office Action which rejected claim 13 (and claim 29) as obvious over U.S. Pat. 6,736,891 to Bice ("Bice"), U.S. Pat. 2,765,851 to Bond ("Bond") and U.S. Pat. 6,586,497 to Gay ("Gay"). *See generally* Ex. 1004 at 81-90, File History of the '469 Application, Final Office Action, Feb. 11, 2011. Examiner Admasu stated that

Bice teaches hydrophobic particulate inorganic oxide that include a reaction product of (1) hydrophobic inorganic oxide selected from the group consisting of particulate and (2) at least one organosilicon reactant in aqueous suspension. Examples of useful organosilicon compounds include 3,3,3-trifluoropropylmethyldichlorosilane, trimethylbutoxysilane and hexamethyldisiloxane. Bice further discloses that the gross particles of the hydrophobic particulate inorganic oxide may be in many forms, as for example, granules, beads and etc. when in the form of beads, the median particle size is in the range of from 150 to 350 microns. The particle size of 150-350 microns is within the ranges of claimed 10 to 100 mesh.

*Id.* at 85.

Examiner Admasu further stated that although Bice does not disclose that the chemical compound for rendering the surface of the particulates hydrophobic is

selected from the group consisting of organo-siloxane, quaternary silanes and fluoro-organic compounds, Bond “teaches the introduction of fluorocarbons into a siliceous or calcareous geological formation will facilitate the recovery of fluid hydrocarbons therefrom.” *Id.* at 85-86. The Examiner further stated that Bond teaches that “the selected fluorocarbon is introduced into the formation . . . which renders the formation surfaces hydrophobic thereby offering less resistance to the flow of the reservoir fluid with a resultant increase in production rate.” *Id.* at 86. Examiner Admasu stated that at the time of the invention, “it would have been obvious to one of ordinary skill in the art to utilize Bond’s surface active fluorocarbon as a chemical compound of Bice for rendering the surface of the particulates hydrophobic. The rationale to do so would have been the motivation provided by the teaching of Bond that to do so would facilitate the recovery of hydrocarbon fluid.” *Id.* Moreover, Examiner Admasu found that “Gay teaches a method for incorporating a foaming agent into mixtures of particulate and then forming a foam within the mixture to increase the flowability of the particulates. . . . Gay further teaches the foaming agent is injected by use of a carrier, which can be water, compressed air, or any other gas.” *Id.* at 87. The Examiner found that the rationale to combine Gay with Bice and Bond “would have been the motivation provided by the teaching of Gay that to do so would increase the flowability of the solid particulate.” *Id.*

99. In response, on July 11, 2011, Trican amended claim 13 as follows:

13. (Currently Amended) An aqueous slurry composition for use in hydraulic fracturing operations comprising:

- (a) water;
- (b) inorganic particulates ranging in size from about 10 to about 100 US mesh; and
- (c) a chemical compound for rendering the surface of the particulates extremely hydrophobic,  
wherein the chemical compound is selected from the group consisting of organo-siloxanes modified with one or more organic cationic groups, organo-siloxanes modified with one or more organic amphoteric groups, quaternary silanes, organosilane, fluoro-organosilane, fluoro-organosiloxanes and combinations thereof;  
wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, carbonate, bauxite, glass beads, shale particulates, subterranean formation particles and combinations thereof; and  
wherein the composition further comprises a gas.

*Id.*, at 115-116.

100. Among other arguments, Trican unsuccessfully tried to distinguish amended claim 13 based on the preamble’s “for use in hydraulic fracturing operations.” Ex. 1004 at 115, File History of the ’469 Application, Amendment and Request for Continued Examination, July 11, 2011. As reflected in the Background section of the ’738 patent, “[h]ydraulic fracturing operations are used extensively in the petroleum industry *to enhance* oil and gas production. In a hydraulic fracturing

operation, a fracturing fluid is injected through a wellbore into a subterranean formation at a pressure sufficient to initiate fractures *to increase* oil and gas production.” Ex. 1001, ’738 patent at 1:26-31 (emphasis added). Thus, as acknowledged by Trican, formation oil exists in the formations in which hydraulic fracturing operations are conducted. Thus, oil would have also be present in the slurry composition of amended claim 13 of the ’469 application. As indicated above, only a small amount of oil is purportedly needed for the invention of the ’738 patent. *Id.* at Abstract.

101. Examiner Admasu issued an Office Action on October 16, 2013, rejecting amended claim 13 (and claim 29) as obvious over Bice in view of Gay. *See generally* Ex. 1004 at 138-144, File History of the ’469 Application, Non-Final Office Action, Oct. 16, 2013. The Examiner based the rejection on the teachings of Bice and Gay disclosed in the February 11, 2011 rejection. Namely that Bice teaches

hydrophobic particulate inorganic oxide that include a reaction product of (1) hydrophobic inorganic oxide selected from the group consisting of particulate and (2) at least one organosilicon reactant in aqueous suspension. Examples of useful organosilicon compounds include 3,3,3-trifluoropropylmethyldichlorosilane, trimethylbutoxysilane and hexamethyldisiloxane. Bice further discloses that the gross particles of the hydrophobic particulate inorganic oxide may be in many forms, as for example, granules, beads and etc., when in the form of beads, the median particle size is in the range of from 150 to 350 microns. The



particle size of 150-350 microns is within the ranges of claimed 10 to 100 mesh.

*Id.* at 141. Moreover and consistent with the February 11, 2011 rejection, Examiner Admasu found that Gay “teaches a method for incorporating a foaming agent into mixtures of particulate and then forming a foam within the mixture to increase the flowability of the particulates. . . . Gay further teaches the foaming agent is injected by use of a carrier, which can be water, compressed air, or any other gas.” *Id.* at 142. The Examiner concluded that it would have been obvious to add the gas which is taught by Gay to Bice’s composition and that “the rationale to do so would have been the motivation provided by the teaching of Gay that to do so would increase the flowability of the solid particulate.” *Id.*

102. On March 17, 2014, in response to the Office Action, Trican submitted the following amendment to claim 13 to distinguish the cited prior art:

13. (Currently Amended) An aqueous hydraulic fracturing slurry composition for use in hydraulic fracturing operations comprising:

- (a) water;
  - (b) inorganic particulates ranging in size from about 10 to about 100 US mesh; and
  - (c) a chemical compound for rendering the surface of the particulates extremely hydrophobic,
- wherein the chemical compound is selected from the group consisting of organo-siloxanes modified with one or more organic cationic groups, organo-siloxanes modified with one or more organic amphoteric

groups, quaternary silanes, organosilane, fluoro-organosilane, fluoro-organosiloxanes and combinations thereof;

wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, carbonate, bauxite, glass beads, shale particulates, subterranean formation particles and combinations thereof; and

wherein the composition further comprises a gas.

103. Trican argued that Bice does not teach rendering of the surface of particulates hydrophobic in a hydraulic fracturing slurry composition for use in a well service operation. Ex. 1004 at 158, File History of the '469 Application, Amendment and Response, Mar. 17, 2014.

104. In the subsequent Office Action of January 2, 2015, the Examiner (Admasu) withdrew the rejection based on Bice and Gay but rejected all pending claims, including claims 13 and 29, in light of newly discovered prior art. Ex. 1004 at 180, File History of the '469 Application, Non-Final Office Action, Jan. 2, 2015. Specifically, claims 13 and 29 were rejected as being anticipated by U.S. Pat. 4,231,428 to Needham ("Needham"), U.S. Pat. App. Pub. 2004/0023818 to Nguyen ("Nguyen"), and U.S. Pat. App. Pub. 2006/0260810 to Weaver ("Weaver"). *Id.* at 182-84.

105. According to Examiner Admasu:

Needham teaches a method of propping a fracture in subterranean formations, comprising placing in the fractures particulate solids such

as sand coated with a water-repellent coating composition such as an oil-soluble organo-silicon compound. The water-repellent coated solid particulate propping agent can be prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with particulate material, and thereafter evaporating the solvent to form a coating of organo-silicon compound on the particulate material (Abstract). The water-repellent material employed to coat inert particulate materials for use as propping agents is an organo-silicon compound. Representative organo-silicon compounds that can be used include polyalkylsiloxanes such as polymethylsiloxanes, and the like. Additionally, chlorosilanes such as chlorotrimethyl silane and other silyl donors can also be used as well as various alkoxysilanes, alkoxysiloxanes and the like, as the water-repellent coating of the propping agent (column 2, lines 25-37). Needham further teaches propping agents used in hydraulic fracturing normally range in particle size from about 8 to about 60 mesh. Sand is by far the most common propping agent currently used because of its low cost and great abundance. . . . Carrier fluid, preferably an aqueous fluid such as salt water having the proppant coated with organo-silicon compound suspended therein is then pumped into the fracture.

*Id* at 182-83.

106. The Examiner also rejected claim 13 as anticipated by Nguyen because: Nguyen teaches dry coated particles with a water-repellent organo-silicon materials. The treated particles are then pumped downhole to fractures as a proppant or to the wellbore-screen annulus to function as a gravel pack ([0005]). Representative organo-silicon compounds that

can be used include polyalkylsiloxane such as polymethylsiloxanes and the like and also include alkoxysilanes, tetraethoxysilane, gammaglycidoxypyrpyltrimethoxysilanes and the like ([0006]). Fracturing or gravel packing fluids which can be utilized include gelled water or oil base liquids, foams and emulsions. The foams utilized are generally comprised of water based foams containing one or more foaming agents foamed with a gas such as nitrogen or air ([0011]).

*Id.* at 183.

107. The Examiner further rejected claim 13 as anticipated by Weaver because:

Weaver teaches method and composition of modifying a stress-activated reactivity of subterranean fracture faces and other surfaces in subterranean formation (Abstract). Weaver further teaches the surface-treating reagent may comprise compounds that are capable of hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include organosilanes, fluoroalkyl group containing silanes and the like where reagents may be present in a treatment fluid ([0047]).

*Id.* at 184.

108. Trican filed another amendment on May 4, 2015, this time amending claim 13 to read:

13. (Currently Amended) An aqueous hydraulic fracturing slurry composition for use in hydraulic fracturing operations comprising:

(a) water;

(b) inorganic particulates ranging in size from about 10 to about 100 US mesh; and

(c) a chemical compound for rendering the surface of the particulates extremely hydrophobic,

wherein the chemical compound is selected from the group consisting of ~~organo-siloxanes~~ polysiloxanes modified with one or more organic cationic groups, ~~organo-siloxanes~~ polysiloxanes modified with one or more organic amphoteric groups, quaternary silanes, organosilane, fluoro-organosilane, fluoro-organosiloxanes and combinations thereof; wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, ~~carbonate~~, bauxite, glass beads, ~~shale particulates~~, ~~subterranean formation particulates~~ and combinations thereof; and

wherein the composition further comprises a gas; and

wherein the hydraulic fracturing slurry composition can suspend the particulates under static and dynamic conditions without viscosifying the slurry composition.

109. Trican attempted to distinguish Nguyen by asserting that “Nguyen teaches the use of a gel-polymer in the fracturing fluid (i.e., the fluid is viscosified in order to transport the proppant). The slurry compositions used in the hydraulic fracturing disclosed and claimed in the present application do not require the use of viscosifiers for transporting particulates into the fractures.” Ex. 1004 at 207-08, File History of the ’469 Application, Amendment and Response, May 4, 2015. Trican also asserted “[a]s mentioned above, in the present invention, the proppants are not

suspended by viscosifying the fluid, but instead, by attaching small bubbles to the proppant surfaces increasing their buoyancy (see attached photographs).” *Id.* at 208.

110. By amending claim 13, Trican acquiesced to Examiner Admasu’s rejection that the March 17, 2014 version of claim 13 was anticipated by Needham and Nguyen. Trican twice asserted that “the subject matter of ***amended*** claims 13 [and 17-29] is novel over Needham.” Ex. 1004, 205-07. Similarly, Trican twice asserted that “the subject matter of ***amended*** claims 13 [and 29] is novel over Nguyen.” See Ex. 1004 at 207-08, File History of the ’469 Application, Amendment and Response, May 4, 2015. Contrast this to Trican’s assertions that “[t]he Applicant respectfully disagrees with the Office Action’s conclusion that Bernard anticipates claims 1, 4 and 6-11” (*Id.* at 201, File History of the ’469 Application, Amendment and Response, May 4, 2015) and “[t]he Applicant respectfully disagrees with this conclusion [the rejection of claims 13 and 17-39 as being anticipated by Weaver (because Weaver was not 102(b) prior art)]” (*Id.* at 203, 209). In response to other rejections during the prosecution of the ’469 application, Trican repeatedly indicated when it disagreed with the rejection. See *e.g.*, Ex. 1004 at 124, File History of the ’469 Application, Amendment and Request for Continued Examination, July 11, 2011 (“The Applicant respectfully disagrees with the Office Action’s conclusion that claims 1, 5 and 10-12 would have been obvious over Bice in view of Bond.”). In other responses, Trican stated “any amendments made herein should not be taken

as an admission that the prior art teaches any part or element of the claims as previously presented.” *See e.g.*, Ex. 1004 at 367, File History of the ’469 Application, Amendment and Response, Oct. 26, 2016. However, no such statements were made before amending claim 13 above. Thus, Trican’s own words and prior conduct suggest that it acknowledged that Needham and Nguyen anticipated the March 17, 2014 version of claim 13.

111. Like previous versions, this version of claim 13 includes virtually every limitation of challenged claim 17 of the ’274 patent as shown in the highlighted text above. The only differences were insubstantial—slightly modifying the mesh size by “about” and substituting “comprises” for “includes” before “a gas.”<sup>4</sup> Furthermore, claim 13 includes every limitation of challenged claim 18 of the ’738 patent, except the “oil” limitation. However, one of ordinary skill in the art would know that formation oil would be mixed with the composition when the slurry was injected into the formation during hydraulic fracturing operations.

112. The Examiner issued a restriction requirement in response to the May 4, 2015 amendment, after which Trican elected to prosecute claim 1 (directed to a composition that did not include a gas) and amended claim 13 to depend from claim 1 to add the gas limitation to the slurry composition. *See generally* Ex. 1004 at 276,

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<sup>4</sup>The Examiner in the Feb. 11, 2011 Office Action indicated that he interpreted “includes” as being synonymous with “comprising,” citing MPEP 2113.03 and *Mars Inc. v H.J. Heinz Co.*, 377 F.3d 1369, 1376 (Fed. Cir. 2004).

File History of the '469 Application, Response to Election Requirement, Dec. 3, 2015.

113. Claim 1 reads as follows:

1. An aqueous hydraulic fracturing slurry composition for use in hydraulic fracturing operations comprising:

- (a) water;
- (b) inorganic particulates ranging in size from about 10 to about 100 US mesh; and
- (c) a chemical compound for rendering the surface of the particulates extremely hydrophobic, wherein the chemical compound is selected from the group consisting of polysiloxanes modified with one or more organic cationic groups, polysiloxanes modified with one or more organic amphoteric groups, quaternary silanes, and combinations thereof. (deletions omitted)

114. Amended claim 13 reads:

13. (amended) The aqueous hydraulic fracturing slurry composition of claim 1 further comprising a gas. (deletions omitted)

115. As seen above, amended claim 13 includes all of the limitations of challenged claim 17 of the '274 patent, and every limitation of claim 18 of the '738 patent when used for the claimed hydraulic fracturing operations due to the presence of formation oil.

116. In the subsequent Office Action, Examiner Bland rejected claim 1 and its dependent claims, including claim 13, as obvious over Nguyen and U.S. Pub.



App. 2004/0023818 to Herzig (“Herzig”). *See* Ex. 1004 at 343-44, File History of the ’469 Application, Non-Final Office Action, July 26, 2016. The Examiner stated that “the limitations ‘aqueous hydraulic fracturing slurry’ and ‘for use in’ are the future intended use of the composition. A recitation of the intended use of the claimed invention must result in a structural difference between the claimed invention and the prior art in order to patentably distinguish the claimed invention from the prior art. If the prior art structure is capable of performing the intended use, then it meets the claim.” *Id.* at 344.

117. In response to the Office Action, Trican withdrew claim 13 without explanation.

118. In sum, despite multiple attempts, Trican was unable to create a patentable claim based on the limitations contained in claim 17 of the ’274 patent, or challenged claim 18 of the ’738 patent. The independent claim 13 submitted in the ’469 application could not survive anticipation rejections under Nguyen or Needham after the Examiner cited those references. The key elements to claim 13 in the ’469 application are exactly those found in challenged claim 18 of the ’738 patent. There is nothing significantly distinguishable between the rejected versions of claim 13 of the ’469 application and challenged claim 18 of the ’738 patent.

119. Trican had five separate statements from the USPTO indicating that claims that included all of the limitations of claim 17 of the ’274 patent were

unpatentable for anticipation and obviousness. Each time, Trican acquiesced to the fact that claim 13 was unpatentable by further amending the claim. Thus, Trican admitted by its actions during the prosecution of the '469 application that a slurry composition comprising water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic, and a gas, along with the method of making this composition, were known in the art and thus not patentable.

120. In summary, the following facts are evident from the prosecution histories of the '738 patent, the '274 patent and the '469 application:

- That a method comprising the steps of mixing water, particulates, and a chemical compound for rendering the surface of the particulates hydrophobic and pumping the mixture into the formation was acknowledged as unpatentable by Trican. *See* Ex. 1003 at 25, File History of '738 Patent, Original Claim 26 of the '370 Application; *Id.* at 885, Non-Final Office Action, July 8, 2011; *Id.* at 910, Response and Amendment, Nov. 8, 2011.
- That formation oil satisfies the “oil” limitation of challenged claim 1 of the '738 patent.<sup>5</sup> *See id.* at 885, File History of the '738 Patent, Non-Final Office Action, July 8, 2011; *see also id.* at 979-80, Final Office

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<sup>5</sup> Moreover the specification of the '738 patent states that only a “small amount of an oil” is required for the purported invention. Ex. 1001, '738 patent at 3:65.

Action, Nov. 29, 2011 (Examiner rejecting the claims under 35 U.S.C. § 112 as failing to comply with the written description requirement because the limitation “‘on the ground surface’ finds no basis in the original disclosure or claims.”); *Id.* at 997, Request for Continued Examination, Mar. 29, 2011 (Trican’s amendment to the claims removing the limitation “on the ground surface”).

- That a slurry composition comprising water, particulates, and a chemical compound for rendering the surface of the particulates hydrophobic was acknowledged as unpatentable by Trican and the Examiner during the prosecution of the ’274 patent. *See* Ex. 1014 at 267-75, File History of the ’274 Patent, Non-Final Office Action, Sept. 5, 2008; *Id.* at 321-29, Final Office Action, July 14, 2009.
- That a slurry composition comprising water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic and a gas was acknowledged as unpatentable by Trican and the Examiner during the prosecution of the ’469 application. *See, e.g.*, Ex. 1004 at 40, File History of the ’469 Application, Office Action, May 25, 2010; *Id.* at 81-90, File History of the ’469 Application, Final Office Action, Feb. 11, 2011; *Id.* at 138-144, File History of the ’469 Application, Non-Final Office Action, Oct. 16, 2013; *Id.* at 180-84, File History of

the '469 Application, Non-Final Office Action, Jan. 2, 2015; *Id.* at 343-44, File History of the '469 Application, Non-Final Office Action, July 26, 2016.

- That slurry composition comprising water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic and a gas, wherein the gas is selected from a group consisting of air, nitrogen, carbon dioxide, methane and mixtures thereof was acknowledged as unpatentable by Trican and the Examiner during the prosecution of the '469 application. *See, e.g.*, Ex. 1004 at 40, File History of the '469 Application, Office Action, May 25, 2010; *Id.* at 81-90, File History of the '469 Application, Final Office Action, Feb. 11, 2011; *id.* at 138-144, File History of the '469 Application, Non-Final Office Action, Oct. 16, 2013; *Id.* at 180-84, File History of the '469 Application, Non-Final Office Action, Jan. 2, 2015; *Id.* at 343-44, File History of the '469 Application, Non-Final Office Action, July 26, 2016.

## **VIII. STATE OF THE ART**

121. All of the various features and elements of the '738 patent were well known in the art at the time of and prior to its priority date of April 2007.

122. For purposes of my analysis all of the cited references are prior art to the '738 patent. This is because each of the cited references have U.S. filing dates or foreign publication dates well before the priority date of the '738 patent. Nguyen has a filing date of August 5, 2002, and a publication date of February 5, 2004. Dawson has an effective filing date of April 1, 2002, and a publication date of October 1, 2003. Vincent has an effective filing date of April 12, 2004, and a publication date of November 3, 2005. Needham has a filing date of December 4, 1978 and issued on November 4, 1980. Weaver has an effective filing date of May 20, 2005, and a publication date of November 23, 2006. Plummer has a a filing date of April 5, 1974 and issued on September 14, 1976. Therefore, all of these references are prior art to the '738 patent because they were all available to the public at least one year before April 26, 2007.

**A. Overview of Nguyen**

123. Nguyen is a published patent application that was abandoned after a final rejection in which the examiner rejected the claims as obvious over Needham in view of other references. The Examiner found that “Needham teaches coating particles with the same organo-silicon compounds and same method of the present invention. . . . Needham teaches that various siloxanes, silanes and silicates may be used to coat the particles.” Ex. 1016, File History of Nguyen, Final Office Action, Sept. 21, 2005 at 2.

124. Nguyen teaches a method to promote and increase hydrocarbon recovery during hydraulic fracture stimulation in which particulates coated with a water-repellent organo-silicon composition, mixed with a carrier fluid, and pumped downhole into an oil and gas well. *See* Ex. 1005, Nguyen at Abstract, [0001], [0005], claim 1, claim 63.

125. Nguyen discloses “[a]n example of the technique for applying the coating on the particles involves dissolving an oil-soluble organo-silicon compound in a solvent, admixing or spraying the resultant solution on the particles, and then evaporating the solvent to form a thin film of siloxane or silane encapsulating the particles. The organo-silicon organo-silane compound is readily absorbed onto the particles from the solvent and the solvent is easily evaporated by drying.” *Id.* at [0007]; *see also* claim 4.

126. Nguyen further teaches that the coated particles “are then pumped downhole to the fractures to function as a proppant or to the wellbore-screen annulus to function as a gravel pack.” *Id.* at [0005].

127. Nguyen teaches that “[v]arious kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like.” *Id.* at [0016].

128. Nguyen further teaches that “[r]epresentative organo-silicon compounds that can be used in this embodiment include polyalkylsiloxane such as

polymethylsiloxanes, polyethylsiloxanes, and the like. Additional organo-silicon compounds that can be used in this embodiment include polyalkylarylsiloxanes . . . chlorosilanes such as ethylchlorosilane, **chlorotrimethylsilane** and other silyl donors. Also, various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes can be used such as tetraethoxysilane, **dimethoxydiphenylsilane**, **dichlorodimethylsilane**, **dichlorodiphenylsilane**, poly[oxy(dimethylsilylene)] and other such materials that will be well known to those skilled in the art. . . . Furthermore, organofunctional silanes can be used in this embodiment.” *Id.* at [0006].

129. Nguyen teaches that “the particles are usually introduced downhole in a carrier fluid.” *Id.* at [0003]. Nguyen further teaches that “fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and emulsions.” *Id.* at [0011]. Nguyen also teaches that “the water utilized to form the fracture fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other aqueous liquid.” *Id.* at [0013]. Moreover, Nguyen teaches that “[o]rganic solvents that can be used as a carrier for the organo-silicon coating material include kerosene, lighter grades of diesel fuel, hexane, xylene, toluene, dipropylene glycol methyl ether . . . and other such solvents that will be well known to those skilled in the art.” *Id.* at [0008].

130. Nguyen additionally teaches that if a foam is used, “the foams utilized are generally comprised of water based liquids containing one or more foaming agents foamed with a gas such as nitrogen or air.” *Id.* at [0011].

131. Nguyen further teaches that “preferred particles are sand having a particle size in the range of from about 10 to about 70 mesh, U.S. Sieve Series.” *Id.* at [0016].

132. Nguyen also discloses that “coating of the particles in the above manner helps to prevent, or at least minimize, the coating, or adsorption, of the above-mentioned gel polymer from the carrier fluid on the surfaces of the particles. Thus, gel polymer or its residue is readily removed from the system during cleanup or flowback of the well. This greatly enhances the conductivity of the particles when used as a proppant and the permeability of the particles when used as a gravel pack.” *Id.* at [0010].

#### **B. Overview of Dawson**

133. Dawson teaches “methods and compositions useful for . . . hydraulic fracturing of subterranean formations that utilize relatively lightweight and/or substantially neutrally buoyant particles as . . . proppant material.” Ex. 1006, Dawson at Abstract. Dawson discloses that his slurry will “provide for substantially improved overall system performance in hydraulic fracturing applications, or in other well treating applications such as sand control.” *Id.*, Dawson at 3:15-16.



134. Dawson teaches that particles “may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material.” *Id.* at 7, l. 3-8. Dawson states that the “[m]odifying agents may be *diluted as so desired in a suitable solvent prior to treating particles* of a naturally-occurring material.” *Id.* at 27, l. 1-2. Dawson further discloses that “[s]pecific examples of *suitable exposure methods* include, but are not limited to, by *soaking* the naturally-occurring material in a modifying agent . . . by *dipping* the naturally-occurring material in a modifying agent, by *spraying* a modifying agent on to the naturally-occurring material . . . by *mixing* naturally-occurring particulate material with liquid, atomized and/or gaseous modifying agent in a vertical mixer . . . by *flowing* modifying agent through a permeable bed or pack of the naturally-occurring particles in a pressurized container. *Drying of particles after exposure* may be accomplished, for example, by continuously dropping through heated air.” *Id.*, at 24, l. 17-26.

135. Specifically, Dawson discloses that “examples of suitable modifying agents include, but are not limited to, agents including polyisocyanates, silanes, siloxanes, and combinations thereof. Selected modifying agent/s may be advantageously used to increase the elastic modulus of a given naturally occurring material, for example, to make particles of a given naturally-occurring material more suitable . . . for use as a relatively lightweight and/or substantially neutrally buoyant

fracture proppant or sand control particulate material having increased effectiveness when exposed to higher closure stresses or other mechanical stresses that may be encountered downhole during a well treatment such as hydraulic fracturing or sand control treatment.” *Id.* at 7, l. 14-23. Sand is a naturally occurring material.

136. Moreover, Dawson incorporates by reference U.S. Patent 6,330,916 to Rickards (“Rickards”). *See* Ex. 1006, Dawson at 13, l. 3-4 (“Additional information on materials and methods for use thereof may be found in U.S. Pat. No. 6,330,916, which is incorporated herein by reference.”). Rickards teaches subterranean fracturing treatments that use deformable particles mixed with fracturing proppants to minimize proppant flowback. Ex. 1011, Rickards at 1:17-22. Rickards also teaches that the “fracture proppant material comprises at least one of silica, resin-coated sand, ceramic particles, synthetic organic particles, glass microspheres, sintered bauxite, or a mixture thereof.” *Id.* at 43:10-13. Specifically, Rickards teaches that in one embodiment, “sand is used as [the] fracture proppant.” *Id.* 12:47-48.

137. Dawson teaches that the modifying agent may contain a carbon chain with “one or more halogen groups (e.g., tetrachlorosilane, *methyltrichlorosilane*, etc.).” *Id.* at 21, l. 10-11. Dawson additionally teaches, examples including

commercially available silane-based products available from CROMPTON include CoatOSil<sup>®</sup> 1706 Silane (vinyl silane monomer), CoatOSil<sup>®</sup> 1757 Silane, CoatOSil<sup>®</sup> 1770 Silane, EURENOR<sup>®</sup> 5020

(liquid isobutylisopropylmethoxysilane), EURENOR<sup>®</sup> 5021 (liquid diisopropylmethoxysilane), EURENOR<sup>®</sup> 5022 (liquid diisobutylmethoxysilane), EURENOR<sup>®</sup> 5023 (liquid dicyclopentylmethoxysilane), Silcat<sup>®</sup> R Silane (vinylsilane with grafting and cross linking catalysts), Sikat<sup>®</sup> RHE Silane (cross linking system of silane, peroxide and catalyst), Silcat<sup>®</sup> RHS Silane (crosslinking system of silane, peroxide catalyst and antioxidants), Sikat<sup>®</sup> VS-735/1 Silane (crosslinking system of silane, peroxide, antioxidants and metal deactivator), Silcat<sup>®</sup> VS-758/0 Silane (stabilized cross linking system of silane, peroxide and catalyst), Sikat<sup>®</sup> VS-870 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat<sup>®</sup> VS-928 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat<sup>®</sup> VS-963 Silane (stabilized cross linking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Silox<sup>®</sup> VS- 911 Silane (liquid silane system), Silquest<sup>®</sup> A-1100 Silane (gammaaminopropyltriethoxysilane), Silquest<sup>®</sup> A-1101 Silane (technical grade amino silane), Silquest<sup>®</sup> A-1102 Silane (technical grade amino silane), Silquest<sup>®</sup> A-1110 Silane (primary amino silane), Silquest<sup>®</sup> A-1120 Silane, Silquest<sup>®</sup> A-1126 Silane (amino silane), Silquest<sup>®</sup> A-1128 Silane (amino silane), Silquest<sup>®</sup> A-1160 Silane (ureido silane), Silquest<sup>®</sup> A-1170 Silane (secondary amino functional silane), Silquest<sup>®</sup> A-1289 Silane, Silquest<sup>®</sup> A-1310 Silane, Silquest<sup>®</sup> A-137 Silane (monomeric alkylalkoxysilane), Silquest<sup>®</sup> A-151 Silane, Silquest<sup>®</sup> A-1524 Silane (100-percent active ureidosilane), Silquest<sup>®</sup> A-1589 Silane (disulfide silane), Silquest<sup>®</sup> A-171 Silane, Silquest<sup>®</sup> A-172 Silane (vinylfunctional coupling agent), Silquest<sup>®</sup> A-174 Silane,

Silquest<sup>®</sup> A-186 Silane (epoxy functional silanes), Silquest<sup>®</sup> A-187 Silane (epoxy functional silanes), Silquest<sup>®</sup> A-189 Silane, Silquest<sup>®</sup> A-2171 Silane (silane compound having vinyl and silane functionality), Silquest<sup>®</sup> Alink 15 Silane (secondary amino silane endcapper), Silquest<sup>®</sup> RC-1 Silane (organosilicone coupling agent), Silquest<sup>®</sup> RC-2 Silane, Silquest<sup>®</sup> Wetlink 78 Silane, Silquest<sup>®</sup> Y-11637 Silane (primary aminosilane), Silquest<sup>®</sup> Y-11683 Silane (di-, tri-functional ethoxy silane), Silquest<sup>®</sup> Y-5997 Silane, XL-PEarl<sup>®</sup> 31 Blend, XL-PEarl<sup>®</sup> 50 Blend, XLPEarl<sup>®</sup> 51 Blend, XL-PEarl<sup>®</sup> 52 Blend, XL-PEarl<sup>®</sup> 60 Blend, XL-PEarl<sup>®</sup> 70 Blend, *etc.*

*Id.* at 21, l. 19- 22, l. 23.

138. Dawson further teaches that the particulates “may be mixed and pumped during any desired portion/s of a well treatment (e.g., hydraulic fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier fluid.” *Id.* at 13, l. 8-10. Accordingly, “any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water, liquid hydrocarbons, and/or nitrogen or other gases.” *Id.* at 13, l. 10-13.

139. Dawson also teaches that his “invention relates generally to methods and compositions useful for subterranean formation treatments, such as hydraulic fracturing treatments and sand control. In particular, this invention relates to use of

relatively lightweight and/or substantially neutrally buoyant particles as proppant materials in hydraulic fracturing treatment and as ***particulate material in sand control methods*** such as gravel packing, ***frac pack treatments, etc.***” Ex. 1006, Dawson at 2, l. 4-8. Dawson also states that his invention “may be directed toward improving wellbore productivity and/or ***controlling the production of fracture proppant*** or formation sand. Particular examples include gravel packing and ‘frac-packs.’” *Id.* at 13, l. 23-25.

140. Moreover, Dawson teaches that “it will be understood with the benefit of this disclosure that the disclosed particulate materials may be used with any method in which a pack of particulate material is formed within a wellbore that it is permeable to fluids produced from a wellbore (*e.g.*, oil, gas, water, etc.) but that substantially prevents or reduces production of formation materials (*e.g.*, formation sand, etc.) from the formation into the wellbore.” *Id.* at 14, l. 31- 15, l. 4.

141. Dawson also incorporates by reference U.S. Patent 3,659,651 to Graham (“Graham”) (Ex. 1012). See Ex. 1006, Dawson at 33, l. 6-8. Graham teaches “[i]n placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or water-in-oil emulsion prepared in accordance with the

teachings of U.S. Pat. No. 3,378,074.” Ex. 1012, Graham at 5:9-16 (citing U.S. Patent 3,378,074 to Kiel at 5:65-72 (Ex. 1013) (disclosing that “high viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either water-in-oil or oil-in-water emulsions. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine or an acid solution containing a suitable surface active agent or emulsifier.”)).

### C. Overview of Vincent

142. Vincent is a published patent application that was abandoned after a final rejection in which the examiner rejected the claims as anticipated by Nguyen. The Examiner found that

Nguyen et al teach particles coated with a water-repellant organo-silicon material including polyalkylsiloxanes such as polymethylsiloxanes and polydimethylsiloxane as well as other silicon resins, wherein the treated particles are then pumped downhole to the fractures to function as a proppant in a carrier fluid or to the wellbore-screen annulus to function as a gravel pack, greatly enhancing the conductivity or permeability, respectively (Abstract; Paragraphs 0004-0006 and 0010.) Nguyen et al teach that the organo-silicon compound can be applied to the particles by dissolving an oil-soluble organo-silicon compound in a solvent, admixing or spraying the resultant solution on the particles, and then evaporating the solvent to form a thin

film of siloxane or silane encapsulating the particles; thereby dry coating the particles (Paragraph 0007; Claims 1 and 25.) Nguyen et al further teach that if the particles are coated when the particles are flowing, then the solvent should be readily miscible in the water-based gel carrier fluid, and that as a non-limiting example, approximately 0.01 % to 3% of organo-silicon by weight of particles can be used to coat onto the particles (hence no drying step and the coated particles are wet upon mixing with the carrier fluid; Paragraph 0008; Claims 63 and 75.) Nguyen et al teach that the proppant particles can be sand, bauxite, and ceramic, and generally have a particle size in the range from about 2 to about 400 mesh, with preferred particles being sand having a particle size of about 10 to about 70 mesh, hence inherently having a surface porosity (Paragraph 0016.) Though Nguyen et al do not specifically teach the bulk density, crush strength and apparent specific gravity of the particles, the Examiner takes the position that the particles taught by Nguyen et al would inherently possess the same properties considering the particles are formed from the same materials.

Ex. 1015, File History of Vincent, Non-Final Office Action, Feb. 27, 2007 at 2-3.

143. Vincent teaches surface-modified proppants with altered wettability for use in oil and gas well hydraulic fracturing of subterranean formations. See Ex. 1007, Vincent at Abstract.

144. Vincent discloses “modifying the surface properties of natural sand, resin-coated sand, and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability, alter the

chemical reactivity, alter the surface topography, impart lubricity, and control relative permeability to flow of fluids such as proppants. Sands, resin coated sands, or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, ***to modify their wettability*** or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.” *Id.* at [0008].

145. Vincent teaches that the above benefits can be achieved by a variety of techniques, including by “coating the proppants with ***a hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes.***” *Id.* at [0009].

146. Vincent teaches that the coatings “affect wettability and provide significant flow benefits under multiphase flow as evidenced by the trapped gas saturation, the altered surface tension/contact angles, and the electrostatic charges on the coated proppant.” *Id.* at [0029].

147. One example taught in Vincent includes coating a proppant with Rain-X<sup>®</sup>, a glass surface treatment material that includes polyalkyl hydrogen siloxane (*id.* at [0043]). *See id.* at [0082]. It was observed that the Rain-X<sup>®</sup> coated proppant trapped air bubbles that accumulated over time and it was clear that the proppant was hydrophobic. *Id.* Vincent teaches that in the presence of a gas well, this coated



proppant “would be expected to preferentially produce a natural gas, while hindering the flow of water which would provide a tremendous economic benefit.” *Id.*

148. Vincent also teaches the use of hydrophobic materials including “silicon containing compounds, including silicone materials and siloxanes, polytetrafluoroethylene (commonly known as Teflon<sup>®</sup>), plant oils, such as linseed oil, soybean oil, corn oil, cottonseed oil, vegetable oil (widely commercially available such as Crisco<sup>®</sup>), and canola oil, and **hydrocarbons such as kerosene, diesel, and crude oil, petroleum distillates such as hydrocarbon liquids comprising a mixture of C<sub>7</sub> -C<sub>12</sub> aliphatic and alicyclic hydrocarbons and aromatic hydrocarbons (C<sub>7</sub>-C<sub>12</sub>)**” (*id.* at [0009]) as well as the specific polysiloxanes “polymethylhydrogen siloxane and polydimethyl siloxane” (*id.* at claim 6).

149. Vincent discloses “pretreating proppant prior to the fracturing treatment.” *Id.* at [0024]. For example, Vincent discloses that the particles can be coated by “**mixing the proppant and the coating** in a beaker for approximately 30 minutes, **then drying it** for approximately 15 to 18 hours in an oven.” *Id.* at [0036]. One example disclosed in Vincent states that “[t]he coating materials were added as follows. Polymethylhydrogen siloxane was added as either a 2 or 5 weight percent **emulsion of siloxane in water**, polydimethyl siloxane was added as a 5 weight percent emulsion of siloxane in water and **Stoddard Solvent** was added without

water dilution. *All samples were dried* at 113° C. for approximately 15 to 18 hours.” *Id.* at [0037].

150. Vincent further teaches that these “chemically coated and/or treated particles of the present invention are useful as propping agent in methods of fracturing subterranean formations to increase the permeability thereof.” *Id.* at [0085]. Vincent discloses that to use the particles as a propping agent in hydraulic fracturing, “the particles may be handled in the same manner as other propping agents. The particles may be delivered to the well site in bags or in bulk form along with the other materials used in fracturing treatment. Conventional equipment and techniques may be used to place the particles as a propping agent.” *Id.* at [0086]. More specifically, Vincent describes that “particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.” *Id.* at [0087]. The fracture fluid taught by Vincent “may be an oil base, water base, acid, emulsion, foam or any other fluid.” *Id.*

#### **D. Overview of Weaver**

151. Weaver teaches a method of coating a plurality of particulates with a surface-treating reagent to modify the surface stress-activated reactivity of the particulate that may be used in a subterranean operation, including in fracturing operations. See Ex. 1008, Weaver at Abstract.

152. Weaver discloses that one way to modify the surface stress-activated reactivity of a particulate is by “depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic.” *Id.* at [0015].

153. Weaver teaches that one “suitable method[] for allowing the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate includes . . . solvent extraction methods.” *Id.* at [0057]. In such an embodiment, “[t]he solution comprising the surface treating reagent then may be coated onto the mineral surface of the particulates by ***adding the solution to the particulates and stirring*** (or mulling) until the mixture is uniform. The treated particulates then ***may be introduced to a water-based fluid that extracts the solvent from the treated particulates***, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface.” *Id.*

154. Weaver teaches that “[s]uitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, [and] Teflon<sup>®</sup> materials.” *Id.* at [0018].

155. Weaver also discloses that “[s]uitable stress-activated reactivity-reducing reagents include, but are not limited to, resins, tackifying agents, and other substances that are capable of hindering or preventing the water-wetting of the mineral surface.” *Id.* at [0022]. Specifically, Weaver discloses that “[e]xamples of suitable compounds include, but are not limited to . . . organosilanes.” *Id.* at [0049].

156. Weaver teaches that in some embodiments, “the surface-treating reagent may comprise a chlorosilyl group containing a compound and an alkylsilane, or the reaction products thereof. In some embodiments, the surface-treating reagent may comprise an organofunctional silane and an aryl acid halide, or the reaction products thereof. Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.” *Id.*

157. Weaver discloses that “particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water [or] salt water . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water

solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.” *Id.* at [0058]. With respect to the emulsions, Weaver specifically discloses that “[s]uitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.” *Id.* at [0059].

158. Moreover, Weaver discloses that foams can be used as the treatment fluid and “can be created by the addition of a gas, such as carbon dioxide or nitrogen.” *Id.*

159. Weaver further discloses that his invention can be used “in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, ***frac-packing operations***, perforation operations, and the like.” Ex. 1008, Weaver at [0058].

#### **E. Overview of Needham**

160. Needham teaches a “method of fracturing an underground zone or formation in oil and gas wells” (Ex. 1009, Needham at 4:1-2) “compris[ing] placing in the fractures particulate solids such as sand coated with a water-repellent coating composition such as an oil-soluble organo-silicon compound” (*id.* at Abstract).

161. Needham teaches “[i]n accordance with a specific embodiment, sand coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, ***admixing the resultant solution with***

*sand, and thereafter evaporating the solvent to form a silicon-coated sand* which can then be used as a propping medium in fractured formations containing hydrocarbons.” *Id.* at 1:61-68; *see also id.* at 2:61-66.

162. Needham discloses that the organo-silicon coating can be useful as a water-repellent for use in hydraulic fracturing operations (*id.* at 1:47-49) and also be useful to increase the strength of the propping agent in the presence of water (*id.* at 1:50-52).

163. Needham teaches that “[r]epresentative organo-silicon compounds that can be used include polyalkylsiloxanes such as ***polymethylsiloxanes***, polyethylsiloxanes, and the like. Additionally, polyalkylarylsiloxanes such as polymethylphenylsiloxane are also usable as coating material of the invention. Chlorosilanes such as ***chlorotrimethylsilane*** and other silyl donors can be used, as well as various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes, and the like, as the water-repellent coating of the propping agent.” *Id.* at 2:28-37.

164. Needham discloses that for use in hydrocarbon-bearing well treatments, “it is preferred that the organo-silicon compound be applied as a coating to a particulate substrate” (*id.* at 2:59-61) and specifically discloses that in hydraulic fracturing, “[s]and is by far the most common propping agent currently used because of its low cost and great abundance. However, under certain conditions light strength

glass beads, as well as bauxite (aluminum oxide), or other propping agents can be used” (*id.* at 3:44-52). *See also id.* at 4:16-17; 4:22-23; 4:48-49; 4:53-54.

165. Moreover, Needham teaches that “[c]arrier fluid, preferably an aqueous fluid such as salt water or a hydrocarbon liquid such as diesel fuel, having the proppant coated with organo-silicon compound suspended therein is then pumped into [a] fracture.” *Id.* at 3:19-23.

166. Thus, Needham teaches “[a] method of treating fractured earthen formations in oil and gas wells comprising the steps of (a) ***mixing together a quantity of particulate solids suitable for use as propping agents and a quantity of an oil-soluble organo-silicon compound dissolved in a solvent and heating the resulting mixture to a temperature sufficient to evaporate said solvent*** and leave said solids coated with an oil-wettable coating of said organo-silicon compound, (b) ***dispersing said coated particulate solids into a carrying fluid***, (c) ***introducing said carrying fluid and dispersed coated solids into a well penetrating a subterranean formation containing fractures***, and (d) applying pressure to the fluid in the well thereby causing the fluid to contact the earthen formations and to be injected into fractures and openings extending into the formation whereby said coated particles are deposited therein. *Id.* at 4:29-47.

## F. Overview of Plummer

167. Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract.

168. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a liquid carrier comprising **one or more liquids** selected from the group consisting of **water and oil**, and a solid proppant comprising one or more materials selected from the group consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that “the liquid component of said foam comprises **at least** one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Plummer also teaches

169. Plummer discloses that “[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling . . . Other friction reducing materials may be added to the fracturing fluid.” *Id.* at 4:28-33.

## IX. OPINIONS ON CLAIM CONSTRUCTION

### A. A Chemical Compound for Rendering the Surface of the Particulates Hydrophobic

170. Claim 1 of the ’738 patent includes the limitation “a chemical compound for rendering the surface of the particulates hydrophobic.” Independent method claim 32 includes the same limitation.



171. A person of ordinary in the art would consider the broadest reasonable construction of this limitation would be “a chemical compound capable of making particulates hydrophobic.” Support for this construction is found in the specification of the ’738 patent.

172. As stated in the ’738 specification, “[t]here are different ways of carrying out the invention. For example, during a fracturing operation, a proppant, for example, sand, which is naturally hydrophilic and can be easily water wetted, is mixed with a fluid containing a chemical agent referred to as a hydrophobizing agent, which *makes* the sand surface hydrophobic.” Ex. 1001, ’738 patent at 3:46-51 (emphasis added).

173. The specification also states that “[t]here are various types of hydrophobizing agents for sand which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” *Id.* at 4:62-67. The specification provides enumerated examples of organosilanes that can be used to make the particulates hydrophobic. *See id.* at 5:66-6:43.

174. The above construction is also consistent with the dictionary definition of “render,” which means “to cause to be or become: make.” Webster’s Third New International Dictionary (2002).

**X. OPINIONS ON VALIDITY OF CLAIMS 1, 21, 22, 32, 40, 41, 51 AND 57 OF THE '738 PATENT UNDER 35 U.S.C. §102**

**A. Claims 1, 21, 22, 32, 40, 41, 51, and 57 Are Anticipated by Nguyen**

175. After considering the Nguyen reference as it would have been understood by a person of ordinary skill in the art in April 2007, it is my opinion that Nguyen discloses each and every element of claims 1, 21, 22, 32, 40, 41, 51 and 57 of the '738 patent. *See generally*, Ex. 1002C, '738 Patent v. Nguyen Claim Chart.<sup>6</sup> Therefore, it is my opinion that Nguyen anticipates claims 1, 21, 22, 32, 40, 41, 51 and 57 of the '738 patent.

**1. Independent Claim 1 Is Anticipated by Nguyen**

176. Independent claim 1 of the '738 patent is a composition claim with four limitations.

177. Claim 1 of the '738 patent recites:

An aqueous slurry composition comprising:

water;

particulates;

a chemical compound for rendering the surface of the particulates hydrophobic; and

an oil.

178. As set forth below on a limitation-by-limitation basis, Nguyen anticipates independent claim 1 of the '738 patent.

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<sup>6</sup>Exhibits 1002C-K are claim charts that are supportive of my opinions herein.

a. **Preamble for Claim 1: “An aqueous slurry composition comprising”**

179. The preamble of claim 1 of the '738 patent states, “An aqueous slurry composition comprising.” Ex. 1001 at 9:46. Nguyen recites “mixing particles in a carrier fluid comprising a water-based polymer to form a mixture.” Ex. 1005, Nguyen at claim 63. Nguyen also states that “[t]he water utilized to form the fracturing fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other aqueous liquid which does not adversely react [to] other components of the fracturing fluid.” *Id.* at [0013]. Thus, Nguyen discloses and aqueous slurry. Therefore, in my opinion, the preamble of claim 1 is met by Nguyen.

b. **Limitation 1A: “water”**

180. Claim 1 recites that the composition comprises “water.” Ex. 1001 at 9:47. Nguyen discloses that “[t]he most common fracturing fluid utilized heretofore which is generally preferred for use in accordance with this invention is comprised of water.” Ex. 1005, Nguyen at [0012]. Nguyen further discloses, in claim 63, “mixing particles in a carrier fluid comprising a water-based polymer to form a mixture.” *Id.* at claim 63. Nguyen further teaches “[t]he method of claim 63 wherein the carrier fluid comprises water and a gelling agent.” *Id.* at claim 65. Nguyen also states that “[t]he water utilized to form the fracturing fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other

aqueous liquid which does not adversely react [to] other components of the fracturing fluid.” *Id.* at [0013]. Moreover, Nguyen discloses that “[f]racturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and **emulsions**.” Ex. 1005, Nguyen at [0011]. A person of ordinary skill in the art would recognize that emulsions used in oil and gas well service operations may be comprised of a mixture of oil and water. *See e.g.*, Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S. Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production).

181. Thus Nguyen clearly describes a composition including “water” as recited in claim 1.

182. Trican previously attempted unsuccessfully to distinguish Nguyen based on Nguyen’s disclosure of an embodiment of a fracturing fluid that contains a gel polymer. However, the challenged claims, including claim 1, are open-ended claims that use the word “comprising” and thus are not limited to the listed ingredients. Furthermore, the ’738 patent discloses that “[t]he most commonly used

fracturing fluids are aqueous fluids including water, brines, water containing polymers or viscoelastic surfactants and foam fluids” (Ex. 1001, ’738 patent at 1:40-42) and that in the instant invention, “different carrying fluids, aqueous or non-aqueous can be used” (*id.* at 4:40-41).

183. Thus, Nguyen anticipates this claim limitation and cannot be distinguished by its disclosure of a gel polymer in the aqueous carrier fluid.

**c.     Limitation 1B: “particulates”**

184. Claim 1 recites that the composition comprises “particulates.” Ex. 1001 at 9:48.

185. Nguyen discloses that “[t]he proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like. . . . The preferred particles are sand.” Ex. 1005, Nguyen at [0016]. Nguyen’s particles are pumped into the well as part of the slurry composition to promote the recovery of hydrocarbons. *Id.* at Abstract.

186. In view of the foregoing excerpt from the disclosure of Nguyen, one of ordinary skill in the art would have understood Nguyen to disclose this limitation.

d. **Limitation 1C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

187. Claim 1 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001 at 9:49-50.

188. The specification of the '738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The '738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51.

189. The '738 patent also recites:

Examples of organosilanes include:  $\text{CH}_3\text{SiCl}_3$ ,  $\text{CH}_3\text{CH}_2\text{SiCl}_3$ ,  $(\text{CH}_3)_2\text{SiCl}_2$ ,  $(\text{CH}_3\text{CH}_2)_2\text{SiCl}_2$ ,  $(\text{C}_6\text{H}_5)_2\text{SiCl}_2$ ,  $(\text{C}_6\text{H}_5)\text{SiCl}_3$ ,  $(\text{CH}_3)_3\text{SiCl}$ ,  $\text{CH}_3\text{HSiCl}_2$ ,  $(\text{CH}_3)_2\text{HSiCl}$ ,  $\text{CH}_3\text{SiBr}_3$ ,  $(\text{C}_6\text{H}_5)\text{SiBr}_3$ ,  $(\text{CH}_3)_2\text{SiBr}_2$ ,  $(\text{CH}_3\text{CH}_2)_2\text{SiBr}_2$ ,  $(\text{C}_6\text{H}_5)_2\text{SiBr}_2$ ,  $(\text{CH}_3)_3\text{SiBr}$ ,  $\text{CH}_3\text{HSiBr}_2$ ,  $(\text{CH}_3)_2\text{HSiBr}$ ,  $\text{Si}(\text{OCH}_3)_4$ ,  $\text{CH}_3\text{Si}(\text{OCH}_3)_3$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_3)_3$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_3$ ,  $\text{CH}_3\text{Si}[\text{O}(\text{CH}_2)_3\text{CH}_3]_3$ ,

$\text{CH}_3\text{CH}_2\text{Si}(\text{OCH}_2\text{CH}_3)_3$ ,  $\text{C}_6\text{H}_5\text{Si}(\text{OCH}_3)_2$ ,  $\text{C}_6\text{H}_5\text{CH}_2\text{Si}(\text{OCH}_3)_3$ ,  
 $\text{C}_6\text{H}_5\text{Si}(\text{OCH}_2\text{CH}_3)_3$ ,  $\text{CH}_2=\text{CHCH}_2\text{Si}(\text{OCH}_3)_3$ ,  $(\text{CH}_3)_2\text{Si}(\text{OCH}_3)_2$ ,  
 $(\text{CH}_2=\text{CH})\text{Si}(\text{CH}_3)_2\text{Cl}$ ,  $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$ ,  
 $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_2$ ,  $(\text{CH}_3)_2\text{Si}[\text{O}(\text{CH}_2)_3\text{CH}_3]_2$ ,  
 $(\text{CH}_3\text{CH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_3)_2$ ,  
 $(\text{C}_6\text{H}_5\text{CH}_2)_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$ ,  
 $(\text{CH}_2=\text{CH})_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{CH}_3)_3\text{SiOCH}_3$ ,  
 $\text{CH}_3\text{HSi}(\text{OCH}_3)_2$ ,  $(\text{CH}_3)_2\text{HSi}(\text{OCH}_3)$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_3$ ,  
 $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $(\text{CH}_2=\text{CH})_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $\text{CH}_3\text{Si}(\text{CH}_3\text{COO})_3$ , 3-  
 aminotriethoxysilane, methyldiethylchlorosilane, butyltrichlorosilane,  
 diphenyldichlorosilane, vinyltrichlorosilane, methyltrimethoxysilane,  
 vinyltriethoxysilane, vinyltris(methoxyethoxy)silane,  
 methacryloxypropyltrimethoxysilane,  
 glycidoxypropyltrimethoxysilane, aminopropyltriethoxysilane,  
 divinyl-di-2-methoxysilane, ethyltributoxysilane,  
 isobutyltrimethoxysilane, hexyltrimethoxysilane, n-  
 octyltriethoxysilane, dihexyldimethoxysilane, octadecyltrichlorosilane,  
 octadecyltrimethoxysilane, octadecyldimethylchlorosilane,  
 octadecyldimethylmethoxysilane and quaternary ammonium silanes  
 including 3-(trimethoxysilyl) propyldimethyloctadecyl ammonium  
 chloride, 3-(trimethoxysilyl)propyldimethyloctadecyl ammonium  
 bromide, 3-(trimethylethoxysilylpropyl)didecylmethyl ammonium  
 chloride, triethoxysilyl soyapropyl dimoniumchloride, 3-  
 (trimethylethoxysilylpropyl)didecylmethyl ammonium bromide, 3-

(trimethylethoxysilylpropyl)didecylmethyl ammonium bromide,  
triethoxysilyl soyapropyl dimonium bromide,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_5)_3\text{Cl}^-$ ,  $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_5)_3\text{Br}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{CH}_3)_3\text{Cl}^-$ ,  $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_{13})_3\text{Cl}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{C}_4\text{H}_9\text{Cl}$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{C}_6\text{H}_5\text{Cl}$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{CH}_2\text{OHCl}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{C}_2\text{H}_5)_3\text{Cl}^-$ ,  
 $(\text{C}_2\text{H}_5\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{C}_{18}\text{H}_{37}\text{Cl}^-$ .

(*id.* at 5:66-6:43) and that “[a]mong different organosiloxane compounds which are useful for the present invention, polysiloxanes modified with organic amphoteric or cationic groups including organic betaine polysiloxanes and organic quaternary siloxanes are examples” (*id.* at 6:44-48).

190. Nguyen teaches “particles [that] are dry coated with a water-repellent organo-silicon material.” Ex. 1005, Nguyen at [0005], claim 1. Nguyen further teaches that “[r]epresentative organo-silicon compounds that can be used in this embodiment include polyalkylsiloxane such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additional organo-silicon compounds that can be used in this embodiment include polyalkylarylsiloxanes . . . chlorosilanes such as ethylchlorosilane, ***chlorotrimethylsilane*** and other silyl donors. Also, various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes can be used such as tetraethoxysilane, ***dimethoxydiphenylsilane***, ***dichlorodimethylsilane***,



**dichlorodiphenylsilane**, poly(dimethylsiloxane, poly[oxy(dimethylsilylene)] and other such materials that will be well known to those skilled in the art. . . . Furthermore, organofunctional silanes can be used in this embodiment.” *Id.* at [0006].

191. Nguyen therefore also discloses this limitation of claim 1 because Nguyen discloses a number of “hydrophobizing agents” listed in the ’738 patent, including **chlorotrimethylsilane** ((CH<sub>3</sub>)<sub>3</sub>SiCl), **dichlorodimethylsilane** ((CH<sub>3</sub>)<sub>2</sub>SiCl<sub>2</sub>), **dichlorodiphenylsilane** ((C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>SiCl<sub>2</sub>), and **dimethoxydiphenylsilane** ((C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>Si(OCH<sub>3</sub>)<sub>2</sub>). Compare Ex. 1005, Nguyen at [0006] with Ex. 1001, ’738 patent at 6:1-2, 6:12; see also NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data: Chlorotrimethylsilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?Name=chlorotrimethylsilane&Units=SI>; NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data: Dimethyldichlorosilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?ID=75-78-5>; NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data: Dichlorodiphenylsilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?Name=dichlorodiphenylsilane&Units=SI>; NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data:

Diphenyldimethoxysilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?Name=dimethoxydiphenylsilane&Units=SI>.

Thus, Nguyen teaches a slurry composition that include particulates coated with hydrophobic rendering compounds.

**e. Limitation 1D: “oil”**

192. Claim 1 further recites that the composition comprises “an oil.” Ex. 1001 at 9:50. The specification of the ’738 patent states that only a “small amount of an oil” is required. *Id.* at 3:65.

193. Nguyen also teaches the inclusion of an oil—“Fracturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and **emulsions**.” Ex. 1005, Nguyen at [0011]. A person of ordinary skill in the art would recognize that emulsions used in oil and gas well service operations may be comprised of a mixture of oil and water. *See e.g.*, Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S. Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production).

194. Additionally, Nguyen teaches that if “the particles are coated when the particles are flowing, the solvent should be readily miscible in the water-based gel carrier fluid. Organic solvents that can be used as a carrier for the organo-silicon coating material including **kerosene** [and] lighter grades of diesel fuel.” *Id.* at [0008]. One of ordinary skill in the art would recognize this disclosure to be an example of an “on the fly” preparation of the slurry taught by Nguyen, where the particles, water-based carrier fluid, and organo-silicon coating in solvent would be mixed at the surface, before being pumped downhole.

195. Nguyen thus teaches the limitation of oil included in the composition.

196. Moreover, the '738 specification also discloses that the oil of the composition may be formation oil. Ex. 1001 at 4:11-15 (“Alternatively the slurry containing a hydrophobizing agent can be pumped into the fracture forming the proppant pack, which can be further consolidated by oil or condensate contained in the formation.”). Thus, the oil limitation in claim 1 can be satisfied by the presence of formation oil. This is consistent with the prosecution history of the '738 patent. *See* Section VII.C, *supra*.

197. Nguyen further discloses that the invention is a “method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to ***promote the recovery of hydrocarbons***. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or

adsorption of any gel polymer from the carrier fluid for the particles.” Ex. 1005, Nguyen at Abstract.

198. One of ordinary skill in the art would understand that hydrocarbon-producing wells referenced by Nguyen contain oil. *See also* Ex. 1005, Nguyen at [0001] (recognizing that oil and gas wells produce hydrocarbons); Ex. 1001, ’738 patent at 1:26-31 (stating that hydraulic fracturing operations are used to “enhance oil and gas production” and to “increase oil and gas production”). Further, one of ordinary skill in the art would understand that injecting the composition containing carrier fluid and water repellent-coated particulate into an oil and gas well or a hydrocarbon-producing well during hydraulic fracturing operations would necessarily mean that oil is present. Because the formation contains oil, the composition of Nguyen must therefore comprise oil.

199. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 1A (water), 1B (particulate), 1C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D (oil), and the preamble of claim 1, is taught by Nguyen. Therefore, independent claim 1 is anticipated by Nguyen.

## **2. Independent Claim 32 Is Also Anticipated by Nguyen**

200. Independent claim 32 of the ’738 patent is a method claim with five limitations.

201. Claim 32 of the '738 patent recites:

A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:

mixing water,

particulates,

a chemical compound for rendering the surface of the particulates hydrophobic, and

an oil;

and pumping the mixture into the formation.

202. As set forth below on a limitation-by-limitation basis, Nguyen anticipates independent claim 32 of the '738 patent.

a. **Preamble for Claim 32: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”**

203. The preamble of claim 32 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 12:5-6. Nguyen recites that “particles [] dry coated with a water-repellent organo-silicon material” “are then pumped downhole . . . to the wellbore-screen annulus to function as a *gravel pack*.” Ex. 1005, Nguyen at [0005]. One of ordinary skill in the art would recognize that gravel packs are used as a method of controlling all types of sand flow, including both formation sand and proppant flowback. Therefore, one of ordinary skill in the art would recognize that Nguyen’s invention was used as a method of controlling proppant flowback.

204. Moreover, Nguyen states that his invention is a method to “enhanc[e] the clean-up of a hydrocarbon-producing well” and to “promote the recovery of hydrocarbons” from an oil and gas well during hydraulic fracturing using a coated particulate slurry. *See* Ex. 1005, Nguyen at Abstract, claim 1. As disclosed in the ’738 patent, using hydrophobic particulates as the proppant in hydraulic fracturing operations is one way to control proppant flowback. *See* Ex. 1001, ’738 patent at 3:30-59. Thus, one of ordinary skill in the art would recognize that the disclosure of Nguyen’s coated proppants would control proppant flowback in accordance with the ’738 patent and therefore meets this preamble.

205. Additionally, because Nguyen discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the ’738 patent. *See, e.g.*, Ex. 1001, ’738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback). Therefore, in my opinion, the preamble of claim 32 is met by Nguyen.

**b. Limitation 32A: “mixing water”**

206. Claim 32 recites that the composition comprises “water.” Ex. 1001 at 12:6-7. Nguyen discloses that “[t]he most common fracturing fluid utilized heretofore which is generally preferred for use in accordance with this invention is comprised of water.” Ex. 1005, Nguyen at [0012]. Nguyen further discloses, in

claim 63, “mixing particles in a carrier fluid comprising a water-based polymer to form a mixture.” *Id.* at claim 63. Nguyen further teaches “[t]he method of claim 63 wherein the carrier fluid comprises water and a gelling agent.” *Id.* at claim 65. Nguyen also states that “[t]he water utilized to form the fracturing fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other aqueous liquid which does not adversely react [to] other components of the fracturing fluids.” *Id.* at [0013]. Nguyen also discloses “[f]racturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and *emulsions*.” Ex. 1005, Nguyen at [0011]. A person of ordinary skill in the art would recognize that emulsions used in oil and gas well service operations may be comprised of a mixture of oil and water.

207. Thus Nguyen clearly describes a composition including “mixing water” as recited in claim 32.

208. Trican previously attempted unsuccessfully to distinguish Nguyen based on Nguyen’s disclosure of an embodiment of a fracturing fluid that contains a gel polymer. However, the challenged claims, including claim 32, are open-ended claims that use the word “comprising” and thus are not limited to the listed ingredients. Furthermore, the ’738 patent discloses that “the most commonly used fracturing fluids are aqueous fluids including water, brines, water containing polymers or viscoelastic surfactants and foam fluids” (Ex. 1001, ’738 patent at 1:40-

42) and that in the instant invention, “different carrying fluids, aqueous or non-aqueous can be used” (*id.* at 4:40-41).

209. Thus, Nguyen anticipates this claim limitation and cannot be distinguished by its disclosure of a gel polymer in the aqueous carrier fluid.

**c. Limitation 32B: “particulates”**

210. Claim 32 recites that the composition comprises “particulates.” Ex. 1001 at 12:7.

211. Nguyen discloses that “[t]he proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like. . . . The preferred particles are sand.” Ex. 1005, Nguyen at [0016]. Nguyen’s particles are pumped into the well as part of the slurry composition to promote the recovery of hydrocarbons. *Id.* at Abstract. Nguyen also discloses a “method . . . comprising mixing particles in a carrier fluid” to “form a mixture.” *Id.* at claim 63. Thus, Nguyen discloses the mixture of proppants with an aqueous carrier fluid to create a slurry composition.

212. In view of the foregoing excerpt from the disclosure of Nguyen, one of ordinary skill in the art would have understood Nguyen to disclose this limitation.



d. **Limitation 32C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

213. Claim 32 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001 at 12:7-8.

214. The specification of the '738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The '738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51.

215. The '738 patent also recites:

Examples of organosilanes include:  $\text{CH}_3\text{SiCl}_3$ ,  $\text{CH}_3\text{CH}_2\text{SiCl}_3$ ,  $(\text{CH}_3)_2\text{SiCl}_2$ ,  $(\text{CH}_3\text{CH}_2)_2\text{SiCl}_2$ ,  $(\text{C}_6\text{H}_5)_2\text{SiCl}_2$ ,  $(\text{C}_6\text{H}_5)\text{SiCl}_3$ ,  $(\text{CH}_3)_3\text{SiCl}$ ,  $\text{CH}_3\text{HSiCl}_2$ ,  $(\text{CH}_3)_2\text{HSiCl}$ ,  $\text{CH}_3\text{SiBr}_3$ ,  $(\text{C}_6\text{H}_5)\text{SiBr}_3$ ,  $(\text{CH}_3)_2\text{SiBr}_2$ ,  $(\text{CH}_3\text{CH}_2)_2\text{SiBr}_2$ ,  $(\text{C}_6\text{H}_5)_2\text{SiBr}_2$ ,  $(\text{CH}_3)_3\text{SiBr}$ ,  $\text{CH}_3\text{HSiBr}_2$ ,  $(\text{CH}_3)_2\text{HSiBr}$ ,  $\text{Si}(\text{OCH}_3)_4$ ,  $\text{CH}_3\text{Si}(\text{OCH}_3)_3$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_3)_3$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_3$ ,  $\text{CH}_3\text{Si}[\text{O}(\text{CH}_2)_3\text{CH}_3]_3$ ,

$\text{CH}_3\text{CH}_2\text{Si}(\text{OCH}_2\text{CH}_3)_3$ ,  $\text{C}_6\text{H}_5\text{Si}(\text{OCH}_3)_2$ ,  $\text{C}_6\text{H}_5\text{CH}_2\text{Si}(\text{OCH}_3)_3$ ,  
 $\text{C}_6\text{H}_5\text{Si}(\text{OCH}_2\text{CH}_3)_3$ ,  $\text{CH}_2=\text{CHCH}_2\text{Si}(\text{OCH}_3)_3$ ,  $(\text{CH}_3)_2\text{Si}(\text{OCH}_3)_2$ ,  
 $(\text{CH}_2=\text{CH})\text{Si}(\text{CH}_3)_2\text{Cl}$ ,  $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$ ,  
 $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_2$ ,  $(\text{CH}_3)_2\text{Si}[\text{O}(\text{CH}_2)_3\text{CH}_3]_2$ ,  
 $(\text{CH}_3\text{CH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_3)_2$ ,  
 $(\text{C}_6\text{H}_5\text{CH}_2)_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$ ,  
 $(\text{CH}_2=\text{CH})_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{CH}_3)_3\text{SiOCH}_3$ ,  
 $\text{CH}_3\text{HSi}(\text{OCH}_3)_2$ ,  $(\text{CH}_3)_2\text{HSi}(\text{OCH}_3)$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_3$ ,  
 $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $(\text{CH}_2=\text{CH})_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
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 aminotriethoxysilane, methyldiethylchlorosilane, butyltrichlorosilane,  
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 including 3-(trimethoxysilyl) propyldimethyloctadecyl ammonium  
 chloride, 3-(trimethoxysilyl)propyldimethyloctadecyl ammonium  
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 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{CH}_3)_3\text{Cl}^-$ ,  $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_{13})_3\text{Cl}^-$ ,  
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 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{C}_6\text{H}_5\text{Cl}$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{CH}_2\text{OHCl}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{C}_2\text{H}_5)_3\text{Cl}^-$ ,  
 $(\text{C}_2\text{H}_5\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{C}_{18}\text{H}_{37}\text{Cl}^-$ .

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216. Nguyen teaches “particles [that] are dry coated with a water-repellent organo-silicon material.” Ex. 1005, Nguyen at [0005], claim 1. Nguyen further teaches that “[r]epresentative organo-silicon compounds that can be used in this embodiment include polyalkylsiloxane such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additional organo-silicon compounds that can be used in this embodiment include polyalkylarylsiloxanes . . . chlorosilanes such as ethylchlorosilane, ***chlorotrimethylsilane*** and other silyl donors. Also, various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes can be used such as tetraethoxysilane, ***dimethoxydiphenylsilane***, ***dichlorodimethylsilane***,

*dichlorodiphenylsilane*, poly(dimethylsiloxane, poly[oxy(dimethylsilylene)] and other such materials that will be well known to those skilled in the art. . . . Furthermore, organofunctional silanes can be used in this embodiment.” *Id.* at [0006].

217. Nguyen therefore also discloses this limitation of claim 32 because Nguyen discloses a number of “hydrophobizing agents” listed in the ’738 patent, including *chlorotrimethylsilane*, *dichlorodimethylsilane*, *dichlorodiphenylsilane*, and *dimethoxydiphenylsilane*. Compare Ex. 1005, Nguyen at [0006] with Ex. 1001, ’738 patent at 6:1-2, 6:12; see also NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data: Chlorotrimethylsilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?Name=chlorotrimethylsilane&Units=SI>; NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data: Dimethyldichlorosilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?ID=75-78-5>; NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data: Dichlorodiphenylsilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?Name=dichlorodiphenylsilane&Units=SI>; NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data: Diphenyldimethoxysilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?Name=dimethoxydiphenylsilane&Units=SI>.

Thus, Nguyen teaches slurry compositions that include particles coated with hydrophobic rendering compounds.

**e. Limitation 32D: “oil”**

218. Claim 32 further recites that the composition comprises “an oil.” Ex. 1001 at 12:8. The specification of the ’738 patent states that only a “small amount of an oil” is required. *Id.* at 3:65.

219. Nguyen also teaches the inclusion of an oil—“Fracturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and **emulsions**.” Ex. 1005, Nguyen at [0011]. One of ordinary skill in the art would recognize that an emulsion may be comprised of oil and water. *See e.g.*, Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S. Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production).

220. Additionally, Nguyen teaches that if “the particles are coated when the particles are flowing, the solvent should be readily miscible in the water-based gel carrier fluid. Organic solvents that can be used as a carrier for the organo-silicon

coating material including **kerosene** [and] lighter grades of diesel fuel.” *Id.* at [0008]. One of ordinary skill in the art would recognize this disclosure to be an example of an “on the fly” preparation of the slurry taught by Nguyen, where the particles, water-based carrier fluid, and organo-silicon coating in solvent would be mixed at the surface, before being pumped downhole. Moreover, one of ordinary skill in the art would recognize that kerosene is an oil. *See e.g.*, Ex. 1001, ’738 patent at 9:11; 11:31-33; 13:1-3 (recognizing that kerosene is a type of oil).

221. Nguyen further teaches that the particulates coated with the water repellent composition are mixed with the carrier fluid to form a mixture (*see e.g., id.* at claim 63, Abstract), which, as disclosed above, may be an emulsion (*id.* at [0011]).

222. Nguyen thus teaches the limitation of oil included in the composition.

f. **Limitation 32E: “pumping the mixture into the formation”**

223. Claim 32 further recites “pumping the mixture into the formation.” Ex. 1001 at 12:8-9.

224. Nguyen teaches “[a] method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid . . . to form a mixture, ***pumping the mixture downhole*** in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid.” Ex. 1005, Nguyen at claim 63. Nguyen also teaches “[a] method for treating a hydrocarbon producing well comprising mixing particles in a carrier

fluid . . . to form a mixture, *pumping the mixture downhole* in a well to a fracture in a formation adjacent to the well to function as a proppant, and coating the particles with a water repellent composition, wherein the particles are coated before being mixed in a carrier fluid.” *Id.* at claim 75.

225. Therefore, Nguyen discloses limitation 32E.

226. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 32A (water), 32B (particulate), 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 32D (oil), 32E (pumping the mixture into the formation), and the preamble of claim 32, is taught by Nguyen. Therefore, independent claim 32 is anticipated by Nguyen.

### 3. Independent Claim 51 Is Also Anticipated by Nguyen

227. Independent claim 51 of the '738 patent is a method claim with four limitations.

228. Claim 51 of the '738 patent recites:

A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:  
contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic,  
separating the particulate from the medium;  
and blending the hydrophobic particulate with water and an oil to form a mixture;

and pumping the mixture into the formation.

229. As set forth below on a limitation-by-limitation basis, Nguyen anticipates independent claim 51 of the '738 patent.

a. **Preamble for Claim 51: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”**

230. The preamble of claim 51 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 13:28-29. Nguyen recites that “particles [] dry coated with a water-repellent organo-silicon material” “are then pumped downhole . . . to the wellbore-screen annulus to function as a *gravel pack*.” Ex. 1005, Nguyen at [0005]. One of ordinary skill in the art would recognize that gravel packs are used as a method of controlling all types of sand flow, including both formation sand and proppant flowback. Therefore, one of ordinary skill in the art would recognize that Nguyen’s invention was used as a method of controlling proppant flowback.

231. Moreover, Nguyen states that his invention is a method to “enhanc[e] the clean-up of a hydrocarbon-producing well” and to “promote the recovery of hydrocarbons” from an oil and gas well during hydraulic fracturing using a coated particulate slurry. See Ex. 1005, Nguyen at Abstract, claim 1. As disclosed in the '738 patent, using hydrophobic particulates as the proppant in hydraulic fracturing operations is one way to control proppant flowback. See Ex. 1001, '738 patent at



3:30-59. Thus, one of ordinary skill in the art would recognize that the disclosure of Nguyen's coated proppants would control proppant flowback in accordance with the '738 patent and therefore meets this preamble.

232. Additionally, because Nguyen discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the '738 patent. *See, e.g.*, Ex. 1001, '738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback). Therefore, in my opinion, the preamble of claim 51 is met by Nguyen.

**b. Limitation 51A: “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic”**

233. Claim 51 recites “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic.” Ex. 1001 at 13:29-31.

234. Nguyen discloses “[a]n example of the technique for applying the coating on the particles involves *dissolving an oil-soluble organo-silicon compound in a solvent, admixing or spraying the resultant solution on the particles*, and then evaporating the solvent to form a thin film of siloxane or silane encapsulating the particles. The organo-silicon organo-silane compound is readily absorbed onto the particles from the solvent and the solvent is easily evaporated by

drying.” *Id.* at [0007]; *see also* claim 4. As discussed, Nguyen discloses examples of organo-silicon compounds that are the same as those disclosed in the ’738 patent, including chlorotrimethylsilane, dichlorodimethylsilane, dichlorodiphenylsilane, and dimethoxydiphenylsilane. *See supra*, ¶¶ 187-191.

235. Thus Nguyen clearly describes contacting the particulates with an organo-silicon compound, which, as stated above, is an example of “a chemical compound to render the surface of the particulates hydrophobic” as required by claim 51.

c. **Limitation 51B: “separating the particulate from the medium”**

236. Claim 51 recites that the now-coated particulates are “separate[ed] . . . from the medium.” Ex. 1001 at 13:32.

237. Nguyen discloses “[a]n example of the technique for applying the coating on the particles involves dissolving an oil-soluble organo-silicon compound in a solvent, admixing or spraying the resultant solution on the particles, ***and then evaporating the solvent to form a thin film of siloxane or silane encapsulating the particles. The organo-silicon organo-silane compound is readily absorbed onto the particles from the solvent and the solvent is easily evaporated by drying.***” *Id.* at [0007]; *see also* claim 4.

238. One of ordinary skill in the art would understand evaporating the solvent is one method of separating the solvent from the organosilane-coated

particle. Thus, Nguyen discloses that the now-coated particulates are separated from the solvent solution.

239. In view of the foregoing excerpt from the disclosure of Nguyen, one of ordinary skill in the art would have understood Nguyen to disclose this limitation.

d. **Limitation 51C: “blending the hydrophobic particulate with water and an oil to form a mixture”**

240. Claim 51 recites that the hydrophobic coated particulate are blended “with water and an oil to form a mixture.” Ex. 1001 at 13:32-34.

241. Nguyen teaches that “[f]racturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and **emulsions**.” Ex. 1005, Nguyen at [0011]. One of ordinary skill in the art would recognize that an emulsion may be comprised of oil and water. *See e.g.*, Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S. Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production).

242. Nguyen teaches that the particulates coated with the water repellent composition are mixed with the carrier fluid “**to form a mixture**” (*see e.g., id.* at claim 63), which, as disclosed above, may be an emulsion (*id.* at [0011]).

243. Thus, Nguyen teaches this limitation.

e. **Limitation 51D: “pumping the mixture into the formation”**

244. Claim 51 further recites “pumping the mixture into the formation.” Ex. 1001 at 13:34.

245. As recited, Nguyen teaches “[a] method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid . . . to form a mixture, ***pumping the mixture downhole*** in a well to a fracture in a formation adjacent to the well to function as a proppant, and coating the particles with a water repellent composition, wherein the particles are coated before being mixed in a carrier fluid.” *Id.* at claim 75. Nguyen also teaches “[a] method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid . . . to form a mixture, ***pumping the mixture downhole*** in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid.” Ex. 1005, Nguyen at claim 63.

246. Therefore, Nguyen discloses limitation 51D.

247. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the

mixture into the formation), and the preamble of claim 51, is taught by Nguyen. Therefore, independent claim 51 is anticipated by Nguyen.

**4. Dependent Claims 21, 22, 40, 41, and 57 Are Anticipated by Nguyen**

248. Dependent claims 21 and 40 recite a near-identical limitation, but depend from different claims. Claim 21 depends from independent claim 1, whereas claim 40 depends from independent claim 32. Similarly, dependent claims 22, 41, and 57 recite the same limitation, but depend from different claims. Claim 22 depends from independent claim 1, whereas claim 41 depends from claim 32, and claim 57 depends from claim 51. Dependent claims 21 and 40 require the particulate be one of an enumerated list of particulates, including sand. *See* Ex. 1001, '738 patent at 11:34-37; 12:63-65 (“wherein the particulates are selected from the group consisting of sand . . .”). Dependent claims 22, 41, and 57 require the particulate to be sands. *Id.* at 11:38-39; 12:66-67 (“wherein the particulates are sands”). Therefore, a prior art reference that discloses the additional limitation of “sand” as the particulate anticipates claims 21, 22, 40, 41, and 57.

249. Claim 21 recites:

The composition from claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates.

250. Claim 40 recites:

The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates.

251. Claim 22 recites:

The composition of claim 1, wherein the particulates are sands.

252. Claim 41 recites:

The method of claim 32, wherein the particulates are sands.

253. Claim 57 recites:

The composition of claim 51, wherein the particulates are sands.

254. As addressed above (*see* Sections X.A.1, X.A.2, and X.A.3) and incorporated herein by reference, all of the limitations of claims 1, 32, and 51, including 1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), and the preambles of claims 1, 32, and 51, are anticipated by Nguyen.

255. Nguyen teaches the additional limitation of claims 21, 22, 40, 41, and 57 because Nguyen teaches “[v]arious kinds of particulates can be utilized as a proppant including sand. . . . The preferred particles are sand.” Ex. 1005, Nguyen at [0016]. Nguyen also teaches “[a] method comprising dry coating particles with a water-repelling composition” (*id.* at claim 1) “wherein the particles are selected from the group consisting of sand and gravel” (*id.* at claim 8).

256. Moreover, Nguyen teaches that the particulates coated with the water repellent composition are mixed with the carrier fluid to form a mixture (*see e.g., id.* at claim 63, Abstract), which, are then pumped “downhole into a well to promote the recovery of hydrocarbons from the well” to function as a gravel pack or a proppant. *Id.* at claims 1-3. Thus, the sand particulates are mixed with the carrier fluid.

257. It is my opinion that because Nguyen discloses the limitations of independent claims 1, 32, and 51, i.e., 1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation) and

the preambles of those claims as well as the additional limitation recited in dependent claims 21, 22, 40, 41, and 57 (sand), claims 21, 22, 40, 41, and 57 are also anticipated by Nguyen.

258. Based on the foregoing analysis of Sections X.A.1-4, all of the elements of claims 1, 21, 22, 32, 40, 41, 51, and 57 (1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), 21A, 22A, 40A, 41A, and 57A (sand), and the preambles of claims 1, 32, and 51) are disclosed by Nguyen.

259. My opinions are consistent with Examiner Admasu's rejection of claim 13 of the '469 patent, which included many of the same limitations as claims 1 and 32 of the '738 patent. Examiner Admasu similarly found that claim 13 was anticipated by Nguyen because

Nguyen teaches dry coated particles with a water-repellent organo-silicon materials. The treated particles are then pumped downhole to fractures as a proppant or to the wellbore-screen annulus to function as a gravel pack ([0005]). Representative organo-silicon compounds that



can be used include polyalkylsiloxane such as polymethylsiloxanes and the like and also include alkoxysilanes, tetraethoxysilane, gammaglycidoxypyrpyltrimethoxysilanes and the like. ([0006]). Fracturing or gravel packing fluids which can be utilized include gelled water or oil base liquids, foams and emulsions. The foams are generally comprised of water based foams containing one or more foaming agents foamed with a gas such as nitrogen or air ([0011]).

Ex. 1004 at 207, File History of the '469 Application, Non Final Rejection, Jan. 2, 2015. I agree with Examiner Admasu that Nguyen anticipates these limitations. Therefore, it is my opinion that one of ordinary skill in the art in April 2007 would understand that Nguyen anticipates claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent.

**B. Claims 1, 21, 22, 32, 40, 41, 51, and 57 Are Anticipated by Dawson**

260. After considering the Dawson reference as it would have been understood by a person of ordinary skill in the art in April 2007, it is my opinion that Dawson discloses each and every element of claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent. *See generally*, Ex. 1002D, '738 Patent v. Dawson Claim Chart. Therefore, it is my opinion that Dawson anticipates claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent.

**1. Independent Claim 1 Is Anticipated by Dawson**

261. As addressed above, independent claim 1 of the '738 patent is a composition claim with four limitations.

262. Claim 1 of the '738 patent recites:

An aqueous slurry composition comprising:

water;

particulates;

a chemical compound for rendering the surface of the particulates hydrophobic; and

an oil.

263. As set forth below on a limitation-by-limitation basis, Dawson anticipates independent claim 1 of the '738 patent.

a. **Preamble for Claim 1: “An aqueous slurry composition comprising”**

264. The preamble of claim 1 of the '738 patent states, “An aqueous slurry composition comprising.” Ex. 1001 at 9:46. Dawson recites “[a] slurry including the disclosed particulate material . . . and a carrier fluid.” Ex. 1006, Dawson at 14, l. 12-15. The carrier fluid may include salt water or fresh water. *Id.* at 13, l. 10-13. Thus, Dawson discloses an aqueous slurry. Therefore, in my opinion, the preamble of claim 1 is met by Dawson.

b. **Limitation 1A: “water”**

265. Claim 1 recites that the composition comprises “water.” Ex. 1001 at 9:47. Dawson discloses that the particles exposed to a modifying agent “may be mixed and pumped during any desired portion/s of a well treatment (*e.g.*, hydraulic fracturing treatment, sand control treatment, *etc.*) and may be mixed in any desired

concentration with a carrier fluid.” Ex. 1006, Dawson at 13, l. 6-10. Dawson also discloses “any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising **salt water, fresh water**, liquid hydrocarbons, and/or nitrogen or other gases.” *Id.* at 13, l. 10-13. Dawson discloses that the particulate is suspended in the carrier fluid (*id.* at 10, l. 1-3), therefore indicating that the carrier fluid such as water is mixed with the particulate to form a slurry.

266. Further, Dawson incorporates by reference U.S. Patent 3,659,651 to Graham (“Graham”). *See* Ex. 1006, Dawson at 33, l. 6-8. Graham teaches “[i]n placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or **water-in-oil emulsion** prepared in accordance with the teachings of U.S. Pat. No. 3,378,074.” Ex. 1012, Graham at 5:9-16 (citing U.S. Patent 3,378,074 to Kiel at 5:65-72 (Ex. 1013) (disclosing that “high viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either **water-in-oil or oil-in-water emulsions**. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with kerosene,

diesel fuel, heating oil, crude oil or a similar hydrocarbon oil **and water, brine** or an acid solution containing a suitable surface active agent or emulsifier.”)).

267. Thus Dawson clearly describes a composition including “water” as recited in claim 1.

**c. Limitation 1B: “particulates”**

268. Claim 1 recites that the composition comprises “particulates.” Ex. 1001 at 9:48.

269. Dawson teaches an invention that “relates to use of relatively lightweight and/or substantially neutrally buoyant **particles as proppant material** in hydraulic fracturing treatments.” Ex. 1006, Dawson at 2, l. 6-7.

270. Dawson additionally teaches that the “particulate material [is] suspended in a carrier fluid.” *Id.* at 10, l. 1-3; 48, claim 13.

271. In view of the foregoing excerpts from the disclosure of Dawson, one of ordinary skill in the art would have understood Dawson to disclose this limitation.

**d. Limitation 1C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

272. Claim 1 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001 at 9:49-50.

273. The specification of the ’738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention.

For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001, ’738 patent at 4:62-67. The ’738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51.

274. The ’738 patent also recites:

Examples of organosilanes include: **CH<sub>3</sub>SiCl<sub>3</sub>**, CH<sub>3</sub>CH<sub>2</sub>SiCl<sub>3</sub>, (CH<sub>3</sub>)<sub>2</sub>SiCl<sub>2</sub>, (CH<sub>3</sub>CH<sub>2</sub>)<sub>2</sub>SiCl<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>SiCl<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>)SiCl<sub>3</sub>, (CH<sub>3</sub>)<sub>3</sub>SiCl, CH<sub>3</sub>HSiCl<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>HSiCl, CH<sub>3</sub>SiBr<sub>3</sub>, (C<sub>6</sub>H<sub>5</sub>)SiBr<sub>3</sub>, (CH<sub>3</sub>)<sub>2</sub>SiBr<sub>2</sub>, (CH<sub>3</sub>CH<sub>2</sub>)<sub>2</sub>SiBr<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>SiBr<sub>2</sub>, (CH<sub>3</sub>)<sub>3</sub>SiBr, CH<sub>3</sub>HSiBr<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>HSiBr, Si(OCH<sub>3</sub>)<sub>4</sub>, CH<sub>3</sub>Si(OCH<sub>3</sub>)<sub>3</sub>, CH<sub>3</sub>Si(OCH<sub>2</sub>CH<sub>3</sub>)<sub>3</sub>, CH<sub>3</sub>Si(OCH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>)<sub>3</sub>, CH<sub>3</sub>Si[O(CH<sub>2</sub>)<sub>3</sub>CH<sub>3</sub>]<sub>3</sub>, CH<sub>3</sub>CH<sub>2</sub>Si(OCH<sub>2</sub>CH<sub>3</sub>)<sub>3</sub>, C<sub>6</sub>H<sub>5</sub>Si(OCH<sub>3</sub>)<sub>2</sub>, C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>Si(OCH<sub>3</sub>)<sub>3</sub>, C<sub>6</sub>H<sub>5</sub>Si(OCH<sub>2</sub>CH<sub>3</sub>)<sub>3</sub>, CH<sub>2</sub>=CHCH<sub>2</sub>Si(OCH<sub>3</sub>)<sub>3</sub>, (CH<sub>3</sub>)<sub>2</sub>Si(OCH<sub>3</sub>)<sub>2</sub>, (CH<sub>2</sub>=CH)Si(CH<sub>3</sub>)<sub>2</sub>Cl, (CH<sub>3</sub>)<sub>2</sub>Si(OCH<sub>2</sub>CH<sub>3</sub>)<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>Si(OCH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>)<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>Si[O(CH<sub>2</sub>)<sub>3</sub>CH<sub>3</sub>]<sub>2</sub>, (CH<sub>3</sub>CH<sub>2</sub>)<sub>2</sub>Si(OCH<sub>2</sub>CH<sub>3</sub>)<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>Si(OCH<sub>3</sub>)<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>)<sub>2</sub>Si(OCH<sub>3</sub>)<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>Si(OCH<sub>2</sub>CH<sub>3</sub>)<sub>2</sub>, (CH<sub>2</sub>=CH)<sub>2</sub>Si(OCH<sub>3</sub>)<sub>2</sub>, (CH<sub>2</sub>=CHCH<sub>2</sub>)<sub>2</sub>Si(OCH<sub>3</sub>)<sub>2</sub>, (CH<sub>3</sub>)<sub>3</sub>SiOCH<sub>3</sub>, CH<sub>3</sub>HSi(OCH<sub>3</sub>)<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>HSi(OCH<sub>3</sub>), CH<sub>3</sub>Si(OCH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>)<sub>3</sub>, (CH<sub>2</sub>=CHCH<sub>2</sub>)<sub>2</sub>Si(OCH<sub>2</sub>CH<sub>2</sub>OCH<sub>3</sub>)<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>Si(OCH<sub>2</sub>CH<sub>2</sub>OCH<sub>3</sub>)<sub>2</sub>,

$(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $(\text{CH}_2=\text{CH})_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $\text{CH}_3\text{Si}(\text{CH}_3\text{COO})_3$ , 3-  
 aminotriethoxysilane, methyldiethylchlorosilane, butyltrichlorosilane,  
 diphenyldichlorosilane, vinyltrichlorosilane, methyltrimethoxysilane,  
 vinyltriethoxysilane, vinyltris(methoxyethoxy)silane,  
 methacryloxypropyltrimethoxysilane,  
 glycidoxypropyltrimethoxysilane, aminopropyltriethoxysilane,  
 divinyl-di-2-methoxysilane, ethyltributoxysilane,  
 isobutyltrimethoxysilane, hexyltrimethoxysilane, n-  
 octyltriethoxysilane, dihexyldimethoxysilane, octadecyltrichlorosilane,  
 octadecyltrimethoxysilane, octadecyldimethylchlorosilane,  
 octadecyldimethylmethoxysilane and quaternary ammonium silanes  
 including 3-(trimethoxysilyl) propyldimethyloctadecyl ammonium  
 chloride, 3-(trimethoxysilyl)propyldimethyloctadecyl ammonium  
 bromide, 3-(trimethylethoxysilylpropyl)didecylmethyl ammonium  
 chloride, triethoxysilyl soyapropyl dimoniumchloride, 3-  
 (trimethylethoxysilylpropyl)didecylmethyl ammonium bromide, 3-  
 (trimethylethoxysilylpropyl)didecylmethyl ammonium bromide,  
 triethoxysilyl soyapropyl dimonium bromide,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_5)_3\text{Cl}^-$ ,  $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_5)_3\text{Br}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{CH}_3)_3\text{Cl}^-$ ,  $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_{13})_3\text{Cl}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{C}_4\text{H}_9\text{Cl}$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{C}_6\text{H}_5\text{Cl}$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{CH}_2\text{OHCl}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{C}_2\text{H}_5)_3\text{Cl}^-$ ,  
 $(\text{C}_2\text{H}_5\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{C}_{18}\text{H}_{37}\text{Cl}^-$ .

(*id.* at 5:66-6:43) and that “[a]mong different organosiloxane compounds which are useful for the present invention, polysiloxanes modified with organic amphoteric or cationic groups including organic betaine polysiloxanes and organic quaternary siloxanes are examples” (*id.* at 6:44-48).

275. Dawson teaches that particles “may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material.” Ex. 1006, Dawson at 7, l. 3-8. Specifically, Dawson discloses that “examples of suitable modifying agents include, but are not limited to, agents including polyisocyanates, *silanes*, *siloxanes*, and combinations thereof. Selected modifying agent/s may be advantageously used to increase the elastic modulus of a given naturally occurring material, for example, to make particles of a given naturally-occurring material more suitable . . . for use as a relatively lightweight and/or substantially neutrally buoyant fracture proppant or sand control particulate material having increased effectiveness when exposed to higher closure stresses or other mechanical stresses that may be encountered downhole during a well treatment such as hydraulic fracturing or sand control treatment.” *Id.* at 7, l. 14-23.

276. Like the '738 patent, Dawson also discloses the use of organosilanes and organosiloxanes to coat the particulate. Specifically, Dawson teaches that the modifying agent may contain “one or more halogen groups, (*e.g.*, tetrachlorosilane, *methyltrichlorosilane*, *etc.*).” *Id.* at 21, l. 10-11. Thus, Dawson teaches the same

methyltrichlorosilane ( $\text{CH}_3\text{SiCl}_3$ ) that is disclosed in the '738 patent. See Ex. 1001, '738 patent at 6:1.

277. Dawson teaches:

Examples of commercially available silane-based products that may be employed as modifying agents in the practice of the disclose methods and compositions include, but are not limited to, silane based products” (Ex. 1006, Dawson at p. 21, l. 19-21) and teaches the specific examples of “commercially available silane-based products available from CROMPTON include CoatOSil<sup>®</sup> 1706 Silane (vinyl silane monomer), CoatOSil<sup>®</sup> 1757 Silane, CoatOSil<sup>®</sup> 1770 Silane, EURENOR<sup>®</sup> 5020 (liquid isobutyldimethoxysilane), EURENOR<sup>®</sup> 5021 (liquid diisopropylmethoxysilane), EURENOR<sup>®</sup> 5022 (liquid diisobutyldimethoxysilane), EURENOR<sup>®</sup> 5023 (liquid dicyclopentylmethoxysilane), Silcat<sup>®</sup> R Silane (vinylsilane with grafting and cross linking catalysts), Sikat<sup>®</sup> RHE Silane (cross linking system of silane, peroxide and catalyst), Silcat<sup>®</sup> RHS Silane (crosslinking system of silane, peroxide catalyst and antioxidants), Sikat<sup>®</sup> VS-735/1 Silane (crosslinking system of silane, peroxide, antioxidants and metal deactivator), Silcat<sup>®</sup> VS-758/0 Silane (stabilized cross linking system of silane, peroxide and catalyst), Sikat<sup>®</sup> VS-870 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat<sup>®</sup> VS-928 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat<sup>®</sup> VS-963 Silane (stabilized cross linking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Silox<sup>®</sup> VS- 911 Silane (liquid silane system), Silquest<sup>®</sup> A-1100 Silane



(gammaaminopropyltriethoxysilane), Silquest<sup>®</sup> A-1101 Silane (technical grade amino silane), Silquest<sup>®</sup> A-1102 Silane (technical grade amino silane), Silquest<sup>®</sup> A-1110 Silane (primary amino silane), Silquest<sup>®</sup> A-1120 Silane, Silquest<sup>®</sup> A-1126 Silane (amino silane), Silquest<sup>®</sup> A-1128 Silane (amino silane), Silquest<sup>®</sup> A-1160 Silane (ureido silane), Silquest<sup>®</sup> A-1170 Silane (secondary amino functional silane), Silquest<sup>®</sup> A-1289 Silane, Silquest<sup>®</sup> A-1310 Silane, Silquest<sup>®</sup> A-137 Silane (monomeric alkylalkoxysilane), Silquest<sup>®</sup> A-151 Silane, Silquest<sup>®</sup> A-1524 Silane (100-percent active ureidosilane), Silquest<sup>®</sup> A-1589 Silane (disulfide silane), Silquest<sup>®</sup> A-171 Silane, Silquest<sup>®</sup> A-172 Silane (vinylfunctional coupling agent), Silquest<sup>®</sup> A-174 Silane, Silquest<sup>®</sup> A-186 Silane (epoxy functional silanes), Silquest<sup>®</sup> A-187 Silane (epoxy functional silanes), Silquest<sup>®</sup> A-189 Silane, Silquest<sup>®</sup> A-2171 Silane (silane compound having vinyl and silane functionality), Silquest<sup>®</sup> Alink 15 Silane (secondary amino silane endcapper), Silquest<sup>®</sup> RC-1 Silane (organosilicone coupling agent), Silquest<sup>®</sup> RC-2 Silane, Silquest<sup>®</sup> Wetlink 78 Silane, Silquest<sup>®</sup> Y-11637 Silane (primary aminosilane), Silquest<sup>®</sup> Y-11683 Silane (di-, tri-functional ethoxy silane), Silquest<sup>®</sup> Y-5997 Silane, XL-PEarl<sup>®</sup> 31 Blend, XL-PEarl<sup>®</sup> 50 Blend, XLPEarl<sup>®</sup> 51 Blend, XL-PEarl<sup>®</sup> 52 Blend, XL-PEarl<sup>®</sup> 60 Blend, XL-PEarl<sup>®</sup> 70 Blend, *etc.*

Ex. 1006, Dawson at 21, l. 23- 22, l. 23. Many of these compounds are organosilanes.

278. Because Dawson teaches coating particles with various silanes and siloxanes, including organosilanes and organosiloxanes, and the '738 patent

identifies organosilanes and organosiloxanes as examples of hydrophobizing agents (Ex. 1001, '738 patent at 4:62-67), one of ordinary skill in the art would understand that Dawson's particulates are coated with hydrophobic rendering compounds. Moreover, Dawson discloses at least one of the same organo-silicon compounds (i.e., methyltrichlorosilane –  $\text{CH}_3\text{SiCl}_3$ ) used to coat the particulates suspended in carrier fluid identified as a “hydrophobic rendering compound” by the '738 patent. Compare Ex. 1006, Dawson at 21, l. 11 with Ex. 1001, '738 patent at 6:1. Thus, Dawson teaches slurry compositions that include hydrophobic rendering compounds. Dawson therefore also discloses this limitation of claim 1.

**e. Limitation 1D: “oil”**

279. Claim 1 further recites that the composition comprises “an oil.” Ex. 1001 at 9:50. The specification of the '738 patent states that only a “small amount of an oil” is required. *Id.* at 3:65.

280. As mentioned above, Dawson discloses that “[t]he particles disclosed herein . . . may be mixed and pumped during any desired portion/s of well treatment (e.g., hydraulic fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not

limited to, carrier fluids comprising salt water, fresh water, **liquid hydrocarbons**, **and/or** nitrogen or other gases.” Ex. 1006, Dawson at 13, l. 6-13.

281. One of ordinary skill in the art would recognize that liquid hydrocarbons can be hydrocarbon oils. Dawson thus teaches the limitation of oil included in the composition and discloses that the carrier fluid may comprise both water **and** a liquid hydrocarbon.

282. Further, Dawson incorporates by reference U.S. Patent 3,659,651 to Graham (“Graham”). See Ex. 1006, Dawson at 33, l. 6-8. Graham teaches “[i]n placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or **water-in-oil emulsion** prepared in accordance with the teachings of U.S. Pat. No. 3,378,074.” Ex. 1012, Graham at 5:9-16 (citing U.S. Patent 3,378,074 to Kiel at 5:65-72 (Ex. 1013) (disclosing that “high viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either **water-in-oil or oil-in-water emulsions**. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with **kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine** or an acid solution containing a suitable surface active agent or emulsifier.”)).

283. Therefore, Dawson discloses the use of water and oil emulsions containing water or brine in kerosene, diesel fuel, crude oil, or a hydrocarbon oil in satisfaction of this limitation.

284. Moreover, the '738 specification also discloses that the oil of the composition may be formation oil. Ex. 1001 at 4:11-15 (“Alternatively the slurry containing a hydrophobizing agent can be pumped into the fracture forming the proppant pack, which can be further consolidated by oil or condensate contained in the formation.”). Thus, the oil limitation in claim 1 can be satisfied by the presence of formation oil. This is consistent with the prosecution history of the '738 patent. *See* Section VII.C, *supra*.

285. Dawson further discloses that “it will be understood with benefit of this disclosure that the disclosed particulate materials may be used with any method in which a pack of particulate material is formed within a wellbore that is permeable to fluids produced from a wellbore (*e.g.*, oil, gas, water, *etc.*).” *Id.* at 14 l. 31- 15 l. 4.

286. One of ordinary skill in the art would understand that hydrocarbon-producing wells contain oil and gas. Further, one of ordinary skill in the art would understand that using the disclosure of Dawson within a wellbore that is producing fluids including oil, gas, and water, would understand that oil is present. Because the disclosure of Dawson specifically teaches that a wellbore produces oil and that

the invention may be used in a wellbore that produces oil, the composition of Dawson must therefore comprise oil.

287. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 1A (water), 1B (particulate), 1C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D (oil), and the preamble of claim 1, is taught by Dawson. Therefore, independent claim 1 is anticipated by Dawson.

## **2. Independent Claim 32 Is Also Anticipated by Dawson**

288. As described, independent claim 32 of the '738 patent is a method claim with five limitations.

289. Claim 32 of the '738 patent recites:

A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:

mixing water,

particulates,

a chemical compound for rendering the surface of the particulates hydrophobic, and

an oil;

and pumping the mixture into the formation.

290. As set forth below on a limitation-by-limitation basis, Dawson anticipates independent claim 32 of the '738 patent.

a. **Preamble for Claim 32: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”**

291. The preamble of claim 32 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 12:5-6. Dawson states that his “invention relates generally to methods and compositions useful for subterranean formation treatments, such as hydraulic fracturing treatments and sand control. In particular, this invention relates to use of relatively lightweight and/or substantially neutrally buoyant particles as proppant materials in hydraulic fracturing treatment and as *particulate material in sand control methods* such as gravel packing, *frac pack treatments*, etc.” Ex. 1006, Dawson at 2, l. 4-8. Dawson also states that his invention “may be directed toward improving wellbore productivity and/or *controlling the production of fracture proppant* or formation sand. Particular examples include gravel packing and ‘frac-packs.’” *Id.* at 13, l. 23-25. One of ordinary skill in the art would recognize, and Dawson discloses (*see, e.g.*, Ex. 1006, Dawson at 13, l. 24-25), that frac packs are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize Dawson’s invention to be directed to a method of controlling proppant flowback.

292. Moreover Dawson states that his invention, which includes a slurry containing particulates coated with methyltrichlorosilane, will “provide for

substantially improved overall system performance in hydraulic fracturing applications, or in other well treating applications such as sand control.” *Id.*, Dawson at 3:15-16. As disclosed in the ’738 patent, using hydrophobic particulates as the proppant in hydraulic fracturing operations is one way to control proppant flowback. See Ex. 1001, ’738 patent at 3:30-59. Thus, one of ordinary skill in the art would recognize that the disclosure of Dawson’s coated particulates would control proppant flowback in accordance with the ’738 patent and therefore meets this preamble.

293. Additionally, because Dawson discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the ’738 patent. See, e.g., Ex. 1001, ’738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback). Therefore, in my opinion, the preamble of claim 32 is met by Dawson.

**b. Limitation 32A: “mixing water”**

294. Claim 32 recites that the composition comprises “water.” Ex. 1001 at 12:6-7. Dawson discloses that the particles exposed to a modifying agent “may be mixed and pumped during any desired portion/s of a well treatment (e.g., hydraulic fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier fluid.” Ex. 1006, Dawson at 13, l. 6-10. Dawson also

discloses “any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising **salt water, fresh water**, liquid hydrocarbons, and/or nitrogen or other gases.” *Id.* at 13, l. 10-13. Dawson discloses that the particulate is suspended in the carrier fluid (*id.* at 10, l. 1-3), therefore indicating that the carrier fluid such as water is mixed with the particulate to form a slurry.

295. Further, Dawson incorporates by reference U.S. Patent 3,659,651 to Graham (“Graham”). *See* Ex. 1006, Dawson at 33, l. 6-8. Graham teaches “[i]n placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or **water-in-oil emulsion** prepared in accordance with the teachings of U.S. Pat. No. 3,378,074.” Ex. 1012, Graham at 5:9-16 (citing U.S. Patent 3,378,074 to Kiel at 5:65-72 (Ex. 1013) (disclosing that “high viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either **water-in-oil or oil-in-water emulsions**. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with



*kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine* or an acid solution containing a suitable surface active agent or emulsifier.”)).

296. Thus Dawson clearly describes a composition including “mixing water” as recited in claim 32.

**c. Limitation 32B: “particulates”**

297. Claim 32 recites that the composition comprises “particulates.” Ex. 1001 at 12:7.

298. Dawson teaches an invention that “relates to use of relatively lightweight and/or substantially neutrally buoyant *particles as proppant material* in hydraulic fracturing treatments.” Ex. 1006, Dawson at 2, l. 6-7.

299. Dawson additionally teaches that the “particulate material [is] suspended in a carrier fluid.” *Id.* at 10, l. 1-3; 48, claim 13. One of ordinary skill in the art would understand that for a particulate to be suspended in a fluid, it would need to be mixed with the fluid.

300. Therefore, one of ordinary skill in the art would have understood Dawson to disclose this limitation.

**d. Limitation 32C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

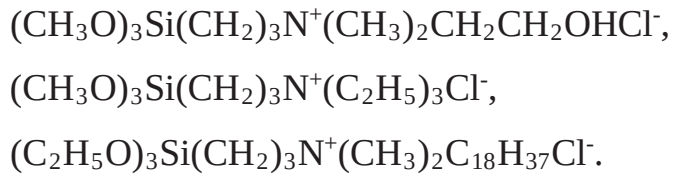
301. Claim 32 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001 at 12:7-8.

302. The specification of the '738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The '738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51.

303. The '738 patent also recites:

Examples of organosilanes include: **CH<sub>3</sub>SiCl<sub>3</sub>**, CH<sub>3</sub>CH<sub>2</sub>SiCl<sub>3</sub>, (CH<sub>3</sub>)<sub>2</sub>SiCl<sub>2</sub>, (CH<sub>3</sub>CH<sub>2</sub>)<sub>2</sub>SiCl<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>SiCl<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>)SiCl<sub>3</sub>, (CH<sub>3</sub>)<sub>3</sub>SiCl, CH<sub>3</sub>HSiCl<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>HSiCl, CH<sub>3</sub>SiBr<sub>3</sub>, (C<sub>6</sub>H<sub>5</sub>)SiBr<sub>3</sub>, (CH<sub>3</sub>)<sub>2</sub>SiBr<sub>2</sub>, (CH<sub>3</sub>CH<sub>2</sub>)<sub>2</sub>SiBr<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>SiBr<sub>2</sub>, (CH<sub>3</sub>)<sub>3</sub>SiBr, CH<sub>3</sub>HSiBr<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>HSiBr, Si(OCH<sub>3</sub>)<sub>4</sub>, CH<sub>3</sub>Si(OCH<sub>3</sub>)<sub>3</sub>, CH<sub>3</sub>Si(OCH<sub>2</sub>CH<sub>3</sub>)<sub>3</sub>, CH<sub>3</sub>Si(OCH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>)<sub>3</sub>, CH<sub>3</sub>Si[O(CH<sub>2</sub>)<sub>3</sub>CH<sub>3</sub>]<sub>3</sub>, CH<sub>3</sub>CH<sub>2</sub>Si(OCH<sub>2</sub>CH<sub>3</sub>)<sub>3</sub>, C<sub>6</sub>H<sub>5</sub>Si(OCH<sub>3</sub>)<sub>2</sub>, C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>Si(OCH<sub>3</sub>)<sub>3</sub>, C<sub>6</sub>H<sub>5</sub>Si(OCH<sub>2</sub>CH<sub>3</sub>)<sub>3</sub>, CH<sub>2</sub>=CHCH<sub>2</sub>Si(OCH<sub>3</sub>)<sub>3</sub>, (CH<sub>3</sub>)<sub>2</sub>Si(OCH<sub>3</sub>)<sub>2</sub>, (CH<sub>2</sub>=CH)Si(CH<sub>3</sub>)<sub>2</sub>Cl, (CH<sub>3</sub>)<sub>2</sub>Si(OCH<sub>2</sub>CH<sub>3</sub>)<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>Si(OCH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>)<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>Si[O(CH<sub>2</sub>)<sub>3</sub>CH<sub>3</sub>]<sub>2</sub>, (CH<sub>3</sub>CH<sub>2</sub>)<sub>2</sub>Si(OCH<sub>2</sub>CH<sub>3</sub>)<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>Si(OCH<sub>3</sub>)<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>)<sub>2</sub>Si(OCH<sub>3</sub>)<sub>2</sub>, (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>Si(OCH<sub>2</sub>CH<sub>3</sub>)<sub>2</sub>,

$(\text{CH}_2=\text{CH})_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{CH}_3)_3\text{SiOCH}_3$ ,  
 $\text{CH}_3\text{HSi}(\text{OCH}_3)_2$ ,  $(\text{CH}_3)_2\text{HSi}(\text{OCH}_3)$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_3$ ,  
 $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $(\text{CH}_2=\text{CH})_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $\text{CH}_3\text{Si}(\text{CH}_3\text{COO})_3$ , 3-  
 aminotriethoxysilane, methyldiethylchlorosilane, butyltrichlorosilane,  
 diphenyldichlorosilane, vinyltrichlorosilane, methyltrimethoxysilane,  
 vinyltriethoxysilane, vinyltris(methoxyethoxy)silane,  
 methacryloxypropyltrimethoxysilane,  
 glycidoxypropyltrimethoxysilane, aminopropyltriethoxysilane,  
 divinyl-di-2-methoxysilane, ethyltributoxysilane,  
 isobutyltrimethoxysilane, hexyltrimethoxysilane, n-  
 octyltriethoxysilane, dihexyldimethoxysilane, octadecyltrichlorosilane,  
 octadecyltrimethoxysilane, octadecyldimethylchlorosilane,  
 octadecyldimethylmethoxysilane and quaternary ammonium silanes  
 including 3-(trimethoxysilyl) propyldimethyloctadecyl ammonium  
 chloride, 3-(trimethoxysilyl)propyldimethyloctadecyl ammonium  
 bromide, 3-(trimethylethoxysilylpropyl)didecylmethyl ammonium  
 chloride, triethoxysilyl soyapropyl dimoniumchloride, 3-  
 (trimethylethoxysilylpropyl)didecylmethyl ammonium bromide, 3-  
 (trimethylethoxysilylpropyl)didecylmethyl ammonium bromide,  
 triethoxysilyl soyapropyl dimonium bromide,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_5)_3\text{Cl}^-$ ,  $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_5)_3\text{Br}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{CH}_3)_3\text{Cl}^-$ ,  $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_{13})_3\text{Cl}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{C}_4\text{H}_9\text{Cl}$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{C}_6\text{H}_5\text{Cl}$ ,



(*id.* at 5:66-6: 43) and that “[a]mong different organosiloxane compounds which are useful for the present invention, polysiloxanes modified with organic amphoteric or cationic groups including organic betaine polysiloxanes and organic quaternary siloxanes are examples” (*id.* at 6:44-48).

304. Dawson teaches that particles “may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material.” Ex. 1006, Dawson at 7, l. 3-8. Specifically, Dawson discloses that “examples of suitable modifying agents include, but are not limited to, agents including polyisocyanates, ***silanes***, ***siloxanes***, and combinations thereof. Selected modifying agent/s may be advantageously used to increase the elastic modulus of a given naturally occurring material, for example, to make particles of a given naturally-occurring material more suitable . . . for use as a relatively lightweight and/or substantially neutrally buoyant fracture proppant or sand control particulate material having increased effectiveness when exposed to higher closure stresses or other mechanical stresses that may be encountered downhole during a well treatment such as hydraulic fracturing or sand control treatment.” *Id.* at 7, l. 14-23.

305. Like the '738 patent, Dawson also discloses the use of organosilanes and organosiloxanes to coat the particulate. Specifically, Dawson teaches that the

modifying agent may contain “one or more halogen groups, (e.g., tetrachlorosilane, *methyltrichlorosilane*, etc.).” *Id.* at 21, l. 10-11. Thus, Dawson teaches the same methyltrichlorosilane (CH<sub>3</sub>SiCl<sub>3</sub>) that is disclosed in the ’738 patent. *See* Ex. 1001, ’738 patent at 6:1.

306. Dawson teaches:

Examples of commercially available silane-based products that may be employed as modifying agents in the practice of the disclose methods and compositions include, but are not limited to, silane based products” (Ex. 1006, Dawson at p. 21, l. 19-21) and teaches the specific examples of “commercially available silane-based products available from CROMPTON include CoatOSil<sup>®</sup> 1706 Silane (vinyl silane monomer), CoatOSil<sup>®</sup> 1757 Silane, CoatOSil<sup>®</sup> 1770 Silane, EURENOR<sup>®</sup> 5020 (liquid isobutyldimethoxysilane), EURENOR<sup>®</sup> 5021 (liquid diisopropylmethoxysilane), EURENOR<sup>®</sup> 5022 (liquid diisobutyldimethoxysilane), EURENOR<sup>®</sup> 5023 (liquid dicyclopentylmethoxysilane), Silcat<sup>®</sup> R Silane (vinylsilane with grafting and cross linking catalysts), Sikat<sup>®</sup> RHE Silane (cross linking system of silane, peroxide and catalyst), Silcat<sup>®</sup> RHS Silane (crosslinking system of silane, peroxide catalyst and antioxidants), Sikat<sup>®</sup> VS-735/1 Silane (crosslinking system of silane, peroxide, antioxidants and metal deactivator), Silcat<sup>®</sup> VS-758/0 Silane (stabilized cross linking system of silane, peroxide and catalyst), Sikat<sup>®</sup> VS-870 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat<sup>®</sup> VS-928 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal

deactivator), Sikat<sup>®</sup> VS-963 Silane (stabilized cross linking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Silox<sup>®</sup> VS- 911 Silane (liquid silane system), Silquest<sup>®</sup> A-1100 Silane (gammaaminopropyltriethoxysilane), Silquest<sup>®</sup> A-1101 Silane (technical grade amino silane), Silquest<sup>®</sup> A-1102 Silane (technical grade amino silane), Silquest<sup>®</sup> A-1110 Silane (primary amino silane), Silquest<sup>®</sup> A-1120 Silane, Silquest<sup>®</sup> A-1126 Silane (amino silane), Silquest<sup>®</sup> A-1128 Silane (amino silane), Silquest<sup>®</sup> A-1160 Silane (ureido silane), Silquest<sup>®</sup> A-1170 Silane (secondary amino functional silane), Silquest<sup>®</sup> A-1289 Silane, Silquest<sup>®</sup> A-1310 Silane, Silquest<sup>®</sup> A-137 Silane (monomeric alkylalkoxysilane), Silquest<sup>®</sup> A-151 Silane, Silquest<sup>®</sup> A-1524 Silane (100-percent active ureidosilane), Silquest<sup>®</sup> A-1589 Silane (disulfide silane), Silquest<sup>®</sup> A-171 Silane, Silquest<sup>®</sup> A-172 Silane (vinylfunctional coupling agent), Silquest<sup>®</sup> A-174 Silane, Silquest<sup>®</sup> A-186 Silane (epoxy functional silanes), Silquest<sup>®</sup> A-187 Silane (epoxy functional silanes), Silquest<sup>®</sup> A-189 Silane, Silquest<sup>®</sup> A-2171 Silane (silane compound having vinyl and silane functionality), Silquest<sup>®</sup> Alink 15 Silane (secondary amino silane endcapper), Silquest<sup>®</sup> RC-1 Silane (organosilicone coupling agent), Silquest<sup>®</sup> RC-2 Silane, Silquest<sup>®</sup> Wetlink 78 Silane, Silquest<sup>®</sup> Y-11637 Silane (primary aminosilane), Silquest<sup>®</sup> Y-11683 Silane (di-, tri-functional ethoxy silane), Silquest<sup>®</sup> Y-5997 Silane, XL-PEarl<sup>®</sup> 31 Blend, XL-PEarl<sup>®</sup> 50 Blend, XLPEarl<sup>®</sup> 51 Blend, XL-PEarl<sup>®</sup> 52 Blend, XL-PEarl<sup>®</sup> 60 Blend, XL-PEarl<sup>®</sup> 70 Blend, *etc.*

Ex. 1006, Dawson at 21, l. 23- 22, l. 23. Many of these compounds are organosilanes.

307. Because Dawson teaches coating particles with various silanes and siloxanes, including organosilanes and organosiloxanes, and the '738 patent identifies organosilanes and organosiloxanes as examples of hydrophobizing agents (Ex. 1001, '738 patent at 4:62-67), one of ordinary skill in the art would understand that Dawson's particulates are coated with hydrophobic rendering compounds. Moreover, Dawson discloses at least one of the same organo-silicon compounds (i.e., methyltrichlorosilane –  $\text{CH}_3\text{SiCl}_3$ ) used to coat the particulates suspended in carrier fluid identified as a "hydrophobic rendering compound" by the '738 patent. *Compare* Ex. 1006, Dawson at 21, l. 11 *with* Ex. 1001, '738 patent at 6:1. Thus, Dawson teaches slurry compositions that include hydrophobic rendering compounds. Dawson therefore also discloses this limitation of claim 1.

**e. Limitation 32D: "oil"**

308. Claim 32 further recites that the composition comprises "an oil." Ex. 1001 at 12:8. The specification of the '738 patent states that only a "small amount of an oil" is required. *Id.* at 3:65.

309. Dawson discloses that "[t]he particles disclosed herein . . . may be mixed and pumped during any desired portion/s of well treatment (*e.g.*, hydraulic fracturing treatment, sand control treatment, *etc.*) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation

fracture in communication therewith may be employed including, but not limited to, carrier fluids **comprising** salt water, fresh water, **liquid hydrocarbons**, **and/or** nitrogen or other gases.” Ex. 1006, Dawson at 13, l. 6-13. Dawson thus teaches the limitation of oil included in the composition and discloses that the carrier fluid may comprise both water **and** a liquid hydrocarbon.

310. One of ordinary skill in the art would recognize that liquid hydrocarbons are an oil (i.e., a hydrocarbon oil). See Ex. 1001, ’738 patent at 13:1-3 (“42. The composition of claim 1, wherein the oil is selected from the group consisting of a **hydrocarbon oil**, a silicone oil, kerosene oil, a frac oil and combinations thereof.”). Thus, one of ordinary skill in the art would also recognize Dawson’s disclosure to mean that the carrier fluid could contain water and oil.

311. Further, Dawson incorporates by reference U.S. Patent 3,659,651 to Graham (“Graham”). See Ex. 1006, Dawson at 33, l. 6-8. Graham teaches “[i]n placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or **water-in-oil emulsion** prepared in accordance with the teachings of U.S. Pat. No. 3,378,074.” Ex. 1012, Graham at 5:9-16 (citing U.S. Patent 3,378,074 at 5:65-72 (Ex. 1013) (disclosing that “high viscosity emulsions suitable for use as viscous



fracturing fluids in carrying out the invention may be either ***water-in-oil or oil-in-water emulsions***. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with ***kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine*** or an acid solution containing a suitable surface active agent or emulsifier.”)).

312. Therefore, Dawson discloses the use of water and oil emulsions containing water or brine and kerosene, diesel fuel, crude oil, or a hydrocarbon oil in satisfaction of this limitation.

313. Dawson thus teaches the limitation of oil included in the mixture that is pumped into the formation.

f. **Limitation 32E: “pumping the mixture into the formation”**

314. Claim 32 further recites “pumping the mixture into the formation.” Ex. 1001 at 12:8-9.

315. Dawson discloses that “[t]he particles disclosed herein . . . ***may be mixed and pumped*** during any desired portion/s of well treatment (*e.g.*, hydraulic fracturing treatment, sand control treatment, *etc.*) and may be mixed in any desired concentration with a carrier fluid.” Ex. 1006, Dawson at 13, l. 6-10. Dawson also teaches that “[i]n another exemplary embodiment, modified particulate material may be formulated as described herein and introduced or pumped into a well.” *Id.* at 29, l. 27-28. As described in Dawson, a person of ordinary skill would recognize that

the slurry comprising particles in a treatment or carrier fluid would be pumped into the formation.

316. Therefore, Dawson discloses limitation 32E.

317. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 32A (water), 32B (particulate), 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 32D (oil), 32E (pumping the mixture into the formation), and the preamble of claim 32, is taught by Dawson. Therefore, independent claim 32 is anticipated by Dawson.

### 3. Independent Claim 51 Is Also Anticipated by Dawson

318. Independent claim 51 of the '738 patent is a method claim with four limitations.

319. Claim 51 of the '738 patent recites:

A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:

contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic,

separating the particulate from the medium;

and blending the hydrophobic particulate with water and an oil to form a mixture;

and pumping the mixture into the formation.

320. As set forth below on a limitation-by-limitation basis, Dawson anticipates independent claim 51 of the '738 patent.

a. **Preamble for Claim 51: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”**

321. The preamble of claim 51 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 13:28-29. Dawson states that his invention “may be directed toward improving wellbore productivity and/or *controlling the production of fracture proppant* or formation sand. Particular examples include gravel packing and ‘frac-packs.’” *Id.* at 13, l. 23-25. Dawson also states that his “invention relates generally to methods and compositions useful for subterranean formation treatments, such as hydraulic fracturing treatments and sand control. In particular, this invention relates to use of relatively lightweight and/or substantially neutrally buoyant particles as proppant materials in hydraulic fracturing treatment and as *particulate material in sand control methods* such as gravel packing, *frac pack treatments, etc.*” Ex. 1006, Dawson at 2, l. 4-8. One of ordinary skill in the art would recognize, and Dawson discloses (*see, e.g.*, Ex. 1006, Dawson at 13, l. 24-25), that frac packs are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize Dawson’s invention to be directed to a method of controlling proppant flowback.

322. Moreover Dawson states that his invention, which includes a slurry containing particulates coated with methyltrichlorosilane, will “provide for

substantially improved overall system performance in hydraulic fracturing applications, or in other well treating applications such as sand control.” *Id.*, Dawson at 3:15-16. As disclosed in the ’738 patent, using hydrophobic particulates as the proppant in hydraulic fracturing operations is one way to control proppant flowback. See Ex. 1001, ’738 patent at 3:30-59. Thus, one of ordinary skill in the art would recognize that the disclosure of Dawson’s coated particulates would control proppant flowback in accordance with the ’738 patent and therefore meets this preamble.

323. Additionally, because Dawson discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the ’738 patent. See, *e.g.*, Ex. 1001, ’738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback). Therefore, in my opinion, the preamble of claim 51 is met by Dawson.

**b. Limitation 51A: “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic”**

324. Claim 51 recites “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic.” Ex. 1001 at 13:29-31.

325. Dawson teaches that particles “may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material.” Ex. 1006, Dawson at 7, l. 7-8. Dawson states that the “[m]odifying agents may be *diluted as so desired in a suitable solvent prior to treating particles* of a naturally-occurring material.” *Id.* at 27, l. 1-2. Dawson further discloses that “[s]pecific examples of *suitable exposure methods* include, but are not limited to, by *soaking* the naturally-occurring material in a modifying agent . . . by *dipping* the naturally-occurring material in a modifying agent, by *spraying* a modifying agent on to the naturally-occurring material . . . by *mixing* naturally-occurring particulate material with liquid, atomized and/or gaseous modifying agent in a vertical mixer . . . by *flowing* modifying agent through a permeable bed or pack of the naturally-occurring particles in a pressurized container. Drying of particles after exposure may be accomplished, for example, by continuously dropping through heated air.” *Id.*, at 24, l. 17-26. As discussed, Dawson discloses examples of organo-silicon compounds that are the same as those disclosed in the ’738 patent, including methyltrichlorosilane. *See supra*, ¶¶ 272-278.

326. Thus Dawson clearly describes contacting the particulates with an organo-silicon compound, which, as stated above, is an example of “a chemical compound to render the surface of the particulates hydrophobic” as required by claim 51.

c. **Limitation 51B: “separating the particulate from the medium”**

327. Claim 51 recites that the now-coated particulates are “separate[ed] . . . from the medium.” Ex. 1001 at 13:32.

328. Dawson teaches that particles “may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material.” Ex. 1006, Dawson at 7, l. 3-8. Dawson states that the “[m]odifying agents may be diluted as so desired in a suitable solvent prior to treating particles of a naturally-occurring material.” *Id.* at 27, l. 1-2. Dawson further discloses that “[s]pecific examples of suitable exposure methods include, but are not limited to, by soaking the naturally-occurring material in a modifying agent . . . by dipping the naturally-occurring material in a modifying agent, by spraying a modifying agent on to the naturally-occurring material . . . by mixing naturally-occurring particulate material with liquid, atomized and/or gaseous modifying agent in a vertical mixer . . . by flowing modifying agent through a permeable bed or pack of the naturally-occurring particles in a pressurized container. ***Drying of particles after exposure*** may be accomplished, for example, by continuously dropping through heated air.” *Id.*, at 24, l. 17-26.

329. One of ordinary skill in the art would understand that drying is a method of separating the coated particles from the modifying agent and solvent solution.

Thus, Dawson discloses that the now-coated particulates are separated from the solvent solution.

330. In view of the foregoing excerpt from the disclosure of Dawson, one of ordinary skill in the art would have understood Dawson to disclose this limitation.

d. **Limitation 51C: “blending the hydrophobic particulate with water and an oil to form a mixture”**

331. Claim 51 recites that the hydrophobic coated particulate are blended “with water and an oil to form a mixture.” Ex. 1001 at 13:32-34.

332. Dawson discloses that “[t]he *particles* disclosed herein . . . may be mixed and pumped during any desired portion/s of well treatment (*e.g.*, hydraulic fracturing treatment, sand control treatment, *etc.*) and ***may be mixed in any desired concentration with a carrier fluid***. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids ***comprising salt water, fresh water, liquid hydrocarbons, and/or nitrogen or other gases***.” Ex. 1006, Dawson at 13, l. 6-13. Dawson thus teaches the limitation of oil included in the composition and discloses that the carrier fluid may comprise both water ***and*** a liquid hydrocarbon.

333. One of ordinary skill in the art would recognize that liquid hydrocarbons are an oil (i.e., a hydrocarbon oil). See Ex. 1001, ’738 patent at 13:1-3 (“42. The composition of claim 1, wherein the oil is selected from the group

consisting of a **hydrocarbon oil**, a silicone oil, kerosene oil, a frac oil and combinations thereof.”). Thus, one of ordinary skill in the art would also recognize Dawson’s disclosure to mean that the carrier fluid could contain water and oil.

334. Further, Dawson incorporates by reference U.S. Patent 3,659,651 to Graham (“Graham”). See Ex. 1006, Dawson at 33, l. 6-8. Graham teaches “[i]n placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or **water-in-oil emulsion** prepared in accordance with the teachings of U.S. Pat. No. 3,378,074.” Ex. 1012, Graham at 5:9-16 (citing U.S. Patent 3,378,074 at 5:65-72 (Ex. 1013) (disclosing that “high viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either **water-in-oil or oil-in-water emulsions**. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with **kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine** or an acid solution containing a suitable surface active agent or emulsifier.”)). Thus, the use of these water-in-oil and oil-in-water emulsions incorporated by reference into Dawson would contain water and oil.



335. Thus, Dawson teaches that the coated particulates are mixed with an oil and water to form a mixture.

e. **Limitation 51D: “pumping the mixture into the formation”**

336. Claim 51 further recites “pumping the mixture into the formation.” Ex. 1001 at 13:34.

337. As recited, Dawson discloses that “[t]he particles disclosed herein . . . *may be mixed and pumped* during any desired portion/s of well treatment (*e.g.*, hydraulic fracturing treatment, sand control treatment, *etc.*) and may be mixed in any desired concentration with a carrier fluid.” Ex. 1006, Dawson at 13, l. 6-10. Dawson also teaches that “[i]n another exemplary embodiment, modified particulate material may be formulated as described herein and introduced or pumped into a well.” *Id.* at 29, l. 27-28. As described in Dawson, a person of ordinary skill would recognize that the slurry comprising particles in a treatment or carrier fluid would be pumped into the formation.

338. Therefore, Dawson discloses limitation 51D.

339. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the

mixture into the formation), and the preamble of claim 51, is taught by Dawson. Therefore, independent claim 51 is anticipated by Dawson.

**4. Dependent Claims 21, 22, 40, 41, and 57 Are Anticipated by Dawson**

340. Dependent claims 21, and 40 recite a near-identical limitation, but depend from different claims. Claim 21 depends from independent claim 1, whereas claim 40 depends from independent claim 32. Similarly, dependent claims 22, 41 and 57 recite the same limitation, but depend from different claims. Claim 22 depends from independent claim 1, whereas claim 41 depends from claim 32, and claim 57 depends from claim 51. Dependent claims 21 and 40 require the particulate be one of an enumerated list of particulates, including sand. *See* Ex. 1001, '738 patent at 11:34-37; 12:63-65 (“wherein the particulates are selected from the group consisting of sand . . .”). Dependent claims 22, 41, and 57 require the particulate to be sands. *Id.* at 11:38-39; 12:66-67; 14:24-25 (“wherein the particulates are sands”). Therefore, a prior art reference that discloses the additional limitation of “sand” as the particulate anticipates claims 21, 22, 40, 41, and 57.

341. Claim 21 recites:

The composition from claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates.

342. Claim 40 recites:

The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates.

343. Claim 22 recites:

The composition of claim 1, wherein the particulates are sands.

344. Claim 41 recites:

The method of claim 32, wherein the particulates are sands.

345. Claim 57 recites:

The composition of claim 51, wherein the particulates are sands.

346. As addressed above (*see* Sections X.B.1, X.B.2, and X.B.3) and incorporated herein by reference, all of the limitations of claims 1, 32, and 57, including 1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), and the preambles of claims 1, 32, and 57, are anticipated by Dawson.

347. Dawson teaches the additional limitation of claims 21, 22, 40, 41, and 57, because Dawson teaches the use of particulate and incorporates by reference U.S.

Patent 6,330,916 (Ex. 1011) (“Rickards”). Ex. 1006, Dawson at 13, l. 3-5. Rickards discloses a subterranean formation treatment method for reducing proppant flowback where a blend of fracturing proppant and deformable particulate material are injected into a formation. Ex. 1011, Rickards at Abstract. Rickards further discloses that the “fracture proppant material may be a material such as sand.” *Id.*

348. Dawson discloses that “[a] slurry including the disclosed particulate material . . . and a carrier fluid may then be introduced into the wellbore.” Ex. 1006, Dawson at 14, l. 12-15. Dawson therefore teaches that the particulates are mixed with the carrier fluid.

349. It is my opinion that because Dawson discloses the limitations of both independent claims 1, 32, and 57, 1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), and the preambles of those claims as well as the additional limitation recited in dependent claims 21, 22, 40, 41, and 57 (sand), claims 21, 22, 40, 41, and 57 are also anticipated by Dawson.

350. Based on the foregoing analysis of Sections X.A.1-4, all of the elements of claims 1, 21, 22, 32, 40, 41, 51, and 57 (1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), 21A, 22A, 40A, 41A, and 57A (sand), and the preambles of claims 1, 32, and 51) are disclosed by Dawson. Therefore, it is my opinion that one of ordinary skill in the art in April 2007 would understand that Dawson anticipates claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent.

**C. Claims 1, 21, 22, 32, 40, 41, 51, and 57 Are Anticipated by Vincent**

351. After considering the Vincent reference as it would have been understood by a person of ordinary skill in the art in April 2007, it is my opinion that Vincent discloses each and every element of claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent. *See generally*, Ex. 1002E, '738 Patent v. Vincent Claim Chart. Therefore, it is my opinion that Vincent anticipates claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent.

**1. Independent Claim 1 Is Anticipated by Vincent**

352. As addressed above, independent claim 1 of the '738 patent is a composition claim with four limitations.

353. Claim 1 of the '738 patent recites:

An aqueous slurry composition comprising:

water;

particulates;

a chemical compound for rendering the surface of the particulates hydrophobic; and

an oil.

354. As set forth below on a limitation-by-limitation basis, Vincent anticipates independent claim 1 of the '738 patent.

a. **Preamble for Claim 1: “An aqueous slurry composition comprising”**

355. The preamble of claim 1 of the '738 patent states, “An aqueous slurry composition comprising.” Ex. 1001 at 9:46. Vincent teaches that “particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or ‘slurry’ into which the particles have previously been introduced and suspended.” Ex. 1007, Vincent at [0087]. Vincent also teaches an aqueous slurry. *Id.* (“The fracturing fluid may be . . . water base . . .”). Therefore, in my opinion, the preamble of claim 1 is met by Vincent.

**b. Limitation 1A: “water”**

356. Claim 1 recites that the composition comprises “water.” Ex. 1001 at 9:47. Vincent discloses that the fracture fluid in which the particulates are suspended “may be an oil base, **water base**, acid, **emulsion**, foam, or any other fluid.” Ex. 1007, Vincent at [0087]. One of ordinary skill in the art would understand that an emulsion may be comprised of oil and water.

357. Thus Vincent clearly describes a composition including “water” as recited in claim 1.

**c. Limitation 1B: “particulates”**

358. Claim 1 recites that the composition comprises “particulates.” Ex. 1001 at 9:48.

359. Vincent teaches that his invention “relates to oil and gas well proppants and, more particularly, to process for physically or chemically modifying the surface characteristics of hydraulic fracturing proppants.” Ex. 1006, Vincent at [0002]. One of ordinary skill in the art would understand that Vincent’s “proppants” are the “particulates” described in the ’738 patent. Vincent further teaches that the particulates would be mixed into the aqueous carrier fluid to form a slurry. *See* Ex. 1007, Vincent at [0087].

360. In view of the foregoing excerpts from the disclosure of Vincent, one of ordinary skill in the art would have understood Vincent to disclose this limitation.

d. **Limitation 1C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

361. Claim 1 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001 at 9:49-50.

362. The specification of the '738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The '738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds” (*id.* at 5:47-51) and that “[a]mong different organosiloxane compounds which are useful for the present invention, polysiloxanes modified with organic amphoteric or cationic groups including organic betaine polysiloxanes and organic quaternary siloxanes are examples” (*id.* at 6:44-48). The '738 patent identifies that “[t]he simplest polysiloxanes are ***polydimethylsiloxanes***.” *Id.* at 5:8-9.



363. Moreover, in Example 1 of the '738 patent, a solution containing “Tegopren 6924, a di-quaternary ***polydimethylsiloxane*** from Degussa Corp.” was used to hydrophobize frac sand. *Id.* at 8:47-50.

364. Vincent discloses “modifying the surface properties of natural sand, resin-coated sand, and manufactured proppants used in oil and gas recovery to achieve one or more of the ***following desirable effects: alter the wettability***, alter the chemical reactivity, alter the surface topography, impart lubricity, and control relative permeability to flow of fluids such as proppants. Sands, resin coated sands, or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.” Ex. 1007, Vincent at [0008].

365. Vincent teaches that the above benefits can be achieved by a variety of techniques, including by “coating the proppants with a ***hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes***.” *Id.* at [0009]. Vincent also discloses a coated proppant “wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and ***polydimethyl siloxane***.” *Id.* at claim 6. Vincent also provides examples of proppant coated with Rain-X<sup>®</sup>, a commercially available product that includes polyalkyl hydrogen siloxane. *See e.g., id.* at [0043]. Vincent teaches that the Rain-X<sup>®</sup> coated

proppant trapped air bubbles that accumulated over time and that the proppant was clearly hydrophobic. *Id.* at [0082]. This is noteworthy because during the prosecution of the '469 application and explicitly stated in the specification of the '738 patent, the Applicant repeatedly described his invention as using micro gas bubbles attached individually to the sand grains to lift the sand grains by buoyancy. *See e.g.*, Ex. 1004 at 310, Declaration Under 37 C.F.R. § 1.132 at ¶ 3, May 10, 2016; Ex. 1004 at 208, File History of the '469 Application, Amendment and Response, May 4, 2015; *see also* Ex. 1001, '738 patent at 3:28-30 (“It is also known that micro-bubbles attached to the hydrophobic particle surfaces also tend to bridge the particles together.”); Ex. 1001, '738 patent at 3:34-40 (“Unlike in conventional approaches, where attention is focused on making proppants or sand particles sticky through formation of chemical bonds between resins coated on the particle surfaces, in the present invention the attention is focused on making particle aggregations by bridging the particles through strong hydrophobic force or micro-bubbles.”). Thus, Vincent explicitly describes Applicant’s self-described inventive concept.

366. Vincent’s published claims clearly disclose coating particles with a hydrophobic material. *See e.g., id.* at claims 1-6, 9-10, 15-17. Vincent also discloses at least one of the source organo-silicon compounds (i.e., polydimethyl siloxane) as listed in the '738 patent as a specific example of a hydrophobic rendering compound.

Compare Ex. 1007, Vincent at claim 6 with Ex. 1001, '738 patent at 8:47-50.

Vincent therefore also discloses this limitation of claim 1.

**e. Limitation 1D: “oil”**

367. Claim 1 further recites that the composition comprises “an oil.” Ex. 1001 at 9:50. The specification of the '738 patent states that only a “small amount of an oil” is required. *Id.* at 3:65.

368. Vincent discloses that the benefits taught by his invention “can be achieved by a variety of techniques, including coating the proppants with a hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes, polytetrafluoroethylene (commonly known as Teflon®), *plant oils, such as linseed oil, soybean oil, corn oil, cottonseed oil, vegetable oil* (widely commercially available such as Crisco®), *and canola oil, and hydrocarbons such as kerosene, diesel, and crude oil, petroleum distillates such as hydrocarbon liquids.*” Ex. 1007, Vincent at [0009].

369. Vincent also discloses proppants coated with a hydrophobic material, “wherein the hydrophobic material *comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, [and] hydrocarbons.*” *Id.* at claim 2; see also *id.* at claims 10 and 21. Thus Vincent teaches the use of two hydrophobic rendering compounds. One, for example, polydimethyl siloxane, satisfies limitation 1C (compound for rendering the

surface of the particulates hydrophobic); the other, for example, plant oil or hydrocarbons satisfies limitation 1D (oil).

370. One of ordinary skill in the art would recognize, and the '738 patent confirms, that “hydrocarbon liquid” or “hydrocarbon” is another term for oil. *See* Ex. 1001, '738 patent at 11:31-33 (“20. The composition of claim 1, wherein the oil is selected from the group consisting of a **hydrocarbon oil**, a silicon oil, kerosene oil, a frac oil and combinations thereof.”); Ex. 1007, Vincent [0009] (“hydrocarbons such as kerosene, diesel, and crude oil, petroleum distillates such as hydrocarbon liquids.”). Vincent thus teaches the limitation of oil included in the composition.

371. Vincent further discloses that the fracture fluid in which the particulates are suspended “may be an oil base, water base, acid, **emulsion**, foam, or any other fluid.” Ex. 1007, Vincent at [0087]. A person of ordinary skill in the art would understand that an emulsion can be comprised of oil and water. *See e.g.*, Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S. Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production).

372. Moreover, the '738 specification also discloses that the oil of the composition may be formation oil. Ex. 1001 at 4:11-15 (“Alternatively the slurry containing a hydrophobizing agent can be pumped into the fracture forming the proppant pack, which can be further consolidated by oil or condensate contained in the formation.”). Thus, the oil limitation in claim 1 can be satisfied by the presence of formation oil. This is consistent with the prosecution history of the '738 patent. *See* Section VII.C, *supra*.

373. Vincent further discloses that “[e]mbodiments of the present invention relate to modifying the surface properties of natural sand, resin-coated sand and manufactured proppants ***used in oil and gas recovery*** to achieve one or more of the following desirable effects: alter the wettability.” Ex. 1007, Vincent at [0008]. And, that “various embodiments of the present invention relate to concepts and techniques to treat fracturing sand and/or proppant to: . . . alter or modify proppant wettability to control the relative permeability to flow of the fluids which may be encountered in the reservoir (***such as oil***, water, gas, chemical treatments, and fracturing fluids).” *Id.* at [0012], [0014].

374. Because the disclosure of Vincent specifically teaches the use of the invention in a reservoir containing oil and that its invention can be used in oil and gas recovery, the composition of Vincent must therefore comprise oil.

375. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 1A (water), 1B (particulate), 1C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D (oil), and the preamble of claim 1, is taught by Vincent. Therefore, independent claim 1 is anticipated by Vincent.

**2. Independent Claim 32 Is Also Anticipated by Vincent**

376. As described, independent claim 32 of the '738 patent is a method claim with five limitations.

377. Claim 32 of the '738 patent recites:

A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:

mixing water,

particulates,

a chemical compound for rendering the surface of the particulates hydrophobic, and

an oil;

and pumping the mixture into the formation.

378. As set forth below on a limitation-by-limitation basis, Vincent anticipates independent claim 32 of the '738 patent.

**a. Preamble for Claim 32: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”**

379. The preamble of claim 32 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 12:5-6.

380. Vincent discloses “concepts and techniques to treat fracturing sand and/or proppant to: . . . alter or modify proppant wettability, to control the relative permeability to flow of the fluids which may be encountered in the reservoir . . . [and] ‘lubricate’ the proppant to allow more efficient proppant arrangement when the fracture closes, effectively increasing packing efficiency.” Ex. 1007, Vincent at [0012], [0014], [0015]. Vincent further discloses that this “‘lubrication’ concept reduces proppant friction, allowing superior proppant redistribution during fracture closing. This redistribution allows more efficient packing of proppant, thereby increasing grain-to-grain contact and effectively increasing proppant pack strength.” *Id.* at [0028].

381. One of ordinary skill in the art would understand that controlling the relative permeability to flow of the fluids would reduce the viscous drag on the proppant particles, which would help control proppant flowback. One of ordinary skill in the art would also understand that increasing grain-to-grain contact to increase proppant pack strength is another method of controlling proppant flowback because more proppant particles would remain in the fracture when it closes, thereby reducing the amount of proppant that would flowback.

382. Moreover, one of ordinary skill in the art would generally understand that to reap the benefits of utilizing a proppant, the proppant would need to remain in the formation fracture—i.e. not be carried out of the fractures and into the wellbore along with produced fluids being flowed back out of the well. Thus, a person of ordinary skill in the art would understand the disclosure of Vincent to be a particulate coated with a hydrophobic material that is better suited than a non-coated particulate to remain in the fracture and not be subject to flowback. This is consistent with the disclosure of the '738 patent that use of hydrophobic proppant helps control proppant flowback. Ex. 1001, '738 patent at 3:30-59.

383. Additionally, because Vincent discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the '738 patent. *See, e.g.*, Ex. 1001, '738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback).

384. Therefore, in my opinion, the preamble of claim 32 is met by Vincent.

**b. Limitation 32A: “mixing water”**

385. Claim 32 recites that the composition comprises “water.” Ex. 1001 at 12:6-7. As recited above, Vincent discloses that the fracture fluid in which the particulates are suspended “may be an oil base, **water base**, acid, **emulsion**, foam, or any other fluid.” Ex. 1007, Vincent at [0087]. One of ordinary skill in the art



would understand that an emulsion may be comprised of oil and water. Vincent teaches the mixing step when describing the fluid or slurry “into which the particles have previously been introduced and suspended.” *Id.* To be suspended, the particles of Vincent would have been mixed with the carrier fluid. Thus Vincent clearly describes a composition including “mixing water” as recited in claim 32.

**c.     Limitation 32B: “particulates”**

386. Claim 32 recites that the composition comprises “particulates.” Ex. 1001 at 12:7.

387. Vincent teaches that his invention “relates to oil and gas well proppants and, more particularly, to process for physically or chemically modifying the surface characteristics of hydraulic fracturing proppants.” Ex. 1006, Vincent at [0002]. One of ordinary skill in the art would understand that Vincent’s “proppants” are particulates, as described in the ’738 patent. Vincent further teaches a “‘slurry’ into which the particles have previously been introduced and suspended.” *Id.* at [0087]. To be suspended, the particles of Vincent would have been mixed with the carrier fluid.

388. Therefore, one of ordinary skill in the art would have understood Dawson to disclose this limitation.

d. **Limitation 32C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

389. Claim 32 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001 at 12:7-8.

390. The specification of the '738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The '738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds” (*id.* at 5:47-51) and that “[a]mong different organosiloxane compounds which are useful for the present invention, polysiloxanes modified with organic amphoteric or cationic groups including organic betaine polysiloxanes and organic quaternary siloxanes are examples” (*id.* at 6:44-48). The '738 patent identifies that “[t]he simplest polysiloxanes are ***polydimethylsiloxanes***.” *Id.* at 5:8-9.

391. Moreover, in Example 1 of the '738 patent, a solution containing “Tegopren 6924, a di-quaternary ***polydimethylsiloxane*** from Degussa Corp.” was used to hydrophobize frac sand. *Id.* at 8:47-50.

392. Vincent discloses “modifying the surface properties of natural sand, resin-coated sand, and manufactured proppants used in oil and gas recovery to achieve one or more of the ***following desirable effects: alter the wettability***, alter the chemical reactivity, alter the surface topography, impart lubricity, and control relative permeability to flow of fluids such as proppants. Sands, resin coated sands, or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.” Ex. 1007, Vincent at [0008].

393. Vincent teaches that the above benefits can be achieved by a variety of techniques, including by “coating the proppants with a ***hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes***.” *Id.* at [0009]. Vincent also discloses a coated proppant “wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and ***polydimethyl siloxane***.” *Id.* at claim 6. Vincent also provides examples of proppant coated with Rain-X<sup>®</sup>, a commercially available product that includes polyalkyl hydrogen siloxane. *See e.g., id.* at [0043]. Vincent teaches that the Rain-X<sup>®</sup> coated

proppant trapped air bubbles that accumulated over time and that the proppant was clearly hydrophobic. *Id.* at [0082]. This is noteworthy because during the prosecution of the '469 application and explicitly stated in the specification of the '738 patent, the Applicant repeatedly described his invention as using micro gas bubbles attached individually to the sand grains to lift the sand grains by buoyancy. *See e.g.*, Ex. 1004 at 310, Declaration Under 37 C.F.R. § 1.132 at ¶ 3, May 10, 2016; Ex. 1004 at 208, File History of the '469 Application, Amendment and Response, May 4, 2015; *see also* Ex. 1001, '738 patent at 3:28-30 (“It is also known that micro-bubbles attached to the hydrophobic particle surfaces also tend to bridge the particles together.”); Ex. 1001, '738 patent at 3:34-40 (“Unlike in conventional approaches, where attention is focused on making proppants or sand particles sticky through formation of chemical bonds between resins coated on the particle surfaces, in the present invention the attention is focused on making particle aggregations by bridging the particles through strong hydrophobic force or micro-bubbles.”). Thus, Vincent explicitly describes Applicant’s self-described inventive concept.

394. Vincent’s published claims clearly disclose coating particles with a hydrophobic material. *See, e.g., id.* at claims 1-6, 9-10, 15-17. Vincent also discloses at least one of the source organo-silicon compounds (i.e., polydimethyl siloxane) as listed in the '738 patent as a specific example of a hydrophobic

rendering compound. Compare Ex. 1007, Vincent at claim 6 with Ex. 1001, '738 patent at 8:47-50. Vincent therefore also discloses this limitation of claim 32.

**e. Limitation 32D: “oil”**

395. Claim 32 further recites that the composition comprises “an oil.” Ex. 1001 at 12:8. The specification of the '738 patent states that only a “small amount of an oil” is required. *Id.* at 3:65.

396. Vincent discloses that the benefits taught by his invention “can be achieved by a variety of techniques, including coating the proppants with a hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes, polytetrafluoroethylene (commonly known as Teflon®), *plant oils, such as linseed oil, soybean oil, corn oil, cottonseed oil, vegetable oil* (widely commercially available such as Crisco®), *and canola oil, and hydrocarbons such as kerosene, diesel, and crude oil, petroleum distillates such as hydrocarbon liquids.*” Ex. 1007, Vincent at [0009].

397. Vincent also discloses proppants coated with a hydrophobic material, “wherein the hydrophobic material *comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, [and] hydrocarbons.*” *Id.* at claim 2; *see also id.* at claims 10 and 21. As stated, one of ordinary skill in the art would understand this disclosure from Vincent to mean that two hydrophobizing agents can be used. Thus, one of the

hydrophobizing agents, i.e., polydimethyl siloxane, would satisfy limitation 32C (chemical compound for rendering the surface of the particulates hydrophobic), while another, i.e., hydrocarbons or plant oil, would satisfy limitation 32D (oil).

398. One of ordinary skill in the art would recognize, and the '738 patent confirms, that “hydrocarbon liquid” or “hydrocarbon” is another term for oil. See Ex. 1001, '738 patent at 11:31-33 (“20. The composition of claim 1, wherein the oil is selected from the group consisting of a **hydrocarbon oil**, a silicon oil, kerosene oil, a frac oil and combinations thereof.”).

399. Moreover, Vincent discloses that the fracture fluid in which the particulates are suspended “may be an oil base, water base, acid, **emulsion**, foam, or any other fluid.” Ex. 1007, Vincent at [0087]. A person of ordinary skill in the art would understand that an emulsion can be comprised of oil and water. See *e.g.*, Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S. Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production).

400. Vincent thus teaches the limitation of oil included in the mixture that is pumped into the formation.

f. **Limitation 32E: “pumping the mixture into the formation”**

401. Claim 32 further recites “pumping the mixture into the formation.” Ex. 1001 at 12:8-9.

402. Vincent teaches “[a] method of fracturing a subterranean formation, comprising: . . . injecting into the fracture a fluid containing a plurality of essentially spherical particles, wherein at least some of the particles are modified to alter the surface characteristics thereof, wherein the particles are modified by coating the particles with a hydrophobic material.” Ex. 1007, Vincent at claim 9.

403. Therefore, Vincent discloses limitation 32E.

404. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 32A (water), 32B (particulate), 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 32D (oil), 32E (pumping the mixture into the formation), and the preamble of claim 32, is taught by Vincent.

405. Therefore, independent claim 32 is anticipated by Vincent.

3. **Independent Claim 51 Is Also Anticipated by Vincent**

406. Independent claim 51 of the '738 patent is a method claim with four limitations.

407. Claim 51 of the '738 patent recites:

A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:  
contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic,  
separating the particulate from the medium;  
and blending the hydrophobic particulate with water and an oil to form a mixture;  
and pumping the mixture into the formation.

408. As set forth below on a limitation-by-limitation basis, Vincent anticipates independent claim 51 of the '738 patent.

a. **Preamble for Claim 51: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”**

409. The preamble of claim 51 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 13:28-29.

410. Vincent discloses “concepts and techniques to treat fracturing sand and/or proppant to: . . . alter or modify proppant wettability, to control the relative permeability to flow of the fluids which may be encountered in the reservoir . . . [and] ‘lubricate’ the proppant to allow more efficient proppant arrangement when the fracture closes, effectively increasing packing efficiency.” Ex. 1007, Vincent at [0012], [0014], [0015]. Vincent further discloses that this “‘lubrication’ concept reduces proppant friction, allowing superior proppant redistribution during fracture



closing. This redistribution allows more efficient packing of proppant, thereby increasing grain-to-grain contact and effectively increasing proppant pack strength.” *Id.* at [0028].

411. One of ordinary skill in the art would understand that controlling the relative permeability to flow of the fluids would reduce the viscous drag on the proppant particles, which would help control proppant flowback. One of ordinary skill in the art would also understand that increasing grain-to-grain contact to increase proppant pack strength is another method of controlling proppant flowback because more proppant particles would remain in the fracture when it closes, thereby reducing the amount of proppant that would flowback.

412. Additionally, because Vincent discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the ’738 patent. *See, e.g.*, Ex. 1001, ’738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback).

**b. Limitation 51A: “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic”**

413. Claim 51 recites “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic.” Ex. 1001 at 13:29-31.

414. Vincent discloses “pretreating proppant prior to the fracturing treatment.” Ex. 1007, Vincent at [0024]. Vincent discloses a variety of techniques for coating the proppant, including spraying, dipping, or soaking the proppant in a liquid solution of the hydrophobic material. *Id.* at [0009], [0036], [0049]. For example, Vincent discloses that the particles may be coated by “***mixing the proppant and the coating in a beaker*** for approximately 30 minutes, then drying it for approximately 15 to 18 hours in an oven.” *Id.* at [0036]. One example disclosed in Vincent states that “[t]he coating materials were added as follows. Polymethylhydrogen siloxane was added as either a 2 or 5 weight percent ***emulsion of siloxane in water***, polydimethyl siloxane was added as a 5 weight percent ***emulsion of siloxane in water*** and ***Stoddard Solvent*** was added without water dilution. All samples were dried at 113° C. for approximately 15 to 18 hours.” *Id.* at [0037]. As discussed, Vincent discloses examples of organo-silicon compounds that are the same as those disclosed in the ’738 patent, including polydimethylsiloxane. *See supra*, ¶¶ 361-366.

415. Thus Vincent clearly describes contacting the particulates with an organo-silicon compound, which, as stated above, is an example of “a chemical compound to render the surface of the particulates hydrophobic” as required by claim 51.

c. **Limitation 51B: “separating the particulate from the medium”**

416. Claim 51 recites that the now-coated particulates are “separate[ed] . . . from the medium.” Ex. 1001 at 13:32.

417. Vincent discloses that the particles are coated by “mixing the proppant and the coating in a beaker for approximately 30 minutes, *then drying it* for approximately 15 to 18 hours in an oven.” *Id.* at [0036]. One example disclosed in Vincent states that “[t]he coating materials were added as follows. Polymethylhydrogen siloxane was added as either a 2 or 5 weight percent emulsion of siloxane in water, polydimethyl siloxane was added as a 5 weight percent emulsion of siloxane in water and Stoddard Solvent was added without water dilution. *All samples were dried* at 113° C. for approximately 15 to 18 hours.” *Id.* at [0037].

418. As stated, one of ordinary skill in the art would understand that drying is a method of separating the siloxane and water solution from the now-coated particles. Thus, Vincent discloses that the now-coated particulates are separated from the solvent solution.

419. In view of the foregoing excerpt from the disclosure of Vincent, one of ordinary skill in the art would have understood Vincent to disclose this limitation.

d. **Limitation 51C: “blending the hydrophobic particulate with water and an oil to form a mixture”**

420. Claim 51 recites that the hydrophobic coated particulate are blended “with water and an oil to form a mixture.” Ex. 1001 at 13:32-34.

421. Vincent discloses that the fracture fluid *in which the particulates are suspended* “may be an oil base, water base, acid, *emulsion*, foam, or any other fluid.” Ex. 1007, Vincent at [0087]. A person of ordinary skill in the art would understand that an emulsion can be comprised of oil and water. See e.g., Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S. Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production). One of ordinary skill in the art would understand that in order for the particulates to be suspended, they must be mixed in the emulsion fracture fluid. Thus, Vincent teaches that the coated particulates are mixed with an oil and water emulsion.

422. Additionally, according to Vincent, “wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, [and] hydrocarbons.” Ex. 1007, Vincent at claim 2. Thus Vincent teaches the use of two hydrophobic rendering compounds: for example, polydimethyl siloxane, as the compound for rendering the surface of the particulates hydrophobic and a plant oil or hydrocarbon

satisfies as the oil. This could then be mixed with the emulsion or other fracture fluid, as described in Vincent. *See id.*, [0087].

e. **Limitation 51D: “pumping the mixture into the formation”**

423. Claim 51 further recites “pumping the mixture into the formation.” Ex. 1001 at 13:34.

424. Vincent discloses “[a] method of fracturing a subterranean formation, comprising: . . . *injecting into the fracture a fluid containing a plurality of essentially spherical particles*, wherein at least some of the particles are modified to alter the surface characteristics thereof, wherein the particles are modified by coating the particles with a hydrophobic material.” Ex. 1007, Vincent at claim 9. Therefore, Vincent discloses limitation 51D.

425. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), and the preamble of claim 51, is taught by Vincent. Therefore, independent claim 51 is anticipated by Vincent.

4. **Dependent Claims 21, 22, 40, 41, and 57 Are Anticipated by Vincent**

426. Dependent claims 21 and 40 recite a near-identical limitation, but depend from different claims. Claim 21 depends from independent claim 1, whereas claim 40 depends from independent claim 32. Similarly, dependent claims 22, 41, and 57 recite the same limitation, but depend from different claims. Claim 22 depends from independent claim 1, whereas claim 41 depends from claim 32, and claim 57 depends from claim 51. Dependent claims 21 and 40 require the particulate be one of an enumerated list of particulates, including sand. *See* Ex. 1001, '738 patent at 11:34-37; 12:63-65 (“wherein the particulates are selected from the group consisting of sand . . .”). Dependent claims 22, 41, and 57 require the particulate to be sands. *Id.* at 11:38-39; 12:66-67; 14:24-25 (“wherein the particulates are sands”). Therefore, a prior art reference that discloses the additional limitation of “sand” as the particulate anticipates claims 21, 22, 40, 41, and 57.

427. Claim 21 recites:

The composition from claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates.

428. Claim 40 recites:

The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates.

429. Claim 22 recites:

The composition of claim 1, wherein the particulates are sands.

430. Claim 41 recites:

The method of claim 32, wherein the particulates are sands.

431. Claim 57 recites:

The composition of claim 51, wherein the particulates are sands.

432. As addressed above (*see* Sections X.C.1, X.C.2, and X.C.3) and incorporated herein by reference, all of the limitations of claims 1, 32, and 51, including 1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation) and the preambles of claims 1, 32, and 51, are anticipated by Vincent.

433. Vincent teaches the additional limitation of claims 21, 22, 40, 41, and 57: “[t]he present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are *natural sands*.” Ex. 1007, Vincent at [0007]. Vincent also teaches that “[e]mbodiments of the present invention relate to

modifying the surface properties of *natural sand*, resin coated sand and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability . . . *Sands*, resin coated sands or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.” *Id.* at [0008].

434. Moreover, Vincent teaches that “particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or ‘slurry’ into which the particles have previously been introduced and suspended.” *Id.* at [0087]. Thus, the particulates are mixed with the carrier fluid before being pumped into the fracture.

435. It is my opinion that because Vincent discloses the limitations of independent claims 1, 32, and 57, 1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), and the preambles of those claims as well as the additional limitation recited in dependent



claims 21, 22, 40, 41, and 51 (sand), claims 21, 22, 40, 41, and 57 are also anticipated by Vincent.

436. Based on the foregoing analysis of Sections X.C.1-4, all of the elements of claims 1, 21, 22, 32, 40, 41, 51, and 57 (1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 21A, 22A, 40A, 41A, and 57A (sand), and the preambles of claims 1, 32, and 51) are disclosed by Vincent. Therefore, it is my opinion that one of ordinary skill in the art in April 2007 would understand that Vincent anticipates claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent.

**D. Claims 1, 21, 22, 32, 40, 41, 51, and 57 Are Anticipated by Weaver**

437. After considering the Weaver reference as it would have been understood by a person of ordinary skill in the art in April 2007, it is my opinion that Weaver discloses each and every element of claims 1, 21, 22, 32, 40, 41, 51 and 57 of the '738 patent. *See generally*, Ex. 1002F, '738 Patent v. Weaver Claim Chart. Therefore, it is my opinion that Weaver anticipates claims 1, 21, 22, 32, 40, 41, 51 and 57 of the '738 patent.

**1. Independent Claim 1 Is Anticipated by Weaver**

438. As addressed above, independent claim 1 of the '738 patent is a composition claim with four limitations.

439. Claim 1 of the '738 patent recites:

An aqueous slurry composition comprising:

water;

particulates;

a chemical compound for rendering the surface of the particulates hydrophobic; and

an oil.

440. As set forth below on a limitation-by-limitation basis, Weaver anticipates independent claim 1 of the '738 patent.

**a. Preamble for Claim 1: “An aqueous slurry composition comprising”**

441. The preamble of claim 1 of the '738 patent states, “An aqueous slurry composition comprising.” Ex. 1001 at 9:46. Weaver teaches that “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous.” Ex. 1008, Weaver at [0058]. Therefore, in my opinion, the preamble of claim 1 is met by Weaver.

**b. Limitation 1A: “water”**

442. Claim 1 recites that the composition comprises “water.” Ex. 1001 at 9:47. As stated above, Weaver discloses “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the

subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be **aqueous** or non-aqueous, **or a mixture thereof**. Where the base fluid is aqueous, it may comprise **fresh water, salt water** (e.g., water containing one or more salts dissolved therein), **brine** (e.g., saturated salt water), **or seawater**.” Ex. 1008, Weaver at [0058].

443. Moreover, Weaver discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including **aqueous gels**, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable **aqueous gels are generally comprised of water** and one or more gelling agents. **Suitable emulsions** can be comprised of two immiscible liquids such as an **aqueous liquid** or gelled liquid and a hydrocarbon.” *Id.* at [0059].

444. Thus Weaver clearly describes a composition including “water” as recited in claim 1.

**c. Limitation 1B: “particulates”**

445. Claim 1 recites that the composition comprises “particulates.” Ex. 1001 at 9:48.

446. Weaver provides “methods of modifying the surface stress-activated reactivity of **proppant particulates** used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates . . .” Ex. 1008, Weaver at Abstract. Weaver also teaches that “particulates suitable for use in

the present invention comprise any particulate that comprises a mineral surface.” *Id.* at [0018].

447. In view of the foregoing excerpt from the disclosure of Weaver, one of ordinary skill in the art would have understood Weaver to disclose this limitation.

d. **Limitation 1C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

448. Claim 1 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001 at 9:49-50.

449. The specification of the ’738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, **organosilane**, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The ’738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51).

450. Weaver discloses “[i]n certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise

depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, ***by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic*** or hydrophilic in nature.” Ex. 1008, Weaver at [0015].

451. Weaver also teaches that “[i]n some embodiments, the surface-treating reagent may comprise other compounds that are capable of ***hindering or preventing the water-wetting of a mineral surface***. Examples of suitable compounds include, but are not limited to, . . . ***organosilanes*** . . . Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.” *Id.* at [0049].

452. Moreover, in the prosecution of the ’469 application, the Examiner found that:

Weaver teaches method and composition of modifying a stress-activated reactivity of subterranean fracture faces and other surfaces in subterranean formation (Abstract). Weaver further teaches the ***surface-treating reagent may comprise compounds that are capable of hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include organosilanes, fluoroalkyl group containing silanes*** and the like where reagents may be present in a treatment fluid ([0047]).

Ex. 1004 at 184, Prosecution History of the '469 Application, Non-Final Office Action, Jan. 2, 2015 (emphasis added). I agree with the Examiner that Weaver teaches this limitation.

**e. Limitation 1D: “oil”**

453. Claim 1 further recites that the composition comprises “an oil.” Ex. 1001 at 9:50. The specification of the '738 patent states that only a “small amount of an oil” is required. *Id.* at 3:65.

454. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, *or a mixture thereof. . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils*, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.” Ex. 1008, Weaver at [0058].

455. Moreover, Weaver discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . *Suitable emulsions* can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid *and a hydrocarbon.*” *Id.* at [0059].

456. One of ordinary skill in the art would recognize, and the '738 patent confirms, oil is a “hydrocarbon”. See Ex. 1001, '738 patent at 11:31-33 (“20. The composition of claim 1, wherein the oil is selected from the group consisting of a **hydrocarbon oil**, a silicon oil, kerosene oil, a frac oil and combinations thereof.”). Weaver thus teaches the limitation of oil included in the composition as part of the treatment fluid emulsion.

457. In addition to inclusion of oils in the treatment fluids disclosed by Weaver, Weaver also discloses the addition of oils to the surface-treating reagent (i.e., organosilanes). See Ex. 1008, Weaver at [0054].

458. Moreover, the '738 specification also discloses that the oil of the composition may be formation oil. Ex. 1001 at 4:11-15 (“Alternatively the slurry containing a hydrophobizing agent can be pumped into the fracture forming the proppant pack, which can be further consolidated by oil or condensate contained in the formation.”). Thus, the oil limitation in claim 1 can be satisfied by the presence of formation oil. This is consistent with the prosecution history of the '738 patent. See Section VII.C, *supra*.

459. Weaver further discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.” Ex. 1008, Weaver at

[0058]. Weaver also discloses a “method of treating a subterranean formation comprising: providing a treatment fluid that comprises: a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; . . . and introducing the treatment fluid into the subterranean formation.”

*Id.* at claim 11.

460. One of ordinary skill in the art would understand that subterranean formations in which subterranean operations were being performed contain oil and gas. This is because one of ordinary skill in the art would recognize that a company would not expend resources on subterranean operations in a subterranean formation, such as fracturing, gravel packing, and perforating, that did not have oil or gas (a/k/a a dry well). Further, one of ordinary skill in the art would understand that “introducing the treatment fluid” containing particulate coated in surface-treating reagent and base fluid “into the subterranean formation” during a subterranean operation would necessarily mean that oil is present. *See id.* Because the subterranean formation contains oil, the composition of Weaver must therefore comprise oil.

461. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 1A (water), 1B (particulate), 1C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D (oil), and



the preamble of claim 1, is taught by Weaver. Therefore, independent claim 1 is anticipated by Weaver.

2. **Independent Claim 32 Is Also Anticipated by Weaver**

462. As described, independent claim 32 of the '738 patent is a method claim with five limitations.

463. Claim 32 of the '738 patent recites:

A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:

mixing water,

particulates,

a chemical compound for rendering the surface of the particulates hydrophobic, and

an oil;

and pumping the mixture into the formation.

464. As set forth below on a limitation-by-limitation basis, Weaver anticipates independent claim 32 of the '738 patent.

a. **Preamble for Claim 32: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”**

465. The preamble of claim 32 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 12:5-6.

466. Weaver discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, ***frac-packing operations***, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. One of ordinary skill in the art would recognize that frac-packing operations are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize that Weaver’s invention is designed to control proppant flowback.

467. Weaver also teaches that “[o]ne problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, ***proppant particulates***, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation. . . . [A] technique used to prevent flow-back problems, commonly referred to as ‘gravel packing,’ involves the placement of a gravel screen in the subterranean formation, which acts as a barrier that prevents particulates from flowing into the well bore.” *Id.* at [0004].

468. One of ordinary skill in the art would understand that Weaver’s teaching of enhancing conductivity within a subterranean formation to maximize the value of a fracturing treatment and to prevent the embedment of particulates into

surfaces of the subterranean formation to be describing operations to reduce particulate flowback. Moreover, Weaver discloses that his invention can be used in frac-packing operations, which is a method of controlling proppant flowback. *See e.g.*, Ex. 1008, Weaver at [0004].

469. Additionally, because Weaver discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the '738 patent. *See, e.g.*, Ex. 1001, '738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback).

470. Moreover, one of ordinary skill in the art would generally understand that to reap the benefits of utilizing a proppant, the proppant would need to remain in the formation fracture—i.e. not be carried out of the fractures and into the wellbore along with produced fluids being flowed back out of the well. Thus, a person of ordinary skill in the art would understand the disclosure of Weaver to be a particulate coated with a hydrophobic surface-treating reagent that is better suited than a non-coated particulate to remain in the fracture and not be subject to flowback.

471. Therefore, in my opinion, the preamble of claim 32 is met by Weaver.

**b. Limitation 32A: “mixing water”**

472. Claim 32 recites that the composition comprises “water.” Ex. 1001 at 12:6-7. As recited above, Weaver discloses “[i]n certain embodiments, particulates

treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be **aqueous** or non-aqueous, or **a mixture thereof**. Where the base fluid is aqueous, it may comprise **fresh water, salt water** (e.g., water containing one or more salts dissolved therein), **brine** (e.g., saturated salt water), **or seawater**.” Ex. 1008, Weaver at [0058].

473. Moreover, Weaver discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including **aqueous gels**, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable **aqueous gels are generally comprised of water** and one or more gelling agents. **Suitable emulsions** can be comprised of two immiscible liquids such as an **aqueous liquid** or gelled liquid and a hydrocarbon.” *Id.* at [0059].

474. Weaver describes that the particulates are mixed with the surface-modifying agents “prior to transporting the particulates to a job site, or in a treatment performed ‘on-the-fly.’ The term ‘on-the-fly’ is used herein to mean that one flowing stream comprising particulates is continuously introduced into another flowing stream comprising the surface-treating reagent so that the streams are combined and mixed while continuing to flow as a single stream as part of the on-going treatment at the job site. Such mixing can also be described as ‘real-time’ mixing.” *Id.* at [0056].

475. Thus Weaver clearly describes a composition including “mixing water” as recited in claim 32.

c. **Limitation 32B: “particulates”**

476. Claim 32 recites that the composition comprises “particulates.” Ex. 1001 at 12:7.

477. Weaver provides “methods of modifying the surface stress-activated reactivity of *proppant particulates* used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates . . .” Ex. 1008, Weaver at Abstract. Weaver also teaches that “particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface.” *Id.* at [0018]. As explained above, Weaver discloses that the particulates are mixed with the surface-treating agent prior to transport to the well site or on-the-fly. *Id.* at [0056].

478. Therefore, one of ordinary skill in the art would have understood Weaver to disclose this limitation.

d. **Limitation 32C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

479. Claim 32 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001 at 12:7-8.

480. The specification of the '738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, **organosilane**, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The '738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51).

481. Weaver discloses “[i]n certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, **by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic** or hydrophilic in nature.” Ex. 1008, Weaver at [0015].

482. Weaver also teaches that “[i]n some embodiments, the surface-treating reagent may comprise other compounds that are capable of **hindering or preventing the water-wetting of a mineral surface**. Examples of suitable compounds include,

but are not limited to, . . . **organosilanes** . . . Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.” *Id.* at [0049].

483. As stated, in the prosecution of the ’469 application, the Examiner found that:

Weaver teaches method and composition of modifying a stress-activated reactivity of subterranean fracture faces and other surfaces in subterranean formation (Abstract). Weaver further teaches the ***surface-treating reagent may comprise compounds that are capable of hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include organosilanes, fluoroalkyl group containing silanes*** and the like where reagents may be present in a treatment fluid ([0047]).

Ex. 1004 at 184, Prosecution History of the ’469 Application, Non-Final Office Action, Jan. 2, 2015 (emphasis added). I agree with the Examiner that Weaver teaches this limitation of claim 32.

**e. Limitation 32D: “oil”**

484. Claim 32 further recites that the composition comprises “an oil.” Ex. 1001 at 12:8. The specification of the ’738 patent states that only a “small amount of an oil” is required. *Id.* at 3:65.

485. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous,

*or a mixture thereof. . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.”* Ex. 1008, Weaver at [0058].

486. Moreover, Weaver discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . **Suitable emulsions** can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid **and a hydrocarbon.**” *Id.* at [0059].

487. One of ordinary skill in the art would recognize, and the ’738 patent confirms, that oil is a type of “hydrocarbon.” *See* Ex. 1001, ’738 patent at 11:31-33 (“20. The composition of claim 1, wherein the oil is selected from the group consisting of a **hydrocarbon oil**, a silicon oil, kerosene oil, a frac oil and combinations thereof.”). Weaver thus teaches the limitation of oil included in the composition in the form of a treatment fluid including a mixture of oil and water, as well as an oil and water emulsion.



488. In addition to inclusion of oils in the treatment fluids disclosed by Weaver, Weaver also discloses the addition of oils to the surface-treating reagent (i.e., organosilanes). *See* Ex. 1008, Weaver at [0054].

489. Vincent thus teaches the limitation of oil included in the mixture that is pumped into the formation.

f. **Limitation 32E: “pumping the mixture into the formation”**

490. Claim 32 further recites “pumping the mixture into the formation.” Ex. 1001 at 12:8-9.

491. Weaver teaches “[a] method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified mineral surface; and *introducing the treatment fluid into the subterranean formation.*” Ex. 1008, Weaver at claim 23; *see also id.* at claim 11.

492. Therefore, Vincent discloses limitation 32E.

493. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 32A (water), 32B (particulate), 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 32D (oil), 32E (pumping the mixture into the formation), and the preamble of claim 32, is taught by Weaver. Therefore, independent claim 32 is anticipated by Weaver.

3. **Independent Claim 51 is Anticipated by Weaver**

494. Independent claim 51 of the '738 patent is a method claim with four limitations.

495. Claim 51 of the '738 patent recites:

A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:

contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic,

separating the particulate from the medium;

and blending the hydrophobic particulate with water and an oil to form a mixture;

and pumping the mixture into the formation.

496. As set forth below on a limitation-by-limitation basis, Weaver anticipates independent claim 51 of the '738 patent.

a. **Preamble for Claim 51: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”**

497. The preamble of claim 51 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 13:28-29. Weaver discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, *frac-packing operations*, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. One of ordinary skill in the art would recognize that frac-

packing operations are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize that Weaver's invention is designed to control proppant flowback.

498. Weaver also teaches that “[o]ne problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, **proppant particulates**, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation. . . . [A] technique used to prevent flow-back problems, commonly referred to as ‘gravel packing,’ involves the placement of a gravel screen in the subterranean formation, which acts as a barrier that prevents particulates from flowing into the well bore.” *Id.* at [0004]. Weaver proceeds that “one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, . . . preventing the embedment of particulates into the surfaces of a subterranean formation.” *Id.* at [0014].

499. One of ordinary skill in the art would understand that Weaver's teaching of enhancing conductivity within a subterranean formation to maximize the value of a fracturing treatment and to prevent the embedment of particulates into

surfaces of the subterranean formation to be describing operations to reduce proppant flowback. Moreover, Weaver discloses that his invention can be used in frac-packing operations, which is a method of controlling proppant flowback. *See e.g.*, Ex. 1008, Weaver at [0004].

500. Additionally, because Weaver discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the '738 patent. *See, e.g.*, Ex. 1001, '738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback).

501. Moreover, one of ordinary skill in the art would generally understand that to reap the benefits of utilizing a proppant, the proppant would need to remain in the formation fracture—i.e. not be carried out of the fractures and into the wellbore along with produced fluids being flowed back out of the well. Thus, a person of ordinary skill in the art would understand the disclosure of Weaver (and Nguyen, Dawson, or Vincent) to be a particulate coated with a hydrophobic surface-treating reagent that is better suited than a non-coated particulate to remain in the fracture and not be subject to flowback.

b. **Limitation 51A: “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic”**

502. Claim 51 recites “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic.” Ex. 1001 at 13:29-31.

503. Weaver teaches that one “suitable method[] for allowing the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate includes . . . solvent extraction methods.” *Id.* at [0057]. In such an embodiment, “[t]he solution comprising the surface treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced to a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface.” *Id.* As discussed, Weaver discloses examples of organo-silicon compounds that are the same as those disclosed in the ’738 patent, including organosilanes. *See supra*, ¶¶ 448-452.

504. Thus Weaver clearly describes contacting the particulates with an organo-silicon compound, which, as stated above, is an example of “a chemical compound to render the surface of the particulates hydrophobic” as required by claim 51.

c. **Limitation 51B: “separating the particulate from the medium”**

505. Claim 51 recites that the now-coated particulates are “separate[ed] . . . from the medium.” Ex. 1001 at 13:32.

506. Weaver teaches that one “suitable method[] for allowing the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate includes . . . solvent extraction methods.” *Id.* at [0057]. In such an embodiment, “[t]he solution comprising the surface treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. *The treated particulates then may be introduced to a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface.*” *Id.*

507. One of ordinary skill in the art would understand that extracting the solvent from the treated particulates is a method of separation. Thus, Weaver discloses that the now-coated particulates are separated from the solvent solution.

508. In view of the foregoing excerpt from the disclosure of Weaver, one of ordinary skill in the art would have understood Weaver to disclose this limitation.

d. **Limitation 51C: “blending the hydrophobic particulate with water and an oil to form a mixture”**

509. Claim 51 recites that the hydrophobic coated particulate are blended “with water and an oil to form a mixture.” Ex. 1001 at 13:32-34.

510. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, ***or a mixture thereof***. . . . Any organic liquid in which a water solution of salts ***can be emulsified also may be suitable for use as a base fluid*** in the methods of the present invention.” Ex. 1008, Weaver at [0058].

511. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . ***Suitable emulsions*** can be comprised of two immiscible liquids such as ***an aqueous liquid*** or gelled liquid ***and a hydrocarbon***.” *Id.* at [0059].

512. One of ordinary skill in the art would recognize, and the ’738 patent confirms, that oil is a type of “hydrocarbon.” *See* Ex. 1001, ’738 patent at 11:31-33 (“20. The composition of claim 1, wherein the oil is selected from the group consisting of a ***hydrocarbon oil***, a silicon oil, kerosene oil, a frac oil and combinations thereof.”). Weaver thus teaches the limitation of oil included in the composition in the form of a treatment fluid including a mixture of oil and water, as well as an oil and water emulsion.

513. Thus, Weaver teaches that the coated particulates are mixed with an oil and water.

e. **Limitation 51D: “pumping the mixture into the formation”**

514. Claim 51 further recites “pumping the mixture into the formation.” Ex. 1001 at 13:34.

515. Weaver teaches “[a] method of treating a subterranean formation comprising: providing a *treatment fluid that comprises a base fluid, and a plurality of particulates*, at least one of which comprises a modified mineral surface; and *introducing the treatment fluid into the subterranean formation.*” Ex. 1008, Weaver at claim 23; *see also id.* at claim 11.

516. Therefore, Weaver discloses limitation 51D.

517. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), and the preamble of claim 51, is taught by Weaver. Therefore, independent claim 51 is anticipated by Weaver.

**4. Dependent Claims 21, 22, 40, 41, and 57 Are Anticipated by Weaver**

518. Dependent claims 21, and 40 recite a near-identical limitation, but depend from different claims. Claim 21 depends from independent claim 1, whereas claim 40 depends from independent claim 32. Similarly, dependent claims 22, 41, and 57 recite the same limitation, but depend from different claims. Claim 22



depends from independent claim 1, whereas claim 41 depends from claim 32, and claim 57 depends from claim 51. Dependent claims 21 and 40 require the particulate be one of an enumerated list of particulates, including sand. *See* Ex. 1001, '738 patent at 11:34-37; 12:63-65 (“wherein the particulates are selected from the group consisting of sand . . .”). Dependent claims 22, 41, and 57 require the particulate to be sands. *Id.* at 11:38-39; 12:66-67; 14:24-25 (“wherein the particulates are sands”). Therefore, a prior art reference that discloses the additional limitation of “sand” as the particulate anticipates claims 21, 22, 40, 41, and 57.

519. Claim 21 recites:

The composition from claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates.

520. Claim 40 recites:

The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates.

521. Claim 22 recites:

The composition of claim 1, wherein the particulates are sands.

522. Claim 41 recites:

The method of claim 32, wherein the particulates are sands.

523. Claim 57 recites:

The claim of claim 51, wherein the particulates are sands.

524. As addressed above (*see* Sections X.D.1, X.D.2, and X.D.3) and incorporated herein by reference, all of the limitations of claims 1, 32, and 57, including 1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), and the preambles of claims 1, 32, and 51, are anticipated by Weaver.

525. Weaver teaches “[t]he particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, *sand*.” Ex. 1008, Weaver at [0018].

526. Weaver also teaches that “the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which

may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.” *Id.* at [0057]. Thus, the sand particulates are mixed with the treatment fluid before being pumped down the formation.

527. It is my opinion that because Weaver discloses the limitations of independent claims 1, 32, and 51, 1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation) and the preambles of those claims as well as the additional limitation recited in dependent claims 21, 22, 40, 41, and 57 (sand), claims 21, 22, 40, 41, and 57 are also anticipated by Weaver.

528. Based on the foregoing analysis of Sections X.D.1-4, all of the elements of claims 1, 21, 22, 32, 40, 41, 51, and 57 [1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting the particulates with a medium containing a

chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), 21A, 22A, 40A, 41A, and 57A (sand), and the preambles of claims 1, 32, and 51] are disclosed by Weaver. Therefore, it is my opinion that one of ordinary skill in the art in April 2007 would understand that Weaver anticipates claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent.

**E. Claims 1, 21 and 22 Are Anticipated by Needham**

529. After considering the Needham reference as it would have been understood by a person of ordinary skill in the art in April 2007, as well as the January 2, 2015 Office Action rejecting claim 13 of the '469 application as anticipated by Needham, it is my opinion that Needham discloses each and every element of claims 1, 21, and 22 of the '738 patent. *See generally*, Ex. 1002G, '738 Patent v. Needham Claim Chart Therefore, it is my opinion that Needham anticipates claims 1, 21, and 22.

**1. Independent Claim 1 Is Anticipated by Needham**

530. Independent claim 1 of the '738 patent is a composition claim with four limitations.

531. Claim 1 of the '738 patent recites:

An aqueous slurry composition comprising:

water;

particulates;  
a chemical compound for rendering the surface of the particulates  
hydrophobic; and  
an oil.

532. As set forth below on a limitation-by-limitation basis, Nguyen anticipates independent claim 1 of the '738 patent.

a. **Preamble for Claim 1: “An aqueous slurry composition comprising”**

533. The preamble of claim 1 of the '738 patent states, “An aqueous slurry composition comprising.” Ex. 1001 at 9:46. Needham teaches that a “[c]arrier fluid, preferably an aqueous fluid such as salt water or a hydrocarbon liquid such as diesel fuel, having the proppant coated with organo-silicon compound suspended therein is then pumped into the fracture.” Ex. 1008, Needham at 3:19-23. The combination of the aqueous carrier fluid with the coated proppant yields the claimed slurry composition. Thus, Needham teaches the preamble of claim 1.

b. **Limitation 1A: “water”**

534. Claim 1 recites that the composition comprises “water.” Ex. 1001 at 9:47. As stated by the Examiner in the prosecution of the '469 application, Needham teaches “[c]arrier fluid, *preferably an aqueous fluid such as salt water* having the proppant coated with organo-silicon compound suspended therein is then pumped into the fracture.” Ex. 1004 at 182, File History of the '469 Application, Non-Final Office Action, January 2, 2015 (citing Needham [Ex. 1009] at 3:19-23). I agree with

the Examiner that Needham discloses a slurry composition that includes water. It is also clear that the water is mixed with the coated proppant because the proppant is suspended in the carrier fluid and then pumped into the fracture.

535. Moreover, a person of ordinary skill in the art would understand that Needham discloses that the water is mixed with the proppant, since it is pumped into the fracture. Ex. 1009, Needham at 4:1-6. Thus, Needham clearly describes a composition including “water” as recited in claim 1.

**c. Limitation 1B: “particulates”**

536. Claim 1 recites that the composition comprises “particulates.” Ex. 1001 at 9:48.

537. The Examiner stated, while rejecting claim 13 of the ’469 patent as anticipated by Needham, that “Needham further teaches propping agents used in hydraulic fracturing normally range in particle size from about 8 to about 60 mesh.” Ex. 1004 at 183, File History of the ’469 Application, Non-Final Office Action, January 2, 2015 (citing Needham [Ex. 1009] at 3:5-12). I agree with the Examiner that Needham discloses particulates as described in this limitation.

538. In view of the foregoing excerpt from the disclosure of Needham and the USPTO’s rejection of a similar limitation as anticipated by Needham, one of ordinary skill in the art would have understood Needham to disclose this limitation.

d. **Limitation 1C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

539. Claim 1 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001 at 9:49-50.

540. The specification of the '738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The '738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51.

541. The '738 patent also recites:

Examples of organosilanes include:  $\text{CH}_3\text{SiCl}_3$ ,  $\text{CH}_3\text{CH}_2\text{SiCl}_3$ ,  $(\text{CH}_3)_2\text{SiCl}_2$ ,  $(\text{CH}_3\text{CH}_2)_2\text{SiCl}_2$ ,  $(\text{C}_6\text{H}_5)_2\text{SiCl}_2$ ,  $(\text{C}_6\text{H}_5)\text{SiCl}_3$ ,  **$(\text{CH}_3)_3\text{SiCl}$** ,  $\text{CH}_3\text{HSiCl}_2$ ,  $(\text{CH}_3)_2\text{HSiCl}$ ,  $\text{CH}_3\text{SiBr}_3$ ,  $(\text{C}_6\text{H}_5)\text{SiBr}_3$ ,  $(\text{CH}_3)_2\text{SiBr}_2$ ,  $(\text{CH}_3\text{CH}_2)_2\text{SiBr}_2$ ,  $(\text{C}_6\text{H}_5)_2\text{SiBr}_2$ ,  $(\text{CH}_3)_3\text{SiBr}$ ,  $\text{CH}_3\text{HSiBr}_2$ ,  $(\text{CH}_3)_2\text{HSiBr}$ ,  $\text{Si}(\text{OCH}_3)_4$ ,  $\text{CH}_3\text{Si}(\text{OCH}_3)_3$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_3)_3$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_3$ ,  $\text{CH}_3\text{Si}[\text{O}(\text{CH}_2)_3\text{CH}_3]_3$ ,

$\text{CH}_3\text{CH}_2\text{Si}(\text{OCH}_2\text{CH}_3)_3$ ,  $\text{C}_6\text{H}_5\text{Si}(\text{OCH}_3)_2$ ,  $\text{C}_6\text{H}_5\text{CH}_2\text{Si}(\text{OCH}_3)_3$ ,  
 $\text{C}_6\text{H}_5\text{Si}(\text{OCH}_2\text{CH}_3)_3$ ,  $\text{CH}_2=\text{CHCH}_2\text{Si}(\text{OCH}_3)_3$ ,  $(\text{CH}_3)_2\text{Si}(\text{OCH}_3)_2$ ,  
 $(\text{CH}_2=\text{CH})\text{Si}(\text{CH}_3)_2\text{Cl}$ ,  $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$ ,  
 $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_2$ ,  $(\text{CH}_3)_2\text{Si}[\text{O}(\text{CH}_2)_3\text{CH}_3]_2$ ,  
 $(\text{CH}_3\text{CH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_3)_2$ ,  
 $(\text{C}_6\text{H}_5\text{CH}_2)_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$ ,  
 $(\text{CH}_2=\text{CH})_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_3)_2$ ,  $(\text{CH}_3)_3\text{SiOCH}_3$ ,  
 $\text{CH}_3\text{HSi}(\text{OCH}_3)_2$ ,  $(\text{CH}_3)_2\text{HSi}(\text{OCH}_3)$ ,  $\text{CH}_3\text{Si}(\text{OCH}_2\text{CH}_2\text{CH}_3)_3$ ,  
 $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{CH}_3)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $(\text{CH}_2=\text{CH})_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{CH}_2=\text{CHCH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  
 $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_2$ ,  $\text{CH}_3\text{Si}(\text{CH}_3\text{COO})_3$ , 3-  
 aminotriethoxysilane, methyldiethylchlorosilane, butyltrichlorosilane,  
 diphenyldichlorosilane, vinyltrichlorosilane, methyltrimethoxysilane,  
 vinyltriethoxysilane, vinyltris(methoxyethoxy)silane,  
 methacryloxypropyltrimethoxysilane,  
 glycidoxypropyltrimethoxysilane, aminopropyltriethoxysilane,  
 divinyl-di-2-methoxysilane, ethyltributoxysilane,  
 isobutyltrimethoxysilane, hexyltrimethoxysilane, n-  
 octyltriethoxysilane, dihexyldimethoxysilane, octadecyltrichlorosilane,  
 octadecyltrimethoxysilane, octadecyldimethylchlorosilane,  
 octadecyldimethylmethoxysilane and quaternary ammonium silanes  
 including 3-(trimethoxysilyl) propyldimethyloctadecyl ammonium  
 chloride, 3-(trimethoxysilyl)propyldimethyloctadecyl ammonium  
 bromide, 3-(trimethylethoxysilylpropyl)didecylmethyl ammonium  
 chloride, triethoxysilyl soyapropyl dimoniumchloride, 3-  
 (trimethylethoxysilylpropyl)didecylmethyl ammonium bromide, 3-



(trimethylethoxysilylpropyl)didecylmethyl ammonium bromide,  
triethoxysilyl soyapropyl dimonium bromide,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_5)_3\text{Cl}^-$ ,  $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_5)_3\text{Br}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{CH}_3)_3\text{Cl}^-$ ,  $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{P}^+(\text{C}_6\text{H}_{13})_3\text{Cl}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{C}_4\text{H}_9\text{Cl}$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{C}_6\text{H}_5\text{Cl}$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{CH}_2\text{OHCl}^-$ ,  
 $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{C}_2\text{H}_5)_3\text{Cl}^-$ ,  
 $(\text{C}_2\text{H}_5\text{O})_3\text{Si}(\text{CH}_2)_3\text{N}^+(\text{CH}_3)_2\text{C}_{18}\text{H}_{37}\text{Cl}^-$ .

(*id.* at 5:66-6:43) and that “[a]mong different organosiloxane compounds which are useful for the present invention, polysiloxanes modified with organic amphoteric or cationic groups including organic betaine polysiloxanes and organic quaternary siloxanes are examples” (*id.* at 6:44-48).

542. Needham teaches that “an object of this invention is to provide improved propping agents for use in hydraulic fracturing of subterranean formations. Another object of this invention is to provide **particulate material coated with a water repellent** useful in hydraulic fracturing of subterranean formations. A further object of this invention is to coat particulate solids useful as propping agents to increase the strength of the propping agent in the presence of water.” Ex. 1009, Needham 1:44-52.

543. Needham further teaches that the propping agent “compris[es] an inert solid particle coated with a **water-repellent organo-silicon material** is displaced

down a well and into a fracture in a subterranean hydrocarbon-bearing formation. In accordance with a specific embodiment, sand coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.” *Id.* at 1:56-67.

544. As stated by the Examiner in the rejection of claim 13 of the ’469 application as anticipated by Needham, Needham teaches that “[t]he water-repellent material employed in the method of the present invention to coat inert particulate materials for use as propping agents is an oil-soluble organo-silicon compound. Representative organo-silicon compounds that can be used include polyalkylsiloxanes such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additionally, polyalkylarylsiloxanes such as polymethylphenylsiloxane are also usable as a coating material of the invention. ***Chlorosilanes such as chlorotrimethylsilane*** and other silyl donors can also be used, as well as various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes, and the like, as the water-repellent coating of the propping agent.” Ex. 1004 at 182, File History of the ’469 Application, Non-Final Office Action, January 2, 2015 (citing Needham [Ex. 1009] at 2:25-41). I agree with the Examiner that Needham anticipates this limitation because chlorotrimethylsilane ((CH<sub>3</sub>)<sub>3</sub>SiCl) is one of the organo-silicon

compounds identified in the '738 specification as a hydrophobic rendering compound. *Compare* Ex. 1009, Needham at 2:33-34 *with* Ex. 1001, '738 patent at 6:2.

**e. Limitation 1D: “oil”**

545. Claim 1 further recites that the composition comprises “an oil.” Ex. 1001 at 9:50. The specification of the '738 patent states that only a “small amount of an oil” is required. *Id.* at 3:65.

546. The '738 specification discloses that the oil of the composition may be formation oil. Ex. 1001 at 4:11-15 (“Alternatively the slurry containing a hydrophobizing agent can be pumped into the fracture forming the proppant pack, which can be further consolidated by oil or condensate contained in the formation.”). Thus, the oil limitation in claim 1 can be satisfied by the presence of formation oil. This is consistent with the prosecution history of the '738 patent. *See* Section VII.C, *supra*.

547. Needham teaches “[i]n a method of fracturing an underground zone or formation in **oil and gas wells**, the steps of (a) injecting into a well a fracturing fluid to form a fracture in said formation, (b) injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound prepared by dissolving the organo-silicon in a solvent therefor, admixing the resultant solution with particulate material, and thereafter evaporating

the solvent to form a silicon-coated particulate material, and (c) closing the well and maintaining the injection pressure on the formation until the fracture approaches the normal formation pressure.” Ex. 1008, Needham at 4:1-15.

548. Needham further discloses that the invention “relates to the treatment of subterranean **hydrocarbon-bearing formations**” (*Id.* at 1:4-5) and that in accordance with the invention, “a novel propping agent comprising an inert solid particle coated with a water-repellent organo-silicon material is displaced down a well and into a fracture **in a subterranean hydrocarbon-bearing formation**” (*Id.* at 1:56-60).

549. One of ordinary skill in the art would understand that hydrocarbon-bearing formations contain oil, as is evidenced by claim 1 of Needham. *See* Ex. 1009, Needham at 4:1-2 (“In a method of fracturing an underground zone or formation in **oil and gas wells**”). Further, one of ordinary skill in the art would understand that injecting the composition containing water and silicon-coated particulate into an oil and gas well or a hydrocarbon-bearing formation would necessarily mean that oil is present. Because the formation contains oil, the composition of Needham must therefore comprise oil.

550. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 1A (water), 1B (particulate), 1C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D (oil), and

the preamble of claim 1, is taught by Needham. Therefore, independent claim 1 is anticipated by Needham.

**2. Dependent Claims 21 and 22 Are Anticipated by Needham**

551. Dependent claims 21 and 22 depend from claim 1. Both dependent claims 21 and 22 recite that the particulate in the composition of claim 1 is sand.

552. Claim 21 recites:

The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates.

553. Claim 22 recites:

The composition of claim 1, wherein the particulates are sands.

554. As addressed above (*see* Section X.E.1) and incorporated here by reference, all of the limitations of claim 1, including 1A (water), 1B (particulates), 1C (a chemical compound for rendering the surface of the particulates hydrophobic), 1D (oil), and the preamble of claim 1, are anticipated by Needham.

555. The only additional limitation recited in claims 21 and 22, limitation 21A and 22A, is that the particulate disclosed in claim 1 is sand. Ex. 1001 at 11:34-39. Dependent claim 21 recites a list of alternative particulates, including sand. Dependent claim 22 recites that the particulate of claim 1 is sand. Thus, if a prior art reference discloses one alternative, i.e. sand, the prior art reference would anticipate both claims 21 and 22.

556. Needham discloses a “method of fracturing an underground zone or formation in oil and gas wells, the steps of . . . injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound” (Ex. 1009, Needham at 4:1-6) “wherein said particulate material comprises **sand**” (*id.* at 4:16-17; 4:48-49). Needham further discloses “[i]n accordance with a specific embodiment, **sand** coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the solvent to form a **silicon-coated sand which can then be used as a propping medium** in fractured formations containing hydrocarbons.” *Id.* at 1:61-68.

557. Needham therefore discloses the additional limitation of claims 21 and 22, limitation 21A and 21A.

558. It is my opinion that because Needham discloses the limitations of independent claim 1 (limitations 1A (water), 1B (particulates), 1C (a chemical compound for rendering the surface of the particulates hydrophobic), 1D (oil) and the preamble of claim 1) as well as the additional limitation recited in dependent claims 21 and 22 (limitations 21A and 22A – sand), claims 21 and 22 are also anticipated by Needham.

559. Based on the foregoing analysis of Sections X.E.1-2, all of the elements of claims 1, 21, and 22 (limitations 1A (water), 1B (particulates), 1C (a chemical

compound for rendering the surface of the particulates hydrophobic), 1D (oil), 21A and 22A (type of particulate), and the preamble of claim 1) are disclosed by Needham. Therefore, it is my opinion that one of ordinary skill in the art in April 2007 would understand that Needham anticipates claims 1, 21, and 22 of the '738 patent.

**XI. OPINIONS ON THE VALIDITY OF CLAIMS 1, 21, 22, 32, 40, 41, 51, AND 57 OF THE '738 PATENT UNDER 35 U.S.C. § 103**

**A. Claims 1, 21, 22, 32, 40, 41, 51, and 57 Are Obvious over Nguyen in view of Weaver or Plummer**

560. As discussed in Sections X.A and X.D above (and incorporated by reference herein), both Nguyen and Weaver anticipate claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent because each reference discloses each and every element of the claims. Furthermore, and in the alternative, the disclosure of Nguyen in view of Weaver or Plummer also renders claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent obvious. *See generally*, Ex. 1002H, '738 Patent v. Nguyen in view of Weaver or Plummer Claim Chart.

561. One of ordinary skill in the art at the time of the invention would have been motivated to combine Nguyen and Weaver because both are directed to methods for modifying sand particulates with a water-repellent compound (specifically organosilanes) for use in hydraulic fracturing operations. Further, one of ordinary skill in the art would be motivated to substitute the aqueous carrier fluid

of Nguyen with the oil and water emulsion treatment fluid of Weaver to arrive at the invention of the '738 patent, since both were well known in the art and there was a high likelihood of success. Additionally, and in the alternative, a person of ordinary skill in the art would be motivated to substitute the aqueous carrier fluid of Nguyen with the oil and water liquid carrier disclosed in Plummer (Ex. 1010 at 7:18-20) for the same reason. Moreover, a person of ordinary skill in the art would be motivated to add oil to the Nguyen slurry as a lubricant friction-reducer, or to reduce clay swelling, as taught by Plummer (Ex. 1010 at 4:28-33) to improve the compositions and methods of Nguyen in hydraulic fracturing operations.

**1. Independent Claim 1 Is Obvious**

562. As discussed in Section X.A.1 and Section X.D.1 above (and incorporated by reference herein), in addition to the preamble of claim 1, claim 1 has four limitations: 1A (water), 1B (particulates), 1C (a chemical compound for rendering the surface of the particulate hydrophobic), and 1D (oil). Nguyen and Weaver each discloses all of those limitations and the preamble. See ¶¶ 176-199; 438-461. The combination of Nguyen in view of Weaver or Plummer further discloses each and every limitation of challenged claim 1 of the '738 patent.

**a. Preamble for Claim 1: “An aqueous slurry composition comprising”**

563. The preamble of claim 1 of the '738 patent states, “An aqueous slurry composition comprising.” Ex. 1001, '738 patent at 9:46.



564. As stated in ¶¶ 179 and 441 (and incorporated by reference herein), Nguyen and Weaver meet this preamble. Nguyen recites “mixing particles in a carrier fluid comprising a water-based polymer to form a mixture.” Ex. 1005, Nguyen at claim 63. Nguyen also states that “[t]he water utilized to form the fracturing fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other aqueous liquid which does not adversely react [to] other components of the fracturing fluid.” *Id.* at [0013].

565. Weaver teaches that “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous.” Ex. 1008, Weaver at [0058].

566. Thus, both Nguyen and Weaver, and accordingly Nguyen in view of Weaver, meet this preamble.

**b. Limitation 1A: “water”**

567. Claim 1 recites that the composition comprises “water.” Ex. 1001 at 9:47. As stated in ¶¶ 180-183 and 442-444 (and incorporated by reference herein), both Nguyen and Weaver teach the use of water.

568. Nguyen discloses that “[t]he most common fracturing fluid utilized heretofore which is generally preferred for use in accordance with this invention is

comprised of water.” Ex. 1005, Nguyen at [0012]. Nguyen further discloses, in claim 63, “mixing particles in a carrier fluid comprising a water-based polymer to form a mixture.” *Id.* at claim 63. Nguyen further teaches “[t]he method of claim 63 wherein the carrier fluid comprises water and a gelling agent.” *Id.* at claim 65. Nguyen also states that “[t]he water utilized to form the fracturing fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other aqueous liquid which does not adversely react [to] other components of the fracturing fluid.” *Id.* at [0013]. Nguyen also teaches that “[f]racturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and **emulsions**.” Ex. 1005, Nguyen at [0011]. A person of ordinary skill in the art would recognize that emulsions used in oil and gas well service operations are typically comprised of a mixture of oil and water. *See, e.g.*, Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S. Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production).

569. Weaver discloses “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a *mixture thereof*. Where the base fluid is aqueous, it may comprise *fresh water, salt water* (e.g., water containing one or more salts dissolved therein), *brine* (e.g., saturated salt water), *or seawater*.” Ex. 1008, Weaver at [0058].

570. Moreover, Weaver discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including *aqueous gels*, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable *aqueous gels are generally comprised of water* and one or more gelling agents. *Suitable emulsions* can be comprised of two immiscible liquids such as an *aqueous liquid* or gelled liquid and a hydrocarbon.” *Id.* at [0059].

571. Thus, both Nguyen and Weaver teach that water can be included in the slurry as the carrier/treatment fluid of the proppant. Limitation 1A would have therefore been obvious over Nguyen in view of Weaver.

**c.     Limitation 1B: “particulates”**

572. Claim 1 recites that the composition comprises “particulates.” Ex. 1001 at 9:48. As stated in ¶¶ 184-186 and 445-447 (and incorporated by reference herein), both Nguyen and Weaver teach the use of particulates.

573. Nguyen discloses that “[t]he proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like. . . . The preferred particles are sand.” Ex. 1005, Nguyen at [0016]. Nguyen’s particles are pumped into the well as part of the slurry composition to promote the recovery of hydrocarbons. *Id.* at Abstract.

574. Weaver provides “methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates . . .” Ex. 1008, Weaver at Abstract. Weaver also teaches that “particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface.” *Id.* at [0018].

575. Thus, both Nguyen and Weaver teach the use of particulates in the slurry. Limitation 1B would have therefore been obvious over Nguyen in view of Weaver.

d. **Limitation 1C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

576. Claim 1 also recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001,

'738 patent at 9:49-50. As stated in ¶¶ 187-191 and 448-452 (and incorporated by reference herein), both Nguyen and Weaver teach this limitation.

577. The specification of the '738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The '738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51.

578. The '738 patent also contains a list of suitable examples of organosilanes, including  $(CH_3)_2SiCl_2$  [dichlorodimethylsilane],  $(C_6H_5)_2SiCl_2$  [dichlorodiphenylsilane],  $(CH_3)_3SiCl$  [chlorotrimethylsilane], and  $(C_6H_5)_2Si(OCH_3)_2$  [dimethoxydiphenylsilane]. *See id.* at 5:66-6:12.

579. Nguyen teaches “particles [that] are dry coated with a water-repellent organo-silicon material.” Ex. 1005, Nguyen at [0005], claim 1. Nguyen further teaches that

[r]epresentative organo-silicon compounds that can be used in this embodiment include polyalkylsiloxane such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additional organo-silicon compounds that can be used in this embodiment include polyalkylarylsiloxanes . . . chlorosilanes such as ethylchlorosilane, **chlorotrimethylsilane** and other silyl donors. Also, various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes can be used such as tetraethoxysilane, **dimethoxydiphenylsilane**, **dichlorodimethylsilane**, **dichlorodiphenylsilane**, poly(dimethylsiloxane, poly[oxy(dimethylsilylene)] and other such materials that will be well known to those skilled in the art. . . . Furthermore, organofunctional silanes can be used in this embodiment.

*Id.* at [0006].

580. Nguyen therefore also discloses this limitation of claim 1 because Nguyen discloses a number of “hydrophobizing agents” listed in the ’738 patent, including **chlorotrimethylsilane**, **dichlorodimethylsilane**, **dichlorodiphenylsilane**, and **dimethoxydiphenylsilane**. Compare Ex. 1005, Nguyen at [0006] with Ex. 1001, ’738 patent at 6:1-2, 6:12; see also NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data: Chlorotrimethylsilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?Name=chlorotrimethylsilane&Units=SI>; NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data: Dimethyldichlorosilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?ID=75-78-5>; NATIONAL INSTITUTE OF

STANDARDS AND TECHNOLOGY, Standard Reference Data: Dichlorodiphenylsilane  
(2017), available at

<http://webbook.nist.gov/cgi/cbook.cgi?Name=dichlorodiphenylsilane&Units=SI>;

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data:  
Diphenyldimethoxysilane (2017), available at

<http://webbook.nist.gov/cgi/cbook.cgi?Name=dimethoxydiphenylsilane&Units=SI>.

581. Weaver discloses “[i]n certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, **by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic** or hydrophilic in nature.” Ex. 1008, Weaver at [0015].

582. Weaver also teaches that “[i]n some embodiments, the surface-treating reagent may comprise other compounds that are capable of **hindering or preventing the water-wetting of a mineral surface**. Examples of suitable compounds include, but are not limited to, . . . **organosilanes** . . . Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.” *Id.* at [0049].

583. Therefore, both Nguyen and Weaver teach particulates coated with a chemical compound for rendering the particulates hydrophobic—and specifically that the chemical compound is an organosilane. This opinion is supported by the USPTO, because in the prosecution of the '469 application, the Examiner rejected claim 13 (which contained a limitation similar to limitation 1C) as anticipated by both Nguyen and Weaver because each taught particles coated in water-repellent compounds. *See* Ex. 1004 at 183-84, File History of the '469 Application, Non-Final Office Action, Jan. 2, 2015 (stating that “Nguyen teaches dry coated particles with a water-repellent organo-silicon materials” and that “Weaver further teaches the surface-treating reagent may comprise compounds that are capable of hindering or preventing the water-wetting of a mineral surface.”). I agree with the Examiner that both Nguyen and Weaver teach this limitation. Thus, the combination of Nguyen in view of Weaver teaches limitation 1C.

**e. Limitation 1D: “oil”**

584. Claim 1 further recites that the composition comprises “an oil.” Ex. 1001 at 9:50. As stated in ¶¶ 192-199 and 453-461 (and incorporated by reference herein), both Nguyen and Weaver teach this limitation.

585. Nguyen teaches that “[f]racturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and *emulsions*.” Ex. 1005, Nguyen at [0011]. A person of ordinary



skill in the art would recognize that emulsions used in oil and gas well service operations are typically comprised of a mixture of oil and water. *See, e.g.*, Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S. Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production).

586. Additionally, Nguyen teaches that if “the particles are coated when the particles are flowing, the solvent should be readily miscible in the water-based gel carrier fluid. Organic solvents that can be used as a carrier for the organo-silicon coating material including *kerosene* [and] lighter grades of diesel fuel.” Ex. 1005, Nguyen at [0008]. One of ordinary skill in the art would recognize this disclosure to be an example of an “on the fly” preparation of the slurry taught by Nguyen, where the particles, water-based carrier fluid, and organo-silicon coating in oil-based, organic solvent would be mixed at the surface, before being pumped downhole.

587. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, *or a mixture thereof. . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids*

*include, but are not limited to, mineral oils, synthetic oils*, esters, and the like. *Any organic liquid in which a water solution of salts can be emulsified* also may be suitable for use as a base fluid in the methods of the present invention.” Ex. 1008, Weaver at [0058].

588. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . *Suitable emulsions* can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid *and a hydrocarbon.*” *Id.* at [0059]. One of ordinary skill in the art would recognize that the term “hydrocarbon” includes “hydrocarbon oil.” Weaver thus teaches the limitation of oil included in the composition.

589. In addition to inclusion of oils in the treatment fluids disclosed by Weaver, Weaver also discloses the addition of oils to the surface-treating reagent (i.e., organosilanes). *See* Ex. 1008, Weaver at [0054].

590. Moreover, the ’738 specification also discloses that the oil of the composition may be formation oil. Ex. 1001 at 4:11-15 (“Alternatively the slurry containing a hydrophobizing agent can be pumped into the fracture forming the proppant pack, which can be further consolidated by oil or condensate contained in the formation.”). Thus, the oil limitation in claim 1 can be satisfied by the presence

of formation oil. This is consistent with the prosecution history of the '738 patent. See Section VII.C, *supra*.

591. Nguyen further discloses that the invention is a “method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to *promote the recovery of hydrocarbons*.” Ex. 1005, Nguyen at Abstract.

592. Weaver further discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. Weaver also discloses a “method of treating a subterranean formation comprising: providing a treatment fluid that comprises: a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; . . . and introducing the treatment fluid into the subterranean formation.” *Id.* at claim 11.

593. One of ordinary skill in the art would understand that hydrocarbon-producing wells referenced by Nguyen and the subterranean formations referenced by Weaver that are treated by fracturing operations, gravel packing operations, frac packing operations and perforating contain oil. See also Ex. 1005, Nguyen at [0001] (recognizing that oil and gas wells produce hydrocarbons); Ex. 1001, '738 patent at

1:26-31 (stating that hydraulic fracturing operations are used to “enhance oil and gas production” and to “increase oil and gas production”). Further, one of ordinary skill in the art would understand that injecting the composition containing carrier fluid and water repellent-coated particulate into an oil and gas well or a hydrocarbon-producing well during hydraulic fracturing operations, as taught by Nguyen, and that “introducing the treatment fluid” containing particulate coated in surface-treating reagent and base fluid “into the subterranean formation” during one of the above-mentioned subterranean operations as taught by Weaver would necessarily mean that oil is present. Because the formation contains oil, the compositions of Nguyen and Weaver must therefore comprise oil.

594. Additionally, Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a liquid carrier comprising *one or more liquids selected from the group consisting of water and oil*, and a solid proppant comprising one or more materials selected from the group consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that “the liquid component of said foam comprises *at least* one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Moreover, Plummer discloses that “[o]il may be added to the foam-water slurry as a lubricant and to prevent clay

swelling . . . Other friction reducing materials may be added to the fracturing fluid.” *Id.* at 4:28-33. Thus, it would have been obvious to one of ordinary skill in the art to substitute the oil and water liquid carrier taught by Plummer for Nguyen’s carrier fluid.

595. As stated, it also would have been obvious to a person of ordinary skill in the art to include Plummer’s oil into the particulate slurry taught by Nguyen and Weaver. The motivation to do so is taught by Plummer: using oil as a lubricant friction-reducer or to control clay swelling can improve hydraulic fracturing operations in wells. *See* Ex. 1010, Plummer at 4:28-33.

596. Thus, the simple substitution of Weaver’s treatment fluid (either the aqueous/non-aqueous mixture or the oil and water emulsion) or Plummer’s carrier (water and oil liquid carrier) for Nguyen’s carrier fluid, and/or the addition of Plummer’s oil to act as a lubricant friction-reducer or to reduce clay swelling would have improved Nguyen’s composition in the same way as described in Weaver or Plummer (i.e., improved hydraulic fracturing operations). This improvement would have been reasonably expected to succeed by a person of ordinary skill in the art, who would have been motivated to make this substitution and/or addition. Limitation 1D is therefore met.

597. Therefore, it is my opinion that challenged independent claim 1 of the ’738 patent would have been obvious over Nguyen in view of Weaver or Plummer.

## 2. Independent Claim 32 Is Obvious

598. As discussed in Section X.A.2 and Section X.D.2 above (and incorporated by reference herein), in addition to the preamble of claim 32, claim 32 has five limitations: 32A (water), 32B (particulates), 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 32D (oil), and 32E (pumping the mixture into the formation). Nguyen and Weaver each discloses all of those limitations and the preamble. See ¶¶ 200-226; 462-493. The combination of Nguyen in view of Weaver or Plummer further discloses each and every limitation of the challenged claim 32 of the '738 patent.

### a. Preamble for Claim 32: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”

599. The preamble of claim 32 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 12:5-6.

600. As stated in ¶¶ 203-205 and 465-471 (and incorporated by reference herein), Nguyen and Weaver meet this preamble. Nguyen recites that “particles [] dry coated with a water-repellent organo-silicon material” “are then pumped downhole . . . to the wellbore-screen annulus to function as a gravel pack.” Ex. 1005, Nguyen at [0005]. One of ordinary skill in the art would recognize that gravel packs are used as a method of controlling all types of sand flow, including both formation

sand and proppant flowback. Therefore, one of ordinary skill in the art would recognize that Nguyen's invention was used as a method of controlling proppant flowback.

601. Moreover, Nguyen states that his invention is a method to “enhanc[e] the clean-up of a hydrocarbon-producing well” and to “promote the recovery of hydrocarbons” from an oil and gas well during hydraulic fracturing using a coated particulate slurry. *See* Ex. 1005, Nguyen at Abstract, claim 1. As disclosed in the '738 patent, using hydrophobic particulates as the proppant in hydraulic fracturing operations is one way to control proppant flowback. *See* Ex. 1001, '738 patent at 3:30-59. Thus, one of ordinary skill in the art would recognize that the disclosure of Nguyen's coated proppants would control proppant flowback in accordance with the '738 patent and therefore meets this preamble.

602. Weaver discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, *frac-packing operations*, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. One of ordinary skill in the art would recognize that frac-packing operations are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize that Weaver's invention is designed to control proppant flowback.

603. Weaver also teaches that “[o]ne problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates . . . to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation.” *Id.* at [0004]. Weaver proceeds that “one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, . . . preventing the embedment of particulates into the surfaces of a subterranean formation.” *Id.* at [0014].

604. One of ordinary skill in the art would understand that Weaver’s teaching of enhancing conductivity within a subterranean formation to maximize the value of a fracturing treatment and to prevent the embedment of particulates into surfaces of the subterranean formation to be describing operations to reduce particulate flowback.

605. Additionally, because the combination of Nguyen in view of Weaver and Plummer discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the ’738 patent. *See, e.g.*, Ex. 1001, ’738 patent at Abstract,



3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback).

606. Thus, both Nguyen and Weaver, and accordingly Nguyen in view of Weaver, meet this preamble.

**b. Limitation 32A: “mixing water”**

607. Claim 32 recites that the composition comprises “mixing water.” Ex. 1001 at 12:6-7. As stated in ¶¶ 206-209 and 472-475 (and incorporated by reference herein), Nguyen and Weaver meet this limitation.

608. Nguyen discloses that “[t]he most common fracturing fluid utilized heretofore which is generally preferred for use in accordance with this invention is comprised of water.” Ex. 1005, Nguyen at [0012]. Nguyen further discloses, in claim 63, “mixing particles in a carrier fluid comprising a water-based polymer to form a mixture.” *Id.* at claim 63. Nguyen further teaches “[t]he method of claim 63 wherein the carrier fluid comprises water and a gelling agent.” *Id.* at claim 65. Nguyen also states that “[t]he water utilized to form the fracturing fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other aqueous liquid which does not adversely react [to] other components of the fracturing fluids.” *Id.* at [0013]. Nguyen further teaches that “[f]racturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and *emulsions*.” Ex. 1005, Nguyen

at [0011]. A person of ordinary skill in the art would recognize that emulsions used in oil and gas well service operations are typically comprised of a mixture of oil and water. Thus, Nguyen discloses mixing the particles in a water-based carrier fluid to form a mixture.

609. Weaver discloses “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or **a mixture thereof**. Where the base fluid is aqueous, it may comprise **fresh water, salt water** (e.g., water containing one or more salts dissolved therein), **brine** (e.g., saturated salt water), **or seawater**.” Ex. 1008, Weaver at [0058].

610. Moreover, Weaver discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including **aqueous gels**, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable **aqueous gels are generally comprised of water** and one or more gelling agents. **Suitable emulsions** can be comprised of two immiscible liquids such as an **aqueous liquid** or gelled liquid and a hydrocarbon.” *Id.* at [0059].

611. Weaver describes that the particulates are mixed with the surface-modifying agents “prior to transporting the particulates to a job site, or in a treatment performed ‘on-the-fly.’ The term ‘on-the-fly’ is used herein to mean that one

flowing stream comprising particulates is continuously introduced into another flowing stream comprising the surface-treating reagent so that the streams are combined and mixed while continuing to flow as a single stream as part of the ongoing treatment at the job site. Such mixing can also be described as ‘real-time’ mixing.” *Id.* at [0056].

612. Therefore, both Nguyen and Weaver, and accordingly Nguyen in view of Weaver, clearly describes a composition including “mixing water” as recited in claim 32.

**c.     Limitation 32B: “particulates”**

613. Claim 32 recites that the composition comprises “particulates.” Ex. 1001 at 12:7. As stated in ¶¶ 210-212 and 476-478 (and incorporated by reference herein), Nguyen and Weaver meet this limitation.

614. Nguyen discloses that “[t]he proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like. . . . The preferred particles are sand.” Ex. 1005, Nguyen at [0016]. Nguyen’s particles are pumped into the well as part of the slurry composition to promote the recovery of hydrocarbons. *Id.* at Abstract. Nguyen also discloses a

“method . . . comprising mixing particles in a carrier fluid” to form a mixture. *Id.* at claim 63. Thus, Nguyen discloses the mixture of proppants with an aqueous carrier fluid to create a slurry composition.

615. Weaver provides “methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates . . .” Ex. 1008, Weaver at Abstract. Weaver also teaches that “particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface.” *Id.* at [0018]. Weaver describes that the particulates are mixed with the surface-modifying agents “prior to transporting the particulates to a job site, or in a treatment performed ‘on-the-fly.’” *Id.* at [0056].

616. Thus, both Nguyen and Weaver teach the use of particulates in the slurry. Limitation 32B would have therefore been obvious over Nguyen in view of Weaver.

d. **Limitation 32C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

617. Claim 32 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001 at 12:7-8. As stated in ¶¶ 213-217 and 479-483 (and incorporated by reference herein), Nguyen and Weaver meet this limitation.

618. The specification of the '738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, **organosilane**, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The '738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51.

619. The '738 patent also contains a list of suitable examples of organosilanes, including  $(CH_3)_2SiCl_2$  [dichlorodimethylsilane],  $(C_6H_5)_2SiCl_2$  [dichlorodiphenylsilane],  $(CH_3)_3SiCl$  [chlorotrimethylsilane], and  $(C_6H_5)_2Si(OCH_3)_2$  [dimethoxydiphenylsilane]. *See id.* at 5:66-6:12.

620. Nguyen teaches “particles [that] are dry coated with a water-repellent organo-silicon material.” Ex. 1005, Nguyen at [0005], claim 1. Nguyen further teaches that

[r]epresentative organo-silicon compounds that can be used in this embodiment include polyalkylsiloxane such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additional organo-silicon compounds that can be used in this embodiment include polyalkylarylsiloxanes . . .

chlorosilanes such as ethylchlorosilane, **chlorotrimethylsilane** and other silyl donors. Also, various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes can be used such as tetraethoxysilane, **dimethoxydiphenylsilane**, **dichlorodimethylsilane**, **dichlorodiphenylsilane**, poly(dimethylsiloxane, poly[oxy(dimethylsilylene)] and other such materials that will be well known to those skilled in the art. . . . Furthermore, organofunctional silanes can be used in this embodiment.

*Id.* at [0006].

621. Nguyen therefore also discloses this limitation of claim 1 because Nguyen discloses a number of “hydrophobizing agents” listed in the ’738 patent, including **chlorotrimethylsilane**, **dichlorodimethylsilane**, **dichlorodiphenylsilane**, and **dimethoxydiphenylsilane**. Compare Ex. 1005, Nguyen at [0006] with Ex. 1001, ’738 patent at 6:1-2, 6:12; see also NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data: Chlorotrimethylsilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?Name=chlorotrimethylsilane&Units=SI>; NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data: Dimethyldichlorosilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?ID=75-78-5>; NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data: Dichlorodiphenylsilane (2017), available at <http://webbook.nist.gov/cgi/cbook.cgi?Name=dichlorodiphenylsilane&Units=SI>;

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Standard Reference Data:  
Diphenyldimethoxysilane (2017), available at  
<http://webbook.nist.gov/cgi/cbook.cgi?Name=dimethoxydiphenylsilane&Units=SI>.

622. Weaver discloses “[i]n certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, **by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic** or hydrophilic in nature.” Ex. 1008, Weaver at [0015].

623. Weaver also teaches that “[i]n some embodiments, the surface-treating reagent may comprise other compounds that are capable of **hindering or preventing the water-wetting of a mineral surface**. Examples of suitable compounds include, but are not limited to, . . . **organosilanes** . . . Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.” *Id.* at [0049].

624. Therefore, both Nguyen and Weaver teach particulates coated with a chemical compound for rendering the particulates hydrophobic—and specifically that the chemical compound is an organosilane. This opinion is supported by the USPTO, because in the prosecution of the ’469 application, the Examiner rejected

claim 13 (which contained a limitation similar to limitation 1C) as anticipated by both Nguyen and Weaver because each taught particles coated in water-repellent compounds. See Ex. 1004 at 183-84, File History of the '469 Application, Non-Final Office Action, Jan. 2, 2015 (stating that “Nguyen teaches dry coated particles with a water-repellent organo-silicon materials” and that “Weaver further teaches the surface-treating reagent may comprise compounds that are capable of hindering or preventing the water-wetting of a mineral surface.”). I agree with the Examiner that both Nguyen and Weaver teach this limitation. Thus, the combination of Nguyen in view of Weaver teaches limitation 32C.

**e. Limitation 32D: “oil”**

625. Claim 32 further recites that the composition comprises “an oil.” Ex. 1001 at 12:8. As stated in ¶¶ 218-222 and 484-489 (and incorporated by reference herein), both Nguyen and Weaver teach this limitation.

626. Nguyen teaches that “[f]racturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and *emulsions*.” Ex. 1005, Nguyen at [0011]. A person of ordinary skill in the art would recognize that emulsions used in oil and gas well service operations are typically comprised of a mixture of oil and water. See, e.g., Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S.



Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production).

627. Additionally, Nguyen teaches that if “the particles are coated when the particles are flowing, the solvent should be readily miscible in the water-based gel carrier fluid. Organic solvents that can be used as a carrier for the organo-silicon coating material including *kerosene* [and] lighter grades of diesel fuel.” Ex. 1005, Nguyen at [0008]. One of ordinary skill in the art would recognize this disclosure to be an example of an “on the fly” preparation of the slurry taught by Nguyen, where the particles, water-based carrier fluid, and organo-silicon coating in oil-based, organic solvent would be mixed at the surface, before being pumped downhole.

628. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, *or a mixture thereof. . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils*, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.” Ex. 1008, Weaver at [0058].

629. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . **Suitable emulsions** can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid **and a hydrocarbon.**” *Id.* at [0059]. One of ordinary skill in the art would recognize that the term “hydrocarbon” includes “hydrocarbon oil.” Weaver thus teaches the limitation of oil included in the composition.

630. In addition to inclusion of oils in the treatment fluids disclosed by Weaver, Weaver also discloses the addition of oils to the surface-treating reagent (i.e., organosilanes). *See* Ex. 1008, Weaver at [0054].

631. Additionally, Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a **liquid carrier comprising one or more liquids selected from the group consisting of water and oil**, and a solid proppant comprising one or more materials selected from the group consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that “the liquid component of said foam comprises **at least** one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Thus, it would have been obvious to one of ordinary skill in the art to substitute the oil and water liquid carrier taught

by Plummer for Nguyen's carrier fluid. Moreover, Plummer discloses that "[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling . . . Other friction reducing materials may be added to the fracturing fluid." *Id.* at 4:28-33.

632. As stated, it would have also been obvious to a person of ordinary skill in the art to include Plummer's oil into the particulate slurry taught by Nguyen and Weaver. The motivation to do so is taught by Plummer: using oil as a lubricant friction-reducer or to reduce clay swelling can improve hydraulic fracturing operations in wells. *See* Ex. 1010, Plummer at 4:28-33.

633. Thus, the simple substitution of Weaver's treatment fluid (either the aqueous/non-aqueous mixture or the oil and water emulsion) or Plummer's carrier (oil and water liquid carrier) for Nguyen's carrier fluid, and/or the addition of Plummer's oil to act as a lubricant friction-reducer or to reduce clay swelling would have improved Nguyen's composition in the same way as described in Weaver or Plummer (i.e., improved hydraulic fracturing operations). This improvement would have been reasonably expected to succeed by a person of ordinary skill in the art, who would have been motivated to make this substitution and/or addition. Thus, motivation existed to make either combination. Limitation 32D is therefore met.

f. **Limitation 32E: "pumping the mixture into the formation"**

634. Claim 32 further recites “pumping the mixture into the formation.” Ex. 1001 at 12:8-9. As stated in ¶¶ 223-226 and 490-493 (and incorporated by reference herein), both Nguyen and Weaver teach pumping the mixture into the formation.

635. Nguyen teaches “[a] method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid . . . to form a mixture, ***pumping the mixture downhole*** in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid.” Ex. 1005, Nguyen at claim 63. Nguyen also teaches “[a] method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid . . . to form a mixture, ***pumping the mixture downhole*** in a well to a fracture in a formation adjacent to the well to function as a proppant, and coating the particles with a water repellent composition, wherein the particles are coated before being mixed in a carrier fluid.” *Id.* at claim 75.

636. Weaver teaches “[a] method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified mineral surface; and ***introducing the treatment fluid into the subterranean formation.***” Ex. 1008, Weaver at claim 23; *see also id.* at claim 11.

637. Thus, both Nguyen and Weaver teach pumping the mixture into the formation. Limitation 32E would have therefore been obvious over Nguyen in view of Weaver.

638. Therefore, in my opinion, challenged independent claim 32 of the '738 patent would have been obvious over Nguyen in view of Weaver or Plummer.

### 3. Independent Claim 51 is Obvious

639. As discussed in Section X.A.3 and Section X.D.3 above (and incorporated by reference herein), in addition to the preamble of claim 51, claim 51 has four limitations: 51A (contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with water and an oil to form a mixture, and 51D (pumping the mixture into the formation). Nguyen and Weaver each discloses all of those limitations and the preamble. See ¶¶ 227-247; 494-517. The combination of Nguyen in view of Weaver or Plummer further discloses each and every limitation of the challenged claim 51 of the '738 patent.

#### a. Preamble for Claim 51: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”

640. The preamble of claim 51 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the

steps of.” Ex. 1001 at 13:28-29. As stated in ¶¶ 230-232 and 497-501 (and incorporated by reference herein), Nguyen and Weaver meet this preamble. Nguyen recites that “particles [] dry coated with a water-repellent organo-silicon material” “are then pumped downhole . . . to the wellbore-screen annulus to function as a gravel pack.” Ex. 1005, Nguyen at [0005]. One of ordinary skill in the art would recognize that gravel packs are used as a method of controlling all types of sand flow, including both formation sand and proppant flowback. Therefore, one of ordinary skill in the art would recognize that Nguyen’s invention was used as a method of controlling proppant flowback.

641. Moreover, Nguyen states that his invention is a method to “enhanc[e] the clean-up of a hydrocarbon-producing well” and to “promote the recovery of hydrocarbons” from an oil and gas well during hydraulic fracturing using a coated particulate slurry. *See* Ex. 1005, Nguyen at Abstract, claim 1. As disclosed in the ’738 patent, using hydrophobic particulates as the proppant in hydraulic fracturing operations is one way to control proppant flowback. *See* Ex. 1001, ’738 patent at 3:30-59. Thus, one of ordinary skill in the art would recognize that the disclosure of Nguyen’s coated proppants would control proppant flowback in accordance with the ’738 patent and therefore meets this preamble.

642. Weaver discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable

subterranean operations include . . . fracturing operations, gravel-packing operations, ***frac-packing operations***, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. One of ordinary skill in the art would recognize that frac-packing operations are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize that Weaver’s invention is designed to control proppant flowback.

643. Weaver also teaches that “[o]ne problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates . . . to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation.” *Id.* at [0004]. Weaver proceeds that “one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, . . . preventing the embedment of particulates into the surfaces of a subterranean formation.” *Id.* at [0014].

644. One of ordinary skill in the art would understand that Weaver’s teaching of enhancing conductivity within a subterranean formation to maximize the value of a fracturing treatment and to prevent the embedment of particulates into

surfaces of the subterranean formation to be describing operations to reduce particulate flowback.

645. Additionally, because the combination of Nguyen in view of Weaver and Plummer discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the '738 patent. *See, e.g.*, Ex. 1001, '738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback).

646. Thus, both Nguyen and Weaver, and accordingly Nguyen in view of Weaver, meet this preamble.

**b. Limitation 51A: “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic”**

647. Claim 51 recites “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic.” Ex. 1001 at 13:29-31.

648. Nguyen discloses “[a]n example of the technique for applying the coating on the particles involves *dissolving an oil-soluble organo-silicon compound in a solvent, admixing or spraying the resultant solution on the particles*, and then evaporating the solvent to form a thin film of siloxane or silane encapsulating the particles. The organo-silicon organo-silane compound is readily



absorbed onto the particles from the solvent and the solvent is easily evaporated by drying.” Ex. 1005, Nguyen at [0007]; *see also id.* claim 4.

649. Moreover, Weaver teaches that one “suitable method[] for allowing the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate includes . . . solvent extraction methods.” *Id.* at [0057]. In such an embodiment, “[t]he solution comprising the surface treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced to a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface.” *Id.* As discussed, Nguyen discloses examples of organo-silicon compounds that are the same as those disclosed in the ’738 patent, including chlorotrimethylsilane, dichlorodimethylsilane, dichlorodiphenylsilane, and dimethoxydiphenylsilane. Moreover, Weaver discloses the use of organosilanes, which the ’738 patent identifies as a chemical compound for rendering the surface of the particulates hydrophobic.

650. Thus Nguyen and Weaver clearly describe contacting the particulates with an organo-silicon compound, which, as stated above, is an example of “a

chemical compound to render the surface of the particulates hydrophobic” as required by claim 51.

c. **Limitation 51B: “separating the particulate from the medium”**

651. Claim 51 recites that the now-coated particulates are “separate[ed] . . . from the medium.” Ex. 1001 at 13:32.

652. Nguyen discloses “[a]n example of the technique for applying the coating on the particles involves dissolving an oil-soluble organo-silicon compound in a solvent, admixing or spraying the resultant solution on the particles, *and then evaporating the solvent to form a thin film of siloxane or silane encapsulating the particles. The organo-silicon organo-silane compound is readily absorbed onto the particles from the solvent and the solvent is easily evaporated by drying.*” Ex. 1005, Nguyen at [0007]; *see also id.* claim 4. As stated, one of ordinary skill in the art would understand evaporation of the solvent to be a method of separating the solvent from the silane- or siloxane-coated particle. Thus, Nguyen discloses that the now-coated particulates are separated from the solvent solution.

653. Weaver teaches that one “suitable method[] for allowing the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate includes . . . solvent extraction methods.” *Id.* at [0057]. In such an embodiment, “[t]he solution comprising the surface treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the

particulates and stirring (or mulling) until the mixture is uniform. *The treated particulates then may be introduced to a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface.*” *Id.* As discussed, one of ordinary skill in the art would understand that “extract[ing] the solvent from the treated particulates” is a method of separating the solvent containing the organo-silicon coating from the coated particulate. Accordingly, Weaver discloses that the now-coated particulates are separated from the solvent solution.

654. In view of the foregoing excerpts from the disclosure of Nguyen and Weaver, one of ordinary skill in the art would have understood Nguyen and Weaver to disclose this limitation.

**d. Limitation 51C: “blending the hydrophobic particulate with water and an oil to form a mixture”**

655. Claim 51 recites that the hydrophobic coated particulate are blended “with water and oil to form a mixture.” Ex. 1001 at 13:32-34.

656. Nguyen teaches that “[f]racturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and *emulsions*.” Ex. 1005, Nguyen at [0011]. One of ordinary skill in the art would recognize that an emulsion may be comprised of oil and water. See *e.g.*, Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic

fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S. Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production).

657. Nguyen teaches that the particulates coated with the water repellent composition are mixed with the carrier fluid to “form a mixture” (*see e.g., id.* at claim 63, Abstract), which, as disclosed above, may be an emulsion (*id.* at [0011]).

658. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, ***or a mixture thereof***. Where the base fluid is aqueous, it may comprise fresh water [or] salt water . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts ***can be emulsified also may be suitable for use as a base fluid*** in the methods of the present invention.” Ex. 1008, Weaver at [0058].

659. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . ***Suitable emulsions*** can be comprised of two

immiscible liquids such as *an aqueous liquid* or gelled liquid *and a hydrocarbon.*”  
*Id.* at [0059].

660. One of ordinary skill in the art would recognize, and the ’738 patent confirms, that oil is a type of “hydrocarbon.” *See* Ex. 1001, ’738 patent at 11:31-33 (“20. The composition of claim 1, wherein the oil is selected from the group consisting of a *hydrocarbon oil*, a silicon oil, kerosene oil, a frac oil and combinations thereof.”). Weaver thus teaches the limitation of oil included in the composition in the form of a treatment fluid including a mixture of oil and water, as well as an oil and water emulsion.

661. Moreover, Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a *liquid carrier comprising one or more liquids selected from the group consisting of water and oil*, and a solid proppant comprising one or more materials selected from the group consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that “the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Thus, it would have been obvious to one of ordinary skill in the art to substitute the oil and water liquid carrier taught by Plummer for Nguyen’s carrier fluid. Moreover, Plummer discloses that “[o]il

may be added to the foam-water slurry as a lubricant and to prevent clay swelling . . . Other friction reducing materials may be added to the fracturing fluid.” *Id.* at 4:28-33.

662. As stated, it would have also been obvious to a person of ordinary skill in the art to include Plummer’s oil into the particulate slurry taught by Nguyen and Weaver. The motivation to do so is taught by Plummer: using oil as a lubricant friction-reducer or to reduce clay swelling can improve hydraulic fracturing operations in wells. *See* Ex. 1010, Plummer at 4:28-33.

663. Thus, the simple substitution of Weaver’s treatment fluid (either the aqueous/non-aqueous mixture or the oil and water emulsion) or Plummer’s carrier (oil and water liquid carrier) for Nguyen’s carrier fluid, and/or the addition of Plummer’s oil to act as a lubricant friction-reducer or to reduce clay swelling would have improved Nguyen’s composition in the same way as described in Weaver or Plummer (i.e., improved hydraulic fracturing operations). This improvement would have been reasonably expected to succeed by a person of ordinary skill in the art, who would have been motivated to make this substitution and/or addition. Limitation 51C is therefore met.

664. Thus, Nguyen in view of Weaver or Plummer teaches this limitation.

e. **Limitation 51D: “pumping the mixture into the formation”**

665. Claim 51 further recites “pumping the mixture into the formation.” Ex. 1001 at 13:34.

666. As recited, Nguyen teaches “[a] method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid . . . to form a mixture, ***pumping the mixture downhole*** in a well to a fracture in a formation adjacent to the well to function as a proppant, and coating the particles with a water repellent composition, wherein the particles are coated before being mixed in a carrier fluid.” *Id.* at claim 75.

667. Nguyen also teaches “[a] method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid . . . to form a mixture, ***pumping the mixture downhole*** in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid.” Ex. 1005, Nguyen at claim 63.

668. Weaver teaches “[a] method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified mineral surface; and ***introducing the treatment fluid into the subterranean formation.***” Ex. 1008, Weaver at claim 23; *see also id.* at claim 11.

669. Therefore, Nguyen and Weaver disclose limitation 51D.

670. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), and the preamble of claim 51, is taught by Nguyen and Weaver. Therefore, independent claim 51 is obvious over Nguyen in view of Weaver or Plummer.

#### 4. Dependent Claims 21, 22, 40, 41, and 57 Are Obvious

671. As addressed above (see Section XI.A.1, XI.A.2, and XI.A.3) and incorporated herein by reference, all of the limitations of claims 1, 32, and 51, including 1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation) and the preambles of claims 1, 32, and 51, are obvious over Nguyen in view of Weaver or Plummer.

672. The only additional limitation in claims 21, 22, 40, 41 relate to the type of particulate. Dependent claims 21, and 40 recite a near-identical limitation, but depend from different claims. Claim 21 depends from independent claim 1, whereas claim 40 depends from independent claim 32. Similarly, dependent claims 22 and 41 recite the same limitation, but depend from different claims. Claim 22 depends



from independent claim 1, whereas claim 41 depends from claim 32, and claim 57 depends from independent claim 51. Dependent claims 21 and 40 require the particulate be one of an enumerated list of particulates, including sand. *See* Ex. 1001, '738 patent at 11:34-37; 12:63-65 (“wherein the particulates are selected from the group consisting of sand . . .”). Dependent claims 22, 41, and 57 require the particulate to be sands. *Id.* at 11:38-39; 12:66-67, 14:24-25 (“wherein the particulates are sands”). Therefore, a prior art reference that discloses the additional limitation of “sand” as the particulate anticipates claims 21, 22, 40, 41, and 57.

673. Nguyen teaches the additional limitation of claims 21, 22, 40, 41, and 57 because Nguyen teaches “[v]arious kinds of particulates can be utilized as a proppant including sand. . . . The preferred particles are sand.” Ex. 1005, Nguyen at [0016]. Nguyen also teaches “[a] method comprising dry coating particles with a water-repelling composition” (*id.* at claim 1) “wherein the particles are selected from the group consisting of sand and gravel” (*id.* at claim 8).

674. Moreover, Nguyen teaches that the particulates coated with the water repellent composition are mixed with the carrier fluid to form a mixture (*see e.g., id.* at claim 63, Abstract), which, are then pumped “downhole into a well to promote the recovery of hydrocarbons from the well” to function as a gravel pack or a proppant. *Id.* at claims 1-3. Thus, the sand particulates are mixed with the carrier fluid.

675. Weaver teaches “[t]he particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand.” Ex. 1008, Weaver at [0018].

676. Weaver also teaches that “the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.” *Id.* at [0057]. Thus, the sand particulates are mixed with the treatment fluid before being pumped down the formation.

677. Thus, Nguyen and Weaver teach the additional limitation of claims 21, 22, 40, 41, and 57. Therefore, it is my opinion that challenged dependent claims 21, 22, 40, 41, and 57 of the ’738 patent would have been obvious over Nguyen in view of Weaver or Plummer.

678. Based on the foregoing analysis of Sections XI.A.1-4, all of the elements of claims 1, 21, 22, 32, 40, 41, 51, and 57 [1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering

the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), 21A, 22A, 40A, 41A, and 57A (sand), and the preambles of claims 1, 32, and 57] are disclosed by the combination of Nguyen in view of Weaver or Plummer. Therefore, it is my opinion that the combination of Nguyen in view of Weaver or Plummer renders challenged claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent obvious.

**B. Claims 1, 21, 22, 32, 40, 41, 51, and 57 Are Obvious over Dawson in view of Weaver or Plummer**

679. As discussed in Sections X.B and X.D above (and incorporated by reference herein), both Dawson and Weaver anticipate claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent because each reference discloses each and every element of the claims. Furthermore, and in the alternative, the disclosure of Dawson in view of Weaver or Plummer also renders claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent obvious. *See generally*, Ex. 1002I, '738 Patent v. Dawson in view of Weaver or Plummer Claim Chart.

680. One of ordinary skill in the art at the time of the invention would have been motivated to combine Dawson and Weaver because both are directed to

methods for modifying particulate with a chemical compound (specifically silanes) for use in hydraulic fracturing operations. Further, one of ordinary skill in the art would be motivated to substitute the carrier fluid of Dawson with the oil and water emulsion treatment fluid of Weaver to arrive at the invention of claim 1 of the '738 patent, since both were well known in the art and there was a high likelihood of success. Additionally, and in the alternative, a person of ordinary skill in the art would be motivated to substitute the aqueous carrier fluid of Dawson with the oil and water liquid carrier disclosed in Plummer for the same reason. Moreover, a person of ordinary skill in the art would be motivated to add oil to the slurry as a lubricant friction-reducer or to reduce clay swelling, as taught by Plummer (Ex. 1010 at 4:28-33) to improve the compositions and methods of Dawson in hydraulic fracturing operations.

#### **1. Independent Claim 1 Is Obvious**

681. As discussed in Section X.B.1 and Section X.D.1 above (and incorporated by reference herein), in addition to the preamble of claim 1, claim 1 has four limitations: 1A (water), 1B (particulates), 1C (a chemical compound for rendering the surface of the particulate hydrophobic), and 1D (oil). Dawson and Weaver each discloses all of those limitations and the preamble. See ¶¶ 261-287; 438-461. The combination of Dawson in view of Weaver or Plummer further discloses each and every limitation of challenged claim 1 of the '738 patent.

a. **Preamble for Claim 1: “An aqueous slurry composition comprising”**

682. The preamble of claim 1 of the '738 patent states, “An aqueous slurry composition comprising.” Ex. 1001, '738 patent at 9:46. As stated in ¶¶ 264 and 441 (and incorporated by reference herein), Dawson and Weaver meet this preamble.

683. Dawson recites “[a] slurry including the disclosed particulate material . . . and a carrier fluid.” Ex. 1006, Dawson at 14, l. 12-15. The carrier fluid may include salt water or fresh water and/or a hydrocarbon. *Id.* at 13, l. 10-13. Thus, Dawson discloses an aqueous slurry.

684. Weaver teaches that “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous.” Ex. 1008, Weaver at [0058].

685. Thus, both Dawson and Weaver, and accordingly Dawson in view of Weaver, meet this preamble.

b. **Limitation 1A: “water”**

686. Claim 1 recites that the composition comprises “water.” Ex. 1001 at 9:47. As stated in ¶¶ 265-267 and 442-444 (and incorporated by reference herein), both Dawson and Weaver teach the use of water.

687. Dawson discloses that the particles exposed to a modifying agent “may be mixed and pumped during any desired portion/s of a well treatment (e.g., hydraulic fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier fluid.” Ex. 1006, Dawson at 13, l. 6-10. Dawson also discloses “any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water, liquid hydrocarbons, and/or nitrogen or other gases.” *Id.* at 13, l. 10-13. Dawson discloses that the particulate is suspended in the carrier fluid (*Id.* at 10, l. 1-3), therefore indicating that the carrier fluid such as water is mixed with the particulate to form a slurry.

688. Weaver discloses “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise *fresh water, salt water* (e.g., water containing one or more salts dissolved therein), *brine* (e.g., saturated salt water), *or seawater*.” Ex. 1008, Weaver at [0058].

689. Moreover, Weaver discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including *aqueous gels*, viscoelastic surfactant gels,

oil gels, foamed gels, and emulsions. Suitable *aqueous gels are generally comprised of water* and one or more gelling agents. *Suitable emulsions* can be comprised of two immiscible liquids such as an *aqueous liquid* or gelled liquid and a hydrocarbon.” *Id.* at [0059].

690. Thus, both Dawson and Weaver teach that water can be included in the slurry as the carrier/treatment fluid of the proppant. Limitation 1A would have therefore been obvious over Dawson in view of Weaver.

**c.     Limitation 1B: “particulates”**

691. Claim 1 recites that the composition comprises “particulates.” Ex. 1001 at 9:48. As stated in ¶¶ 268-271 and 445-447 (and incorporated by reference herein), both Dawson and Weaver teach the use of particulates.

692. Dawson teaches an invention that “relates to use of relatively lightweight and/or substantially neutrally buoyant *particles as proppant material* in hydraulic fracturing treatments.” Ex. 1006, Dawson at 2, l. 6-7. Dawson additionally teaches that the “particulate material [is] suspended in a carrier fluid.” *Id.* at 10, l. 1-3; 48, claim 13.

693. Weaver provides “methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates . . .” Ex. 1008, Weaver at Abstract. Weaver also teaches that “particulate suitable for use in

the present invention comprise any particulate that comprises a mineral surface.” *Id.* at [0018].

694. Thus, both Dawson and Weaver teach the use of particulates in the slurry. Limitation 1B would have therefore been obvious over Dawson in view of Weaver.

d. **Limitation 1C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

695. Claim 1 also recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001, ’738 patent at 9:49-50. As stated in ¶¶ 272-278 and 448-452 (and incorporated by reference herein), both Dawson and Weaver teach this limitation.

696. The specification of the ’738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The ’738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51.



697. The '738 patent also contains a list of suitable examples of organosilanes, including **CH<sub>3</sub>SiCl<sub>3</sub>** [methyltrichlorosilane]. *See id.* at 5:66-6:1.

698. Dawson teaches that particles “may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material.” Ex. 1006, Dawson at 7, l. 3-8. Specifically, Dawson discloses that “examples of suitable modifying agents include, but are not limited to, agents including polyisocyanates, **silanes**, **siloxanes**, and combinations thereof. Selected modifying agent/s may be advantageously used to increase the elastic modulus of a given naturally occurring material, for example, to make particles of a given naturally-occurring material more suitable . . . for use as a relatively lightweight and/or substantially neutrally buoyant fracture proppant or sand control particulate material having increased effectiveness when exposed to higher closure stresses or other mechanical stresses that may be encountered downhole during a well treatment such as hydraulic fracturing or sand control treatment.” *Id.* at 7, l. 14-23.

699. Like the '738 patent, Dawson also discloses the use of organosilanes and organosiloxanes to coat the particulate. Specifically, Dawson teaches that the modifying agent may contain “one or more halogen groups, (*e.g.*, tetrachlorosilane, **methyltrichlorosilane**, *etc.*).” *Id.* at 21, l. 10-11. Thus, Dawson teaches the same methyltrichlorosilane (CH<sub>3</sub>SiCl<sub>3</sub>) that is disclosed in the '738 patent. *See* Ex. 1001, '738 patent at 6:1.

700. Dawson teaches:

Examples of commercially available silane-based products that may be employed as modifying agents in the practice of the disclose methods and compositions include, but are not limited to, silane based products” (Ex. 1006, Dawson at p. 21, l. 19-21) and teaches the specific examples of “commercially available silane-based products available from CROMPTON include CoatOSil<sup>®</sup> 1706 Silane (vinyl silane monomer), CoatOSil<sup>®</sup> 1757 Silane, CoatOSil<sup>®</sup> 1770 Silane, EURENOR<sup>®</sup> 5020 (liquid isobutyldimethoxysilane), EURENOR<sup>®</sup> 5021 (liquid diisopropylmethoxysilane), EURENOR<sup>®</sup> 5022 (liquid diisobutyldimethoxysilane), EURENOR<sup>®</sup> 5023 (liquid dicyclopentylmethoxysilane), Silcat<sup>®</sup> R Silane (vinylsilane with grafting and cross linking catalysts), Sikat<sup>®</sup> RHE Silane (cross linking system of silane, peroxide and catalyst), Silcat<sup>®</sup> RHS Silane (crosslinking system of silane, peroxide catalyst and antioxidants), Sikat<sup>®</sup> VS-735/1 Silane (crosslinking system of silane, peroxide, antioxidants and metal deactivator), Silcat<sup>®</sup> VS-758/0 Silane (stabilized cross linking system of silane, peroxide and catalyst), Sikat<sup>®</sup> VS-870 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat<sup>®</sup> VS-928 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat<sup>®</sup> VS-963 Silane (stabilized cross linking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Silox<sup>®</sup> VS- 911 Silane (liquid silane system), Silquest<sup>®</sup> A-1100 Silane (gammaaminopropyltriethoxysilane), Silquest<sup>®</sup> A-1101 Silane (technical grade amino silane), Silquest<sup>®</sup> A-1102 Silane (technical

grade amino silane), Silquest<sup>®</sup> A-1110 Silane (primary amino silane), Silquest<sup>®</sup> A-1120 Silane, Silquest<sup>®</sup> A-1126 Silane (amino silane), Silquest<sup>®</sup> A-1128 Silane (amino silane), Silquest<sup>®</sup> A-1160 Silane (ureido silane), Silquest<sup>®</sup> A-1170 Silane (secondary amino functional silane), Silquest<sup>®</sup> A-1289 Silane, Silquest<sup>®</sup> A-1310 Silane, Silquest<sup>®</sup> A-137 Silane (monomeric alkylalkoxysilane), Silquest<sup>®</sup> A-151 Silane, Silquest<sup>®</sup> A-1524 Silane (100-percent active ureidosilane), Silquest<sup>®</sup> A-1589 Silane (disulfide silane), Silquest<sup>®</sup> A-171 Silane, Silquest<sup>®</sup> A-172 Silane (vinylfunctional coupling agent), Silquest<sup>®</sup> A-174 Silane, Silquest<sup>®</sup> A-186 Silane (epoxy functional silanes), Silquest<sup>®</sup> A-187 Silane (epoxy functional silanes), Silquest<sup>®</sup> A-189 Silane, Silquest<sup>®</sup> A-2171 Silane (silane compound having vinyl and silane functionality), Silquest<sup>®</sup> Alink 15 Silane (secondary amino silane endcapper), Silquest<sup>®</sup> RC-1 Silane (organosilicone coupling agent), Silquest<sup>®</sup> RC-2 Silane, Silquest<sup>®</sup> Wetlink 78 Silane, Silquest<sup>®</sup> Y-11637 Silane (primary aminosilane), Silquest<sup>®</sup> Y-11683 Silane (di-, tri-functional ethoxy silane), Silquest<sup>®</sup> Y-5997 Silane, XL-PEarl<sup>®</sup> 31 Blend, XL-PEarl<sup>®</sup> 50 Blend, XLPEarl<sup>®</sup> 51 Blend, XL-PEarl<sup>®</sup> 52 Blend, XL-PEarl<sup>®</sup> 60 Blend, XL-PEarl<sup>®</sup> 70 Blend, *etc.*

Ex. 1006, Dawson at 21, l. 23- 22, l. 23. Many of these compounds are organosilanes.

701. Because Dawson teaches coating particles with various silanes and siloxanes, including organosilanes and organosiloxanes, and the '738 patent identifies organosilanes and organosiloxanes as examples of hydrophobizing agents (Ex. 1001, '738 patent at 4:62-67), one of ordinary skill in the art would understand

that Dawson's particulates are coated with hydrophobic rendering compounds. Moreover, Dawson discloses at least one of the same organo-silicon compounds (i.e., methyltrichlorosilane) used to coat the particulates suspended in carrier fluid identified as a "hydrophobic rendering compound" by the '738 patent. *Compare* Ex. 1006, Dawson at 21, l. 11 *with* Ex. 1001, '738 patent at 6:1.

702. Weaver discloses "[i]n certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, ***by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic*** or hydrophilic in nature." Ex. 1008, Weaver at [0015].

703. Weaver also teaches that "[i]n some embodiments, the surface-treating reagent may comprise other compounds that are capable of ***hindering or preventing the water-wetting of a mineral surface***. Examples of suitable compounds include, but are not limited to, . . . ***organosilanes*** . . . Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes." *Id.* at [0049].

704. Therefore, both Dawson and Weaver teach particulates coated with a chemical compound for rendering the particulates hydrophobic—and specifically

that the chemical compound is a silane. This opinion is supported by the USPTO, because in the prosecution of the '469 application, the Examiner rejected claim 13 (which contained a limitation similar to limitation 1C) as anticipated by Weaver because each taught particles coated in water-repellent compounds. See Ex. 1004 at 184, File History of the '469 Application, Non-Final Office Action, Jan. 2, 2015 (stating that “Weaver further teaches the surface-treating reagent may comprise compounds that are capable of hindering or preventing the water-wetting of a mineral surface.”). I agree with the Examiner that Weaver teaches this limitation. Moreover, it is my opinion that Dawson additionally teaches this limitation by disclosing methyltrichlorosilane, which is disclosed as a hydrophobizing agent in the '738 patent. See Ex. 1001, '738 patent at 6:1. Thus, the combination of Dawson in view of Weaver teaches limitation 1C.

**e. Limitation 1D: “oil”**

705. Claim 1 further recites that the composition comprises “an oil.” Ex. 1001 at 9:50. As stated in ¶¶ 279-287 and 453-461 (and incorporated by reference herein), both Dawson and Weaver teach this limitation.

706. As mentioned, Dawson discloses that “[t]he particles disclosed herein . . . may be mixed and pumped during any desired portion/s of well treatment (*e.g.*, hydraulic fracturing treatment, sand control treatment, *etc.*) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable

for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water, **liquid hydrocarbons, and/or** nitrogen or other gases.” Ex. 1006, Dawson at 13, l. 6-13.

707. One of ordinary skill in the art would recognize that liquid hydrocarbons can be hydrocarbon oils. Dawson thus teaches the limitation of oil included in the composition and discloses that the carrier fluid may comprise both water **and** a liquid hydrocarbon.

708. Further, Dawson incorporates by reference U.S. Patent 3,659,651 to Graham (“Graham”). See Ex. 1006, Dawson at 33, l. 6-8. Graham teaches “[i]n placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or **water-in-oil emulsion** prepared in accordance with the teachings of U.S. Pat. No. 3,378,074.” Ex. 1012, Graham at 5:9-16 (citing U.S. Patent 3,378,074 to Kiel at 5:65-72 (Ex. 1013) (disclosing that “high viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either **water-in-oil or oil-in-water emulsions**. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with

*kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine* or an acid solution containing a suitable surface active agent or emulsifier.”)).

709. Therefore, Dawson discloses the use of water-in-oil emulsions containing water or brine in kerosene, diesel fuel, crude oil, or a hydrocarbon oil in satisfaction of this limitation.

710. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, *or a mixture thereof. . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils*, esters, and the like. Any *organic liquid in which a water solution of salts can be emulsified also may be suitable* for use as a base fluid in the methods of the present invention.” Ex. 1008, Weaver at [0058].

711. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . *Suitable emulsions* can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid *and a hydrocarbon.*” *Id.* at [0059]. One of ordinary skill in the art would recognize that the term “hydrocarbon” includes “hydrocarbon oil.” Weaver thus teaches the limitation of oil included in the composition.

712. In addition to inclusion of oils in the treatment fluids disclosed by Weaver, Weaver also discloses the addition of oils to the surface-treating reagent (i.e., organosilanes). *See* Ex. 1008, Weaver at [0054].

713. Moreover, the '738 specification also discloses that the oil of the composition may be formation oil. Ex. 1001 at 4:11-15 (“Alternatively the slurry containing a hydrophobizing agent can be pumped into the fracture forming the proppant pack, which can be further consolidated by oil or condensate contained in the formation.”). Thus, the oil limitation in claim 1 can be satisfied by the presence of formation oil. This is consistent with the prosecution history of the '738 patent. *See* Section VII.C, *supra*.

714. Dawson further discloses that “it will be understood with benefit of this disclosure that the disclosed particulate materials may be used with any method in which a pack of particulate material is formed within a wellbore that is permeable to fluids produced from a wellbore (*e.g.*, oil, gas, water, *etc.*).” *Id.* at 14 l. 31- 15 l. 4.

715. Weaver further discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. Weaver also discloses a “method of treating a subterranean formation comprising: providing a treatment fluid that comprises: a base fluid, a plurality of



particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; . . . and introducing the treatment fluid into the subterranean formation.”

*Id.* at claim 11.

716. One of ordinary skill in the art would understand that wellbores referenced by Dawson and the subterranean formations referenced by Weaver that are treated by fracturing operations, gravel packing operations, frac packing operations and perforating contain oil and gas. *See, e.g.*, Ex. 1001, '738 patent at 1:26-31 (stating that hydraulic fracturing operations are used to “enhance oil and gas production” and to “increase oil and gas production”). Further, one of ordinary skill in the art would understand that using the disclosure of Dawson within a wellbore that is producing fluids including oil and gas, would understand that oil is present. Because the disclosure of Dawson specifically teaches that a wellbore produces oil and that the invention may be used in a wellbore permeable to oil, the composition of Dawson must therefore comprise oil. Similarly, one of ordinary skill in the art would understand that “introducing the treatment fluid” containing particulate coated in surface-treating reagent and base fluid “into the subterranean formation” during a subterranean operation as taught by Weaver would necessarily mean that oil is present. Because the formation contains oil, the composition of Weaver must also therefore comprise oil.

717. Additionally, Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a **liquid carrier comprising one or more liquids selected from the group consisting of water and oil**, and a solid proppant comprising one or more materials selected from the group consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that “the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Thus, it would have been obvious to one of ordinary skill in the art to substitute the oil and water liquid carrier taught by Plummer for Dawson’s carrier fluid. Moreover, Plummer discloses that “[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling . . . Other friction reducing materials may be added to the fracturing fluid.” *Id.* at 4:28-33.

718. As stated, it would have been obvious to a person of ordinary skill in the art to include Plummer’s oil into the particulate slurry taught by Dawson and Weaver. The motivation to do so is taught by Plummer: using oil as a lubricant friction-reducer or to control clay swelling can improve hydraulic fracturing operations in wells. *See* Ex. 1010, Plummer at 4:28-33.

719. Thus, the simple substitution of Weaver's treatment fluid (either the aqueous/non-aqueous mixture or the oil and water emulsion) or Plummer's carrier (oil and water liquid carrier) for Dawson's carrier fluid, and/or the addition of Plummer's oil to act as a lubricant friction-reducer or to reduce clay swelling would have improved Dawson's composition in the same way as described in Weaver or Plummer (i.e., improved hydraulic fracturing operations). This improvement would have been reasonably expected to succeed by a person of ordinary skill in the art, who would have been motivated to make this substitution and/or addition. Limitation 1D is therefore met.

720. Therefore, it is my opinion that challenged independent claim 1 of the '738 patent would have been obvious over Dawson in view of Weaver or Plummer.

## **2. Independent Claim 32 Is Obvious**

721. As discussed in Section X.B.2 and Section X.D.2 above (and incorporated by reference herein), in addition to the preamble of claim 32, claim 32 has five limitations: 32A (water), 32B (particulates), 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 32D (oil), and 32E (pumping the mixture into the formation). Dawson and Weaver each discloses all of those limitations and the preamble. See ¶¶ 288-317; 462-493. The combination of Dawson in view of Weaver or Plummer further discloses each and every limitation of the challenged claim 32 of the '738 patent.

a. **Preamble for Claim 32: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”**

722. The preamble of claim 32 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 12:5-6.

723. As stated in ¶¶ 291-293 and 465-471 (and incorporated by reference herein), Dawson and Weaver meet this preamble. Dawson states that his “invention relates generally to methods and compositions useful for subterranean formation treatments, such as hydraulic fracturing treatments and sand control. In particular, this invention relates to use of relatively lightweight and/or substantially neutrally buoyant particles as proppant materials in hydraulic fracturing treatment and as *particulate material in sand control methods* such as gravel packing, *frac pack treatments, etc.*” Ex. 1006, Dawson at 2:4-8. Dawson also states that his invention “may be directed toward improving wellbore productivity and/or *controlling the production of fracture proppant* or formation sand. Particular examples include gravel packing and ‘frac-packs.’” *Id.* at 13, l. 23-25. One of ordinary skill in the art would recognize, and Dawson discloses (*see, e.g., id.*), that frac packs are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize Dawson’s invention to be directed to a method of controlling proppant flowback.

724. Moreover Dawson states that his invention, which includes a slurry containing particulates coated with methyltrichlorosilane, will “provide for substantially improved overall system performance in hydraulic fracturing applications, or in other well treating applications such as sand control.” *Id.*, Dawson at 3:15-16. As disclosed in the ’738 patent, using hydrophobic particulates as the proppant in hydraulic fracturing operations is one way to control proppant flowback. See Ex. 1001, ’738 patent at 3:30-59. Thus, one of ordinary skill in the art would recognize that the disclosure of Dawson’s coated particulates would control proppant flowback in accordance with the ’738 patent and therefore meets this preamble.

725. Weaver discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, ***frac-packing operations***, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. One of ordinary skill in the art would recognize that frac-packing operations are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize that Weaver’s invention is designed to control proppant flowback.

726. Weaver also teaches that “[o]ne problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for

particulates . . . to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation.” *Id.* at [0004]. Weaver proceeds that “one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, . . . preventing the embedment of particulates into the surfaces of a subterranean formation.” *Id.* at [0014].

727. One of ordinary skill in the art would understand that Weaver’s teaching of enhancing conductivity within a subterranean formation to maximize the value of a fracturing treatment and to prevent the “clogging” of the particulates flowing back from the fracture as a means to reduce particulate flowback.

728. Additionally, because the combination of Dawson in view of Weaver and Plummer discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the ’738 patent. *See, e.g.*, Ex. 1001, ’738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback).

729. Thus, both Dawson and Weaver, and accordingly Dawson in view of Weaver, meet this preamble.

**b. Limitation 32A: “mixing water”**

730. Claim 32 recites that the composition comprises “mixing water.” Ex. 1001, ’738 patent at 12:6-7. As stated in ¶¶ 294-296 and 472-475 (and incorporated by reference herein), Dawson and Weaver meet this limitation.

731. Dawson discloses that the particles exposed to a modifying agent “may be mixed and pumped during any desired portion/s of a well treatment (e.g., hydraulic fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier fluid.” Ex. 1006, Dawson at 13, l. 6-10 Dawson also discloses “any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising ***salt water, fresh water, liquid hydrocarbons, and/or*** nitrogen or other gases.” *Id.* at 13, l. 10-13. Dawson discloses that the particulate is suspended in the carrier fluid (*id.* at 10, l. 1-3), therefore indicating that the carrier fluid including water is mixed with the particulate to form a slurry.

732. Weaver discloses “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise

***fresh water, salt water*** (e.g., water containing one or more salts dissolved therein), ***brine*** (e.g., saturated salt water), ***or seawater.***” Ex. 1008, Weaver at [0058].

733. Moreover, Weaver discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including ***aqueous gels***, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable ***aqueous gels are generally comprised of water*** and one or more gelling agents. ***Suitable emulsions*** can be comprised of two immiscible liquids such as an ***aqueous liquid*** or gelled liquid and a hydrocarbon.” *Id.* at [0059].

734. Weaver describes that the particulates are mixed with the surface-modifying agents “prior to transporting the particulates to a job site, or in a treatment performed ‘on-the-fly.’ The term ‘on-the-fly’ is used herein to mean that one flowing stream comprising particulates is continuously introduced into another flowing stream comprising the surface-treating reagent so that the streams are combined and mixed while continuing to flow as a single stream as part of the ongoing treatment at the job site. Such mixing can also be described as ‘real-time’ mixing.” *Id.* at [0056].

735. Therefore, both Dawson and Weaver, and accordingly Dawson in view of Weaver, clearly describes a composition including “mixing water” as recited in claim 32.



c. **Limitation 32B: “particulates”**

736. Claim 32 recites that the composition comprises “particulates.” Ex. 1001 at 12:7. As stated in ¶¶ 297-300 and 476-478 (and incorporated by reference herein), Dawson and Weaver meet this limitation.

737. Dawson teaches an invention that “relates to use of relatively lightweight and/or substantially neutrally buoyant *particles as proppant material* in hydraulic fracturing treatments.” Ex. 1006, Dawson at 2, l. 6-7.

738. Dawson additionally teaches that the “particulate material [is] suspended in a carrier fluid.” *Id.* at 10, l. 1-3; 48, claim 13. One of ordinary skill in the art would understand that for a particulate to be suspended in a fluid, it would need to be mixed with the fluid.

739. Weaver provides “methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates . . .” Ex. 1008, Weaver at Abstract. Weaver also teaches that “particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface.” *Id.* at [0018]. Weaver describes that the particulates are mixed with the surface-modifying agents “prior to transporting the particulates to a job site, or in a treatment performed ‘on-the-fly.’” *Id.* at [0056].

740. Thus, both Dawson and Weaver teach the use of particulates in the slurry. Limitation 32B would have therefore been obvious over Dawson in view of Weaver.

d. **Limitation 32C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

741. Claim 32 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001, ’738 patent at 12:7-8. As stated in ¶¶ 301-307 and 479-483 (and incorporated by reference herein), Dawson and Weaver meet this limitation.

742. The specification of the ’738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The ’738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51.

743. The ’738 patent also contains a list of suitable examples of organosilanes, including  $CH_3SiCl_3$  [methyltrichlorosilane]. *See id.* at 5:66-6:1.

744. Dawson teaches that particles “may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material.” Ex. 1006, Dawson at 7, l. 3-8. Specifically, Dawson discloses that “examples of suitable modifying agents include, but are not limited to, agents including polyisocyanates, *silanes*, *siloxanes*, and combinations thereof. Selected modifying agent/s may be advantageously used to increase the elastic modulus of a given naturally occurring material, for example, to make particles of a given naturally-occurring material more suitable . . . for use as a relatively lightweight and/or substantially neutrally buoyant fracture proppant or sand control particulate material having increased effectiveness when exposed to higher closure stresses or other mechanical stresses that may be encountered downhole during a well treatment such as hydraulic fracturing or sand control treatment.” *Id.* at 7, l. 14-23.

745. Like the '738 patent, Dawson also discloses the use of organosilanes and organosiloxanes to coat the particulate. Specifically, Dawson teaches that the modifying agent may contain “one or more halogen groups, (*e.g.*, tetrachlorosilane, *methyltrichlorosilane*, *etc.*).” *Id.* at 21, l. 10-11. Thus, Dawson teaches the same methyltrichlorosilane ( $\text{CH}_3\text{SiCl}_3$ ) that is disclosed in the '738 patent. See Ex. 1001, '738 patent at 6:1.

746. Dawson teaches:

Examples of commercially available silane-based products that may be employed as modifying agents in the practice of the disclose methods and compositions include, but are not limited to, silane based products” (Ex. 1006, Dawson at p. 21, l. 19-21) and teaches the specific examples of “commercially available silane-based products available from CROMPTON include CoatOSil<sup>®</sup> 1706 Silane (vinyl silane monomer), CoatOSil<sup>®</sup> 1757 Silane, CoatOSil<sup>®</sup> 1770 Silane, EURENOR<sup>®</sup> 5020 (liquid isobutyldimethoxysilane), EURENOR<sup>®</sup> 5021 (liquid diisopropylmethoxysilane), EURENOR<sup>®</sup> 5022 (liquid diisobutyldimethoxysilane), EURENOR<sup>®</sup> 5023 (liquid dicyclopentylmethoxysilane), Silcat<sup>®</sup> R Silane (vinylsilane with grafting and cross linking catalysts), Sikat<sup>®</sup> RHE Silane (cross linking system of silane, peroxide and catalyst), Silcat<sup>®</sup> RHS Silane (crosslinking system of silane, peroxide catalyst and antioxidants), Sikat<sup>®</sup> VS-735/1 Silane (crosslinking system of silane, peroxide, antioxidants and metal deactivator), Silcat<sup>®</sup> VS-758/0 Silane (stabilized cross linking system of silane, peroxide and catalyst), Sikat<sup>®</sup> VS-870 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat<sup>®</sup> VS-928 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat<sup>®</sup> VS-963 Silane (stabilized cross linking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Silox<sup>®</sup> VS- 911 Silane (liquid silane system), Silquest<sup>®</sup> A-1100 Silane (gammaaminopropyltriethoxysilane), Silquest<sup>®</sup> A-1101 Silane (technical grade amino silane), Silquest<sup>®</sup> A-1102 Silane (technical grade amino silane), Silquest<sup>®</sup> A-1110 Silane (primary amino silane), Silquest<sup>®</sup> A-1120 Silane, Silquest<sup>®</sup> A-1126 Silane (amino silane),

Silquest<sup>®</sup> A-1128 Silane (amino silane), Silquest<sup>®</sup> A-1160 Silane (ureido silane), Silquest<sup>®</sup> A-1170 Silane (secondary amino functional silane), Silquest<sup>®</sup> A-1289 Silane, Silquest<sup>®</sup> A-1310 Silane, Silquest<sup>®</sup> A-137 Silane (monomeric alkylalkoxysilane), Silquest<sup>®</sup> A-151 Silane, Silquest<sup>®</sup> A-1524 Silane (100-percent active ureidosilane), Silquest<sup>®</sup> A-1589 Silane (disulfide silane), Silquest<sup>®</sup> A-171 Silane, Silquest<sup>®</sup> A-172 Silane (vinylfunctional coupling agent), Silquest<sup>®</sup> A-174 Silane, Silquest<sup>®</sup> A-186 Silane (epoxy functional silanes), Silquest<sup>®</sup> A-187 Silane (epoxy functional silanes), Silquest<sup>®</sup> A-189 Silane, Silquest<sup>®</sup> A-2171 Silane (silane compound having vinyl and silane functionality), Silquest<sup>®</sup> Alink 15 Silane (secondary amino silane endcapper), Silquest<sup>®</sup> RC-1 Silane (organosilicone coupling agent), Silquest<sup>®</sup> RC-2 Silane, Silquest<sup>®</sup> Wetlink 78 Silane, Silquest<sup>®</sup> Y-11637 Silane (primary aminosilane), Silquest<sup>®</sup> Y-11683 Silane (di-, tri-functional ethoxy silane), Silquest<sup>®</sup> Y-5997 Silane, XL-PEarl<sup>®</sup> 31 Blend, XL-PEarl<sup>®</sup> 50 Blend, XLPEarl<sup>®</sup> 51 Blend, XL-PEarl<sup>®</sup> 52 Blend, XL-PEarl<sup>®</sup> 60 Blend, XL-PEarl<sup>®</sup> 70 Blend, *etc.*

Ex. 1006, Dawson at 21, l. 23- 22, l. 23. Many of these compounds are organosilanes.

747. Because Dawson teaches coating particles with various silanes and siloxanes, including organosilanes and organosiloxanes, and the '738 patent identifies organosilanes and organosiloxanes as examples of hydrophobizing agents (Ex. 1001, '738 patent at 4:62-67), one of ordinary skill in the art would understand that Dawson's particulates are coated with hydrophobic rendering compounds.

Moreover, Dawson discloses at least one of the same organo-silicon compounds (i.e., methyltrichlorosilane) used to coat the particulates suspended in carrier fluid identified as a “hydrophobic rendering compound” by the ’738 patent. *Compare* Ex. 1006, Dawson at 21, l. 11 *with* Ex. 1001, ’738 patent at 6:1. Thus, Dawson teaches slurry compositions that include hydrophobic rendering compounds.

748. Weaver discloses “[i]n certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, ***by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic*** or hydrophilic in nature.” Ex. 1008, Weaver at [0015].

749. Weaver also teaches that “[i]n some embodiments, the surface-treating reagent may comprise other compounds that are capable of ***hindering or preventing the water-wetting of a mineral surface***. Examples of suitable compounds include, but are not limited to, . . . ***organosilanes*** . . . Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.” *Id.* at [0049].

750. Therefore, both Dawson and Weaver teach particulates coated with a chemical compound for rendering the particulates hydrophobic—and specifically

that the chemical compound is a silane. This opinion is supported by the USPTO, because in the prosecution of the '469 application, the Examiner rejected claim 13 (which contained a limitation similar to limitation 1C) as anticipated by Weaver because each taught particles coated in water-repellent compounds. See Ex. 1004 at 184, File History of the '469 Application, Non-Final Office Action, Jan. 2, 2015 (stating that “Weaver further teaches the surface-treating reagent may comprise compounds that are capable of hindering or preventing the water-wetting of a mineral surface.”). I agree with the Examiner that Weaver teaches this limitation. Moreover, it is my opinion that Dawson additionally teaches this limitation by disclosing methyltrichlorosilane, which is disclosed as a hydrophobizing agent in the '738 patent. See Ex. 1001, '738 patent at 6:1. Thus, the combination of Dawson in view of Weaver teaches limitation 32C.

**e. Limitation 32D: “oil”**

751. Claim 32 further recites that the composition comprises “an oil.” Ex. 1001, '738 patent at 12:8. As stated in ¶¶ 308-313 and 484-489 (and incorporated by reference herein), both Dawson and Weaver teach this limitation.

752. Dawson discloses that “[t]he particles disclosed herein . . . may be mixed and pumped during any desired portion/s of well treatment (*e.g.*, hydraulic fracturing treatment, sand control treatment, *etc.*) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable for

transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids **comprising** salt water, fresh water, **liquid hydrocarbons, and/or** nitrogen or other gases.” Ex. 1006, Dawson at 13, l. 6-13. One of ordinary skill in the art would recognize this disclosure from Dawson to mean that the carrier fluid could comprise water, oil, and gas.

753. One of ordinary skill in the art would recognize that oil is a liquid hydrocarbon. See Ex. 1001, '738 patent at 11:31-33 (“20. The composition of claim 1, wherein the oil is selected from the group consisting of a **hydrocarbon oil**, a silicon oil, kerosene oil, a frac oil and combinations thereof.”). One of ordinary skill in the art would also recognize Dawson’s disclosure to mean that the carrier fluid could contain water and liquid hydrocarbon.

754. Further, Dawson incorporates by reference U.S. Patent 3,659,651 to Graham (“Graham”). See Ex. 1006, Dawson at 33, l. 6-8. Graham teaches “[i]n placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or **water-in-oil emulsion** prepared in accordance with the teachings of U.S. Pat. No. 3,378,074.” Ex. 1012, Graham at 5:9-16 (citing U.S. Patent 3,378,074 at 5:65-72



(Ex. 1013) (disclosing that “high viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either ***water-in-oil or oil-in-water emulsions***. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with ***kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine*** or an acid solution containing a suitable surface active agent or emulsifier.”)).

755. Therefore, Dawson discloses the use of water-in-oil emulsions containing water or brine in kerosene, diesel fuel, crude oil, or a hydrocarbon oil in satisfaction of this limitation.

756. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, ***or a mixture thereof. . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils***, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.” Ex. 1008, Weaver at [0058].

757. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . ***Suitable emulsions*** can be comprised of two

immiscible liquids such as an aqueous liquid or gelled liquid ***and a hydrocarbon.***” *Id.* at [0059]. One of ordinary skill in the art would recognize that the term “hydrocarbon” includes “hydrocarbon oil.” Weaver thus teaches the limitation of oil included in the composition.

758. In addition to inclusion of oils in the treatment fluids disclosed by Weaver, Weaver also discloses the addition of oils to the surface-treating reagent (i.e., organosilanes). *See* Ex. 1008, Weaver at [0054].

759. Additionally, Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a ***liquid carrier comprising one or more liquids selected from the group consisting of water and oil***, and a solid proppant comprising one or more materials selected from the group consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that “the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Thus, it would have been obvious to one of ordinary skill in the art to substitute the oil and water liquid carrier taught by Plummer for Dawson’s carrier fluid. Moreover, Plummer discloses that “[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling . .

. Other friction reducing materials may be added to the fracturing fluid.” *Id.* at 4:28-33.

760. As stated, it would have also been obvious to a person of ordinary skill in the art to include Plummer’s oil into the particulate slurry taught by Dawson and Weaver. The motivation to do so is taught by Plummer: using oil as a lubricant friction-reducer or to control clay swelling can improve hydraulic fracturing operations in wells. See Ex. 1010, Plummer at 4:28-33.

761. Thus, the simple substitution of Weaver’s treatment fluid (either the aqueous/non-aqueous mixture or the oil and water emulsion) or Plummer’s carrier (oil and water liquid carrier) for Dawson’s carrier fluid, and/or the addition of Plummer’s oil to act as a lubricant friction-reducer or as a clay stabilizer would have improved Dawson’s composition in the same way as described in Weaver or Plummer (i.e., improved hydraulic fracturing operations). This improvement would have been reasonably expected to succeed by a person of ordinary skill in the art, who would have been motivated to make this substitution and/or addition. Limitation 32D is therefore met.

f. **Limitation 32E: “pumping the mixture into the formation”**

762. Claim 32 further recites “pumping the mixture into the formation.” Ex. 1001, ’738 patent at 12:8-9. As stated in ¶¶ 314-317 and 490-493 (and incorporated

by reference herein), both Dawson and Weaver teach pumping the mixture into the formation.

763. Dawson discloses that “[t]he particles disclosed herein . . . may be ***mixed and pumped during any desired portion/s of well treatment*** (e.g., hydraulic fracturing treatment, sand control treatment, *etc.*) and may be mixed in any desired concentration with a carrier fluid.” Ex. 1006, Dawson at 13, l. 6-10. Dawson also teaches that “[i]n another exemplary embodiment, modified particulate material may be formulated as described herein and ***introduced or pumped into a well.***” *Id.* at 29, l. 27-28. A person of ordinary skill in the art would recognize that to have a sand control treatment or a hydraulic fracturing treatment, the slurry containing the sand and the carrier fluid would need to be pumped downhole, into the formation.

764. Weaver teaches “[a] method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified mineral surface; and ***introducing the treatment fluid into the subterranean formation.***” Ex. 1008, Weaver at claim 23; *see also id.* at claim 11.

765. Thus, both Dawson and Weaver teach pumping the mixture into the formation. Limitation 32E would have therefore been obvious over Dawson in view of Weaver.

766. Therefore, in my opinion, challenged independent claim 32 of the '738 patent would have been obvious over Dawson in view of Weaver or Plummer.

### 3. Independent Claim 51 is Obvious

767. As discussed in Section X.B.3 and Section X.D.3 above (and incorporated by reference herein), in addition to the preamble of claim 51, claim 51 has four limitations: 51A (contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with water and an oil to form a mixture), and 51D (pumping the mixture into the formation). Dawson and Weaver each discloses all of those limitations and the preamble. See ¶¶ 318-338; 494-517. The combination of Dawson in view of Weaver or Plummer further discloses each and every limitation of the challenged claim 51 of the '738 patent.

#### a. Preamble for Claim 51: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”

768. The preamble of claim 51 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 13:28-29. Dawson states that his invention “may be directed toward improving wellbore productivity and/or *controlling the production of fracture proppant* or formation sand. Particular examples include gravel packing

and ‘frac-packs.’” *Id.* at 13, l. 23-25. Dawson states that his “invention relates generally to methods and compositions useful for subterranean formation treatments, such as hydraulic fracturing treatments and sand control. In particular, this invention relates to use of relatively lightweight and/or substantially neutrally buoyant particles as proppant materials in hydraulic fracturing treatment and as ***particulate material in sand control methods*** such as gravel packing, ***frac pack treatments***, *etc.*” Ex. 1006, Dawson at 2, l. 4-8. One of ordinary skill in the art would recognize, and Dawson discloses (*see, e.g.*, Ex. 1006, Dawson at 13, l. 24-25), that frac packs are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize Dawson’s invention to be directed to a method of controlling proppant flowback.

769. Moreover Dawson states that his invention, which includes a slurry containing particulates coated with methyltrichlorosilane, will “provide for substantially improved overall system performance in hydraulic fracturing applications, or in other well treating applications such as sand control.” *Id.*, Dawson at 3:15-16. As disclosed in the ’738 patent, using hydrophobic particulates as the proppant in hydraulic fracturing operations is one way to control proppant flowback. *See* Ex. 1001, ’738 patent at 3:30-59. Thus, one of ordinary skill in the art would recognize that the disclosure of Dawson’s coated particulates would

control proppant flowback in accordance with the '738 patent and therefore meets this preamble.

770. Weaver discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, ***frac-packing operations***, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. One of ordinary skill in the art would recognize that frac-packing operations are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize that Weaver’s invention is designed to control proppant flowback.

771. Weaver also teaches that “[o]ne problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, ***proppant particulates***, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation. . . . [A] technique used to prevent flow-back problems, commonly referred to as ‘gravel packing,’ involves the placement of a gravel screen in the subterranean formation, which acts as a barrier that prevents particulates from flowing into the well bore.” *Id.* at [0004]. Weaver proceeds that “one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of

a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, . . . preventing the embedment of particulates into the surfaces of a subterranean formation.” *Id.* at [0014].

772. One of ordinary skill in the art would understand that Weaver’s teaching of enhancing conductivity within a subterranean formation to maximize the value of a fracturing treatment and to prevent the embedment of particulates into surfaces of the subterranean formation to be describing operations to reduce particulate flowback. Moreover, Weaver discloses that his invention can be used in frac-packing operations, which is a method of controlling proppant flowback. *See e.g.*, Ex. 1008, Weaver at [0004].

773. Additionally, because Dawson in view of Weaver or Plummer discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the ’738 patent. *See, e.g.*, Ex. 1001, ’738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback).

774. Therefore, in my opinion, the preamble of claim 51 is met by the combination of Dawson in view of Weaver or Plummer.



b. **Limitation 51A: “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic”**

775. Claim 51 recites “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic.” Ex. 1001 at 13:29-31.

776. Dawson teaches that particles “may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material.” *Id.* at 7, l. 3-8. Dawson states that the “[m]odifying agents may be *diluted as so desired in a suitable solvent prior to treating particles* of a naturally-occurring material.” *Id.* at 27, l. 1-2. Dawson further discloses that “[s]pecific examples of *suitable exposure methods* include, but are not limited to, by *soaking* the naturally-occurring material in a modifying agent . . . by *dipping* the naturally-occurring material in a modifying agent, by *spraying* a modifying agent on to the naturally-occurring material . . . by *mixing* naturally-occurring particulate material with liquid, atomized and/or gaseous modifying agent in a vertical mixer . . . by *flowing* modifying agent through a permeable bed or pack of the naturally-occurring particles in a pressurized container. Drying of particles after exposure may be accomplished, for example, by continuously dropping through heated air.” *Id.*, at 24, l. 17-26.

777. Weaver teaches that one “suitable method[] for allowing the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate includes . . . solvent extraction methods.” *Id.* at [0057]. In such an embodiment, “[t]he solution comprising the surface treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced to a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface.” *Id.* As discussed, Dawson discloses examples of organo-silicon compounds that are the same as those disclosed in the ’738 patent, including methyltrichlorosilane. Moreover, Weaver discloses the use of organosilanes, which the ’738 patent identifies as a chemical compound for rendering the surface of the particulates hydrophobic.

778. Thus Dawson and Weaver clearly describe contacting the particulates with an organo-silicon compound, which, as stated above, is an example of “a chemical compound to render the surface of the particulates hydrophobic” as required by claim 51.

c. **Limitation 51B: “separating the particulate from the medium”**

779. Claim 51 recites that the now-coated particulates are “separate[ed] . . . from the medium.” Ex. 1001 at 13:32.

780. Dawson teaches that particles “may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material.” Id. at 7, l. 3-8. Dawson states that the “[m]odifying agents may be diluted as so desired in a suitable solvent prior to treating particles of a naturally-occurring material.” Id. at 27, l. 1-2. Dawson further discloses that “[s]pecific examples of suitable exposure methods include, but are not limited to, by soaking the naturally-occurring material in a modifying agent . . . by dipping the naturally-occurring material in a modifying agent, by spraying a modifying agent on to the naturally-occurring material . . . by mixing naturally-occurring particulate material with liquid, atomized and/or gaseous modifying agent in a vertical mixer . . . by flowing modifying agent through a permeable bed or pack of the naturally-occurring particles in a pressurized container. *Drying of particles after exposure* may be accomplished, for example, by continuously dropping through heated air.” Id., at 24, l. 17-26. As recited, drying is one method of separating the particles. Thus, Dawson discloses that the now-coated particulates are separated from the solvent solution.

781. Weaver teaches that one “suitable method[] for allowing the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a

particulate includes . . . solvent extraction methods.” *Id.* at [0057]. In such an embodiment, “[t]he solution comprising the surface treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. ***The treated particulates then may be introduced to a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface.***” *Id.* As discussed, one of ordinary skill in the art would understand Weaver’s disclosure of “extract[ing] the solvent from the treated particulates” to be a method of separating the particulate from the solvent containing the organo-silicon coating. Thus, Weaver discloses that the now-coated particulates are separated from the solvent solution.

782. In view of the foregoing excerpts from the disclosure of Dawson and Weaver, one of ordinary skill in the art would have understood the combination of Dawson in view of Weaver or Plummer to disclose this limitation.

**d. Limitation 51C: “blending the hydrophobic particulate with water and an oil to form a mixture”**

783. Claim 51 recites that the hydrophobic coated particulate are blended “with water and oil to form a mixture.” Ex. 1001 at 13:32-34.

784. Dawson discloses that “[t]he ***particles*** disclosed herein . . . may be mixed and pumped during any desired portion/s of well treatment (*e.g.*, hydraulic

fracturing treatment, sand control treatment, *etc.*) and ***may be mixed in any desired concentration with a carrier fluid.*** In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids ***comprising salt water, fresh water, liquid hydrocarbons, and/or*** nitrogen or other gases.” Ex. 1006, Dawson at 13, l. 6-13. Dawson thus teaches the limitation of oil included in the composition and discloses that the carrier fluid may comprise both water ***and*** a liquid hydrocarbon.

785. One of ordinary skill in the art would recognize that liquid hydrocarbons are an oil (i.e., a hydrocarbon oil). See Ex. 1001, ’738 patent at 13:1-3 (“42. The composition of claim 1, wherein the oil is selected from the group consisting of a ***hydrocarbon oil***, a silicone oil, kerosene oil, a frac oil and combinations thereof.”). Thus, one of ordinary skill in the art would also recognize Dawson’s disclosure to mean that the carrier fluid could contain water and oil.

786. Further, Dawson incorporates by reference U.S. Patent 3,659,651 to Graham (“Graham”). See Ex. 1006, Dawson at 33, l. 6-8. Graham teaches “[i]n placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil

or ***water-in-oil emulsion*** prepared in accordance with the teachings of U.S. Pat. No. 3,378,074.” Ex. 1012, Graham at 5:9-16 (citing U.S. Patent 3,378,074 at 5:65-72 (Ex. 1013) (disclosing that “high viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either ***water-in-oil or oil-in-water emulsions***. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with ***kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine*** or an acid solution containing a suitable surface active agent or emulsifier.”)).

787. Weaver discloses that the ***treatment fluids containing the suspended proppant*** “generally comprise a base fluid, which may be aqueous or non-aqueous, ***or a mixture thereof***. Where the base fluid is aqueous, it may comprise fresh water [or] salt water . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts ***can be emulsified also may be suitable for use as a base fluid*** in the methods of the present invention.” Ex. 1008, Weaver at [0058].

788. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . ***Suitable emulsions*** can be comprised of two

immiscible liquids such as an *aqueous liquid* or gelled liquid *and a hydrocarbon.*”  
*Id.* at [0059].

789. One of ordinary skill in the art would recognize, and the ’738 patent confirms, that oil is a type of “hydrocarbon.” *See* Ex. 1001, ’738 patent at 11:31-33 (“20. The composition of claim 1, wherein the oil is selected from the group consisting of a *hydrocarbon oil*, a silicon oil, kerosene oil, a frac oil and combinations thereof.”). Weaver thus teaches the limitation of oil included in the composition in the form of a treatment fluid including a mixture of oil and water, as well as an oil and water emulsion.

790. Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a *liquid carrier comprising one or more liquids selected from the group consisting of water and oil*, and a solid proppant comprising one or more materials selected from the group consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that “the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Thus, it would have been obvious to one of ordinary skill in the art to substitute the oil and water liquid carrier taught by Plummer for Dawson’s carrier fluid. Moreover, Plummer discloses that “[o]il may

be added to the foam-water slurry as a lubricant and to prevent clay swelling . . . Other friction reducing materials may be added to the fracturing fluid.” *Id.* at 4:28-33.

791. As stated, it would have also been obvious to a person of ordinary skill in the art to include Plummer’s oil into the particulate slurry taught by Nguyen and Weaver. The motivation to do so is taught by Plummer: using oil as a lubricant friction-reducer or to reduce clay swelling can improve hydraulic fracturing operations in wells. *See* Ex. 1010, Plummer at 4:28-33.

792. Thus, the simple substitution of Weaver’s treatment fluid (either the aqueous/non-aqueous mixture or the oil and water emulsion) or Plummer’s carrier (oil and water liquid carrier) for Dawson’s carrier fluid, and/or the addition of Plummer’s oil to act as a lubricant friction-reducer or to reduce clay swelling would have improved Dawson’s composition in the same way as described in Weaver or Plummer (i.e., improved hydraulic fracturing operations). This improvement would have been reasonably expected to succeed by a person of ordinary skill in the art, who would have been motivated to make this substitution and/or addition. Limitation 51C is therefore met.

e. **Limitation 51D: “pumping the mixture into the formation”**

793. Claim 51 further recites “pumping the mixture into the formation.” Ex. 1001 at 13:34.



794. As recited, Dawson discloses that “[t]he particles disclosed herein . . . ***may be mixed and pumped*** during any desired portion/s of well treatment (*e.g.*, hydraulic fracturing treatment, sand control treatment, *etc.*) and may be mixed in any desired concentration with a carrier fluid.” Ex. 1006, Dawson at 13, l. 6-10. Dawson also teaches that “[i]n another exemplary embodiment, modified particulate material may be formulated as described herein and introduced or pumped into a well.” *Id.* at 29, l. 27-28. As described in Dawson, a person of ordinary skill would recognize that the slurry comprising particles in a treatment or carrier fluid would be pumped into the formation.

795. Weaver teaches “[a] method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified mineral surface; and ***introducing the treatment fluid into the subterranean formation.***” Ex. 1008, Weaver at claim 23; *see also id.* at claim 11.

796. Therefore, Dawson and Weaver disclose limitation 51D.

797. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the

mixture into the formation), and the preamble of claim 51, is taught by the combination of Dawson in view of Weaver or Plummer. Therefore, independent claim 51 is obvious over Dawson in view of Weaver or Plummer.

#### 4. Dependent Claims 21, 22, 40, 41, and 57 Are Obvious

798. As addressed above (*see* Section XI.B.1, XI.B.2, and XI.B.3) and incorporated herein by reference, all of the limitations of claims 1, 32, and 51, including 1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium); 51C (blending the hydrophobic particulate with water and an oil to form a mixture), 51D (pumping the mixture into the formation), and the preambles of claims 1, 32, and 57, are obvious over Dawson in view of Weaver or Plummer.

799. The only additional limitation in claims 21, 22, 40, 41, and 57 relate to the type of particulate. Dependent claims 21, and 40 recite a near-identical limitation, but depend from different claims. Claim 21 depends from independent claim 1, whereas claim 40 depends from independent claim 32. Similarly, dependent claims 22, 41, and 57 recite the same limitation, but depend from different claims. Claim 22 depends from independent claim 1, whereas claim 41 depends from claim

32, and claim 57 depends from independent claim 51. Dependent claims 21 and 40 require the particulate be one of an enumerated list of particulates, including sand. *See* Ex. 1001, '738 patent at 11:34-37; 12:63-65 (“wherein the particulates are selected from the group consisting of sand . . .”). Dependent claims 22, 41, and 57 require the particulate to be sands. *Id.* at 11:38-39; 12:66-67; 14:24-25 (“wherein the particulates are sands”). Therefore, a prior art reference that discloses the additional limitation of “sand” as the particulate anticipates claims 21, 22, 40, 41, and 57.

800. Dawson teaches the additional limitation of claims 21, 22, 40, 41, and 57 because Dawson teaches the use of particulate and incorporates by reference U.S. Patent 6,330,916 (Ex. 1011) (“Rickards”). Ex. 1006, Dawson at 13, l. 3-5. Rickards discloses a subterranean formation treatment method for reducing proppant flowback. Ex. 1011, Rickards at Abstract. Rickards further discloses that the “fracture proppant material may be a material such as *sand*.” *Id.*

801. Dawson discloses that “[a] slurry including the disclosed particulate material . . . and a carrier fluid may then be introduced into the wellbore.” Dawson at p. 14, l. 12-15. Dawson therefore teaches that the particulates are mixed with the carrier fluid.

802. Weaver teaches the additional limitation of claims 21, 22, 40, 41 and 57 because Weaver teaches “[t]he particulates suitable for use in the present

invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, **sand.**” Ex. 1008, Weaver at [0018].

803. Weaver also teaches that “the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.” *Id.* at [0057]. Thus, the sand particulates are mixed with the treatment fluid before being pumped down the formation.

804. Thus, Dawson and Weaver teach the additional limitation of claims 21, 22, 40, 41, and 57. Therefore, it is my opinion that challenged dependent claims 21, 22, 40, 41, and 57 of the ’738 patent would have been obvious over Dawson in view of Weaver or Plummer.

805. Based on the foregoing analysis of Sections XI.B.1-4, all of the elements of claims 1, 21, 22, 32, 40, 41, 51, and 57 [1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the

mixture into the formation), 51A (contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium); 51C (blending the hydrophobic particulate with water and an oil to form a mixture), 51D (pumping the mixture into the formation), 21A, 22A, 40A, 41A, and 57A (sand), and the preambles of claims 1, 32, and 51] are disclosed by the combination of Dawson in view of Weaver or Plummer. Therefore, it is my opinion that the combination of Dawson in view of Weaver or Plummer renders challenged claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent obvious.

**C. Claims 1, 21, 22, 32, 40, 41, 51, and 57 Are Obvious over Vincent in view of Weaver or Plummer**

806. As discussed in Sections X.C and X.D above (and incorporated by reference herein), both Vincent and Weaver anticipate claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent because each reference discloses each and every element of the claims. Furthermore, and in the alternative, the disclosure of Vincent in view of Weaver or Plummer also renders claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent obvious. *See generally*, Ex. 1002J, '738 Patent v. Vincent in view of Weaver or Plummer Claim Chart.

807. One of ordinary skill in the art at the time of the invention would have been motivated to combine Vincent and Weaver because both are directed to methods for modifying sand particulates with a water-repellent compound for use in

hydraulic fracturing operations. Further, one of ordinary skill in the art would be motivated to substitute the aqueous fracture fluid of Vincent with the oil and water emulsion treatment fluid of Weaver to arrive at the invention of claim 1 of the '738 patent, since both were well known in the art and there was a high likelihood of success. Additionally, and in the alternative, a person of ordinary skill in the art would be motivated to substitute the aqueous fracture fluid of Vincent with the oil and water liquid carrier disclosed in Plummer for the same reason. Moreover, a person of ordinary skill in the art would be motivated to add oil to the slurry as a lubricant friction-reducer or to reduce clay swelling, as taught by Plummer (Ex. 1010 at 4:28-33) to improve the compositions and methods of Vincent in hydraulic fracturing operations.

**1. Independent Claim 1 Is Obvious**

808. As discussed in Section X.C.1 and Section X.D.1 above (and incorporated by reference herein), in addition to the preamble of claim 1, claim 1 has four limitations: 1A (water), 1B (particulates), 1C (a chemical compound for rendering the surface of the particulate hydrophobic), and 1D (oil). Vincent and Weaver each discloses all of those limitations and the preamble. See ¶¶ 352-375; 438-461. The combination of Vincent in view of Weaver or Plummer further discloses each and every limitation of challenged claim 1 of the '738 patent.

**a. Preamble for Claim 1: “An aqueous slurry composition comprising”**

809. The preamble of claim 1 of the '738 patent states, “An aqueous slurry composition comprising.” Ex. 1001, '738 patent at 9:46.

810. As stated in ¶¶ 355 and 441 (and incorporated by reference herein), Vincent and Weaver meet this preamble. Vincent teaches that “particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or ‘slurry’ into which the particles have previously been introduced and suspended.” Ex. 1007, Vincent at [0087]. Vincent also teaches an aqueous slurry. *Id.* (“The fracturing fluid may be . . . **water base** . . .”).

811. Weaver teaches that “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be **aqueous**.” Ex. 1008, Weaver at [0058].

812. Thus, both Vincent and Weaver, and accordingly Vincent in view of Weaver, meet this preamble.

**b. Limitation 1A: “water”**

813. Claim 1 recites that the composition comprises “water.” Ex. 1001 at 9:47. As stated in ¶¶ 356-357 and 442-444 (and incorporated by reference herein), both Vincent and Weaver teach the use of water.

814. Vincent discloses that the fracture fluid in which the particulates are suspended “may be an oil base, **water base**, acid, **emulsion**, foam, or any other fluid.” Ex. 1007, Vincent at [0087]. One of ordinary skill in the art would understand that an emulsion may contain oil and water.

815. Weaver discloses “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or **a mixture thereof**. Where the base fluid is aqueous, it may comprise **fresh water, salt water** (e.g., water containing one or more salts dissolved therein), **brine** (e.g., saturated salt water), **or seawater**.” Ex. 1008, Weaver at [0058].

816. Moreover, Weaver discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including **aqueous gels**, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable **aqueous gels are generally comprised of water** and one or more gelling agents. **Suitable emulsions** can be comprised of two immiscible liquids such as an **aqueous liquid** or gelled liquid and a hydrocarbon.” *Id.* at [0059].

817. Thus, both Vincent and Weaver teach that water can be included in the slurry as the fracturing/treatment fluid of the proppant. Limitation 1A would have therefore been obvious over Vincent in view of Weaver.



c. **Limitation 1B: “particulates”**

818. Claim 1 recites that the composition comprises “particulates.” Ex. 1001 at 9:48. As stated in ¶¶ 358-360 and 445-447 (and incorporated by reference herein), both Vincent and Weaver teach the use of particulates.

819. Vincent teaches that his invention “relates to oil and gas well proppants and, more particularly, to process for physically or chemically modifying the surface characteristics of hydraulic fracturing proppants.” Ex. 1007, Vincent at [0002]. Vincent further teaches that the particulates would be mixed into the aqueous carrier fluid to form a slurry. *See id.*, Vincent at [0087].

820. Weaver provides “methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates . . .” Ex. 1008, Weaver at Abstract. Weaver also teaches that “particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface.” *Id.* at [0018].

821. Both Vincent and Weaver teach the use of particulates in the slurry. Limitation 1B would have therefore been obvious over Vincent in view of Weaver.

d. **Limitation 1C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

822. Claim 1 of the ’738 patent recites that the composition includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex.

1001, '738 patent at 9:49-50. As stated in ¶¶ 361-366 and 448-452 (and incorporated by reference herein), both Vincent and Weaver teach this limitation.

823. The specification of the '738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including **organosiloxane**, **organosilane**, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The '738 patent further states that “[a]mong different organosiloxane compounds which are useful for the present invention, polysiloxanes modified with organic amphoteric or cationic groups including organic betaine polysiloxanes and organic quaternary siloxanes are examples” (*id.* at 6:44-48). The '738 patent identifies that “[t]he simplest polysiloxanes are **polydimethylsiloxanes**.” *Id.* at 5:8-9.

824. Moreover, in Example 1 of the '738 patent, a solution containing “Tegopren 6924, a di-quaternary **polydimethylsiloxane** from Degussa Corp.” was used to hydrophobize frac sand. *Id.* at 8:47-50.

825. Vincent discloses “modifying the surface properties of natural sand, resin-coated sand, and manufactured proppants used in oil and gas recovery to achieve one or more of the **following desirable effects: alter the wettability**, alter the chemical reactivity, alter the surface topography, impart lubricity, and control

relative permeability to flow of fluids such as proppants. Sands, resin coated sands, or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.” Ex. 1007, Vincent at [0008].

826. Vincent teaches that the above benefits can be achieved by a variety of techniques, including by “coating the proppants with a **hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes.**” *Id.* at [0009]. Vincent also discloses a coated proppant “wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and **polydimethyl siloxane.**” *Id.* at claim 6. Vincent also provides examples of proppant coated with Rain-X<sup>®</sup>, a commercially available product that includes polyalkyl hydrogen siloxane. *See e.g., id.* at [0043]. Vincent teaches that the Rain-X<sup>®</sup> coated proppant trapped air bubbles that accumulated over time and that the proppant was clearly hydrophobic. *Id.* at [0082]. This is noteworthy because during the prosecution of the ’469 application and explicitly stated in the specification of the ’738 patent, the Applicant repeatedly described his invention as using micro gas bubbles attached individually to the sand grains to lift the sand grains by buoyancy. *See e.g.,* Ex. 1004 at 310, Declaration Under 37 C.F.R. § 1.132 at ¶ 3, May 10, 2016; Ex. 1004 at 208, File History of the ’469 Application, Amendment and Response,

May 4, 2015; *see also* Ex. 1001, '738 patent at 3:28-30 (“It is also known that micro-bubbles attached to the hydrophobic particle surfaces also tend to bridge the particles together.”); Ex. 1001, '738 patent at 3:34-40 (“Unlike in conventional approaches, where attention is focused on making proppants or sand particles sticky through formation of chemical bonds between resins coated on the particle surfaces, in the present invention the attention is focused on making particle aggregations by bridging the particles through strong hydrophobic force or micro-bubbles.”). Thus, Vincent explicitly describes Applicant’s self-described inventive concept.

827. Vincent’s published claims clearly disclose coating particles with a hydrophobic material. *See e.g., id.* at claims 1-6, 9-10, 15-17. Vincent also discloses at least one of the source organo-silicon compounds (i.e., polydimethyl siloxane) as listed in the '738 patent as a specific example of a hydrophobic rendering compound. *Compare* Ex. 1007, Vincent at claim 6 *with* Ex. 1001, '738 patent at 8:47-50.

828. Weaver discloses “[i]n certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, ***by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic*** or hydrophilic in nature.” Ex. 1008, Weaver at [0015].

829. Weaver also teaches that “[i]n some embodiments, the surface-treating reagent may comprise other compounds that are capable of *hindering or preventing the water-wetting of a mineral surface*. Examples of suitable compounds include, but are not limited to, . . . *organosilanes* . . . Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.” *Id.* at [0049].

830. Therefore, both Vincent and Weaver teach particulates coated with a chemical compound for rendering the particulates hydrophobic—and specifically that the chemical compound is an organo-silicon compound. This opinion is supported by the USPTO, because in the prosecution of the ’469 application, the Examiner rejected claim 13 (which contained a limitation similar to limitation 1C) as anticipated by Weaver because each taught particles coated in water-repellent compounds. *See* Ex. 1004 at 184, File History of the ’469 Application, Non-Final Office Action, Jan. 2, 2015 (stating that “Weaver further teaches the surface-treating reagent may comprise compounds that are capable of hindering or preventing the water-wetting of a mineral surface.”). I agree with the Examiner that Weaver teaches this limitation. Moreover, it is my opinion that Vincent additionally teaches this limitation by disclosing polydimethylsiloxanes, an organosiloxane, which is disclosed as a hydrophobizing agent in the ’738 patent. *See* Ex. 1001, ’738 patent

at 4:62-67. Thus, the combination of Vincent in view of Weaver teaches limitation 1C.

**e. Limitation 1D: “oil”**

831. Claim 1 further recites that the composition comprises “an oil.” Ex. 1001, ’738 patent at 9:50. As stated in ¶¶ 367-375 and 453-461 (and incorporated by reference herein), both Vincent and Weaver teach this limitation.

832. Vincent discloses that the benefits taught by his invention “can be achieved by a variety of techniques, including coating the proppants with a hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes, polytetrafluoroethylene (commonly known as Teflon®), *plant oils, such as linseed oil, soybean oil, corn oil, cottonseed oil, vegetable oil* (widely commercially available such as Crisco®), *and canola oil, and hydrocarbons such as kerosene, diesel, and crude oil, petroleum distillates such as hydrocarbon liquids.*” Ex. 1007, Vincent at [0009]. Moreover, Vincent teaches that the “fracturing fluid may be an oil base, water base, acid, *emulsion*, foam, or any other fluid.” *Id.* at [0087]. One of ordinary skill in the art would understand that an emulsion may be comprised of a water and an oil.

833. Vincent also discloses proppants coated with a hydrophobic material, “wherein the hydrophobic material *comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene,*

*plant oils*, [and] *hydrocarbons*.” *Id.* at claim 2; *see also id.* at claims 10 and 21. Thus Vincent teaches the use of two hydrophobic rendering compounds. One, for example, polydimethyl siloxane, satisfies limitation 1C (compound for rendering the surface of the particulates hydrophobic); the other, for example, plant oil or hydrocarbons satisfies limitation 1D (oil).

834. One of ordinary skill in the art would recognize, and the ’738 patent confirms, that oil is a “hydrocarbon liquid” or “hydrocarbon.” *See* Ex. 1001, ’738 patent at 11:31-33 (“20. The composition of claim 1, wherein the oil is selected from the group consisting of a **hydrocarbon oil**, a silicon oil, kerosene oil, a frac oil and combinations thereof.”). Vincent thus teaches the limitation of oil included in the composition.

835. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, **or a mixture thereof. . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils**, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.” Ex. 1008, Weaver at [0058].

836. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . **Suitable emulsions** can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid **and a hydrocarbon.**” *Id.* at [0059]. One of ordinary skill in the art would recognize that the term “hydrocarbon” includes “hydrocarbon oil.” Weaver thus teaches the limitation of oil included in the composition.

837. In addition to inclusion of oils in the treatment fluids disclosed by Weaver, Weaver also discloses the addition of oils to the surface-treating reagent (i.e., organosilanes). *See* Ex. 1008, Weaver at [0054].

838. Moreover, the ’738 specification also discloses that the oil of the composition may be formation oil. Ex. 1001 at 4:11-15 (“Alternatively the slurry containing a hydrophobizing agent can be pumped into the fracture forming the proppant pack, which can be further consolidated by oil or condensate contained in the formation.”). Thus, the oil limitation in claim 1 can be satisfied by the presence of formation oil. This is consistent with the prosecution history of the ’738 patent. *See* Section VII.C, *supra*.

839. Vincent further discloses that “[e]mbodiments of the present invention relate to modifying the surface properties of natural sand, resin-coated sand and manufactured proppants **used in oil and gas recovery** to achieve one or more of the



following desirable effects: alter the wettability.” Ex. 1007, Vincent at [0008]. And, that “various embodiments of the present invention relate to concepts and techniques to treat fracturing sand and/or proppant to: . . . alter or modify proppant wettability to control the relative permeability to flow of the fluids which may be encountered in the reservoir (*such as oil*, water, gas, chemical treatments, and fracturing fluids).” *Id.* at [0012], [0014].

840. Weaver further discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. Weaver also discloses a “method of treating a subterranean formation comprising: providing a treatment fluid that comprises: a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; . . . and introducing the treatment fluid into the subterranean formation.” *Id.* at claim 11.

841. Because the disclosure of Vincent specifically teaches the use of the invention in a reservoir containing oil and that its invention can be used in oil and gas recovery, the composition of Vincent must therefore comprise oil. Similarly, one of ordinary skill in the art would recognize that the subterranean formations

referenced by Weaver that are treated by fracturing operations, gravel packing operations, frac packing operations and perforating contain oil. *See, e.g.*, Ex. 1001, '738 patent at 1:26-31 (stating that hydraulic fracturing operations are used to “enhance oil and gas production” and to “increase oil and gas production”). Further, one of ordinary skill in the art would understand that “introducing the treatment fluid” containing particulate coated in surface-treating reagent and base fluid “into the subterranean formation” during a subterranean operation as taught by Weaver would necessarily mean that oil is present. Because the formation contains oil, the composition of Weaver must therefore comprise oil.

842. Additionally, Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising *a liquid carrier comprising one or more liquids selected from the group consisting of water and oil*, and a solid proppant comprising one or more materials selected from the group consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that “the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Thus, it would have been obvious to one of ordinary skill in the art to substitute the oil and water liquid carrier taught by Plummer for Vincent’s fracturing fluid. Moreover, Plummer discloses that “[o]il

may be added to the foam-water slurry as a lubricant and to prevent clay swelling . . . Other friction reducing materials may be added to the fracturing fluid.” *Id.* at 4:28-33.

843. As stated, it would have been obvious to a person of ordinary skill in the art to include Plummer’s oil into the particulate slurry taught by Vincent and Weaver. The motivation to do so is taught by Plummer: using oil as a lubricant friction-reducer or to control clay swelling can improve hydraulic fracturing operations in wells. *See* Ex. 1010, Plummer at 4:28-33.

844. Thus, the simple substitution of Weaver’s treatment fluid (either the aqueous/non-aqueous mixture or the oil and water emulsion) or Plummer’s carrier (oil and water liquid carrier) for Vincent’s fracturing fluid, and/or the addition of Plummer’s oil to act as a lubricant friction-reducer or to prevent clay swelling would have improved Vincent’s composition in the same way as described in Weaver or Plummer (i.e., improved hydraulic fracturing operations). This improvement would have been reasonably expected to succeed by a person of ordinary skill in the art, who would have been motivated to make this substitution and/or addition. Limitation 1D is therefore met.

845. Therefore, it is my opinion that challenged independent claim 1 of the ’738 patent would have been obvious over Vincent in view of Weaver or Plummer.

## 2. Independent Claim 32 Is Obvious

846. As discussed in Section X.C.2 and Section X.D.2 above (and incorporated by reference herein), in addition to the preamble of claim 32, claim 32 has five limitations: 32A (water), 32B (particulates), 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 32D (oil), and 32E (pumping the mixture into the formation). Vincent and Weaver each discloses all of those limitations and the preamble. See ¶¶ 376-405; 462-493. The combination of Vincent in view of Weaver or Plummer further discloses each and every limitation of the challenged claim 32 of the '738 patent.

a. **Preamble for Claim 32: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”**

847. The preamble of claim 32 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 12:5-6. As stated in ¶¶ 379-384 and 465-471 (and incorporated by reference herein), Vincent and Weaver meet this preamble.

848. The preamble of claim 51 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 13:28-29.

849. Vincent discloses “concepts and techniques to treat fracturing sand and/or proppant to: . . . alter or modify proppant wettability, to control the relative permeability to flow of the fluids which may be encountered in the reservoir . . .

[and] ‘lubricate’ the proppant to allow more efficient proppant arrangement when the fracture closes, effectively increasing packing efficiency.” Ex. 1007, Vincent at [0012], [0014], [0015]. Vincent further discloses that this “‘lubrication’ concept reduces proppant friction, allowing superior proppant redistribution during fracture closing. This redistribution allows more efficient packing of proppant, thereby increasing grain-to-grain contact and effectively increasing proppant pack strength.” *Id.* at [0028].

850. One of ordinary skill in the art would understand that controlling the relative permeability to flow of the fluids would reduce the viscous drag on the proppant particles, which would help control proppant flowback. One of ordinary skill in the art would also understand that increasing grain-to-grain contact to increase proppant pack strength is another method of controlling proppant flowback because more proppant particles would remain in the fracture when it closes, thereby reducing the amount of proppant that would flowback.

851. Further, Weaver discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. One of ordinary skill in the art would recognize that frac-packing operations are a type of gravel pack that are especially designed to control proppant flowback.

Thus, one of ordinary skill in the art would recognize that Weaver's invention is designed to control proppant flowback.

852. Weaver also teaches that “[o]ne problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates . . . to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation.” *Id.* at [0004]. Weaver proceeds that “one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, . . . preventing the embedment of particulates into the surfaces of a subterranean formation.” *Id.* at [0014].

853. One of ordinary skill in the art would understand that Weaver's teaching of enhancing conductivity within a subterranean formation to maximize the value of a fracturing treatment and to prevent “clogging” of particulates to reduce particulate flowback.

854. Additionally, because the combination of Vincent in view of Weaver and Plummer discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the '738 patent. *See, e.g.*, Ex. 1001, '738 patent at Abstract,

3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback).

855. Thus, both Vincent and Weaver, and accordingly Vincent in view of Weaver, meet this preamble.

**b. Limitation 32A: “mixing water”**

856. Claim 32 recites that the composition comprises “mixing water.” Ex. 1001 at 12:6-7. As stated in ¶¶ 385 and 472-475 (and incorporated by reference herein), Vincent and Weaver meet this limitation.

857. Vincent discloses that the fracture fluid in which the particulates are suspended “may be an oil base, **water base**, acid, **emulsion**, foam, or any other fluid.” Ex. 1007, Vincent at [0087]. One of ordinary skill in the art would understand that an emulsion can comprise oil and water. Vincent teaches the mixing step when describing the fluid or slurry “into which the particles have previously been introduced and suspended.” *Id.* To be suspended, the particles of Vincent would have been mixed with the carrier fluid. Thus, Vincent teaches the mixing water step.

858. Weaver discloses “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or

non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise ***fresh water, salt water*** (e.g., water containing one or more salts dissolved therein), ***brine*** (e.g., saturated salt water), ***or seawater.***” Ex. 1008, Weaver at [0058].

859. Moreover, Weaver discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including ***aqueous gels***, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable ***aqueous gels are generally comprised of water*** and one or more gelling agents. ***Suitable emulsions*** can be comprised of two immiscible liquids such as an ***aqueous liquid*** or gelled liquid and a hydrocarbon.” *Id.* at [0059].

860. Weaver describes that the particulates are mixed with the surface-modifying agents “prior to transporting the particulates to a job site, or in a treatment performed ‘on-the-fly.’ The term ‘on-the-fly’ is used herein to mean that one flowing stream comprising particulates is continuously introduced into another flowing stream comprising the surface-treating reagent so that the streams are combined and mixed while continuing to flow as a single stream as part of the ongoing treatment at the job site. Such mixing can also be described as ‘real-time’ mixing.” *Id.* at [0056].

861. Therefore, both Vincent and Weaver, and accordingly Vincent in view of Weaver, clearly describes a composition including “mixing water” as recited in claim 32.



c. **Limitation 32B: “particulates”**

862. Claim 32 recites that the composition comprises “particulates.” Ex. 1001 at 12:7. As stated in ¶¶ 386-388 and 476-478 (and incorporated by reference herein), Vincent and Weaver meet this limitation.

863. Vincent teaches that his invention “relates to oil and gas well proppants and, more particularly, to process for physically or chemically modifying the surface characteristics of hydraulic fracturing proppants.” Ex. 1006, Vincent at [0002]. Vincent further teaches a “‘slurry’ into which the particles have previously been introduced and suspended.” *Id.* at [0087].

864. Weaver provides “methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates . . .” Ex. 1008, Weaver at Abstract. Weaver also teaches that “particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface.” *Id.* at [0018]. Weaver describes that the particulates are mixed with the surface-modifying agents “prior to transporting the particulates to a job site, or in a treatment performed ‘on-the-fly.’” *Id.* at [0056].

865. Thus, both Vincent and Weaver teach the use of particulates in the slurry. Limitation 32B would have therefore been obvious over Vincent in view of Weaver.

d. **Limitation 32C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

866. Claim 32 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001 at 12:7-8. As stated in ¶¶ 389-394 and 479-483 (and incorporated by reference herein), Vincent and Weaver meet this limitation.

867. The specification of the ’738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including *organosiloxane*, *organosilane*, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The ’738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51.

868. Vincent discloses “modifying the surface properties of natural sand, resin-coated sand, and manufactured proppants used in oil and gas recovery to achieve one or more of the *following desirable effects: alter the wettability*, alter the chemical reactivity, alter the surface topography, impart lubricity, and control relative permeability to flow of fluids such as proppants. Sands, resin coated sands,

or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.” Ex. 1007, Vincent at [0008].

869. Vincent teaches that the above benefits can be achieved by a variety of techniques, including by “coating the proppants with a **hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes.**” *Id.* at [0009]. Vincent also discloses a coated proppant “wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and **polydimethyl siloxane.**” *Id.* at claim 6. Vincent also provides examples of proppant coated with Rain-X<sup>®</sup>, a commercially available product that includes polyalkyl hydrogen siloxane. *See e.g., id.* at [0043]. Vincent teaches that the Rain-X<sup>®</sup> coated proppant trapped air bubbles that accumulated over time and that the proppant was clearly hydrophobic. *Id.* at [0082]. This is noteworthy because during the prosecution of the ‘469 application and explicitly stated in the specification of the ’738 patent, the Applicant repeatedly described his invention as using micro gas bubbles attached individually to the sand grains to lift the sand grains by buoyancy. *See e.g.,* Ex. 1004 at 310, Declaration Under 37 C.F.R. § 1.132 at ¶ 3, May 10, 2016; Ex. 1004 at 208, File History of the ’469 Application, Amendment and Response, May 4, 2015; *see also* Ex. 1001, ’738 patent at 3:28-30 (“It is also known that micro-

bubbles attached to the hydrophobic particle surfaces also tend to bridge the particles together.”); Ex. 1001, ’738 patent at 3:34-40 (“Unlike in conventional approaches, where attention is focused on making proppants or sand particles sticky through formation of chemical bonds between resins coated on the particle surfaces, in the present invention the attention is focused on making particle aggregations by bridging the particles through strong hydrophobic force or micro-bubbles.”). Thus, Vincent explicitly describes Applicant’s self-described inventive concept.

870. Vincent’s published claims clearly disclose coating particles with a hydrophobic material. *See, e.g., id.* at claims 1-6, 9-10, 15-17. Vincent also discloses at least one of the source organo-silicon compounds (i.e., polydimethyl siloxane) as listed in the ’738 patent as a specific example of a hydrophobic rendering compound. Compare Ex. 1007, Vincent at claim 6 with Ex. 1001, ’738 patent at 8:47-50. Vincent therefore also discloses this limitation of claim 32.

871. Weaver discloses “[i]n certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, *by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic* or hydrophilic in nature.” Ex. 1008, Weaver at [0015].

872. Weaver also teaches that “[i]n some embodiments, the surface-treating reagent may comprise other compounds that are capable of *hindering or preventing the water-wetting of a mineral surface*. Examples of suitable compounds include, but are not limited to, . . . *organosilanes* . . . Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.” *Id.* at [0049].

873. Therefore, both Vincent and Weaver teach particulates coated with a chemical compound for rendering the particulates hydrophobic—and specifically that the chemical compound is a silane. This opinion is supported by the USPTO, because in the prosecution of the ’469 application, the Examiner rejected claim 13 (which contained a limitation similar to limitation 1C) as anticipated by Weaver because each taught particles coated in water-repellent compounds. *See* Ex. 1004 at 184, File History of the ’469 Application, Non-Final Office Action, Jan. 2, 2015 (stating that “Weaver further teaches the surface-treating reagent may comprise compounds that are capable of hindering or preventing the water-wetting of a mineral surface.”). I agree with the Examiner that Weaver teaches this limitation. Moreover, it is my opinion that Vincent additionally teaches this limitation by disclosing polydimethylsiloxanes, an organosiloxane, which is disclosed as a hydrophobizing agent in the ’738 patent. *See* Ex. 1001, ’738 patent at 4:62-67. Thus, the combination of Vincent in view of Weaver teaches limitation 1C.

e. **Limitation 32D: “oil”**

874. Claim 32 further recites that the composition comprises “an oil.” Ex. 1001 at 12:8. As stated in ¶¶ 395-400 and 484-489 (and incorporated by reference herein), both Vincent and Weaver teach this limitation.

875. Vincent discloses that the benefits taught by his invention “can be achieved by a variety of techniques, including coating the proppants with a hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes, polytetrafluoroethylene (commonly known as Teflon®), *plant oils, such as linseed oil, soybean oil, corn oil, cottonseed oil, vegetable oil* (widely commercially available such as Crisco®), *and canola oil, and hydrocarbons such as kerosene, diesel, and crude oil, petroleum distillates such as hydrocarbon liquids.*” Ex. 1007, Vincent at [0009].

876. Vincent also discloses proppants coated with a hydrophobic material, “wherein the hydrophobic material *comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, [and] hydrocarbons.*” *Id.* at claim 2; *see also id.* at claims 10 and 21. Thus Vincent teaches the use of two hydrophobic rendering compounds. One, for example, polydimethyl siloxane, satisfies limitation 32C (compound for rendering the surface of the particulates hydrophobic); the other, for example, plant oil or hydrocarbons satisfies limitation 32D (oil).

877. One of ordinary skill in the art would recognize, and the '738 patent confirms, that “hydrocarbon liquid” or “hydrocarbon” is another term for oil. See Ex. 1001, '738 patent at 11:31-33 (“20. The composition of claim 1, wherein the oil is selected from the group consisting of a **hydrocarbon oil**, a silicon oil, kerosene oil, a frac oil and combinations thereof.”).

878. Moreover, Vincent discloses that the fracture fluid in which the particulates are suspended “may be an oil base, water base, acid, **emulsion**, foam, or any other fluid.” Ex. 1007, Vincent at [0087]. A person of ordinary skill in the art would understand that an emulsion can be comprised of oil and water. See *e.g.*, Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S. Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production).

879. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. . . . ***Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils***, esters, and the like. Any

organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.” Ex. 1008, Weaver at [0058].

880. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . **Suitable emulsions** can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid **and a hydrocarbon.**” *Id.* at [0059]. One of ordinary skill in the art would recognize that the term “hydrocarbon” includes “hydrocarbon oil.” Weaver thus teaches the limitation of oil included in the composition.

881. In addition to inclusion of oils in the treatment fluids disclosed by Weaver, Weaver also discloses the addition of oils to the surface-treating reagent (i.e., organosilanes). *See* Ex. 1008, Weaver at [0054].

882. Additionally, Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a **liquid carrier comprising one or more liquids selected from the group consisting of water and oil**, and a solid proppant comprising one or more materials selected from the group consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that



“the liquid component of said foam comprises *at least* one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Thus, it would have been obvious to one of ordinary skill in the art to substitute the oil and water liquid carrier taught by Plummer for Vincent’s fracturing fluid. Moreover, Plummer discloses that “[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling . . . Other friction reducing materials may be added to the fracturing fluid.” *Id.* at 4:28-33.

883. As stated, it would have been obvious to a person of ordinary skill in the art to include Plummer’s oil into the particulate slurry taught by Vincent and Weaver. The motivation to do so is taught by Plummer: using oil as a lubricant friction-reducer or to control clay swelling can improve hydraulic fracturing operations in wells. *See* Ex. 1010, Plummer at 4:28-33.

884. Thus, the simple substitution of Weaver’s treatment fluid (either the aqueous/non-aqueous mixture or the oil and water emulsion) or Plummer’s carrier (oil and water liquid carrier) for Vincent’s fracturing fluid, and/or the addition of Plummer’s oil to act as a lubricant friction-reducer or to control clay swelling would have improved Vincent’s composition in the same way as described in Weaver or Plummer (i.e., improved hydraulic fracturing operations). This improvement would have been reasonably expected to succeed by a person of ordinary skill in the art,

who would have been motivated to make this substitution and/or addition. Limitation 32D is therefore met.

f. **Limitation 32E: “pumping the mixture into the formation”**

885. Claim 32 further recites “pumping the mixture into the formation.” Ex. 1001, ’738 patent at 12:8-9. As stated in ¶¶ 401-405 and 490-493 (and incorporated by reference herein), both Vincent and Weaver teach pumping the mixture into the formation.

886. Vincent teaches “[a] method of fracturing a subterranean formation, comprising: . . . *injecting into the fracture a fluid containing a plurality of essentially spherical particles*, wherein at least some of the particles are modified to alter the surface characteristics thereof, wherein the particles are modified by coating the particles with a hydrophobic material.” Ex. 1007, Vincent at claim 9.

887. Weaver teaches “[a] method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified mineral surface; and *introducing the treatment fluid into the subterranean formation.*” Ex. 1008, Weaver at claim 23; *see also id.* at claim 11.

888. Thus, both Vincent and Weaver teach pumping the mixture into the formation. Limitation 32E would have therefore been obvious over Vincent in view of Weaver.

889. Therefore, in my opinion, challenged independent claim 32 of the '738 patent would have been obvious over Vincent in view of Weaver or Plummer.

### 3. Independent Claim 51 is Obvious

890. As discussed in Section X.C.2 and Section X.D.2 above (and incorporated by reference herein), in addition to the preamble of claim 51, claim 51 has four limitations: 51A (contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with water and an oil to form a mixture), 51D (pumping the mixture into the formation). Vincent and Weaver each discloses all of those limitations and the preamble. See ¶¶ 406-425; 494-517. The combination of Vincent in view of Weaver or Plummer further discloses each and every limitation of the challenged claim 51 of the '738 patent.

#### a. Preamble for Claim 51: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”

891. The preamble of claim 51 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 13:28-29. As stated in ¶¶ 409-412 and 497-501 (and incorporated by reference herein), Vincent and Weaver meet this preamble.

892. The preamble of claim 51 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 13:28-29.

893. Vincent discloses “concepts and techniques to treat fracturing sand and/or proppant to: . . . alter or modify proppant wettability, to control the relative permeability to flow of the fluids which may be encountered in the reservoir . . . [and] ‘lubricate’ the proppant to allow more efficient proppant arrangement when the fracture closes, effectively increasing packing efficiency.” Ex. 1007, Vincent at [0012], [0014], [0015]. Vincent further discloses that this “‘lubrication’ concept reduces proppant friction, allowing superior proppant redistribution during fracture closing. This redistribution allows more efficient packing of proppant, thereby increasing grain-to-grain contact and effectively increasing proppant pack strength.” *Id.* at [0028].

894. One of ordinary skill in the art would understand that controlling the relative permeability to flow of the fluids would reduce the viscous drag on the proppant particles, which would help control proppant flowback. One of ordinary skill in the art would also understand that increasing grain-to-grain contact to increase proppant pack strength is another method of controlling proppant flowback because more proppant particles would remain in the fracture when it closes, thereby reducing the amount of proppant that would flowback.

895. Moreover, one of ordinary skill in the art would generally understand that to reap the benefits of utilizing a proppant, the proppant would need to remain in the formation fracture—i.e. not be carried out of the fractures and into the wellbore along with produced fluids being flowed back out of the well. Thus, a person of ordinary skill in the art would understand the disclosure of Vincent to be a particulate coated with a hydrophobic material that is better suited than a non-coated particulate to remain in the fracture and not be subject to flowback.

896. Weaver discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, ***frac-packing operations***, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. One of ordinary skill in the art would recognize that frac-packing operations are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize that Weaver’s invention is designed to control proppant flowback.

897. Weaver also teaches that “[o]ne problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, ***proppant particulates***, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation. . . . [A]

technique used to prevent flow-back problems, commonly referred to as ‘gravel packing,’ involves the placement of a gravel screen in the subterranean formation, which acts as a barrier that prevents particulates from flowing into the well bore.” *Id.* at [0004]. Weaver proceeds that “one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, . . . preventing the embedment of particulates into the surfaces of a subterranean formation.” *Id.* at [0014].

898. One of ordinary skill in the art would understand that Weaver’s teaching of enhancing conductivity within a subterranean formation to maximize the value of a fracturing treatment and to prevent the embedment of particulates into surfaces of the subterranean formation to be describing operations to reduce particulate flowback. Moreover, Weaver discloses that his invention can be used in frac-packing operations, which is a method of controlling proppant flowback. *See e.g.*, Ex. 1008, Weaver at [0004].

899. Additionally, because Vincent and Weaver disclose the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the ’738 patent. *See, e.g.*, Ex. 1001, ’738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil

increases hydrophobic aggregation, which in turn controls proppant flowback). Thus, Vincent and Weaver disclose this preamble.

**b. Limitation 51A: “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic”**

900. Claim 51 recites “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic.” Ex. 1001 at 13:29-31. As stated in ¶¶ 413-415 and 502-504 (and incorporated by reference herein), Vincent and Weaver meet this limitation. Vincent discloses “pretreating proppant prior to the fracturing treatment.” Ex. 1007, Vincent at [0024]. Vincent discloses a variety of techniques for coating the proppant, including spraying, dipping, or soaking the proppant in a liquid solution of the hydrophobic material. *Id.* at [0009], [0036], [0049]. For example, Vincent discloses that the particles are coated by “*mixing the proppant and the coating in a beaker* for approximately 30 minutes, then drying it for approximately 15 to 18 hours in an oven.” *Id.* at [0036]. One example disclosed in Vincent states that “[t]he coating materials were added as follows. Polymethylhydrogen siloxane was added as either a 2 or 5 weight percent *emulsion of siloxane in water*, polydimethyl siloxane was added as a 5 weight percent *emulsion of siloxane in water* and *Stoddard Solvent* was added without water dilution. All samples were dried at 113° C. for approximately 15 to 18 hours.” *Id.* at [0037].

901. Weaver teaches that one “suitable method[] for allowing the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate includes . . . solvent extraction methods.” *Id.* at [0057]. In such an embodiment, “[t]he solution comprising the surface treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced to a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface.” *Id.* As discussed, Vincent discloses examples of organo-silicon compounds that are the same as those disclosed in the ’738 patent, including polydimethyl siloxane. Moreover, Weaver discloses the use of organosilanes, which the ’738 patent identifies as a chemical compound for rendering the surface of the particulates hydrophobic.

902. Thus Vincent and Weaver clearly describe contacting the particulates with an organo-silicon compound, which, as stated above, is an example of “a chemical compound to render the surface of the particulates hydrophobic” as required by claim 51.

c. **Limitation 51B: “separating the particulate from the medium”**



903. Claim 51 recites that the now-coated particulates are “separate[ed] . . . from the medium.” Ex. 1001 at 13:32. As stated in ¶¶ 416-419 and 505-508 (and incorporated by reference herein), Vincent and Weaver meet this limitation.

904. Vincent discloses that the particles are coated by “mixing the proppant and the coating in a beaker for approximately 30 minutes, *then drying it* for approximately 15 to 18 hours in an oven.” *Id.* at [0036]. One example disclosed in Vincent states that “[t]he coating materials were added as follows. Polymethylhydrogen siloxane was added as either a 2 or 5 weight percent emulsion of siloxane in water, polydimethyl siloxane was added as a 5 weight percent emulsion of siloxane in water and Stoddard Solvent was added without water dilution. *All samples were dried* at 113° C. for approximately 15 to 18 hours.” *Id.* at [0037]. As stated, a person of ordinary skill in the art would understand that drying is one method of separating the particulates from the siloxane and water solution. Thus, Vincent discloses that the now-coated particulates are separated from the solvent solution.

905. Weaver teaches that one “suitable method[] for allowing the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate includes . . . solvent extraction methods.” *Id.* at [0057]. In such an embodiment, “[t]he solution comprising the surface treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the

particulates and stirring (or mulling) until the mixture is uniform. *The treated particulates then may be introduced to a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface.*” *Id.* As discussed, one of ordinary skill in the art would understand Weaver’s disclosure of “extract[ing] the solvent from the treated particulates” to be a method of separating the particulate from the solvent containing the organo-silicon coating. Thus, Weaver discloses that the now-coated particulates are separated from the solvent solution.

906. In view of the foregoing excerpts from the disclosure of Vincent and Weaver, one of ordinary skill in the art would have understood Vincent and Weaver to disclose this limitation.

**d. Limitation 51C: “blending the hydrophobic particulate with water and an oil to form a mixture”**

907. Claim 51 recites that the hydrophobic coated particulate are blended “with water and oil to form a mixture.” Ex. 1001 at 13:32-34. As stated in ¶¶ 420-422 and 509-513 (and incorporated by reference herein), the combination of Vincent in view of Weaver or Plummer meet this limitation.

908. Vincent discloses that the fracture fluid in which the particulates are suspended “may be an oil base, water base, acid, *emulsion*, foam, or any other fluid.” Ex. 1007, Vincent at [0087]. A person of ordinary skill in the art would understand

that an emulsion can be comprised of oil and water. *See e.g.*, Ex. 1017, U.S. Patent 3,977,472 at Abstract (disclosing a method of hydraulic fracturing wherein a viscous oil-in-water emulsion is injected into a formation); Ex. 1018, U.S. Patent 5,633,220 at Abstract (using a water-in-oil emulsion as a fracturing fluid in the hydraulic fracturing of a subterranean formation); Ex. 1019, U.S. Patent 2,999,063 at 1:10-12 (teaching water-in-oil emulsions for use in well drilling and for use in fracturing producing horizons to promote oil production). Additionally, Vincent discloses the use of multiple hydrophobic rendering compounds: “wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, [and] hydrocarbons.” *Id.*, claim 2. Thus, for example, polydimethyl siloxane, would satisfy a compound for rendering the surface of the particulates hydrophobic, and a plant oil or hydrocarbons would satisfy the oil requirement.

909. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, ***or a mixture thereof***. Where the base fluid is aqueous, it may comprise fresh water [or] salt water . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in

which a water solution of salts *can be emulsified also may be suitable for use as a base fluid* in the methods of the present invention.” Ex. 1008, Weaver at [0058].

910. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . *Suitable emulsions* can be comprised of two immiscible liquids such as *an aqueous liquid* or gelled liquid *and a hydrocarbon.*” *Id.* at [0059].

911. One of ordinary skill in the art would recognize, and the ’738 patent confirms, that oil is a type of “hydrocarbon.” See Ex. 1001, ’738 patent at 11:31-33 (“20. The composition of claim 1, wherein the oil is selected from the group consisting of a *hydrocarbon oil*, a silicon oil, kerosene oil, a frac oil and combinations thereof.”). Weaver thus teaches the limitation of oil included in the composition in the form of a treatment fluid including a mixture of oil and water, as well as an oil and water emulsion.

912. Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising *a liquid carrier comprising one or more liquids selected from the group consisting of water and oil*, and a solid proppant comprising one or more materials selected from the group

consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that “the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Thus, it would have been obvious to one of ordinary skill in the art to substitute the oil and water liquid carrier taught by Plummer for Vincent’s fracturing fluid. Moreover, Plummer discloses that “[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling . . . Other friction reducing materials may be added to the fracturing fluid.” *Id.* at 4:28-33.

913. As stated, it would have been obvious to a person of ordinary skill in the art to include Plummer’s oil into the particulate slurry taught by Vincent and Weaver. The motivation to do so is taught by Plummer: using oil as a lubricant friction-reducer or to reduce clay swelling can improve hydraulic fracturing operations in wells. *See* Ex. 1010, Plummer at 4:28-33.

914. Thus, the simple substitution of Weaver’s treatment fluid (either the aqueous/non-aqueous mixture or the oil and water emulsion) or Plummer’s carrier (water and oil liquid carrier) for Vincent’s fracturing fluid, and/or the addition of Plummer’s oil to act as a lubricant friction-reducer or to reduce clay swelling would have improved Vincent’s composition in the same way as described in Weaver or Plummer (i.e., improved hydraulic fracturing operations). This improvement would have been reasonably expected to succeed by a person of ordinary skill in the art,

who would have been motivated to make this substitution and/or addition. Limitation 51C is therefore met.

e. **Limitation 51D: “pumping the mixture into the formation”**

915. Claim 51 further recites “pumping the mixture into the formation.” Ex. 1001 at 13:34.

916. Vincent teaches “[a] method of fracturing a subterranean formation, comprising: . . . *injecting into the fracture a fluid containing a plurality of essentially spherical particles*, wherein at least some of the particles are modified to alter the surface characteristics thereof, wherein the particles are modified by coating the particles with a hydrophobic material.” Ex. 1007, Vincent at claim 9.

917. Weaver teaches “[a] method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified mineral surface; and *introducing the treatment fluid into the subterranean formation.*” Ex. 1008, Weaver at claim 23; *see also id.* at claim 11.

918. Therefore, Vincent and Weaver disclose limitation 51D.

919. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 51A (contacting the particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the

hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), and the preamble of claim 51, is taught by Vincent in view of Weaver or Plummer. Therefore, independent claim 51 is obvious over Vincent in view of Weaver or Plummer.

#### **4. Dependent Claims 21, 22, 40, 41, and 57 Are Obvious**

920. As addressed above (*see* Section XI.C.1, XI.C.2, XI.C.3) and incorporated herein by reference, all of the limitations of claims 1, 32, and 57, including 1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulates from the medium), 51C (blending the hydrophobic particulate with water and oil to form a mixture); 51D (pumping the mixture into the formation), and the preambles of claims 1, 32, and 51, are obvious over Vincent in view of Weaver or Plummer.

921. The only additional limitation in claims 21, 22, 40, 41, and 51 relate to the type of particulate. Dependent claims 21, and 40 recite a near-identical limitation, but depend from different claims. Claim 21 depends from independent claim 1, whereas claim 40 depends from independent claim 32. Similarly, dependent claims 22, 41, and 57 recite the same limitation, but depend from different claims.

Claim 22 depends from independent claim 1, whereas claim 41 depends from claim 32, and claim 57 depends from claim 51. Dependent claims 21 and 40 require the particulate be one of an enumerated list of particulates, including sand. *See* Ex. 1001, '738 patent at 11:34-37; 12:63-65 (“wherein the particulates are selected from the group consisting of sand . . .”). Dependent claims 22 and 41 require the particulate to be sands. *Id.* at 11:38-39; 12:66-67; 14:24-25 (“wherein the particulates are sands”). Therefore, a prior art reference that discloses the additional limitation of “sand” as the particulate anticipates claims 21, 22, 40, 41, and 57.

922. Vincent teaches the additional limitation of claims 21, 22, 40, 41, and 57: “[t]he present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are ***natural sands***.” Ex. 1007, Vincent at [0007]. Vincent also teaches that “[e]mbodiments of the present invention relate to modifying the surface properties of ***natural sand***, resin coated sand and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability . . . Sands, resin coated sands or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.” *Id.* at [0008].



923. Additionally, Vincent teaches that “particles as hereinbefore describe are placed in the fracture by injecting into the fracture a fluid or ‘slurry’ into which the particles have previously been introduced and suspended.” *Id.* at [0087]. Thus, the particulates are mixed with the carrier fluid before being pumped into the fracture.

924. Weaver teaches “[t]he particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, **sand**.” Ex. 1008, Weaver at [0018].

925. Weaver also teaches that “the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.” *Id.* at [0057]. Thus, the sand particulates are mixed with the treatment fluid before being pumped down the formation. Thus, Vincent and Weaver teach the additional limitation of claims 21, 22, 40, 41, and 57. Therefore, it is my opinion that challenged dependent claims 21,

22, 40, 41, and 57 of the '738 patent would have been obvious over Vincent in view of Weaver or Plummer.

926. Based on the foregoing analysis of Sections XI.C.1-4, all of the elements of claims 1, 21, 22, 32, 40, 41, 51, and 57 [1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulates from the medium), 51C (blending the hydrophobic particulate with water and oil to form a mixture); 51D (pumping the mixture into the formation), 21A, 22A, 40A, 41A, and 57A (sand), and the preambles of claims 1, 32, and 51] are disclosed by the combination of Vincent in view of Weaver or Plummer. Therefore, it is my opinion that the combination of Vincent in view of Weaver or Plummer renders challenged claims 1, 21, 22, 32, 40, 41, 51 and 57 of the '738 patent obvious.

**D. Claims 1, 21, 22, 32, 40, 41, 51, and 57 Are Obvious over Needham in view of Weaver or Plummer**

927. As discussed in Sections X.E and X.D above (and incorporated by reference herein), both Needham and Weaver anticipate claims 1, 21, and 22 of the '738 patent because each reference discloses each and every element of the claims. As discussed in Section X.D.2-4 above (and incorporated by reference herein),

Weaver also anticipates claims 32, 40, 41, 51 and 57 of the '738 patent. Furthermore, the disclosure of Needham in view of Weaver or Plummer also renders claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent obvious. *See generally*, Ex. 1002K, '738 Patent v. Needham in view of Weaver or Plummer Claim Chart.

928. One of ordinary skill in the art at the time of the invention would have been motivated to combine Needham and Weaver because both are directed to methods for modifying sand particulates with a water-repellent compound (specifically organosilanes) for use in hydraulic fracturing operations. Further, one of ordinary skill in the art would be motivated to substitute the aqueous carrier fluid of Needham with the oil and water emulsion treatment fluid of Weaver to arrive at the invention of claim 32 of the '738 patent, since both were well known in the art and there was a high likelihood of success. Additionally, and in the alternative, a person of ordinary skill in the art would be motivated to substitute the aqueous carrier fluid of Needham with the oil and water liquid carrier disclosed in Plummer for the same reason. Moreover, a person of ordinary skill in the art would be motivated to add oil to the slurry as a lubricant friction-reducer or to reduce clay swelling, as taught by Plummer (Ex. 1010 at 4:28-33) to improve the compositions and methods of Needham in hydraulic fracturing operations.

# **1. Independent Claim 1 Is Obvious**

929. As discussed in Section X.E.1 and Section X.D.1 above (and incorporated by reference herein), in addition to the preamble of claim 1, claim 1 has four limitations: 1A (water), 1B (particulates), 1C (a chemical compound for rendering the surface of the particulate hydrophobic), and 1D (oil). Needham and Weaver each discloses all of those limitations and the preamble. See ¶¶ 530-550; 438-461. The combination of Needham in view of Weaver or Plummer further discloses each and every limitation of challenged claim 1 of the '738 patent.

**a. Preamble for Claim 1: “An aqueous slurry composition comprising”**

930. The preamble of claim 1 of the '738 patent states, “An aqueous slurry composition comprising.” Ex. 1001, '738 patent at 9:46.

931. As stated in ¶¶ 533 and 441 (and incorporated by reference herein), Needham and Weaver meet this preamble. Needham teaches that a “[c]arrier fluid, preferably an aqueous fluid such as salt water or a hydrocarbon liquid such as diesel fuel, having the proppant coated with organo-silicon compound suspended herein is then pumped into the fracture.” Ex. 1009, Needham at 3:19-23. The combination of the aqueous carrier fluid with the coated proppant yields the claimed slurry composition.

932. Weaver teaches that “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid).

These treatment fluids generally comprise a base fluid, which may be aqueous.” Ex. 1008, Weaver at [0058].

933. Thus, both Needham and Weaver, and accordingly Needham in view of Weaver, meet this preamble.

**b. Limitation 1A: “water”**

934. Claim 1 recites that the composition comprises “water.” Ex. 1001 at 9:47. As stated in ¶¶ 534-35 and 442-444 (and incorporated by reference herein), both Needham and Weaver teach the use of water.

935. As stated by the Examiner in the prosecution of the ’469 application, Needham teaches “[c]arrier fluid, *preferably an aqueous fluid such as salt water* having the proppant coated with organo-silicon compound suspended therein is then pumped into the fracture.” Ex. 1004 at 182, File History of the ’469 Application, Non-Final Office Action, January 2, 2015 (citing Needham [Ex. 1009] at 3:19-23). I agree with the Examiner that Needham discloses a slurry composition that includes water. It is also clear that the water is mixed with the coated proppant because the proppant is suspended in the carrier fluid and then pumped into the fracture.

936. A person of ordinary skill in the art would understand that Needham discloses that the water is mixed with the proppant, since it is pumped into the fracture. Ex. 1009, Needham at 4:1-6.

937. Weaver discloses “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise **fresh water, salt water** (e.g., water containing one or more salts dissolved therein), **brine** (e.g., saturated salt water), **or seawater.**” Ex. 1008, Weaver at [0058].

938. Moreover, Weaver discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including **aqueous gels**, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable **aqueous gels are generally comprised of water** and one or more gelling agents. **Suitable emulsions** can be comprised of two immiscible liquids such as an **aqueous liquid** or gelled liquid and a hydrocarbon.” *Id.* at [0059].

939. Thus, both Needham and Weaver teach that water can be included in the slurry as the carrier/treatment fluid of the proppant. Limitation 1A would have therefore been obvious over Needham in view of Weaver.

**c.     Limitation 1B: “particulates”**

940. Claim 1 recites that the composition comprises “particulates.” Ex. 1001 at 9:48. As stated in ¶¶ 536-538 and 445-447 (and incorporated by reference herein), both Needham and Dawson teach the use of particulates.

941. The Examiner stated, while rejecting claim 13 of the '469 application as anticipated by Needham, that “Needham further teaches propping agents used in hydraulic fracturing normally range in particle size from about 8 to about 60 mesh.” Ex. 1004 at 183, File History of the '469 Application, Non-Final Office Action, January 2, 2015 (citing Needham [Ex. 1009] at 3:5-12). I agree with the Examiner that Needham discloses particulates as described in this limitation. Needham further teaches that an “object of this invention is to coat particulate solids useful as propping agents.” Ex. 1009, Needham at 1:50-51.

942. Weaver provides “methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates . . .” Ex. 1008, Weaver at Abstract. Weaver also teaches that “particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface.” *Id.* at [0018].

943. Thus, both Needham and Weaver teach the use of particulates in the slurry. Limitation 1B would have therefore been obvious over Needham in view of Weaver.

d. **Limitation 1C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

944. Claim 1 of the '738 patent recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.”

Ex. 1001 at 9:49-50. As stated in ¶¶ 539-544 and 448-452 (and incorporated by reference herein), both Needham and Weaver teach this limitation.

945. The specification of the '738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The '738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51.

946. The '738 patent also recites: “[e]xamples of organosilanes include:  $\text{CH}_3\text{SiCl}_3$ ,  $\text{CH}_3\text{CH}_2\text{SiCl}_3$ ,  $(\text{CH}_3)_2\text{SiCl}_2$ ,  $(\text{CH}_3\text{CH}_2)_2\text{SiCl}_2$ ,  $(\text{C}_6\text{H}_5)_2\text{SiCl}_2$ ,  $(\text{C}_6\text{H}_5)\text{SiCl}_3$ ,  **$(\text{CH}_3)_3\text{SiCl}$** ,  $\text{CH}_3\text{HSiCl}_2 \dots$ ” (*id.* at 5:66-6:2).

947. Needham teaches that “an object of this invention is to provide improved propping agents for use in hydraulic fracturing of subterranean formations. Another object of this invention is to provide ***particulate material coated with a water repellent*** useful in hydraulic fracturing of subterranean formations. A further object of this invention is to coat particulate solids useful as propping agents to



increase the strength of the propping agent in the presence of water.” Ex. 1009, Needham 1:44-52.

948. Needham further teaches that the propping agent “compris[es] an inert solid particle coated with a ***water-repellent organo-silicon material*** is displaced down a well and into a fracture in a subterranean hydrocarbon-bearing formation. In accordance with a specific embodiment, sand coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.” *Id.* at 1:56-67.

949. As stated by the Examiner in the rejection of claim 13 of the ’469 application as anticipated by Needham, Needham teaches that “[t]he water-repellent material employed in the method of the present invention to coat inert particulate materials for use as propping agents is an oil-soluble organo-silicon compound. Representative organo-silicon compounds that can be used include polyalkylsiloxanes such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additionally, polyalkylarylsiloxanes such as polymethylphenylsiloxane are also usable as a coating material of the invention. ***Chlorosilanes such as chlorotrimethylsilane*** and other silyl donors can also be used, as well as various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes, and the like, as

the water-repellent coating of the propping agent.” Ex. 1004 at 182, File History of the ’469 Application, Non-Final Office Action, January 2, 2015 (citing Needham [Ex. 1009] at 2:25-41). I agree with the Examiner that Needham discloses this limitation because chlorotrimethylsilane ((CH<sub>3</sub>)<sub>3</sub>SiCl) is one of the organo-silicon compounds identified in the ’738 specification as a hydrophobic rendering compound.

950. Weaver discloses “[i]n certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, *by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic* or hydrophilic in nature.” Ex. 1008, Weaver at [0015].

951. Weaver also teaches that “[i]n some embodiments, the surface-treating reagent may comprise other compounds that are capable of *hindering or preventing the water-wetting of a mineral surface*. Examples of suitable compounds include, but are not limited to, . . . *organosilanes* . . . Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.” *Id.* at [0049].

952. Therefore, both Needham and Weaver teach particulates coated with a chemical compound for rendering the particulates hydrophobic—and specifically that the chemical compound is an organosilane. This opinion is supported by the USPTO, because in the prosecution of the '469 application, the Examiner rejected claim 13 (which contained a limitation similar to limitation 1C) as anticipated by both Needham and Weaver because each taught particles coated in water-repellent compounds. *See* Ex. 1004 at 182-84, File History of the '469 Application, Non-Final Office Action, Jan. 2, 2015 (stating that “Needham teaches a method of propping a fracture in subterranean formations, comprising placing in the fractures particulate solids such as sand coated with a water-repellent coating composition” and that “Weaver further teaches the surface-treating reagent may comprise compounds that are capable of hindering or preventing the water-wetting of a mineral surface.”). I agree with the Examiner that both Needham and Weaver teach this limitation. Thus, the combination of Needham in view of Weaver teaches limitation 1C.

**e. Limitation 1D: “oil”**

953. Claim 1 further recites that the composition comprises “an oil.” Ex. 1001 at 9:50. As stated in ¶¶ 545-550 and 453-461 (and incorporated by reference herein), both Needham and Weaver teach this limitation.

954. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, **or a mixture thereof. . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils**, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.” Ex. 1008, Weaver at [0058].

955. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . **Suitable emulsions** can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid **and a hydrocarbon.**” *Id.* at [0059]. One of ordinary skill in the art would recognize that the term “hydrocarbon” includes “hydrocarbon oil.” Weaver thus teaches the limitation of oil included in the composition.

956. In addition to inclusion of oils in the treatment fluids disclosed by Weaver, Weaver also discloses the addition of oils to the surface-treating reagent (i.e., organosilanes). *See id.*, Weaver at [0054].

957. Moreover, the ’738 specification discloses that the oil of the composition may be formation oil. Ex. 1001 at 4:11-15 (“Alternatively the slurry

containing a hydrophobizing agent can be pumped into the fracture forming the proppant pack, which can be further consolidated by oil or condensate contained in the formation.”). Thus, the oil limitation in claim 1 can be satisfied by the presence of formation oil. This is consistent with the prosecution history of the ’738 patent. See Section VII.C, *supra*.

958. Needham teaches “[i]n a method of fracturing an underground zone or formation in **oil and gas wells**, the steps of (a) injecting into a well a fracturing fluid to form a fracture in said formation, (b) injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound prepared by dissolving the organo-silicon in a solvent therefor, admixing the resultant solution with particulate material, and thereafter evaporating the solvent to form a silicon-coated particulate material, and (c) closing the well and maintaining the injection pressure on the formation until the fracture approaches the normal formation pressure.” Ex. 1009, Needham at 4:1-15.

959. Needham further discloses that the invention “relates to the treatment of subterranean **hydrocarbon-bearing formations**” (*id.* at 1:4-5) and that in accordance with the invention, “a novel propping agent comprising an inert solid particle coated with a water-repellent organo-silicon material is displaced down a well and into a fracture in a subterranean hydrocarbon-bearing formation” (*id.* at 1:56-60).

960. Weaver further discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. Weaver also discloses a “method of treating a **subterranean formation** comprising: providing a treatment fluid that comprises: a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; . . . and introducing the treatment fluid into the subterranean formation.” *Id.* at claim 11.

961. One of ordinary skill in the art would understand that hydrocarbon-bearing formations and treated subterranean formations contain oil, as is evidenced by claim 1 of Needham. *See* Ex. 1009, Needham at 4:1-2 (“In a method of fracturing an underground zone or formation in **oil and gas wells**”); Ex. 1001, ’738 patent at 1:26-31 (stating that hydraulic fracturing operations are used to “enhance oil and gas production” and to “increase oil and gas production”). Further, one of ordinary skill in the art would understand that injecting the composition containing water and silicon-coated particulate into a subterranean formation to be treated or a hydrocarbon-bearing formation would necessarily mean that oil is present. Because

the formation contains oil, the compositions of Needham and Weaver must therefore comprise oil.

962. Additionally, Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising *a liquid carrier comprising one or more liquids selected from the group consisting of water and oil*, and a solid proppant comprising one or more materials selected from the group consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that “the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Thus, it would have been obvious to one of ordinary skill in the art to substitute the oil and water liquid carrier taught by Plummer for Needham’s carrier fluid.

963. Moreover, Plummer discloses that “[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling . . . Other friction reducing materials may be added to the fracturing fluid.” *Id.* at 4:28-33.

964. As stated, it would have been obvious to a person of ordinary skill in the art to include Plummer’s oil into the particulate slurry taught by Needham and Weaver. The motivation to do so is taught by Plummer: using oil as a lubricant

friction-reducer or to control clay swelling can improve hydraulic fracturing operations in wells. See Ex. 1010, Plummer at 4:28-33.

965. Thus, the simple substitution of Weaver's treatment fluid (either the aqueous/non-aqueous mixture or the oil and water emulsion) or Plummer's carrier (oil and water liquid carrier) for Needham's carrier fluid, and/or the addition of Plummer's oil to act as a lubricant friction-reducer or as a clay stabilizer would have improved Needham's composition in the same way as described in Weaver or Plummer (i.e., improved hydraulic fracturing operations). This improvement would have been reasonably expected to succeed by a person of ordinary skill in the art, who would have been motivated to make this substitution and/or addition. Limitation 1D is therefore met.

966. Therefore, it is my opinion that challenged independent claim 1 of the '738 patent would have been obvious over Needham in view of Weaver or Plummer.

## **2. Independent Claim 32 Is Obvious**

967. As discussed in Section X.D.2 above (and incorporated by reference herein), in addition to the preamble of claim 32, claim 32 has five limitations: 32A (water), 32B (particulates), 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 32D (oil), and 32E (pumping the mixture into the formation). Weaver discloses all of those limitations and the preamble. See ¶¶ 462-



493. The combination of Needham in view of Weaver or Plummer further discloses each and every limitation of the challenged claim 32 of the '738 patent.

a. **Preamble for Claim 32: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”**

968. The preamble of claim 32 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 12:5-6.

969. As stated in ¶¶ 465-471 (and incorporated by reference herein), Weaver meets this preamble. Weaver discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, *frac-packing operations*, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. One of ordinary skill in the art would recognize that frac-packing operations are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize that Weaver’s invention is designed to control proppant flowback.

970. Weaver also teaches that “[o]ne problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates . . . to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the

interior of the formation.” *Id.* at [0004]. Weaver proceeds that “one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, . . . preventing the embedment of particulates into the surfaces of a subterranean formation.” *Id.* at [0014].

971. One of ordinary skill in the art would understand that Weaver’s teaching of enhancing conductivity within a subterranean formation to maximize the value of a fracturing treatment and to prevent the “clogging” of the particulates in the subterranean formation to be describing operations to reduce particulate flowback.

972. Additionally, because the combination of Needham in view of Weaver and Plummer discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent with the specification of the ’738 patent. *See, e.g.*, Ex. 1001, ’738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback).

973. Thus, Weaver, and accordingly Needham in view of Weaver, meets this preamble.

**b. Limitation 32A: “mixing water”**

974. Claim 32 recites that the composition comprises “mixing water.” Ex. 1001, ’738 patent at 12:6-7. Needham and Weaver meet this limitation.

975. As stated by the Examiner in the prosecution of the ’469 application, Needham teaches “[c]arrier fluid, *preferably an aqueous fluid such as salt water* having the proppant coated with organo-silicon compound suspended therein is then pumped into the fracture.” Ex. 1004 at 182, File History of the ’469 Application, Non-Final Office Action, January 2, 2015 (citing Needham [Ex. 1009] at 3:19-23). I agree with the Examiner that Needham discloses a slurry composition that includes water. It is also clear that the water is mixed with the coated proppant because the proppant is suspended in the carrier fluid and then pumped into the fracture.

976. Moreover, a person of ordinary skill in the art would understand that Needham discloses that the water is mixed with the proppant, since it is pumped into the fracture. Ex. 1009, Needham at 4:1-6.

977. Weaver discloses “[i]n certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise *fresh water, salt water* (e.g., water containing one or more salts dissolved therein), *brine* (e.g., saturated salt water), *or seawater*.” Ex. 1008, Weaver at [0058].

978. Moreover, Weaver discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including **aqueous gels**, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable **aqueous gels are generally comprised of water** and one or more gelling agents. **Suitable emulsions** can be comprised of two immiscible liquids such as an **aqueous liquid** or gelled liquid and a hydrocarbon.” *Id.* at [0059].

979. Weaver describes that the particulates are mixed with the surface-modifying agents “prior to transporting the particulates to a job site, or in a treatment performed ‘on-the-fly.’ The term ‘on-the-fly’ is used herein to mean that one flowing stream comprising particulates is continuously introduced into another flowing stream comprising the surface-treating reagent so that the streams are combined and mixed while continuing to flow as a single stream as part of the ongoing treatment at the job site. Such mixing can also be described as ‘real-time’ mixing.” *Id.* at [0056].

980. Therefore, Needham in view of Weaver clearly describes a composition including “mixing water” as recited in claim 32.

**c. Limitation 32B: “particulates”**

981. Claim 32 recites that the composition comprises “particulates.” Ex. 1001 at 12:7. Needham and Weaver meet this limitation.

982. The Examiner stated, while rejecting claim 13 of the '469 application as anticipated by Needham, that "Needham further teaches propping agents used in hydraulic fracturing normally range in particle size from about 8 to about 60 mesh." Ex. 1004 at 183, File History of the '469 Application, Non-Final Office Action, January 2, 2015 (citing Needham [Ex. 1009] at 3:5-12). I agree with the Examiner that Needham discloses particulates as described in this limitation. Needham also discloses that an "object of this invention is to coat particulate solids useful as propping agents." Ex. 1009, 1:50-51.

983. Moreover, Needham teaches that these particulates are coated with an organo-silicon compound and "dispersed into a carrying fluid" which is then introduced "into a well penetrating a subterranean formation containing fractures." Ex. 1009, Needham at 4:29-42.

984. Weaver provides "methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates . . ." Ex. 1008, Weaver at Abstract. Weaver also teaches that "particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface." *Id.* at [0018]. Weaver describes that the particulates are mixed with the surface-modifying agents "prior to transporting the particulates to a job site, or in a treatment performed 'on-the-fly.'" *Id.* at [0056].

985. Thus, both Needham and Weaver teach the use of particulates mixed in the slurry. Limitation 32B would have therefore been obvious over Needham in view of Weaver.

d. **Limitation 32C: “a chemical compound for rendering the surface of the particulates hydrophobic”**

986. Claim 32 recites that the composition further includes “a chemical compound for rendering the surface of the particulates hydrophobic.” Ex. 1001 at 12:7-8. Needham and Weaver satisfy this limitation.

987. The specification of the '738 patent discloses that “[t]here are various types of hydrophobizing agents for sand, which can be used in the present invention. For example, it is known that many organosilicon compounds including organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds are commonly used to render various surfaces hydrophobic.” Ex. 1001 at 4:62-67. The '738 patent further states that “[o]ther types of chemical compounds, which are not organosilicon compounds, which can be used to render particulate surface hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds including cationic fluoro-organic compounds.” *Id.* at 5:47-51.

988. The '738 patent also recites: “[e]xamples of organosilanes include:  $\text{CH}_3\text{SiCl}_3$ ,  $\text{CH}_3\text{CH}_2\text{SiCl}_3$ ,  $(\text{CH}_3)_2\text{SiCl}_2$ ,  $(\text{CH}_3\text{CH}_2)_2\text{SiCl}_2$ ,  $(\text{C}_6\text{H}_5)_2\text{SiCl}_2$ ,  $(\text{C}_6\text{H}_5)\text{SiCl}_3$ , ***(CH<sub>3</sub>)<sub>3</sub>SiCl***,  $\text{CH}_3\text{HSiCl}_2$  . . .” (*id.* at 5:66-6:2).

989. Needham teaches that “an object of this invention is to provide improved propping agents for use in hydraulic fracturing of subterranean formations. Another object of this invention is to provide ***particulate material coated with a water repellent*** useful in hydraulic fracturing of subterranean formations. A further object of this invention is to coat particulate solids useful as propping agents to increase the strength of the propping agent in the presence of water.” Ex. 1009, Needham 1:44-52.

990. Needham further teaches that the propping agent “compris[es] an inert solid particle coated with a ***water-repellent organo-silicon material*** is displaced down a well and into a fracture in a subterranean hydrocarbon-bearing formation. In accordance with a specific embodiment, sand coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.” *Id.* at 1:56-67.

991. As stated by the Examiner in the rejection of claim 13 of the ’469 application as anticipated by Needham, Needham teaches that “[t]he water-repellent material employed in the method of the present invention to coat inert particulate materials for use as propping agents is an oil-soluble organo-silicon compound. Representative organo-silicon compounds that can be used include

polyalkylsiloxanes such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additionally, polyalkylarylsiloxanes such as polymethylphenylsiloxane are also usable as a coating material of the invention. ***Chlorosilanes such as chlorotrimethylsilane*** and other silyl donors can also be used, as well as various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes, and the like, as the water-repellent coating of the propping agent.” Ex. 1004 at 182, File History of the ’469 Application, Non-Final Office Action, January 2, 2015 (citing Needham [Ex. 1009] at 2:25-41). I agree with the Examiner that Needham discloses this limitation because chlorotrimethylsilane ((CH<sub>3</sub>)<sub>3</sub>SiCl) is one of the organo-silicon compounds identified in the ’738 specification as a hydrophobic rendering compound.

992. Weaver discloses “[i]n certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, ***by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic*** or hydrophilic in nature.” Ex. 1008, Weaver at [0015].

993. Weaver also teaches that “[i]n some embodiments, the surface-treating reagent may comprise other compounds that are capable of ***hindering or preventing***



*the water-wetting of a mineral surface.* Examples of suitable compounds include, but are not limited to, . . . **organosilanes** . . . Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.” *Id.* at [0049].

994. Therefore, both Needham and Weaver teach particulates coated with a chemical compound for rendering the particulates hydrophobic—and specifically that the chemical compound is an organosilane. This opinion is supported by the USPTO, because in the prosecution of the ’469 application, the Examiner rejected claim 13 (which contained a limitation similar to limitation 32C) as anticipated by both Needham and Weaver because each taught particles coated in water-repellent compounds. *See* Ex. 1004 at 182-84, File History of the ’469 Application, Non-Final Office Action, Jan. 2, 2015 (stating that “Needham teaches a method of propping a fracture in subterranean formations, comprising placing in the fractures particulate solids such as sand coated with a water-repellent coating composition” and that “Weaver further teaches the surface-treating reagent may comprise compounds that are capable of hindering or preventing the water-wetting of a mineral surface.”). I agree with the Examiner that both Needham and Weaver teach this limitation. Thus, the combination of Needham in view of Weaver teaches limitation 32C.

e. **Limitation 32D: “oil”**

995. Claim 32 further recites that the composition comprises “an oil.” Ex. 1001, ’738 patent at 12:8. Needham in view of Weaver and Plummer meet this limitation.

996. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. . . . *Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils*, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.” Ex. 1008, Weaver at [0058].

997. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . *Suitable emulsions* can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid *and a hydrocarbon.*” *Id.* at [0059]. One of ordinary skill in the art would recognize that the term “hydrocarbon” includes “hydrocarbon oil.” Weaver thus teaches the limitation of oil included in the composition.

998. In addition to inclusion of oils in the treatment fluids disclosed by Weaver, Weaver also discloses the addition of oils to the surface-treating reagent (i.e., organosilanes). *See* Ex. 1008, Weaver at [0054].

999. Additionally, Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising ***a liquid carrier comprising one or more liquids selected from the group consisting of water and oil***, and a solid proppant comprising one or more materials selected from the group consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that “the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Thus, it would have been obvious to one of ordinary skill in the art to substitute the oil and water liquid carrier taught by Plummer for Needham’s carrier fluid. Moreover, Plummer discloses that “[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling . . . Other friction reducing materials may be added to the fracturing fluid.” *Id.* at 4:28-33.

1000. As stated, it would have been obvious to a person of ordinary skill in the art to include Plummer’s oil into the particulate slurry taught by Needham and Weaver. The motivation to do so is taught by Plummer: using oil as a lubricant

friction-reducer or to control clay swelling can improve hydraulic fracturing operations in wells. See Ex. 1010, Plummer at 4:28-33.

1001. Thus, the simple substitution of Weaver’s aqueous/nonaqueous mixture or emulsion-based treatment fluid, or Plummer’s oil and water liquid carrier for Needham’s aqueous carrier fluid, and/or the addition of Plummer’s oil to act as a lubricant friction-reducer or to reduce clay swelling would have improved Needham’s composition in the same way as described in Weaver or Plummer (i.e., improved hydraulic fracturing operations). This improvement would have been reasonably expected to succeed by a person of ordinary skill in the art, who would have been motivated to make this substitution and/or addition. Limitation 32D is therefore met.

f. **Limitation 32E: “pumping the mixture into the formation”**

1002. Claim 32 further recites “pumping the mixture into the formation.” Ex. 1001, ’738 patent at 12:8-9. Needham and Weaver both teach pumping the mixture into the formation.

1003. Needham teaches “a method of fracturing an underground zone or formation in oil and gas wells, the steps of . . . ***injecting into said formation*** a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound.” Ex. 1009, Needham at 4:1-7. Needham also teaches that “[c]arrier fluid, preferably an aqueous fluid such as salt water or a hydrocarbon

liquid such as diesel fuel, having the proppant coated with organo-silicon compound suspended therein is then ***pumped into the fracture.***” *Id.* at 3:19-23.

1004. Weaver teaches “[a] method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified mineral surface; and ***introducing the treatment fluid into the subterranean formation.***” Ex. 1008, Weaver at claim 23; *see also id.* at claim 11.

1005. Thus, both Needham and Weaver teach pumping the mixture into the formation. Limitation 32E would have therefore been obvious over Needham in view of Weaver.

1006. Therefore, in my opinion, challenged independent claim 32 of the ’738 patent would have been obvious over Needham in view of Weaver or Plummer.

### **3. Independent Claim 51 is Obvious**

1007. As discussed in Section X.E.2 and Section X.D.2 above (and incorporated by reference herein), in addition to the preamble of claim 51, claim 51 has four limitations: 51A (contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with water and an oil to form a mixture), 51D (pumping the mixture into the formation). Weaver discloses all of those limitations and the preamble.

Moreover, Needham in view of Weaver or Plummer discloses each and every limitation of challenged claim 51.

a. **Preamble for Claim 51: “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of”**

1008. The preamble of claim 51 of the '738 patent states, “A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of.” Ex. 1001 at 13:28-29. As stated in ¶¶497-501 (and incorporated by reference herein), Weaver meets this preamble. Weaver discloses that “[t]he methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include . . . fracturing operations, gravel-packing operations, *frac-packing operations*, perforation operations, and the like.” Ex. 1008, Weaver at [0058]. One of ordinary skill in the art would recognize that frac-packing operations are a type of gravel pack that are especially designed to control proppant flowback. Thus, one of ordinary skill in the art would recognize that Weaver’s invention is designed to control proppant flowback.

1009. Weaver also teaches that “[o]ne problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, *proppant particulates*, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog

the conductive channels and/or damage the interior of the formation. . . . [A] technique used to prevent flow-back problems, commonly referred to as ‘gravel packing,’ involves the placement of a gravel screen in the subterranean formation, which acts as a barrier that prevents particulates from flowing into the well bore.” *Id.* at [0004]. Weaver proceeds that “one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, . . . preventing the embedment of particulates into the surfaces of a subterranean formation.” *Id.* at [0014].

1010. One of ordinary skill in the art would understand that Weaver’s teaching of enhancing conductivity within a subterranean formation to maximize the value of a fracturing treatment and to prevent the embedment of particulates into surfaces of the subterranean formation to be describing operations to reduce particulate flowback. Moreover, Weaver discloses that his invention can be used in frac-packing operations, which is a method of controlling proppant flowback. *See e.g.*, Ex. 1008, Weaver at [0004].

1011. Additionally, because the combination of Needham in view of Weaver or Plummer discloses the use of all of the claimed ingredients, including hydrophobic proppant and oil, it necessarily controls proppant flowback, consistent

with the specification of the '738 patent. *See, e.g.*, Ex. 1001, '738 patent at Abstract, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which in turn controls proppant flowback).

1012. Moreover, one of ordinary skill in the art would generally understand that to reap the benefits of utilizing a proppant, the proppant would need to remain in the formation fracture—i.e. not be carried out of the fractures and into the wellbore along with produced fluids being flowed back out of the well. Thus, a person of ordinary skill in the art would understand the disclosures of Weaver and Needham to be a particulate coated with a hydrophobic surface-treating reagent that is better suited than a non-coated particulate to remain in the fracture and not be subject to flowback. Thus, one of ordinary skill in the art would have been motivated to combine the teachings of Weaver with Needham in order to control proppant flowback because to do so would enhance hydrocarbon production.

**b. Limitation 51A: “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic”**

1013. Claim 51 recites “contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic.” Ex. 1001 at 13:29-31.

1014. Needham discloses “[a] method of treating fractured earthen formations in oil and gas wells comprising the steps of (a) *mixing together a quantity of*



*particulate solids suitable for use as propping agents and a quantity of an oil-soluble organo-silicon compound dissolved in a solvent* and heating the resulting mixture to a temperature sufficient to evaporate said solvent and leave said solids coated with an oil-wettable coating of said organo-silicon compound, (b) dispersing said coated particulate solids into a carrying fluid, (c) introducing said carrying fluid and dispersed coated solids into a well penetrating a subterranean formation containing fractures, and (d) applying pressure to the fluid in the well thereby causing the fluid to contact the earthen formations and to be injected into fractures and openings extending into the formation whereby said coated particles are deposited therein.” *Id.* at 4:29-47.

1015. Weaver teaches that one “suitable method[] for allowing the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate includes . . . solvent extraction methods.” *Id.* at [0057]. In such an embodiment, “[t]he solution comprising the surface treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced to a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface.” *Id.* As discussed, Needham discloses examples of organo-silicon

compounds that are the same as those disclosed in the '738 patent, including chlorotrimethylsilane. Moreover, Weaver discloses the use of organosilanes, which the '738 patent identifies as a chemical compound for rendering the surface of the particulates hydrophobic.

1016. Thus Needham and Weaver clearly describe contacting the particulates with an organo-silicon compound, which, as stated above, is an example of “a chemical compound to render the surface of the particulates hydrophobic” as required by claim 51.

c. **Limitation 51B: “separating the particulate from the medium”**

1017. Claim 51 recites that the now-coated particulates are “separate[ed] . . . from the medium.” Ex. 1001 at 13:32.

1018. Needham teaches “[a] method of treating fractured earthen formations in oil and gas wells comprising the steps of (a) mixing together a quantity of particulate solids suitable for use as propping agents and a quantity of an oil-soluble organo-silicon compound dissolved in a solvent and ***heating the resulting mixture to a temperature sufficient to evaporate said solvent and leave said solids coated with an oil-wettable coating of said organo-silicon compound***, (b) dispersing said coated particulate solids into a carrying fluid, (c) introducing said carrying fluid and dispersed coated solids into a well penetrating a subterranean formation ***containing fractures***, and (d) applying pressure to the fluid in the well thereby causing the fluid

to contact the earthen formations and to be injected into fractures and openings extending into the formation whereby said coated particles are deposited therein. *Id.* at 4:29-47. A person of ordinary skill in the art would understand that heating the solvent containing dissolved organo-silicon compound to a temperature sufficient to evaporate the solvent and leave a coated particulate is a method of separation.

1019. Weaver teaches that one “suitable method[] for allowing the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate includes . . . solvent extraction methods.” *Id.* at [0057]. In such an embodiment, “[t]he solution comprising the surface treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. ***The treated particulates then may be introduced to a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface.***” *Id.* As discussed, one of ordinary skill in the art would understand Weaver’s disclosure of “extract[ing] the solvent from the treated particulates” to be a method of separating the particulate from the solvent containing the organo-silicon coating. Thus, Weaver discloses that the now-coated particulates are separated from the solvent solution.

1020. In view of the foregoing excerpts from the disclosures of Needham and Weaver, one of ordinary skill in the art would have understood Needham and Weaver to disclose this limitation.

d. **Limitation 51C: “blending the hydrophobic particulate with water and an oil to form a mixture”**

1021. Claim 51 recites that the hydrophobic coated particulate are blended “with water and oil to form a mixture.” Ex. 1001 at 13:32-34.

1022. Needham discloses “dispersing said coated particulate solids into a carrying fluid.” Ex. 1009, Needham at 4:38-39.

1023. Weaver discloses that the treatment fluids containing the suspended proppant “generally comprise a base fluid, which may be aqueous or non-aqueous, ***or a mixture thereof***. Where the base fluid is aqueous, it may comprise fresh water [or] salt water . . . Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts ***can be emulsified also may be suitable for use as a base fluid*** in the methods of the present invention.” Ex. 1008, Weaver at [0058].

1024. Weaver also discloses that “[s]uitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . ***Suitable emulsions*** can be comprised of two

immiscible liquids such as *an aqueous liquid* or gelled liquid *and a hydrocarbon.*”  
*Id.* at [0059].

1025. One of ordinary skill in the art would recognize, and the ’738 patent confirms, that oil is a type of “hydrocarbon.” *See* Ex. 1001, ’738 patent at 11:31-33 (“20. The composition of claim 1, wherein the oil is selected from the group consisting of a *hydrocarbon oil*, a silicon oil, kerosene oil, a frac oil and combinations thereof.”). Weaver thus teaches the limitation of oil included in the composition in the form of a treatment fluid including a mixture of oil and water, as well as an oil and water emulsion.

1026. Plummer teaches “[f]oam fracturing fluids and methods for fracturing well formations using foam as the carrier for the proppant agent.” Ex. 1010, Plummer at Abstract. Plummer teaches that his “invention provides a formation fracturing fluid, comprising a pressured stable foam comprising *a liquid carrier comprising one or more liquids selected from the group consisting of water and oil*, and a solid proppant comprising one or more materials selected from the group consisting of sand . . .” *Id.* at 7:16-20. Specifically, Plummer teaches that “the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.” *Id.* at 8:30-32. Thus, it would have been obvious to one of ordinary skill in the art to substitute the oil and water liquid carrier taught by Plummer for Nguyen’s carrier fluid. Moreover, Plummer discloses that “[o]il may

be added to the foam-water slurry as a lubricant and to prevent clay swelling . . . Other friction reducing materials may be added to the fracturing fluid.” *Id.* at 4:28-33.

1027. As stated, it would have been obvious to a person of ordinary skill in the art to include Plummer’s oil into the particulate slurry taught by Nguyen and Weaver. The motivation to do so is taught by Plummer: using oil as a lubricant friction-reducer or to reduce clay swelling can improve hydraulic fracturing operations in wells. *See* Ex. 1010, Plummer at 4:28-33.

1028. Thus, the simple substitution of Weaver’s treatment fluid (either the aqueous/non-aqueous mixture or the oil and water emulsion) or Plummer’s carrier (oil and water liquid carrier) for Needham’s carrier fluid, and/or the addition of Plummer’s oil to act as a lubricant friction-reducer or to reduce clay swelling would have improved Needham’s composition in the same way as described in Weaver or Plummer (i.e., improved hydraulic fracturing operations). This improvement would have been reasonably expected to succeed by a person of ordinary skill in the art, who would have been motivated to make this substitution and/or addition. Limitation 51C is therefore met.

e. **Limitation 51D: “pumping the mixture into the formation”**

1029. Claim 51 further recites “pumping the mixture into the formation.” Ex. 1001 at 13:34.

1030. Needham teaches “[a] method of treating fractured earthen formations in oil and gas wells comprising the steps of (a) mixing together a quantity of particulate solids suitable for use as propping agents and a quantity of an oil-soluble organo-silicon compound dissolved in a solvent and heating the resulting mixture to a temperature sufficient to evaporate said solvent and leave said solids coated with an oil-wettable coating of said organo-silicon compound, (b) *dispersing said coated particulate solids into a carrying fluid*, (c) *introducing said carrying fluid and dispersed coated solids into a well penetrating a subterranean formation containing fractures*, and (d) applying pressure to the fluid in the well thereby causing the fluid to contact the earthen formations and to be injected into fractures and openings extending into the formation whereby said coated particles are deposited therein. *Id.* at 4:29-47.

1031. Weaver teaches “[a] method of treating a subterranean formation comprising: providing a *treatment fluid that comprises a base fluid, and a plurality of particulates*, at least one of which comprises a modified mineral surface; and *introducing the treatment fluid into the subterranean formation.*” Ex. 1008, Weaver at claim 23; *see also id.* at claim 11.

1032. Therefore, Needham and Weaver disclose limitation 51D.

1033. Based on the foregoing analysis, it is my opinion that each and every limitation, including limitations 51A (contacting the particulates with a medium

containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulate from the medium), 51C (blending the hydrophobic particulate with oil and water to form a mixture), 51D (pumping the mixture into the formation), and the preamble of claim 51, is taught by the combination of Needham in view of Weaver or Plummer. Therefore, independent claim 51 is obvious over Needham in view of Weaver or Plummer.

#### 4. Dependent Claims 21, 22, 40, 41, and 57 Are Obvious

1034. As addressed above (*see* Section XI.D.1, XI.D.2, and XI.D.3) and incorporated herein by reference, all of the limitations of claims 1, 32, and 51, including 1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the mixture into the formation), 51A (contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulates from the medium), 51C (blending the hydrophobic particulate with water and oil to form a mixture); 51D (pumping the mixture into the formation), and the preambles of claims 1, 32, and 51, are obvious over Needham in view of Weaver or Plummer.

1035. The only additional limitation in claims 21, 22, 40, 41 and 57 relate to the type of particulate. Dependent claims 21 and 40 recite a near-identical limitation, but depend from different claims. Claim 21 depends from independent claim 1,



whereas claim 40 depends from independent claim 32. Similarly, dependent claims 22, 41, and 57 recite the same limitation, but depend from different claims. Claim 22 depends from independent claim 1, whereas claim 41 depends from claim 32, and claim 57 depends from claim 51. Dependent claims 21 and 40 require the particulate be one of an enumerated list of particulates, including sand. See Ex. 1001, '738 patent at 11:34-37; 12:63-65 (“wherein the particulates are selected from the group consisting of sand . . .”). Dependent claims 22, 41, and 57 require the particulate to be sands. *Id.* at 11:38-39; 12:66-67, 14:24-25 (“wherein the particulates are sands”). Therefore, a prior art reference that discloses the additional limitation of “sand” as the particulate anticipates claims 21, 22, 40, 41, and 57.

1036. Needham discloses a “method of fracturing an underground zone or formation in oil and gas wells, the steps of . . . injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound” (Ex. 1009, Needham at 4:1-6) “wherein said particulate material comprises sand” (*id.* at 4:16-17; 4:48-49). Needham further discloses “[i]n accordance with a specific embodiment, **sand** coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the solvent to form a **silicon-coated sand which can then be used as a propping medium** in fractured formations containing hydrocarbons.” *Id.* at 1:61-68.

1037. Weaver teaches “[t]he particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, *sand*.” Ex. 1008, Weaver at [0018].

1038. Weaver also teaches that “the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.” *Id.* at [0057]. Thus, the sand particulates are mixed with the treatment fluid before being pumped down the formation. Thus, Needham and Weaver teach the additional limitation of claims 21, 22, 40, 41 and 57. Therefore, it is my opinion that challenged dependent claims 21, 22, 40, 41, and 57 of the ’738 patent would have been obvious over Needham in view of Weaver or Plummer.

1039. Based on the foregoing analysis of Sections XI.D.1-4, all of the elements of claims 1, 21, 22, 32, 40, 41, 51, and 57 [1A and 32A (water/mixing water), 1B and 32B (particulates), 1C and 32C (a chemical compound for rendering the surface of the particulate hydrophobic), 1D and 32D (oil), 32E (pumping the

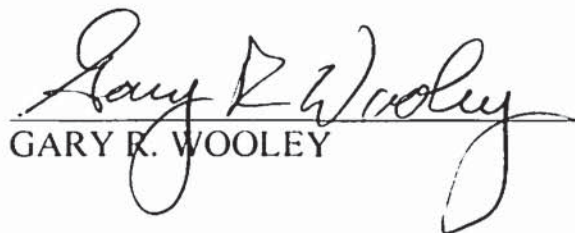
mixture into the formation), 51A (contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic), 51B (separating the particulates from the medium), 51C (blending the hydrophobic particulate with water and oil to form a mixture); 51D (pumping the mixture into the formation), 21A, 22A, 40A, 41A and 57A (sand), and the preambles of claims 1, 32 and 51] are disclosed by the combination of Needham in view of Weaver or Plummer. Therefore, it is my opinion that the combination of Needham in view of Weaver or Plummer renders challenged claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent obvious.

**E. Secondary Considerations of Non-Obviousness**

1040. At this time, I have not formed an opinion regarding any objective factors that the Board might consider in making a determination as to whether the claimed invention would have been obvious to a person of ordinary skill in the art since none appear in the various prosecution histories and I am not otherwise aware of any such secondary considerations. I understand that if the Patent Owner presents evidence of objective factors that it argues support a finding of non-obviousness, I may be asked to review such evidence and to formulate an opinion.

1041. As required under 28 U.S.C. § 1746, I declare under penalty of perjury that the foregoing is true and correct.

Executed on this 29th day of September, 2017.

  
GARY R. WOOLEY

# EXHIBIT A

## Resumé for Gary R. Wooley

### Personal

Born New Orleans, Louisiana, 1946  
Married with three children  
Member, director, board member or officer of  
various educational, civic and religious  
organizations



### Education

Louisiana State University:

BS: Mechanical Engineering, January 1969

MS: Engineering Mechanics, May 1970

PhD: Engineering Science, December 1972, with Minors in  
Applied Mathematics and Mechanical Engineering

Honors: NSF Fellow, LSU Dissertation Fellow, Dean's List,  
Phi Kappa Phi (Phi Beta Kappa), Sigma Xi, Tau Beta Pi,  
Pi Tau Sigma

Various Petroleum Professional Short Courses

### Technical Experience

|              |                                                |
|--------------|------------------------------------------------|
| 1966-68      | Shell Oil and Chevron Oil - Summer Engineer    |
| 1969         | Humble Oil (Exxon) - Offshore Field Engineer   |
| 1970-72      | Louisiana State University - Instructor        |
| 1972-78      | Atlantic Richfield Co. - Sr. Research Engineer |
| 1978-86      | Enertech Engineering - Vice President          |
| 1986-Present | Wooley & Associates, Inc. - President          |

### Professional

ASME, SPE, API, ASM: Member, Invited Speaker, Technical  
Committee Chairman, Standardization Committees.

Registered Professional Engineer in State of Texas

Numerous publications in professional literature

Advisory Board to LSU Dean of Engineering

[LSU Engineering Hall of Distinction](#)

LSU ME Alumni Achievement Lecture Club

Listed in various publications of Who's Who

5 January 2017

# EXHIBIT B

## Recent Wooley Testimony

28 July 2017

The following list of recent testimony by Gary R. Wooley is compiled from available information. It is possible there may be additional testimony not included in this list:

1. 8/23/2013 and 3/11/2014 depositions, Houston, Texas, and 4/11/2014 trial, Jena, Louisiana, Justiss Oil Company, Inc. vs. Oil Country Tubular Corporation, North American Interpipe (Tim Hightower, Hightower & Russo), No. C-2009-1338”A”. 36<sup>th</sup> Judicial District Court, Beauregard Parish, Louisiana.
2. 9/5/2013 deposition, Houston, Texas, Cenco, Inc. vs. CETCO Oilfield Services Company (David Leefe, Liskow & Lewis), et al, No. 91945-C, 15<sup>th</sup> Judicial District, Vermilion Parish, Louisiana.
3. 10/15/2013 deposition, Houston, Texas, Angelica Cepeda, et al vs. Mobile Crane, Hess (John Anaipakos, Baker Botts), et al, No. C-2481-12B, 93<sup>rd</sup> Judicial District, Hidalgo County, Texas.
4. 10/28/2013, 5/16/2014 and 10/9/2014 depositions, Houston, Texas, and 5/21/2014 hearing testimony, Corpus Christi, Texas, Magnum Oil Tools vs. Tony D. McClinton (Robert Barz, K&L Gates), et al, No. 2:12-CV-00099, U. S. District Court, Corpus Christi Division, Texas.
5. 3/26/2014 deposition, Houston, Texas, and 4/8/2014 trial, Midland, Texas, Houston, Texas, Peak Completion, et al vs. Team Oil Tools (Robert Bowick, Raley & Bowick), et al, No. CV-48,344, 441<sup>st</sup> Judicial District, Midland County, Texas.
6. 4/30/2014 deposition, Houston, Texas, Dan A. Hughes, Company, LP, et al vs. Gulf Coast Tubulars, LP, Lincoln Manufacturing, Inc. (H. Kent Twining, Litchfield, Cavo, LLP), No. 12-03-00056-CVK, District Court, Karnes County, Texas, 218<sup>th</sup> Judicial District.
7. 5/15/2014 deposition, Houston, Texas, Hayward Industries (Petitioner) vs. Pentair Water Pool and Spa (C. J. Fahy, Quarles & Brady) (Patent Owner), US Patent and Trademark Office Case IPR2013-00285, U. S. Patent 8,019,479.



8. 10/14/2014 deposition, Corpus Christi, Texas, Leonard May, et al vs. Duke Energy Operating Company, LLC, et al (Clay Hoblit, Hoblit Ferguson Darling), No. 07-62775-1, County Court at Law No. One, Nueces County, Texas.
9. 11/12/2014 deposition, Houston, Texas, Greene's Energy Group, LLC, Petitioner vs. Oil States Energy Services, LLC, Patent Owner, (Erik Hawes, Morgan Lewis Brockius), IPR2014-00216 Patent 6,179,053, IPR2014-00364 Patent 6,289,993.
10. 11/20/2014 deposition, Houston, Texas, 4/16/2013 hearing, Austin, Texas, National Oilwell Varco, L.P. (Bobby Bowick, Raley & Bowick) vs. Omron Oilfield & Marine, Inc., No. CA 1:12-CV-00773-SS, U. S. District Court for the Southern District of Texas, Houston Division.
11. 12/16/2014 and 3/4/2015 deposition, Houston, Texas, Oil States Energy Service, LLC (Erik Hawes, Morgan Lewis & Brockius), et al vs. Trojan Wellhead Protection, Inc., et al, Civil Action No.: 6:12-CV-611, U. S. District Court for the Eastern District of Texas, Tyler Division.
12. 5/17/2015 deposition, Houston, Texas, Tammy Manning, et al (Bill Dubanovich, Parker & Waichman) vs. WPX Energy Appalachia, et al, No. 3:12-cv-00646, U. S. District Court for Middle District of Pennsylvania.
13. 9/10/2015 deposition, Houston, Texas, Flow Valve, LLC (Mick McCarthy, Hall Estill) vs. Forum Energy Technologies, Inc., et al, No. 5:13-CV-01261-F, U. S. Western District of Oklahoma.
14. 11/12/2015 deposition, Houston, Texas, Cenco, Inc. vs. CETCO Oilfield Services Company, et al, (Davis Leefe, Liskow & Lewis), No. 91945-C, 15th District, Vermilion Parish, Louisiana.
15. 9/21/2016 trial, Calgary, Alberta, Canada, Excalibre Oil Tools, Ltd., et al, (Doak Horne, Gowling WLG) vs. Advantage Products Inc., et al, Court File No. T-1741-08, Canadian Federal Court.
16. 10/12/2016, 10/18/2016, 2/1/2017 and 3/2/2017 deposition, Houston, Texas, Statoil Oil & Gas LP vs. North American Interpipe, Inc. (Tim Hightower, Hightower-Capellan), No. D-1-GN-13-003407, 126<sup>th</sup> Judicial District, Travis County, Texas.
17. 12/8/2016 deposition, Houston, Texas, Blackbrush Oil & Gas LP vs. GB

Tubulars, Inc., et al (Brian Zimmerman, Zimmerman, Axelrad) No. 13-09-00195-CVK, 81<sup>st</sup> District, Karnes County, Texas.

18. 4/5/2017 deposition, Houston, Texas, Legacy Separators, LLC (Pam Hohensee, Yetter, Coleman) vs. Halliburton Energy Services, Inc., et al, No. H-14-2081 (H-14-3110), U.S. Southern District of Texas, Houston Division.
19. 5/18/2017 deposition, Houston, Texas and 7/24/2017 hearing, Marshall, Texas, Multilift, et al vs. C&J Energy Services, Inc. (Erik Hawes, Morgan, Lewis), et al, No. 2:16-cv-01187, U.S. Eastern District, Marshall Division.

# EXHIBIT C

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2004/0023818 (Nguyen)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

| Claim 1 of U.S. Patent 8,236,738             | Reference to U.S. Publication 2004/0023818 (Nguyen)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |
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| 1. An aqueous slurry composition comprising: | <p>Claim 63: ... mixing particles in a carrier fluid comprising a water-based polymer to form a mixture . . .</p> <p>[0013] The water utilized to form the fracturing fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other aqueous liquid which does not adversely react [to] other components of the fracturing fluid.</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |
| water;                                       | <p>[0011] Fracturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and emulsions. The foams utilized are generally comprised of water based liquids containing one or more foaming agents foamed with a gas such as nitrogen or air. Emulsions formed with two or more immiscible liquids have also been utilized.</p> <p>[0012] The most common fracturing fluid utilized heretofore which is generally preferred for use in accordance with this invention is comprised of water . . .</p> <p>[0013] The water utilized to form the fracturing fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other aqueous liquid which does not adversely react [to] other components of the fracturing fluid.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the</p> |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2004/0023818 (Nguyen)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                    | <p>particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surface of the particles.</p> <p>Claim 65: The method of claim 63 wherein the carrier fluid comprises water and a gelling agent.</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |
| particulates;                                                                      | <p>[0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like. Generally the particles used have a particle size in the range of from about 2 to about 400 mesh, U.S. Sieve Series. The preferred particles are sand having a particle size in the range of from about 10 to about 70 mesh, U.S. Sieve Series.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of hydrocarbons.</p> |
| a chemical compound for rendering the surface of the particulates hydrophobic; and | <p>[0005] According to an embodiment, the particles are dry coated with a water-repellent organo-silicon material.</p> <p>[0006] Representative organo-silicon compounds that can be used in this embodiment include polyalkylsiloxane such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additional organo-silicon compounds that can be used in this embodiment include polyalkylarylsiloxanes, [] chlorosilanes [], chlorotrimethylsilane and other silyl donors. Also, various</p>                                                                                                                                                                                                                                                                                                                                              |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2004/0023818 (Nguyen)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|         | <p>alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes can be used such as tetraethoxysilane, dimethoxydiphenylsilane, dichlorodimethylsilane, dichlorodiphenylsilane, poly[oxy(dimethylsilylene)] and other such materials that will be well known to those skilled in the art. . . . Furthermore, organofunctional silanes can be used in this embodiment...</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p>                                                                                                                                                                                                                                                                                                                                                                           |
| an oil. | <p>[0011] Fracturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and emulsions.</p> <p>[0008] If the particles are coated when the particles are flowing, the solvent should be readily miscible in the water-based gel carrier fluid. Organic solvents that can be used as a carrier for the organo-silicon coating material include kerosene, lighter grades of diesel fuel, hexane, xylene, toluene . . . and other such solvents that will be well known to those skilled in the art.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of the hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p> |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2004/0023818 (Nguyen)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                                                                                                    | <p>[0001] After most oil and gas wells are drilled, they do not produce hydrocarbons at a rate to provide satisfactory economic return. Therefore, the oil industry uses a process known as hydraulic fracture stimulation to generate fractures deep into the hydrocarbon-bearing rock formations, which provides highly conductive flow channels to the well.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> |
| <b>Claim 21 of U.S. Patent 8,236,738</b>                                                                                                                                                                           | <b>Reference to U.S. Publication 2004/0023818 (Nguyen)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
| 21. The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates. | <p>[0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like.</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p>                                                                                                                                                                                                                            |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2004/0023818 (Nguyen)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                             | Claim 8: The method of claim 1 wherein the particles are selected from the group consisting of sand and gravel.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |
| <b>Claim 22 of U.S. Patent 8,236,738</b>                                                                    | <b>Reference to U.S. Publication 2004/0023818 (Nguyen)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| 22. The composition of claim 1, wherein the particulates are sands.                                         | <p>[0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like.</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Claim 8: The method of claim 1 wherein the particles are selected from the group consisting of sand and gravel.</p> |
| <b>Claim 32 of U.S. Patent 8,236,738</b>                                                                    | <b>Reference to U.S. Publication 2004/0023818 (Nguyen)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| 32. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: | <p>[0005] According to an embodiment, the particles are dry coated with a water-repellent organo-silicon material. The treated particles are then pumped down to the fractures to function as a proppant or to the wellbore-screen annulus to function as a gravel pack.</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p>                                                                                                                                                                                                                                                                |



U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2004/0023818 (Nguyen)  
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|                                                                                                                                  | <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of the hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |
| <p>mixing water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic and an oil; and</p> | <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of the hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p> <p>[0005] According to an embodiment, the particles are dry coated with a water-repellent organo-silicon material.</p> <p>[0006] Representative organo-silicon compounds that can be used in this embodiment include polyalkylsiloxane such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additional organo-silicon compounds that can be used in this embodiment include polyalkylarylsiloxanes, . . . chlorosilanes such as ethylchlorosilane, chlorotrimethylsilane and other silyl donors. Also, various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes can be used such as tetraethoxysilane, dimethoxydiphenylsilane, dichlorodimethylsilane, dichlorodiphenylsilane, poly[oxy(dimethylsilylene)] and other such materials that will be well known to those skilled in the art. . . . Furthermore, organofunctional silanes can be used in this embodiment...</p> |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>[0008] If the particles are coated when the particles are flowing, the solvent should be readily miscible in the water-based gel carrier fluid. Organic solvents that can be used as a carrier for the organo-silicon coating material include kerosene, lighter grade of diesel fuel . . . and other such solvents that will be well known to those in the art.</p> <p>[0011] Fracturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and emulsions.</p> <p>[0012] The most common fracturing fluid utilized heretofore which is generally preferred for use in accordance with this invention is comprised of water. . .</p> <p>[0013] The water utilized to form the fracturing fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other aqueous liquid which does not adversely react to the other components of the fracturing fluids.</p> <p>[0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like. Generally the particles used have a particle size in the range of from about 2 to about 400 mesh, U.S. Sieve Series. The preferred particles are sand having a</p> |
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U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2004/0023818 (Nguyen)  
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|                                         | <p>particle size in the range of from about 10 to about 70 mesh, U.S. Sieve Series.</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> <p>Claim 65: The method of claim 63 wherein the carrier fluid comprises water and a gelling agent.</p> |
| pumping the mixture into the formation. | <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> <p>Claim 75: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture</p>                                                                                                                                                                                                         |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2004/0023818 (Nguyen)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                                                                      | downhole in a well to a fracture in a formation adjacent the well to function as a proppant, and coating the particles with a water repellent composition, wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |
| <b>Claim 40 of U.S. Patent 8,236,738</b>                                                                                                                                             | <b>Reference to U.S. Publication 2004/0023818 (Nguyen)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
| 40. The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates. | <p>[0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Claim 2: The method of claim 1 wherein the particles are pumped downhole to a screen and function as a gravel pack.</p> |

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|                                                                 | <p>Claim 3: The method of claim 1 wherein the particles are pumped downhole to a fracture in a formation adjacent the well and function as a proppant.</p> <p>Claim 8: The method of claim 1 wherein the particles are selected from the group consisting of sand and gravel.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p>                    |
| <b>Claim 41 of U.S. Patent 8,236,738</b>                        | <b>Reference to U.S. Publication 2004/0023818 (Nguyen)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |
| 41. The method of claim 32, wherein the particulates are sands. | <p>[0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p> |

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|                                                                                                             | <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Claim 2: The method of claim 1 wherein the particles are pumped downhole to a screen and function as a gravel pack.</p> <p>Claim 3: The method of claim 1 wherein the particles are pumped downhole to a fracture in a formation adjacent the well and function as a proppant.</p> <p>Claim 8: The method of claim 1 wherein the particles are selected from the group consisting of sand and gravel.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> |
| <b>Claim 51 of U.S. Patent 8,236,738</b>                                                                    | <b>Reference to U.S. Publication 2004/0023818 (Nguyen)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |
| 51. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: | [0005] According to an embodiment, the particles are dry coated with a water-repellent organo-silicon material. The treated particles are then pumped down to the fractures to function as a proppant or to the wellbore-screen annulus to function as a gravel pack.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |

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|                                                                                                                                 | <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of the hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p>                                                                                                                                                                                                                         |
| Contacting particulates with a medium containing a chemical compound for rendering the surface of the particulates hydrophobic, | <p>[0007]: An example of the technique for applying the coating on the particles involves dissolving an oil-soluble organo-silicon compound in a solvent, admixing or spraying the resultant solution on the particles, and then evaporating the solvent to form a thin film of siloxane or silane encapsulating the particles. The organo-silicon organo-silane coating is readily absorbed onto the particles from the solvent and the solvent is easily evaporated by drying.</p> <p>Claim 4: The method of claim 1, wherein the composition is oil-soluble, and wherein the particles are coated by dissolving the composition in a solvent, admixing or spraying the resultant solution the particulates, and then evaporating the solvent to form a film encapsulating the particles.</p> |
| Separating the particulate from the medium;                                                                                     | <p>[0007]: An example of the technique for applying the coating on the particles involves dissolving an oil-soluble organo-silicon compound in a solvent, admixing or spraying the resultant solution on the particles, and then evaporating the solvent to form a thin film of siloxane or silane encapsulating the</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2004/0023818 (Nguyen)  
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|                                                                                   | <p>particles. The organo-silicon organo-silane coating is readily absorbed onto the particles from the solvent and the solvent is easily evaporated by drying.</p> <p>Claim 4: The method of claim 1, wherein the composition is oil-soluble, and wherein the particles are coated by dissolving the composition in a solvent, admixing or spraying the resultant solution the particulates, and then evaporating the solvent to form a film encapsulating the particles.</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |
| And blending the hydrophobic particulate with water and an oil to form a mixture; | <p>[0011] Fracturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and emulsions.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p> |
| And pumping the mixture into the formation.                                       | <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |



U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2004/0023818 (Nguyen)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                      | <p>downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> <p>Claim 75: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to a fracture in a formation adjacent the well to function as a proppant, and coating the particles with a water repellent composition, wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> |
| <b>Claim 57 of U.S. Patent 8,236,738</b>                             | <b>Reference to U.S. Publication 2004/0023818 (Nguyen)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| 57. The composition of claim 51, wherein the particulates are sands. | <p>[0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p>                                            |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2004/0023818 (Nguyen)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Claim 2: The method of claim 1 wherein the particles are pumped downhole to a screen and function as a gravel pack.</p> <p>Claim 3: The method of claim 1 wherein the particles are pumped downhole to a fracture in a formation adjacent the well and function as a proppant.</p> <p>Claim 8: The method of claim 1 wherein the particles are selected from the group consisting of sand and gravel.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> |
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# EXHIBIT D

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

| Claim 1 of U.S. Patent 8,236,738             | Reference to Canadian Patent 2,423,031 (Dawson)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
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| 1. An aqueous slurry composition comprising: | <p>P. 14, l. 12-15: A slurry including the disclosed particulate material (<i>e.g.</i>, relatively lightweight and/or substantially neutrally buoyant particulates, and/or particulates of naturally-occurring material that have been exposed to a modifying agent) and a carrier fluid may then be introduced into the wellbore . . .</p> <p>P. 13, l. 10-13: In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water . . .</p>                                                                                                                                                                                                                             |
| water;                                       | <p>P. 13, l. 6-13: The particles disclosed herein . . . may be mixed and pumped during any desired portion/s of a well treatment (<i>e.g.</i>, hydraulic fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water. . .</p> <p>P. 10, l. 1-3: In another respect, disclosed herein is a method of fracturing a subterranean formation including introducing a particulate material suspended in a carrier fluid into the subterranean formation. . .</p> <p>P. 33, l. 6-8: Incorporates by reference Graham (U.S. Patent 3,659,651)</p> |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|               | <p>Graham, Col. 5, l. 9-16: In placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or <b>water-in-oil emulsion</b> prepared in accordance with the teachings of U.S. Pat. No. 3,378,074 (Kiel)</p> <ul style="list-style-type: none"> <li>• Kiel, Col. 5, l. 66-73: High viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either water-in-oil or oil-in-water emulsions. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine or an acid solution containing a suitable surface active agent or emulsifier.</li> </ul> |
| particulates; | <p>P. 2, l. 5-8: In particular, this invention relates to use of relatively lightweight and/or substantially neutrally buoyant particles as proppant material in hydraulic fracturing treatments and as particulate material in sand control methods such as gravel packing, frac pack treatments, <i>etc.</i></p> <p>P. 10, l. 1-3: In another respect, disclosed herein is a method of fracturing a subterranean formation, including introducing a particulate material suspended in a carrier fluid into the subterranean formation at a pressure above a fracturing pressure of the subterranean formation.</p> <p>P. 48, claim 13: A method of fracturing a subterranean formation, comprising: introducing a particulate material</p>                                                                                                                                                                                                                                                                                                 |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                    | suspended in a carrier fluid into said subterranean formation at a pressure above a fracturing pressure of said subterranean formation; . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |
| a chemical compound for rendering the surface of the particulates hydrophobic; and | <p>P. 7, l. 3-23: In a further embodiment, individual particles . . . may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material . . . Specific examples of suitable modifying agents include, but are not limited to, agents including polyisocyanates, silanes, siloxanes, and combinations thereof. Selected modifying agent/s may be advantageously used to increase the elastic modulus of a given naturally occurring material, for example, to make particles of a given naturally-occurring material more suitable . . . for use as a relatively lightweight and/or substantially neutrally buoyant fracture proppant or sand control particulate material having increased effectiveness when exposed to higher closure stresses or other mechanical stresses that may be encountered downhole during a well treatment such as hydraulic fracturing or sand control treatment.</p> <p>P. 21, l. 8-14: In a further embodiment, one or more of the carbon chains R and/or R' of formula (I) may be optionally and independently derivatized, <i>e.g.</i>, the R carbon chain and/or one or more of the R' carbon chains may each contain one or more amino functional groups, one or more halogen groups (<i>e.g.</i>, tetrachlorosilane, methyltrichlorosilane, <i>etc.</i>), two or more isocyanate functional groups, two or more epoxy groups, <i>etc.</i> Where halogen groups (<i>e.g.</i> such as chlorine groups) are present, it may be desirable to rapidly neutralize any acidic by-products that may be created during a modification reaction.</p> |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>P. 21, l. 19- P. 22, l. 23: Examples of commercially available silane-based products that may be employed as modifying agents in the practice of the disclose methods and compositions include, but are not limited to, silane based products. . . . Specific examples of commercially available silane-based products available from CROMPTON include CoatOSil® 1706 Silane (vinyl silane monomer), CoatOSil® 1757 Silane, CoatOSil® 1770 Silane, EURENOR® 5020 (liquid isobutylisopropylmethoxysilane), EURENOR® 5021 (liquid diisopropylmethoxysilane), EURENOR® 5022 (liquid diisobutylmethoxysilane), EURENOR® 5023 (liquid dicyclopentylmethoxysilane), Silcat® R Silane (vinylsilane with grafting and cross linking catalysts), Sikat® RHE Silane (cross linking system of silane, peroxide and catalyst), Silcat® RHS Silane (crosslinking system of silane, peroxide catalyst and antioxidants), Sikat® VS-735/1 Silane (crosslinking system of silane, peroxide, antioxidants and metal deactivator), Silcat® VS-758/0 Silane (stabilized cross linking system of silane, peroxide and catalyst), Sikat® VS-870 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat® VS-928 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat® VS-963 Silane (stabilized cross linking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Silox® VS- 911 Silane (liquid silane system), Silquest® A-1100 Silane (gammaaminopropyltriethoxysilane), Silquest® A-1101 Silane (technical grade amino silane), Silquest® A-1102 Silane (technical grade amino silane), Silquest® A-1110 Silane (primary amino silane), Silquest® A-1120 Silane, Silquest® A-1126 Silane (amino silane), Silquest® A-1128 Silane (amino silane), Silquest® A-1160 Silane (ureido silane), Silquest® A-1170 Silane (secondary amino functional silane), Silquest® A-1289 Silane, Silquest® A-1310 Silane,</p> |
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U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|         | <p>Silquest® A-137 Silane (monomeric alkylalkoxysilane), Silquest® A-151 Silane, Silquest® A-1524 Silane (100-percent active ureidosilane), Silquest® A-1589 Silane (disulfide silane), Silquest® A-171 Silane, Silquest® A-172 Silane (vinylfunctional coupling agent), Silquest® A-174 Silane, Silquest® A-186 Silane (epoxy functional silanes), Silquest® A-187 Silane (epoxy functional silanes), Silquest® A-189 Silane, Silquest® A-2171 Silane (silane compound having vinyl and silane functionality), Silquest® Alink 15 Silane (secondary amino silane endcapper), Silquest® RC-1 Silane (organosilicone coupling agent), Silquest® RC-2 Silane, Silquest® Wetlink 78 Silane, Silquest® Y-11637 Silane (primary aminosilane), Silquest® Y-11683 Silane (di-, tri-functional ethoxy silane), Silquest® Y-5997 Silane, XL-PEarl® 31 Blend, XL-PEarl® 50 Blend, XLPEarl® 51 Blend, XL-PEarl® 52 Blend, XL-PEarl® 60 Blend, XL-PEarl® 70 Blend, <i>etc.</i></p> |
| an oil. | <p>P. 13, l. 6-13: The particles disclosed herein . . . may be mixed and pumped during any desired portion/s of a well treatment (e.g., hydraulic fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water, liquid hydrocarbons, and/or nitrogen or other gases.</p> <p>P. 33, l. 6-8: Incorporates by reference Graham (U.S. Patent 3,659,651)</p> <p>Graham, Col. 5, l. 9-16: In placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high</p>                |



U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                                                                                                    | <p>viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or <b>water-in-oil emulsion</b> prepared in accordance with the teachings of U.S. Pat. No. 3,378,074 (Kiel)</p> <ul style="list-style-type: none"> <li>Kiel, Col. 5, l. 66-73: High viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either water-in-oil or oil-in-water emulsions. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine or an acid solution containing a suitable surface active agent or emulsifier.</li> </ul> <p>P. 14, l. 31- 15, l. 4: [I]t will be understood with benefit of this disclosure that the disclosed particulate materials may be used with any method in which a pack of particulate material is formed within a wellbore that it is permeable to fluids produced from a wellbore (e.g., oil, gas, water, etc.) but that substantially prevents or reduces production of formation materials (e.g., formation sand, etc.) from the formation into the wellbore.</p> |
| <b>Claim 21 of U.S. Patent 8,236,738</b>                                                                                                                                                                           | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                |
| 21. The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates. | <p>P. 13, l. 3-5: Additional information on materials and methods for use thereof may be found in U.S. Pat. No. 6,330,916, which is incorporated herein by reference. Further information on materials and methods may also be found in U.S. Patent No. 6,364,018.</p> <p>U.S. Pat. No 6,330,916 (Rickards), Abstract: A subterranean formation is treated by injecting a blend of fracture proppant material and deformable particulate material into the formation.</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                             | The deformable particulate material may combine with the fracture proppant material to increase fracture conductivity, reduce fines generation, and/or reduce proppant flowback. The fracture proppant material may be a material such as sand . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |
| <b>Claim 22 of U.S. Patent. 8,236,738</b>                                                                   | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |
| 22. The composition of claim 1, wherein the particulates are sands.                                         | <p>P. 13, l. 3-5: Additional information on materials and methods for use thereof may be found in U.S. Pat. No. 6,330,916, which is incorporated herein by reference. Further information on materials and methods may also be found in U.S. patent No. 6,364,018.</p> <p>U.S. Pat. No 6,330,916 (Rickards), Abstract: A subterranean formation is treated by injecting a blend of fracture proppant material and deformable particulate material into the formation. The deformable particulate material may combine with the fracture proppant material to increase fracture conductivity, reduce fines generation, and/or reduce proppant flowback. The fracture proppant material may be a material such as sand . . .</p> |
| <b>Claim 32 of U.S. Patent 8,236,738</b>                                                                    | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |
| 32. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: | P. 2, l. 4-8: This invention relates generally to methods and compositions useful for subterranean formation treatments, such as hydraulic fracturing treatments and sand control. In particular, this invention relates to the use of relatively lightweight and/or substantially neutrally buoyant particles as proppant materials in hydraulic fracturing treatment and as particulate material in sand control methods such as gravel packing, frac pack treatments, etc.                                                                                                                                                                                                                                                  |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                  | <p>P. 13, l. 22-25: Such other treatments may be near wellbore in nature (affecting near wellbore regions) and may be directed towards improving wellbore productivity and/or controlling the production of fracture proppant or formation sand. Particular examples include gravel packing and “frac-packs.”</p> <p>P. 3, l. 13-16: In the disclosed method, the application of relatively lightweight and/or substantially neutrally buoyant particulate material as a fracture proppant particulate advantageously may provide for substantially improved overall system performance in hydraulic fracturing applications, or in other well treating applications such as sand control.</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| <p>mixing water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic and an oil; and</p> | <p>P. 13, l. 6-13: The particles disclosed herein (<i>e.g.</i>, relatively lightweight and/or substantially neutrally buoyant proppant or sand control particulates, and/or particulates of naturally-occurring material that have been exposed to a modifying agent) may be mixed and pumped during any desired portion/s of well treatment (<i>e.g.</i>, hydraulic fracturing treatment, sand control treatment, <i>etc.</i>) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water, liquid hydrocarbons, and/or nitrogen or other gases.</p> <p>P. 2, l. 5-8: In particular, this invention relates to use of relatively lightweight and/or substantially neutrally buoyant particles as proppant material in hydraulic fracturing treatments and as particulate material in sand control methods such as gravel packing, frac pack treatments, <i>etc.</i></p> |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>P. 10, l. 1-3: In another respect, disclosed herein is a method of fracturing a subterranean formation, including introducing a particulate material suspended in a carrier fluid in the subterranean formation at a pressure above a fracturing pressure of the subterranean formation...</p> <p>P. 48, claim 13: A method of fracturing a subterranean formation, comprising: introducing a particulate material suspended in a carrier fluid into said subterranean formation at a pressure above a fracturing pressure of said subterranean formation ;. . .</p> <p>P. 7, l. 3-23: In a further embodiment, individual particles . . . may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material . . . Specific examples of suitable modifying agents include, but are not limited to, agents including polyisocyanates, silanes, siloxanes, and combinations thereof. Selected modifying agent/s may be advantageously used to increase the elastic modulus of a given naturally occurring material, for example, to make particles of a given naturally-occurring material more suitable . . . for use as a relatively lightweight and/or substantially neutrally buoyant fracture proppant or sand control particulate material having increased effectiveness when exposed to higher closure stresses or other mechanical stresses that may be encountered downhole during a well treatment such as hydraulic fracturing or sand control treatment.</p> |
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U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>P. 21, l. 8-14: In a further embodiment, one or more of the carbon chains R and/or R' of formula (I) may be optionally and independently derivatized, e.g., the R carbon chain and/or one or more of the R' carbon chains may each contain one or more amino functional groups, one or more halogen groups (e.g., tetrachlorosilane, methyltrichlorosilane, etc.), two or more isocyanate functional groups, two or more epoxy groups, etc. Where halogen groups (e.g., such as chlorine groups) are present, it may be desirable to rapidly neutralize any acidic by-products that may be created during a modification reaction.</p> <p>P. 21, l. 23- P. 22, l. 23: Examples of commercially available silane-based products that may be employed as modifying agents in the practice of the disclose methods and compositions include, but are not limited to, silane based products” (Ex. 1006, Dawson at p. 21, l. 19-21) and teaches the specific examples of “commercially available silane-based products available from CROMPTON include CoatOSil® 1706 Silane (vinyl silane monomer), CoatOSil® 1757 Silane, CoatOSil® 1770 Silane, EURENOR® 5020 (liquid isobuytlisopropyldimethoxysilane), EURENOR® 5021 (liquid diisopropylmethoxysilane), EURENOR® 5022 (liquid diisobutyldimethoxysilane), EURENOR® 5023 (liquid dicyclopentyldimethoxysilane), Silcat® R Silane (vinylsilane with grafting and cross linking catalysts), Sikat® RHE Silane (cross linking system of silane, peroxide and catalyst), Silcat® RHS Silane (crosslinking system of silane, peroxide catalyst and antioxidants), Sikat® VS-735/1 Silane (crosslinking system of silane, peroxide, antioxidants and metal deactivator), Silcat® VS-758/0 Silane (stabilized cross linking system of silane, peroxide and catalyst), Sikat® VS-870 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat® VS-928 Silane (stabilized crosslinking system of silane, peroxide,</p> |
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U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>catalyst, antioxidants and metal deactivator), Sikat® VS-963 Silane (stabilized cross linking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Silox® VS- 911 Silane (liquid silane system), Silquest® A-1100 Silane (gammaaminopropyltriethoxysilane), Silquest® A-1101 Silane (technical grade amino silane), Silquest® A-1102 Silane (technical grade amino silane), Silquest® A-1110 Silane (primary amino silane), Silquest® A-1120 Silane, Silquest® A-1126 Silane (amino silane), Silquest® A-1128 Silane (amino silane), Silquest® A-1160 Silane (ureido silane), Silquest® A-1170 Silane (secondary amino functional silane), Silquest® A-1289 Silane, Silquest® A-1310 Silane, Silquest® A-137 Silane (monomeric alkylalkoxysilane), Silquest® A-151 Silane, Silquest® A-1524 Silane (100-percent active ureidosilane), Silquest® A-1589 Silane (disulfide silane), Silquest® A-171 Silane, Silquest® A-172 Silane (vinylfunctional coupling agent), Silquest® A-174 Silane, Silquest® A-186 Silane (epoxy functional silanes), Silquest® A-187 Silane (epoxy functional silanes), Silquest® A-189 Silane, Silquest® A-2171 Silane (silane compound having vinyl and silane functionality), Silquest® Alink 15 Silane (secondary amino silane endcapper), Silquest® RC-1 Silane (organosilicone coupling agent), Silquest® RC-2 Silane, Silquest® Wetlink 78 Silane, Silquest® Y-11637 Silane (primary aminosilane), Silquest® Y-11683 Silane (di-, tri-functional ethoxy silane), Silquest® Y-5997 Silane, XL-PEarl® 31 Blend, XL-PEarl® 50 Blend, XLPEarl® 51 Blend, XL-PEarl® 52 Blend, XL-PEarl® 60 Blend, XL-PEarl® 70 Blend, <i>etc.</i></p> <p>P. 13, l. 6-13: The particles disclosed herein . . . may be mixed and pumped during any desired portion/s of a well treatment (e.g., hydraulic fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier</p> |
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U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                         | <p>fluid. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water, liquid hydrocarbons, and/or nitrogen or other gases.</p> <p>P. 33, l. 6-8: Incorporates by reference Graham (U.S. Patent 3,659,651)</p> <p>Graham, Col. 5, l. 9-16: In placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or <b>water-in-oil emulsion</b> prepared in accordance with the teachings of U.S. Pat. No. 3,378,074 (Kiel)</p> <ul style="list-style-type: none"> <li>Kiel, Col. 5, l. 66-73: High viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either water-in-oil or oil-in-water emulsions. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine or an acid solution containing a suitable surface active agent or emulsifier.</li> </ul> |
| pumping the mixture into the formation. | <p>P. 13, l. 6-10: The particles disclosed herein (<i>e.g.</i>, relatively lightweight and/or substantially neutrally buoyant proppant or sand control particulates, and/or particulates of naturally-occurring material that have been exposed to a modifying agent) may be mixed and pumped during any desired portion/s of well treatment (<i>e.g.</i>, hydraulic fracturing treatment, sand control</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                                                                      | <p>treatment, <i>etc.</i>) and may be mixed in any desired concentration with a carrier fluid.</p> <p>P. 29, l. 27-28: In another exemplary embodiment, modified particulate material may be formulated as described herein and introduced or pumped into a well...</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                        |
| <b>Claim 40 of U.S. Patent 8,236,738</b>                                                                                                                                             | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |
| 40. The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates. | <p>P. 13, l. 3-5: Additional information on materials and methods for use thereof may be found in U.S. Pat. No. 6,330,916, which is incorporated herein by reference. Further information on materials and methods may also be found in U.S. Patent No. 6,364,018.</p> <p>U.S. Pat. No 6,330,916 (Rickards), Abstract: A subterranean formation is treated by injecting a blend of fracture proppant material and deformable particulate material into the formation. The deformable particulate material may combine with the fracture proppant material to increase fracture conductivity, reduce fines generation, and/or reduce proppant flowback. The fracture proppant material may be a material such as sand . . .</p> |
| <b>Claim 41 of U.S. Patent 8,236,738</b>                                                                                                                                             | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |
| 41. The method of claim 32, wherein the particulates are sands.                                                                                                                      | <p>P. 13, l. 3-5: Additional information on materials and methods for use thereof may be found in U.S. Pat. No. 6,330,916, which is incorporated herein by reference. Further information on materials and methods may also be found in U.S. Patent No. 6,364,018.</p> <p>U.S. Pat. No 6,330,916 (Rickards), Abstract: A subterranean formation is treated by injecting a blend of fracture proppant</p>                                                                                                                                                                                                                                                                                                                       |



U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                 | material and deformable particulate material into the formation. The deformable particulate material may combine with the fracture proppant material to increase fracture conductivity, reduce fines generation, and/or reduce proppant flowback. The fracture proppant material may be a material such as sand . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |
| <b>Claim 51 of U.S. Patent 8,236,738</b>                                                                                        | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |
| 51. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:                     | <p>P. 2, l. 4-8: This invention relates generally to methods and compositions useful for subterranean formation treatments, such as hydraulic fracturing treatments and sand control. In particular, this invention relates to the use of relatively lightweight and/or substantially neutrally buoyant particles as proppant materials in hydraulic fracturing treatment and as particulate material in sand control methods such as gravel packing, frac pack treatments, etc.</p> <p>P. 13, l. 22-25: Such other treatments may be near wellbore in nature (affecting near wellbore regions) and may be directed towards improving wellbore productivity and/or controlling the production of fracture proppant or formation sand. Particular examples include gravel packing and “frac-packs.”</p> <p>P. 3, l. 13-16: In the disclosed method, the application of relatively lightweight and/or substantially neutrally buoyant particulate material as a fracture proppant particulate advantageously may provide for substantially improved overall system performance in hydraulic fracturing applications, or in other well treating applications such as sand control.</p> |
| Contacting particulates with a medium containing a chemical compound for rendering the surface of the particulates hydrophobic, | P. 7, l. 3-8: In a further embodiment, individual particles . . . may be optionally treated by exposure to a modifying agent that                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                             | <p>is capable of interacting with compounds present in or on a natural material . . .</p> <p>P. 27, l. 1-2: Modifying agents may be diluted as so desired in a suitable solvent prior to treating particles of a naturally-occurring material.</p> <p>P. 24, l. 17-26: Specific examples of suitable exposure methods include, but are not limited to, by soaking the naturally-occurring material in a modifying agent . . . by dipping the naturally-occurring material in a modifying agent, by spraying a modifying agent on to the naturally-occurring material . . . by mixing naturally-occurring particulate material with liquid, atomized and/or gaseous modifying agent in a vertical mixer . . . by flowing modifying agent through a permeable bed or pack of the naturally-occurring particles in a pressurized container. Drying of particles after exposure may be accomplished, for example, by continuously dropping through heated air...</p> |
| Separating the particulate from the medium; | <p>P. 24, l. 17-26: Specific examples of suitable exposure methods include, but are not limited to, by soaking the naturally-occurring material in a modifying agent . . . by dipping the naturally-occurring material in a modifying agent, by spraying a modifying agent on to the naturally-occurring material . . . by mixing naturally-occurring particulate material with liquid, atomized and/or gaseous modifying agent in a vertical mixer . . . by flowing modifying agent through a permeable bed or pack of the naturally-occurring particles in a pressurized container. Drying of particles after exposure may be accomplished, for example, by continuously dropping through heated air...</p>                                                                                                                                                                                                                                                    |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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| <p>And blending the hydrophobic particulate with water and an oil to form a mixture;</p> | <p>P. 13, l. 6-13: The particles disclosed herein . . . may be mixed and pumped during any desired portion/s of a well treatment (e.g., hydraulic fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water, liquid hydrocarbons, and/or nitrogen or other gases.</p> <p>P. 33, l. 6-8: Incorporates by reference Graham (U.S. Patent 3,659,651)</p> <p>Graham, Col. 5, l. 9-16: In placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or <b>water-in-oil emulsion</b> prepared in accordance with the teachings of U.S. Pat. No. 3,378,074 (Kiel)</p> <ul style="list-style-type: none"> <li>Kiel, Col. 5, l. 66-73: High viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either water-in-oil or oil-in-water emulsions. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine or an acid solution containing a suitable surface active agent or emulsifier.</li> </ul> |
| <p>And pumping the mixture into the formation.</p>                                       | <p>P. 13, l. 6-10: The particles disclosed herein (e.g., relatively lightweight and/or substantially neutrally buoyant proppant or sand control particulates, and/or particulates of naturally-</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                      | <p>occurring material that have been exposed to a modifying agent) may be mixed and pumped during any desired portion/s of well treatment (<i>e.g.</i>, hydraulic fracturing treatment, sand control treatment, <i>etc.</i>) and may be mixed in any desired concentration with a carrier fluid.</p> <p>P. 29, l. 27-28: In another exemplary embodiment, modified particulate material may be formulated as described herein and introduced or pumped into a well...</p>                                                                                                                                                                                                                                                      |
| <b>Claim 57 of U.S. Patent 8,236,738</b>                             | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |
| 57. The composition of claim 51, wherein the particulates are sands. | <p>P. 13, l. 3-5: Additional information on materials and methods for use thereof may be found in U.S. Pat. No. 6,330,916, which is incorporated herein by reference. Further information on materials and methods may also be found in U.S. Patent No. 6,364,018.</p> <p>U.S. Pat. No 6,330,916 (Rickards), Abstract: A subterranean formation is treated by injecting a blend of fracture proppant material and deformable particulate material into the formation. The deformable particulate material may combine with the fracture proppant material to increase fracture conductivity, reduce fines generation, and/or reduce proppant flowback. The fracture proppant material may be a material such as sand . . .</p> |

# EXHIBIT E

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2005/0244641 (Vincent)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

| Claim 1 of U.S. Patent 8,236,738                                                   | Reference to U.S. Publication 2005/0244641 (Vincent)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |
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| 1. An aqueous slurry composition comprising:                                       | [0087] The fracturing fluid may be . . . water base . . . [P]articles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |
| water;                                                                             | [0087] The fracturing fluid may be an oil base, water base, acid emulsion, foam, or any other fluid.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |
| particulates;                                                                      | [0002] The present invention relates to oil and gas well proppants and, more particularly, to processes for physically or chemically modifying surface characteristics of hydraulic fracturing proppants.<br><br>[0087] The fracturing fluid may be . . . water base . . . [P]articles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.                                                                                                                                                                                                                                                      |
| a chemical compound for rendering the surface of the particulates hydrophobic; and | [0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin-coated sand, and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability, alter the chemical reactivity, alter the surface topography, impart lubricity, and control relative permeability to flow of fluids such as proppants. Sands, resin coated sands, or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, the modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2005/0244641 (Vincent)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>[0009] These benefits can be achieved by a variety of techniques, including coating the proppants with a hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes...</p> <p>[0043] Coated samples of a sintered bauxite proppant commercially available from CARBO Ceramics Inc. . . . were prepared by coating the proppant with a product that is commercially available from SOPUS Products under the tradename "Rain-X®". Rain-X® is a glass surface treatment material that includes polyalkyl hydrogen siloxane, ethanol and isopropanol.</p> <p>[0082] First, in both gel-contaminated and uncontaminated packs, the Rain-X® treated proppant initially allowed reduced flowtimes. Secondly, both samples treated with Rain-X® showed significantly increasing flowtimes to water with subsequent flushes. It was visually observed that the Rain-X® coated proppant trapped air bubbles that accumulated over time. It was clear that the proppant was hydrophobic.</p> <p>Claim 1: A gas and oil well proppant comprising: a plurality of essentially spherical particles, wherein the particles are coated with a hydrophobic material.</p> <p>Claim 6: The proppant of claim 2, wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and polydimethyl siloxane.</p> <p>Claims 3-5, 9-10, and 15-17 also meet this limitation.</p> |
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U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2005/0244641 (Vincent)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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| <p>an oil.</p> | <p>[0009] These benefits can be achieved by a variety of techniques, including coating the proppants with a hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes, polytetrafluoroethylene (commonly known as Teflon®), plant oils, such as linseed oil, soybean oil, corn oil, cottonseed oil, vegetable oil (widely commercially available such as Crisco®), and canola oil, and hydrocarbons such as kerosene, diesel, and crude oil, petroleum distillates such as hydrocarbon liquids comprising a mixture of C<sub>7</sub>-C<sub>12</sub> aliphatic and alicyclic hydrocarbons and aromatic hydrocarbons (C<sub>7</sub>-C<sub>12</sub>), commonly known as Stoddard Solvent, aliphatic solvents, solvent naphtha (medium aliphatic and light aromatic), and paraffin, such as solvent dewaxed heavy paraffinic petroleum distillate.</p> <p>[0087] The fracturing fluid may be an oil base, water base, acid emulsion, foam, or any other fluid.</p> <p>Claim 1: A gas and oil well proppant comprising: a plurality of essentially spherical particles, wherein the particles are coated with a hydrophobic material.</p> <p>Claim 2: The proppant of claim 1, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, hydrocarbons, copolymerized polyvinylidene chloride, glass frit and enamel.</p> <p>Claim 10: The method of claim 9, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes,</p> |
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U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2005/0244641 (Vincent)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                                                                                                    | <p>polytetrafluoroethylene, plant oil, hydrocarbons, copolymerized, polyvinylidene chloride, glass frit and enamel.</p> <p>Claim 21: The method of claim 20, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, hydrocarbons, copolymerized polyvinylidene chloride, glass frit and enamel.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin-coated sand and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability . . .</p> <p>[0012], [0014] Thus, various embodiments of the present invention relate to concepts and techniques to treat fracturing sand and/or proppant to: . . . 2) alter or modify proppant wettability to control the relative permeability to flow of the fluids which may be encountered in the reservoir (such as oil, water, gas, chemical treatments, and fracturing fluids).</p> |
| <b>Claim 21 of U.S. Patent 8,236,738</b>                                                                                                                                                                           | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |
| 21. The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates. | <p>[0007] The present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are natural sands or ceramic granules used in the hydraulic fracturing of oil and gas wells.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin coated sand and manufactured proppants used in oil and gas recovery to</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2005/0244641 (Vincent)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                     | <p>achieve one or more of the following desirable effects: alter the wettability . . . Sands, resin coated sands or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.</p> <p>[0087] Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.</p>                                                                                                                                                                                                                                                                                                                                                                                                              |
| <b>Claim 22 of U.S. Patent 8,236,738</b>                            | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |
| 22. The composition of claim 1, wherein the particulates are sands. | <p>[0007] The present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are natural sands or ceramic granules used in the hydraulic fracturing of oil and gas wells.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin coated sand and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability . . . Sands, resin coated sands or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.</p> <p>[0087] Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.</p> |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2005/0244641 (Vincent)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

| Claim 32 of U.S. Patent 8,236,738                                                                                                | Reference to U.S. Publication 2005/0244641 (Vincent)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |
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| <p>32. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:</p>               | <p>[0012], [0014], [0015] Thus, various embodiments of the present invention relate to concepts and techniques to treat fracturing sand and/or proppant to: . . . 2) alter or modify proppant wettability, to control the relative permeability to flow of the fluids which may be encountered in the reservoir (such as oil, water, gas, chemical treatments, and fracturing fluids), 3) “lubricate” the proppant to allow more efficient proppant arrangement when the fracture closes, effectively increasing packing efficiency . . .</p> <p>[0028] The “lubrication” concept reduces proppant friction, allowing superior proppant redistribution during fracture closing. This redistribution allows more efficient packing of proppant, thereby increasing grain-to-grain contact and effectively increasing proppant pack strength and reducing proppant crush.</p> |
| <p>mixing water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic and an oil; and</p> | <p>[0087] The fracturing fluid may be an oil base, water base, acid emulsion, foam, or any other fluid. . . . Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which particles have previously been introduced and suspended.</p> <p>[0002] The present invention relates to oil and gas well proppants and, more particularly, to processes for physically or chemically modifying the surface characteristics of hydraulic fracturing proppants.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin-coated sand, and manufactured proppants used in oil and gas recovery</p>                                                                                                                                          |

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 Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>to achieve one or more of the following desirable effects: alter the wettability, alter the chemical reactivity, alter the surface topography, impart lubricity, and control relative permeability to flow of fluids such as proppants. Sands, resin coated sands, or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, the modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties</p> <p>[0009] These benefits can be achieved by a variety of techniques, including coating the proppants with a hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes, polytetrafluoroethylene (commonly known as Teflon®), plant oils, such as linseed oil, soybean oil, corn oil, cottonseed oil, vegetable oil (widely commercially available such as Crisco®), and canola oil, and hydrocarbons such as kerosene, diesel, and crude oil, petroleum distillates such as hydrocarbon liquids comprising a mixture of C<sub>7</sub>-C<sub>12</sub> aliphatic and alicyclic hydrocarbons and aromatic hydrocarbons (C<sub>7</sub>-C<sub>12</sub>), commonly known as Stoddard Solvent, aliphatic solvents, solvent naphtha (medium aliphatic and light aromatic), and paraffin, such as solvent dewaxed heavy paraffinic petroleum distillate.</p> <p>[0029] According to embodiments of the present invention, coatings affect wettability and provide significant flow benefits under multiphase flow as evidenced by the trapped gas saturation, the altered surface tension/contact angles, and the electrostatic charges on the coated proppant.</p> <p>[0043] Coated samples of a sintered bauxite proppant commercially available from CARBO Ceramics Inc. . . . were</p> |
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U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2005/0244641 (Vincent)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>prepared by coating the proppant with a product that is commercially available from SOPUS Products under the tradename "Rain-X®". Rain-X® is a glass surface treatment material that includes polyalkyl hydrogen siloxane, ethanol and isopropanol.</p> <p>[0082] First, in both gel-contaminated and uncontaminated packs, the Rain-X® treated proppant initially allowed reduced flowtimes. Secondly, both samples treated with Rain-X® showed significantly increasing flowtimes to water with subsequent flushes. It was visually observed that the Rain-X® coated proppant trapped air bubbles that accumulated over time. It was clear that the proppant was hydrophobic.</p> <p>Claim 2: The proppant of claim 1, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, hydrocarbons, copolymerized polyvinylidene chloride, glass frit and enamel.</p> <p>Claim 6: The proppant of claim 2, wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and polydimethyl siloxane.</p> <p>Claim 10: The method of claim 9, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, hydrocarbons, copolymerized, polyvinylidene chloride, glass frit and enamel.</p> |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                                                                      | <p>Claim 21: The method of claim 20, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, hydrocarbons, copolymerized polyvinylidene chloride, glass frit and enamel.</p> <p>Claims 1-6, 9-10, and 15-17 also meet this limitation.</p>                                                                                                                                                                                                                                                                                                                                                                                                                                        |
| pumping the mixture into the formation.                                                                                                                                              | <p>Claim 9: A method of fracturing a subterranean formation, comprising: injecting a hydraulic fluid into a subterranean formation at a rate and pressure sufficient to open a fracture therein; and injecting into the fracture a fluid containing a plurality of essentially spherical particles, wherein at least some of the particles are modified to alter the surface characteristics thereof, wherein the particles are modified by coating the particles with a hydrophobic material.</p>                                                                                                                                                                                                                                                                                                      |
| <b>Claim 40 of U.S. Patent 8,236,738</b>                                                                                                                                             | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |
| 40. The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates. | <p>[0007] The present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are natural sands or ceramic granules used in the hydraulic fracturing of oil and gas wells.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin coated sand and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability . . . Sands, resin coated sands or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.</p> |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                             | [0087] Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                |
| <b>Claim 41 of U.S. Patent 8,236,738</b>                                                                    | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |
| 41. The method of claim 32, wherein the particulates are sands.                                             | <p>[0007] The present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are natural sands or ceramic granules used in the hydraulic fracturing of oil and gas wells.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin coated sand and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability . . . Sands, resin coated sands or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.</p> <p>[0087] Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.</p> |
| <b>Claim 51 of U.S. Patent 8,236,738</b>                                                                    | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |
| 51. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: | [0012], [0014], [0015] Thus, various embodiments of the present invention relate to concepts and techniques to treat fracturing sand and/or proppant to: . . . 2) alter or modify proppant wettability, to control the relative permeability to flow of the fluids which may be encountered in the reservoir (such as oil,                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |

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|                                                                                                                                        | <p>water, gas, chemical treatments, and fracturing fluids), 3) “lubricate” the proppant to allow more efficient proppant arrangement when the fracture closes, effectively increasing packing efficiency . . .</p> <p>[0028] The “lubrication” concept reduces proppant friction, allowing superior proppant redistribution during fracture closing. This redistribution allows more efficient packing of proppant, thereby increasing grain-to-grain contact and effectively increasing proppant pack strength and reducing proppant crush.</p>                                                                                                                                                                                                    |
| <p>Contacting particulates with a medium containing a chemical compound for rendering the surface of the particulates hydrophobic,</p> | <p>[0023], [0024] Thus, exemplary techniques for treating proppant include but are not limited to: 1) pretreating proppant prior to the fracturing treatment,</p> <p>[0036] In each case, the coating was applied by mixing the proppant and the coating in a beater for approximately 30 minutes, then drying it for approximately 15 to 18 hours in an oven.</p> <p>[0037] The coating materials were added as follows. Polymethylhydrogen siloxane was added as either a 2 or 5 weight percent emulsion of siloxane in water, polydimethyl siloxane was added as a 5 weight percent emulsion of siloxane in water and Stoddard Solvent was added without water dilution. All samples were dried at 113° C. for approximately 15 to 18 hours.</p> |
| <p>Separating the particulate from the medium;</p>                                                                                     | <p>[0036] In each case, the coating was applied by mixing the proppant and the coating in a beaker for approximately 30 minutes, then drying it for approximately 15 to 18 hours in an oven.</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |



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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                   | <p>[0037] The coating materials were added as follows. Polymethylhydrogen siloxane was added as either a 2 or 5 weight percent emulsion of siloxane in water, polydimethyl siloxane was added as a 5 weight percent emulsion of siloxane in water and Stoddard Solvent was added without water dilution. All samples were dried at 113° C. for approximately 15 to 18 hours.</p>                                                                                                                   |
| And blending the hydrophobic particulate with water and an oil to form a mixture; | <p>[0087] The fracturing fluid may be an oil base, water base, acid emulsion, foam, or any other fluid. . . . Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.</p>                                                                                                                                                                            |
| And pumping the mixture into the formation.                                       | <p>Claim 9: A method of fracturing a subterranean formation, comprising: injecting a hydraulic fluid into a subterranean formation at a rate and pressure sufficient to open a fracture therein; and injecting into the fracture a fluid containing a plurality of essentially spherical particles, wherein at least some of the particles are modified to alter the surface characteristics thereof, wherein the particles are modified by coating the particles with a hydrophobic material.</p> |
| <b>Claim 57 of U.S. Patent 8,236,738</b>                                          | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                        |
| 57. The composition of claim 51, wherein the particulates are sands               | <p>[0007] The present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are natural sands or ceramic granules used in the hydraulic fracturing of oil and gas wells.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin coated sand and manufactured proppants used in oil and gas recovery to</p>                                                                                |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2005/0244641 (Vincent)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>achieve one or more of the following desirable effects: alter the wettability . . . Sands, resin coated sands or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.</p> <p>[0087] Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.</p> |
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# EXHIBIT F

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2006/0260808 (Weaver)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

| Claim 1 of U.S. Patent 8,236,738             | Reference to U.S. Publication 2006/0260808 (Weaver)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |
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| 1. An aqueous slurry composition comprising: | [0058] In certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |
| water;                                       | <p>[0058] In certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprise of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> |
| particulates;                                | Abstract: Provided are methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates. . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                    | [0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |
| a chemical compound for rendering the surface of the particulates hydrophobic; and | <p>[0015] A mineral surface whose reactivity has been modified according to this definition is herein referred to as “a modified mineral surface.” In certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic or hydrophilic in nature.</p> <p>[0049] In some embodiments, the surface-treating reagent may comprise other compounds that are capable of hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include, but are not limited to, vinyl monomers, dienes, keto esters, amines, substituted amine hydrochlorides, amides, alcohols, organosilanes, organotitanates, organozirconates, trivalent metal cations, tetravalent metal cations, ammonium halides, quaternary ammonium halides, ammonium salts of inorganic acids, ammonium salts of carboxylic acids, oligomeric materials, monomeric materials, oil-wetting compounds, and derivatives thereof. In some embodiments, the surface-treating reagent may comprise lecithin. In some embodiments, the surface-treating reagent may comprise a chlorosilyl group containing compound and an alkylsilane, or the reaction products thereof. In some embodiments, the surface-treating reagent may comprise an organofunctional silane and an aryl acid halide, or the reaction</p> |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|         | products thereof. Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |
| an oil. | <p>[0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> <p>[0054] In certain embodiments of the present invention, the surface-treating reagent may be encapsulated with various materials, which, among other things, delays its reaction with the mineral surface and/or other substances present. Solid surface-treating reagents can be encapsulated by spray coating a variety of materials thereon. Such coating materials include, but are not limited to, waxes, drying oils such as tung oil and linseed oil,</p> |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2006/0260808 (Weaver)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                                                                                                           | <p>polyurethanes and cross-linked partially hydrolyzed polyacrylics.</p> <p>[0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.</p> <p>Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid into the subterranean formation.</p> |
| <b>Claim 21 of U.S. Patent 8,236,738</b>                                                                                                                                                                                  | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |
| <p>21. The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates.</p> | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, cured resinous particulates comprising fruit pit</p>                                                                                                                                                                                         |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2006/0260808 (Weaver)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                     | <p>pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p> |
| <b>Claim 22 of U.S. Patent 8,236,738</b>                            | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| 22. The composition of claim 1, wherein the particulates are sands. | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p>                                                                   |



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| Claim 32 of U.S. Patent 8,236,738                                                                                                | Reference to U.S. Publication 2006/0260808 (Weaver)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |
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| <p>32. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of:</p>               | <p>[0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.</p> <p>[0004] One problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, proppant particulates, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channel and/or damage the interior of the formation or equipment placed in the formation.</p> <p>[0014] Thus, one object of the present invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, preventing build-up of filter cake and/or gel residue in conductive channels (e.g., fractures) within subterranean formation, and preventing the embedment of particulates into the surfaces of a subterranean formation.</p> |
| <p>mixing water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic and an oil; and</p> | <p>Abstract: Provided are methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates, at least one of which comprises a mineral surface; providing a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; and allowing the surface-treating reagent</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |

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|  | <p>modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate.</p> <p>[0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprised of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> <p>[0056] This may be accomplished in treatments performed prior to transporting the particulates to a job site, or in a treatment performed “on-the-fly.” The term “on-the-fly” is used herein to mean that one flowing stream comprising particulates is continuously introduced into another flowing stream comprising the surface-treating reagent so that the streams are combined and mixed while continuing to flow as a single stream as part of the</p> |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>on-going treatment at the job site. Such mixing can also be described as “real-time” mixing.</p> <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.</p> <p>[0015] A mineral surface whose reactivity has been modified according to this definition is herein referred to as “a modified mineral surface.” In certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, by</p> |
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U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2006/0260808 (Weaver)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                         | <p>hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic or hydrophilic in nature.</p> <p>[0049] In some embodiments, the surface-treating reagent may comprise other compounds that are capable of hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include, but are not limited to, vinyl monomers, dienes, keto esters, amines, substituted amine hydrochlorides, amides, alcohols, organosilanes, organotitanates, organozirconates, trivalent metal cations, tetravalent metal cations, ammonium halides, quaternary ammonium halides, ammonium salts of inorganic acids, ammonium salts of carboxylic acids, oligomeric materials, monomeric materials, oil-wetting compounds, and derivatives thereof. In some embodiments, the surface-treating reagent may comprise lecithin. In some embodiments, the surface-treating reagent may comprise a chlorosilyl group containing compound and an alkylsilane, or the reaction products thereof. In some embodiments, the surface-treating reagent may comprise an organofunctional silane and an aryl acid halide, or the reaction products thereof. Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.</p> |
| pumping the mixture into the formation. | <p>Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2006/0260808 (Weaver)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                                                                      | <p>surface of at least one particulate; and introducing the treatment fluid into the subterranean formation.</p> <p>Claim 23: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified material surface; and introducing the treatment fluid into the subterranean formation.</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |
| <b>Claim 40 of U.S. Patent 8,236,738</b>                                                                                                                                             | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |
| 40. The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates. | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.</p> |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2006/0260808 (Weaver)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                             | [0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
| <b>Claim 41 of U.S. Patent 8,236,738</b>                                                                    | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |
| 41. The method of claim 32, wherein the particulates are sands.                                             | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p> |
| <b>Claim 51 of U.S. Patent 8,236,738</b>                                                                    | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |
| 51. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: | <p>[0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.</p> <p>[0004] One problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for</p>                                                                                                                                                                                                                                                                                                                                                                                                |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2006/0260808 (Weaver)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                 | <p>particulates (e.g., formation fines, proppant particulates, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channel and/or damage the interior of the formation or equipment placed in the formation.</p> <p>[0014] Thus, one object of the present invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, preventing build-up of filter cake and/or gel residue in conductive channels (e.g., fractures) within subterranean formation, and preventing the embedment of particulates into the surfaces of a subterranean formation.</p> |
| Contacting particulates with a medium containing a chemical compound for rendering the surface of the particulates hydrophobic, | <p>[0057] Other suitable methods for allow the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate include, but are not limited to, methods utilizing a drive mixer and solvent extraction methods. . . . The solution comprising the surface-treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced into a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface...</p>                                          |
| Separating the particulate from the medium;                                                                                     | <p>[0057] Other suitable methods for allow the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate include, but are not limited to, methods utilizing a drive mixer and solvent extraction methods. . . . The solution comprising the surface-treating reagent then may be</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2006/0260808 (Weaver)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                   | coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced into a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface...                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| And blending the hydrophobic particulate with water and an oil to form a mixture; | <p>[0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprised of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> |
| And pumping the mixture into the formation.                                       | Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |



U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2006/0260808 (Weaver)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                      | <p>modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid into the subterranean formation.</p> <p>Claim 23: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified material surface; and introducing the treatment fluid into the subterranean formation.</p>                                                                                                                                                                                                                                 |
| <b>Claim 57 of U.S. Patent 8,236,738</b>                             | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |
| 57. The composition of claim 51, wherein the particulates are sands. | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p> |

# EXHIBIT G

U.S. Patent 8,236,738 (Zhang) v. U.S. Patent 4,231,428 (Needham)  
Challenged Claims: 1, 21, 22

| Claim 1 of U.S. Patent 8,236,738                                                   | Reference to U.S. Patent 4,231,428 (Needham)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |
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| 1. An aqueous slurry composition comprising:                                       | Col. 3, l. 19-23: Carrier fluid, preferably an aqueous fluid such as salt water or a hydrocarbon liquid such as diesel fuel, having the proppant coated with organo-silicon compound suspended therein is then pumped into the fracture.                                                                                                                                                                                                                                                                                                                                                                  |
| water;                                                                             | Col. 3, l. 19-23: Carrier fluid, preferably an aqueous fluid such as salt water or a hydrocarbon liquid such as diesel fuel, having the proppant coated with organo-silicon compound suspended therein is then pumped into the fracture.<br><br>Col. 4, l. 1-7: In a method of fracturing an underground zone or formation in oil and gas wells, the steps of (a) injecting into a well a fracturing fluid to form a fracture in said formation, (b) injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound . . . |
| particulates;                                                                      | Col. 3, l. 5-12: Propping agents used in hydraulic fracturing normally range in particle size from about 8 to about 60 mesh, with the most common range being between 20 and 40 mesh. Sand is by far the most common propping agent currently used because of its low cost and great abundance. However, under certain conditions, light strength glass beads, as well as bauxite (aluminum oxide), or other propping agents can be used.                                                                                                                                                                 |
| a chemical compound for rendering the surface of the particulates hydrophobic; and | Col. 1, l. 44-52: Accordingly, an object of this invention is to provide improved propping agents for use in hydraulic fracturing of subterranean formations. Another object of this invention is to provide particulate material coated with a water repellent useful in hydraulic fracturing of subterranean formations. A further object of this invention is to coat particulate solids useful                                                                                                                                                                                                        |

U.S. Patent 8,236,738 (Zhang) v. U.S. Patent 4,231,428 (Needham)  
Challenged Claims: 1, 21, 22

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|  | <p>as propping agents to increase the strength of the propping agent in the presence of water.</p> <p>Col. 1, l. 56-68: In accordance with the invention, a novel propping agent comprising an inert solid particle coated with a water-repellent organo-silicon material is displaced down a well and into a fracture in a subterranean hydrocarbon-bearing formation. In accordance with a specific embodiment, sand coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.</p> <p>Col. 2, l. 25-41: The water-repellent material employed in the method of the present invention to coat inert particulate materials for use as propping agents is an oil-soluble organo-silicon compound. Representative organo-silicon compounds that can be used include polyalkylsiloxanes such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additionally, polyalkylarylsiloxanes such as polymethylphenylsiloxane are also usable as a coating material of the invention. Chlorosilanes such as chlorotrimethylsilane and other silyl donors can also be used, as well as various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes, and the like, as the water-repellent coating of the propping agent. Other organo-silicon oil-soluble compounds which can be used include the ethyl silicates, methyl sodium silanolate, and other silicon resins such as mixtures of silane esters, silyl amines, and the like.</p> |
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U.S. Patent 8,236,738 (Zhang) v. U.S. Patent 4,231,428 (Needham)  
Challenged Claims: 1, 21, 22

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| an oil.                                                                                                                                                                                                            | <p>Col. 4, l. 1-2: In a method of fracturing an underground zone or formation in oil and gas wells . . .</p> <p>Col. 1, l. 4-5: This invention relates to the treatment of subterranean hydrocarbon-bearing formations.</p> <p>Col. 1, l. 56-60: In accordance with the invention, a novel propping agent comprising an inert solid particle coated with a water-repellent organo-silicon material is displaced down a well and into a fracture in a subterranean hydrocarbon-bearing formation.</p>                                                                                                                                                                                                                                                                                                                                                                                                  |
| <b>Claim 21 of U.S. Patent 8,236,738</b>                                                                                                                                                                           | <b>Reference to U.S. Patent 4,231,428 (Needham)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |
| 21. The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates. | <p>Col. 1, l. 61-68: In accordance with a specific embodiment, <i>sand</i> coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.</p> <p>Col. 4:1-7: In a method of fracturing an underground zone or formation in oil and gas wells, the steps of (a) injecting into a well a fracturing fluid to form a fracture in said formation, (b) injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound . . .</p> <p>Col. 4, l. 16-17: A method according to claim 1 wherein said particulate material comprises sand . . .</p> |

U.S. Patent 8,236,738 (Zhang) v. U.S. Patent 4,231,428 (Needham)  
Challenged Claims: 1, 21, 22

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|                                                                     | Col. 4, l. 48-49: A method according to claim 5 wherein said particles comprise sand . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |
| <b>Claim 22 of U.S. Patent 8,236,738</b>                            | <b>Reference to U.S. Patent 4,231,428 (Needham)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |
| 22. The composition of claim 1, wherein the particulates are sands. | <p>Col. 1, l. 61-68: In accordance with a specific embodiment, <i>sand</i> coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.</p> <p>Col. 4:1-7: In a method of fracturing an underground zone or formation in oil and gas wells, the steps of (a) injecting into a well a fracturing fluid to form a fracture in said formation, (b) injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound . . .</p> <p>Col. 4, l. 16-17: A method according to claim 1 wherein said particulate material comprises sand . . .</p> <p>Col. 4, l. 48-49: A method according to claim 5 wherein said particles comprise sand . . .</p> |

# EXHIBIT H

U.S. Patent 8,236,738 v. U.S. Publication 2004/0023818 (Nguyen) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

| Claim 1 of U.S. Patent 8,236,738             | Reference to U.S. Publication 2004/0023818 (Nguyen)                                                                                                                                                                                                                                                                                                                                    | Reference to U.S. Publication 2006/0260808 (Weaver)                                                                                                                                                                                                                                                                                                             | Reference to U.S. Patent 3,980,136 (Plummer) |
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| 1. An aqueous slurry composition comprising: | <p>Claim 63: ... mixing particles in a carrier fluid comprising a water-based polymer to form a mixture . . .</p> <p>[0013] The water utilized to form the fracturing fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other aqueous liquid which does not adversely react [to] other components of the fracturing fluid.</p> | [0058] In certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof.                         |                                              |
| water;                                       | <p>[0011] Fracturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and emulsions.</p> <p>[0012] The most common fracturing fluid utilized heretofore which is generally preferred for use in accordance with this invention is comprised of water . . .</p>                                  | [0058] In certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is |                                              |



U.S. Patent 8,236,738 v. U.S. Publication 2004/0023818 (Nguyen) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>[0013] The water utilized to form the fracturing fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other aqueous liquid which does not adversely react [to] other components of the fracturing fluid.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surface of the particles.</p> <p>Claim 65: The method of claim 63 wherein the carrier fluid</p> | <p>aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprise of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> |  |
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U.S. Patent 8,236,738 v. U.S. Publication 2004/0023818 (Nguyen) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|               | comprises water and a gelling agent.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                                                                                                                                                                                                                                                                                                                                                                              |  |
| particulates; | <p>[0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like. Generally the particles used have a particle size in the range of from about 2 to about 400 mesh, U.S. Sieve Series. The preferred particles are sand having a particle size in the range of from about 10 to about 70 mesh, U.S. Sieve Series.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped</p> | <p>Abstract: Provided are methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates. . .</p> <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface.</p> |  |

U.S. Patent 8,236,738 v. U.S. Publication 2004/0023818 (Nguyen) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                    | into the well to promote the recovery of hydrocarbons.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |  |
| a chemical compound for rendering the surface of the particulates hydrophobic; and | <p>[0005] According to an embodiment, the particles are dry coated with a water-repellent organo-silicon material.</p> <p>[0006] Representative organo-silicon compounds that can be used in this embodiment include polyalkylsiloxane such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additional organo-silicon compounds that can be used in this embodiment include polyalkylarylsiloxanes, [] chlorosilanes [], chlorotrimethylsilane and other silyl donors. Also, various alkoxy silanes, aroxy silanes, alkoxy siloxanes, and aroxy siloxanes can be used such as tetraethoxysilane, dimethoxydiphenylsilane, dichlorodimethylsilane, dichlorodiphenylsilane, poly[oxy(dimethylsilylene)] and other such materials that will be well known to those</p> | <p>[0015] A mineral surface whose reactivity has been modified according to this definition is herein referred to as “a modified mineral surface.” In certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic or hydrophilic in nature.</p> <p>[0049] In some embodiments, the surface-treating reagent may comprise other compounds that are capable of</p> |  |

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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>skilled in the art. . . .<br/> Furthermore, organofunctional silanes can be used in this embodiment...</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> | <p>hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include, but are not limited to, vinyl monomers, dienes, keto esters, amines, substituted amine hydrochlorides, amides, alcohols, organosilanes, organotitanates, organozirconates, trivalent metal cations, tetravalent metal cations, ammonium halides, quaternary ammonium halides, ammonium salts of inorganic acids, ammonium salts of carboxylic acids, oligomeric materials, monomeric materials, oil-wetting compounds, and derivatives thereof. In some embodiments, the surface-treating reagent may comprise lecithin. In some embodiments, the surface-treating reagent may comprise a chlorosilyl group containing compound and an alkylsilane, or the reaction products thereof. In some embodiments, the surface-treating reagent may comprise an organofunctional silane and an</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|         |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | aryl acid halide, or the reaction products thereof. Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |
| an oil. | <p>[0011] Fracturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and emulsions. The foams utilized are generally comprised of water based liquids containing one or more foaming agents foamed with a gas such as nitrogen or air. Emulsions formed with two or more immiscible liquids have also been utilized.</p> <p>[0008] If the particles are coated when the particles are flowing, the solvent should be readily miscible in the water-based gel carrier fluid. Organic solvents that can be used as a carrier for the organo-silicon coating material</p> | <p>[0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the</p> | <p>Abstract: Foam fracturing fluids and methods for fracturing well formations using foam as the carrier for the propping agents are disclosed.</p> <p>Col. 4, l. 28-33: Oil may be added to the foam-water slurry as a lubricant and to prevent clay swelling; oil may replace all of the water in the sand slurry, in which case a chemical suitable for foaming oil is used in place of the surfactant. Other friction reducing materials may be added to the fracturing fluid.</p> <p>Col. 7, l. 16-20: To summarize, the invention provides a formation fracturing fluid, comprising a pressured stable</p> |

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|  | <p>include kerosene, lighter grades of diesel fuel, hexane, xylene . . . and other such solvents that will be well known to those skilled in the art.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of the hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p> <p>[0001] After most oil and gas wells are drilled, they do not produce hydrocarbons at a rate to provide satisfactory economic return. Therefore, the oil industry uses a process known as hydraulic fracture stimulation to generate fractures deep into the hydrocarbon-bearing rock formations, which provides</p> | <p>like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> <p>[0054] In certain embodiments of the present invention, the surface-treating reagent may be encapsulated with various materials, which, among other things, delays its reaction with the mineral surface and/or other substances present. Solid surface-treating reagents can be encapsulated by spray coating a variety of materials thereon. Such coating</p> | <p>foam comprising a liquid carrier comprising one or more liquids selecting of the group consisting of water and oil, and a solid proppant . . .</p> <p>Col. 8, l. 30-32: Method according to claim 1, wherein the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.</p> |
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|  | highly conductive flow channels to the well. | <p>materials include, but are not limited to, waxes, drying oils such as tung oil and linseed oil, polyurethanes and cross-linked partially hydrolyzed polyacrylics.</p> <p>[0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.</p> <p>Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a</p> |  |
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|                                                                                                                                                                                                                    |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid into the subterranean formation.                                                                                                                                                                                                                                                                                                                                                      |                                                     |
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| <b>Claim 21 of U.S. Patent 8,236,738</b>                                                                                                                                                                           | <b>Reference to U.S. Publication 2004/0023818 (Nguyen)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 21. The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates. | <p>[0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the</p> | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell</p> |                                                     |



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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>recovery of hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Claim 2: The method of claim 1 wherein the particles are pumped downhole to a screen and function as a gravel pack.</p> <p>Claim 3: The method of claim 1 wherein the particles are pumped downhole to a fracture in a formation adjacent the well and function as a proppant.</p> <p>Claim 8: The method of claim 1 wherein the particles are</p> | <p>pieces, cured resinous particulates comprising nut shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a</p> |  |
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|                                                                     | <p>selected from the group consisting of sand and gravel.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> | <p>solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p>                                                                         |                                                     |
| <b>Claim 22 of U.S. Patent 8,236,738</b>                            | <b>Reference to U.S. Publication 2004/0023818 (Nguyen)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                             | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 22. The composition of claim 1, wherein the particulates are sands. | [0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand,                                                                                                                                                                                                                                              | [0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical |                                                     |

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|  | <p>bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Claim 2: The method of claim 1 wherein the particles are pumped downhole to a screen and function as a gravel pack.</p> | <p>materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>Claim 3: The method of claim 1 wherein the particles are pumped downhole to a fracture in a formation adjacent the well and function as a proppant.</p> <p>Claim 8: The method of claim 1 wherein the particles are selected from the group consisting of sand and gravel.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> |  |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

| Claim 32 of U.S. Patent 8,236,738                                                                           | Reference to U.S. Publication 2004/0023818 (Nguyen)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | Reference to U.S. Publication 2006/0260808 (Weaver)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | Reference to U.S. Patent 3,980,136 (Plummer) |
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| 32. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: | <p>[0005] According to an embodiment, the particles are dry coated with a water-repellent organo-silicon material. The treated particles are then pumped down to the fractures to function as a proppant or to the wellbore-screen annulus to function as a gravel pack.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of hydrocarbons.</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> | <p>[0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.</p> <p>[0004] One problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, proppant particulates, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation or equipment placed in the formation.</p> |                                              |

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|                                                                                                                           |                                                                                                                                                                                                                                                                                                                                     | [0014] Thus, one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, preventing build-up of filter cake and/or gel residue in conductive channels (e.g., fractures) within a subterranean formation, and preventing the embedment of particulates into the surfaces of a subterranean formation. |                                                                                                                                                                                                                                                                                                                                          |
| mixing water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic and an oil; and | <p>[0005] According to an embodiment, the particles are dry coated with a water-repellent organo-silicon material.</p> <p>[0006] Representative organo-silicon compounds that can be used in this embodiment include polyalkylsiloxane such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additional organo-silicon</p> | Abstract: Provided are methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates, at least one of which comprises a mineral surface; providing a surface-treating reagent capable of modifying the stress-activated                                                                                                                                                                  | <p>Abstract: Foam fracturing fluids and methods for fracturing well formations using foam as the carrier for the propping agents are disclosed.</p> <p>Col. 4, l. 28-33: Oil may be added to the foam-water slurry as a lubricant and to prevent clay swelling; oil may replace all of the water in the sand slurry, in which case a</p> |

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|  | <p>compounds that can be used in this embodiment include polyalkylarylsiloxanes, . . . chlorosilanes such as ethylchlorosilane, chlorotrimethylsilane and other silyl donors. Also, various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes can be used such as tetraethoxysilane, dimethoxydiphenylsilane, dichlorodimethylsilane, dichlorodiphenylsilane, poly[oxy(dimethylsilylene)] and other such materials that will be well known to those skilled in the art. . . . Furthermore, organofunctional silanes can be used in this embodiment...</p> <p>[0008] If the particles are coated when the particles are flowing, the solvent should be readily miscible in the water-based gel carrier fluid. Organic solvents that can be used as a carrier for the organo-silicon coating material include kerosene, lighter grade of diesel fuel . . . and other</p> | <p>reactivity of a mineral surface of a particulate; and allowing the surface-treating reagent modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate.</p> <p>[0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in</p> | <p>chemical suitable for foaming oil is used in place of the surfactant. Other friction reducing materials may be added to the fracturing fluid.</p> <p>Col. 7, l. 16-20: To summarize, the invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a liquid carrier comprising one or more liquids selecting of the group consisting of water and oil, and a solid proppant . . .</p> <p>Col. 8, l. 30-32: Method according to claim 1, wherein the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.</p> |
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|  | <p>such solvents that will be well known to those in the art.</p> <p>[0011] Fracturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and emulsions.</p> <p>[0012] The most common fracturing fluid utilized heretofore which is generally preferred for use in accordance with this invention is comprised of water. . .</p> <p>[0013] The water utilized to form the fracturing fluids used in accordance with the methods of this invention can be fresh water, salt water, brine or any other aqueous liquid which does not adversely react to the other components of the fracturing fluids.</p> <p>[0016] The proppant or gravel utilized is of a size such that formation particulate solids</p> | <p>which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprised of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> <p>[0056] This may be accomplished in treatments performed prior to transporting the particulates to a job site, or in a treatment performed “on-the-fly.” The term “on-the-fly” is used herein to mean that one flowing stream comprising particulates is continuously introduced into another</p> |  |
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|  | <p>which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like. Generally the particles used have a particle size in the range of from about 2 to about 400 mesh, U.S. Sieve Series. The preferred particles are sand having a particle size in the range of from about 10 to about 70 mesh, U.S. Sieve Series.</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Claim 63: A method for treating a hydrocarbon</p> | <p>flowing stream comprising the surface-treating reagent so that the streams are combined and mixed while continuing to flow as a single stream as part of the on-going treatment at the job site. Such mixing can also be described as “real-time” mixing.</p> <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut</p> |  |
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U.S. Patent 8,236,738 v. U.S. Publication 2004/0023818 (Nguyen) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> <p>Claim 65: The method of claim 63 wherein the carrier fluid comprises water and a gelling agent.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of hydrocarbons.</p> | <p>shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.</p> <p>[0015] A mineral surface whose reactivity has been modified according to this definition is herein referred to as “a modified mineral surface.” In certain embodiments of the present invention, modifying the</p> |  |
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U.S. Patent 8,236,738 v. U.S. Publication 2004/0023818 (Nguyen) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  |  | <p>stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic or hydrophilic in nature.</p> <p>[0049] In some embodiments, the surface-treating reagent may comprise other compounds that are capable of hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include, but are not limited to, vinyl monomers, dienes, keto esters, amines, substituted amine hydrochlorides, amides, alcohols, organosilanes, organotitanates, organozirconates, trivalent</p> |  |
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U.S. Patent 8,236,738 v. U.S. Publication 2004/0023818 (Nguyen) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  |  | metal cations, tetravalent metal cations, ammonium halides, quaternary ammonium halides, ammonium salts of inorganic acids, ammonium salts of carboxylic acids, oligomeric materials, monomeric materials, oil-wetting compounds, and derivatives thereof. In some embodiments, the surface-treating reagent may comprise lecithin. In some embodiments, the surface-treating reagent may comprise a chlorosilyl group containing compound and an alkylsilane, or the reaction products thereof. In some embodiments, the surface-treating reagent may comprise an organofunctional silane and an aryl acid halide, or the reaction products thereof. Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes. |  |
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U.S. Patent 8,236,738 v. U.S. Publication 2004/0023818 (Nguyen) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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| <p>pumping the mixture into the formation.</p> | <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> <p>Claim 75: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to a fracture in a formation adjacent the well to function as a proppant, and coating the particles with a water repellent composition, wherein the particles are coated before being mixed in</p> | <p>Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid into the subterranean formation.</p> <p>Claim 23: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified material surface; and introducing the treatment fluid</p> |  |
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U.S. Patent 8,236,738 v. U.S. Publication 2004/0023818 (Nguyen) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                                                                      | the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | into the subterranean formation.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |                                                     |
| <b>Claim 40 of U.S. Patent 8,236,738</b>                                                                                                                                             | <b>Reference to U.S. Publication 2004/0023818 (Nguyen)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 40. The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates. | <p>[0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of</p> | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces,</p> |                                                     |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>any gel polymer from the carrier fluid for the particles.</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Claim 2: The method of claim 1 wherein the particles are pumped downhole to a screen and function as a gravel pack.</p> <p>Claim 3: The method of claim 1 wherein the particles are pumped downhole to a fracture in a formation adjacent the well and function as a proppant.</p> <p>Claim 8: The method of claim 1 wherein the particles are selected from the group consisting of sand and gravel.</p> <p>Claim 63: A method for treating a hydrocarbon</p> | <p>cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution</p> |  |
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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                 | producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles. | and/or fluid into a subterranean formation or well bore.                                                                                                                                                                                                                                                                                                                                                       |                                                     |
| <b>Claim 41 of U.S. Patent 8,236,738</b>                        | <b>Reference to U.S. Publication 2004/0023818 (Nguyen)</b>                                                                                                                                                                                                                                                                                                                                                                 | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                     | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 41. The method of claim 32, wherein the particulates are sands. | [0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like.              | [0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates |                                                     |



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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Claim 2: The method of claim 1 wherein the particles are pumped downhole to a screen and function as a gravel pack.</p> <p>Claim 3: The method of claim 1 wherein the particles are pumped downhole to a fracture in a formation adjacent the</p> | <p>include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p> |  |
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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                          | <p>well and function as a proppant.</p> <p>Claim 8: The method of claim 1 wherein the particles are selected from the group consisting of sand and gravel.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> |                                                                                                           |                                                     |
| <b>Claim 51 of U.S. Patent 8,236,738</b>                                 | <b>Reference to U.S. Publication 2004/0023818 (Nguyen)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 51. A method of controlling proppant flowback in a hydrocarbon producing | [0005] According to an embodiment, the particles are dry coated with a water-repellent organo-silicon                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | [0058] The methods of the present invention may be utilized in a variety of subterranean operations known |                                                     |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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| formation comprising the steps of: | <p>material. The treated particles are then pumped down to the fractures to function as a proppant or to the wellbore-screen annulus to function as a gravel pack.</p> <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of the hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p> | <p>in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.</p> <p>[0004] One problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, proppant particulates, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation or equipment placed in the formation.</p> <p>[0014] Thus, one object of the present invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                 |                                                                                                                                                                                                                                                                                                                                                                                                                                                                               | increased conductivity may be realized. Other objects of the present invention include, but are not limited to, preventing build-up of filter cake and/or gel residue in conductive channels (e.g., fractures) within subterranean formation, and preventing the embedment of particulates into the surfaces of a subterranean formation.                                                                                                                                                                                                                                                                 |  |
| Contacting particulates with a medium containing a chemical compound for rendering the surface of the particulates hydrophobic, | [0007]: An example of the technique for applying the coating on the particles involves dissolving an oil-soluble organo-silicon compound in a solvent, admixing or spraying the resultant solution on the particles, and then evaporating the solvent to form a thin film of siloxane or silane encapsulating the particles. The organo-silicon organo-silane coating is readily absorbed onto the particles from the solvent and the solvent is easily evaporated by drying. | [0057] Other suitable methods for allow the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate include, but are not limited to, methods utilizing a drive mixer and solvent extraction methods. . . . The solution comprising the surface-treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced into a water-based fluid that extracts the solvent from the treated |  |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                             | Claim 4: The method of claim 1, wherein the composition is oil-soluble, and wherein the particles are coated by dissolving the composition in a solvent, admixing or spraying the resultant solution the particulates, and then evaporating the solvent to form a film encapsulating the particles.                                                                                                                                                                           | particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface...                                                                                                                                                                                                                                                                                                                                                                                                                            |  |
| Separating the particulate from the medium; | [0007]: An example of the technique for applying the coating on the particles involves dissolving an oil-soluble organo-silicon compound in a solvent, admixing or spraying the resultant solution on the particles, and then evaporating the solvent to form a thin film of siloxane or silane encapsulating the particles. The organo-silicon organo-silane coating is readily absorbed onto the particles from the solvent and the solvent is easily evaporated by drying. | [0057] Other suitable methods for allow the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate include, but are not limited to, methods utilizing a drive mixer and solvent extraction methods. . . . The solution comprising the surface-treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced into a water-based fluid that extracts the solvent from the treated |  |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                   | Claim 4: The method of claim 1, wherein the composition is oil-soluble, and wherein the particles are coated by dissolving the composition in a solvent, admixing or spraying the resultant solution the particulates, and then evaporating the solvent to form a film encapsulating the particles.                                                                                                                                                                                                                                                                    | particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface...                                                                                                                                                                                                                                                                                                                                                                                                                               |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |
| And blending the hydrophobic particulate with water and an oil to form a mixture; | <p>[0011] Fracturing or gravel packing fluids which can be utilized in accordance with the present invention include gelled water or oil base liquids, foams and emulsions.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the</p> | [0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic | <p>Abstract: Foam fracturing fluids and methods for fracturing well formations using foam as the carrier for the propping agents are disclosed.</p> <p>Col. 4, l. 28-33: Oil may be added to the foam-water slurry as a lubricant and to prevent clay swelling; oil may replace all of the water in the sand slurry, in which case a chemical suitable for foaming oil is used in place of the surfactant. Other friction reducing materials may be added to the fracturing fluid.</p> |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                             | <p>coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p> | <p>liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprised of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> | <p>Col. 7, l. 16-20: To summarize, the invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a liquid carrier comprising one or more liquids selecting of the group consisting of water and oil, and a solid proppant . . .</p> <p>Col. 8, l. 30-32: Method according to claim 1, wherein the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.</p> |
| And pumping the mixture into the formation. | Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole                                                                                                                                                                                                                                                                    | Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                                                                                                                                                                                                                                                                                                                                                                                                                                                             |

U.S. Patent 8,236,738 v. U.S. Publication 2004/0023818 (Nguyen) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>in a well to form a gravel pack, and coating the particles with a water repellent composition wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> <p>Claim 75: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based polymer to form a mixture, pumping the mixture downhole in a well to a fracture in a formation adjacent the well to function as a proppant, and coating the particles with a water repellent composition, wherein the particles are coated before being mixed in the carrier fluid whereby the coating prevents absorption of the water-based polymer on the surfaces of the particles.</p> | <p>surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid into the subterranean formation.</p> <p>Claim 23: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified material surface; and introducing the treatment fluid into the subterranean formation.</p> |  |
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U.S. Patent 8,236,738 v. U.S. Publication 2004/0023818 (Nguyen) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

| Claim 57 of U.S. Patent 8,236,738                                    | Reference to U.S. Publication 2004/0023818 (Nguyen)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | Reference to U.S. Publication 2006/0260808 (Weaver)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | Reference to U.S. Patent 3,980,136 (Plummer) |
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| 57. The composition of claim 51, wherein the particulates are sands. | <p>[0016] The proppant or gravel utilized is of a size such that formation particulate solids which migrate with produced fluids are prevented from flowing through the fractures or through the gravel pack in the annulus. Various kinds of particles can be utilized as proppant including sand, bauxite, ceramic materials, glass materials, TEFLON™ materials, curable resin-coated proppant, and the like.</p> <p>Abstract: A method for enhancing the clean-up of a hydrocarbon-producing well, in which particles are pumped into the well to promote the recovery of hydrocarbons. The particles are coated with a water-repellent composition to prevent, or at least minimize, the coating or adsorption of any gel polymer from the carrier fluid for the particles.</p> | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution</p> |                                              |

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|  | <p>Claim 1: A method comprising dry coating particles with a water-repelling composition, and pumping the particles downhole into a well to promote the recovery of hydrocarbons from the well.</p> <p>Claim 2: The method of claim 1 wherein the particles are pumped downhole to a screen and function as a gravel pack.</p> <p>Claim 3: The method of claim 1 wherein the particles are pumped downhole to a fracture in a formation adjacent the well and function as a proppant.</p> <p>Claim 8: The method of claim 1 wherein the particles are selected from the group consisting of sand and gravel.</p> <p>Claim 63: A method for treating a hydrocarbon producing well comprising mixing particles in a carrier fluid comprising a water-based</p> | and/or fluid into a subterranean formation or well bore. |  |
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|  | polymer to form a mixture,<br>pumping the mixture downhole<br>in a well to form a gravel pack,<br>and coating the particles with a<br>water repellent composition<br>wherein the particles are<br>coated before being mixed in<br>the carrier fluid whereby the<br>coating prevents absorption of<br>the water-based polymer on<br>the surfaces of the particles. |  |  |
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# EXHIBIT I

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

| Claim 1 of U.S. Patent 8,236,738             | Reference to Canadian Patent 2,423,031 (Dawson)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | Reference to U.S. Publication 2006/0260808 (Weaver)                                                                                                                                                                                                                                                                                              | Reference to U.S. Patent 3,980,136 (Plummer) |
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| 1. An aqueous slurry composition comprising: | <p>P. 14, l. 12-15: A slurry including the disclosed particulate material (<i>e.g.</i>, relatively lightweight and/or substantially neutrally buoyant particulates, and/or particulates of naturally-occurring material that have been exposed to a modifying agent) and a carrier fluid may then be introduced into the wellbore . . .</p> <p>P. 13, l. 10-13: In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water . . .</p> | [0058] In certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid ( <i>e.g.</i> , a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. |                                              |
| water;                                       | P. 13, l. 6-13: The particles disclosed herein . . . may be mixed and pumped during any desired portion/s of a well treatment ( <i>e.g.</i> , hydraulic                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | [0058] In certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the                                                                                                                                                                                                                    |                                              |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson) in view of  
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water, liquid hydrocarbons, and/or nitrogen or other gases.</p> <p>P. 10, l. 1-3: In another respect, disclosed herein is a method of fracturing a subterranean formation, including introducing a particulate material suspended in a carrier fluid into the subterranean formation at a pressure above a fracturing pressure of the subterranean formation.</p> | <p>subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprise of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an</p> |  |
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|               |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | aqueous liquid or gelled liquid and a hydrocarbon.                                                                                                                                                                                                                                                                                                                           |  |
| particulates; | <p>P. 2, l. 5-8: In particular, this invention relates to use of relatively lightweight and/or substantially neutrally buoyant particles as proppant material in hydraulic fracturing treatments and as particulate material in sand control methods such as gravel packing, frac pack treatments, <i>etc.</i></p> <p>P. 10, l. 1-3: In another respect, disclosed herein is a method of fracturing a subterranean formation, including introducing a particulate material suspended in a carrier fluid into the subterranean formation at a pressure above a fracturing pressure of the subterranean formation.</p> <p>P. 48, claim 13: A method of fracturing a subterranean formation, comprising: introducing a particulate material suspended in a carrier</p> | <p>Abstract: Provided are methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates. . .</p> <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface.</p> |  |

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|                                                                                    | fluid into said subterranean formation at a pressure above a fracturing pressure of said subterranean formation; . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |  |
| a chemical compound for rendering the surface of the particulates hydrophobic; and | P. 7, l. 3-23, generally: In a further embodiment, individual particles . . . may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material . . . Specific examples of suitable modifying agents include, but are not limited to, agents including polyisocyanates, silanes, siloxanes, and combinations thereof. Selected modifying agent/s may be advantageously used to increase the elastic modulus of a given naturally occurring material, for example, to make particles of a given naturally-occurring material more suitable . . . for use as a relatively lightweight and/or substantially neutrally buoyant fracture proppant or sand control particulate material having increased effectiveness when exposed to | <p>[0015] A mineral surface whose reactivity has been modified according to this definition is herein referred to as “a modified mineral surface.” In certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic or hydrophilic in nature.</p> <p>[0049] In some embodiments, the surface-treating reagent</p> |  |



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|  | <p>higher closure stresses or other mechanical stresses that may be encountered downhole during a well treatment such as hydraulic fracturing or sand control treatment.</p> <p>P. 21, l. 8-14: In a further embodiment, one or more of the carbon chains R and/or R' of formula (I) may be optionally and independently derivatized, <i>e.g.</i>, the R carbon chain and/or one or more of the R' carbon chains may each contain one or more amino functional groups, one or more halogen groups (<i>e.g.</i>, tetrachlorosilane, methyltrichlorosilane, <i>etc.</i>), two or more isocyanate functional groups, two or more epoxy groups, <i>etc.</i> Where halogen groups (<i>e.g.</i> such as chlorine groups) are present, it may be desirable to rapidly neutralize any acidic by-products that may be created during a modification reaction.</p> | <p>may comprise other compounds that are capable of hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include, but are not limited to, vinyl monomers, dienes, keto esters, amines, substituted amine hydrochlorides, amides, alcohols, organosilanes, organotitanates, organozirconates, trivalent metal cations, tetravalent metal cations, ammonium halides, quaternary ammonium halides, ammonium salts of inorganic acids, ammonium salts of carboxylic acids, oligomeric materials, monomeric materials, oil-wetting compounds, and derivatives thereof. In some embodiments, the surface-treating reagent may comprise lecithin. In some embodiments, the surface-treating reagent may comprise a chlorosilyl group containing compound and an alkylsilane, or the reaction products thereof. In some embodiments, the surface-treating reagent</p> |  |
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|  | <p>P. 21, l. 19- P. 22, l. 23:<br/> Examples of commercially available silane-based products that may be employed as modifying agents in the practice of the disclose methods and compositions include, but are not limited to, silane based products. . . .<br/> Specific examples of commercially available silane-based products available from CROMPTON include CoatOSil® 1706 Silane (vinyl silane monomer), CoatOSil® 1757 Silane, CoatOSil® 1770 Silane, EURENOR® 5020 (liquid isobutylisopropylmethoxysilane), EURENOR® 5021 (liquid diisopropylmethoxysilane), EURENOR® 5022 (liquid diisobutylmethoxysilane), EURENOR® 5023 (liquid dicyclopentylmethoxysilane) , Silcat® R Silane (vinylsilane with grafting and cross linking catalysts), Sikat® RHE Silane (cross linking system of silane, peroxide and catalyst), Silcat® RHS Silane (crosslinking</p> | <p>may comprise an organofunctional silane and an aryl acid halide, or the reaction products thereof. Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.</p> |  |
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|  | system of silane, peroxide<br>catalyst and antioxidants),<br>Sikat® VS-735/1 Silane<br>(crosslinking system of silane,<br>peroxide, antioxidants and<br>metal deactivator), Silcat®<br>VS-758/0 Silane (stabilized<br>cross linking system of silane,<br>peroxide and catalyst), Sikat®<br>VS-870 Silane (stabilized<br>crosslinking system of silane,<br>peroxide, catalyst, antioxidants<br>and metal deactivator), Sikat®<br>VS-928 Silane (stabilized<br>crosslinking system of silane,<br>peroxide, catalyst, antioxidants<br>and metal deactivator), Sikat®<br>VS-963 Silane (stabilized cross<br>linking system of silane,<br>peroxide, catalyst, antioxidants<br>and metal deactivator), Silox®<br>VS- 911 Silane (liquid silane<br>system), Silquest® A-1100<br>Silane<br>(gammaaminopropyltriethoxys<br>ilane), Silquest® A-1101<br>Silane (technical grade amino<br>silane), Silquest® A-1102<br>Silane (technical grade amino<br>silane), Silquest® A-1110<br>Silane (primary amino silane),<br>Silquest® A-1120 Silane, |  |  |
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|  | <p>Silquest® A-1126 Silane (amino silane), Silquest® A-1128 Silane (amino silane), Silquest® A-1160 Silane (ureido silane), Silquest® A-1170 Silane (secondary amino functional silane), Silquest® A-1289 Silane, Silquest® A-1310 Silane, Silquest® A-137 Silane (monomeric alkylalkoxysilane), Silquest® A-151 Silane, Silquest® A-1524 Silane (100-percent active ureidosilane), Silquest® A-1589 Silane (disulfide silane), Silquest® A-171 Silane, Silquest® A-172 Silane (vinylfunctional coupling agent), Silquest® A-174 Silane, Silquest® A-186 Silane (epoxy functional silanes), Silquest® A-187 Silane (epoxy functional silanes), Silquest® A-189 Silane, Silquest® A-2171 Silane (silane compound having vinyl and silane functionality), Silquest® Alink 15 Silane (secondary amino silane endcapper), Silquest® RC-1 Silane (organosilicone coupling agent), Silquest® RC-2 Silane, Silquest®</p> |  |  |
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|         | Wetlink 78 Silane, Silquest® Y-11637 Silane (primary aminosilane), Silquest® Y-11683 Silane (di-, tri-functional ethoxy silane), Silquest® Y-5997 Silane, XL-PEarl® 31 Blend, XL-PEarl® 50 Blend, XLPEarl® 51 Blend, XL-PEarl® 52 Blend, XL-PEarl® 60 Blend, XL-PEarl® 70 Blend, <i>etc.</i>                                                                                                                                                                                                                                                                                                    |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |
| an oil. | P. 13, l. 6-13: The particles disclosed herein . . . may be mixed and pumped during any desired portion/s of a well treatment (e.g., hydraulic fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water, liquid hydrocarbons, and/or nitrogen or other gases. | [0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic | Abstract: Foam fracturing fluids and methods for fracturing well formations using foam as the carrier for the propping agents are disclosed.<br><br>Col. 4, l. 28-33: Oil may be added to the foam-water slurry as a lubricant and to prevent clay swelling; oil may replace all of the water in the sand slurry, in which case a chemical suitable for foaming oil is used in place of the surfactant. Other friction reducing materials may be added to the fracturing fluid. |

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|  | <p>P. 33, l. 6-8: Incorporates by reference Graham (U.S. Patent 3,659,651)</p> <p>Graham, Col. 5, l. 9-16: In placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or <b>water-in-oil emulsion</b> prepared in accordance with the teachings of U.S. Pat. No. 3,378,074 (Kiel)</p> <ul style="list-style-type: none"> <li>Kiel, Col. 5, l. 66-73: High viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either water-in-oil or oil-in-water emulsions. Emulsions which have</li> </ul> | <p>liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> <p>[0054] In certain embodiments of the present invention, the surface-treating reagent may be encapsulated with various materials, which, among other things, delays its reaction with the mineral surface and/or other substances present. Solid surface-treating reagents can</p> | <p>Col. 7, l. 16-20: To summarize, the invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a liquid carrier comprising one or more liquids selecting of the group consisting of water and oil, and a solid proppant . . .</p> <p>Col. 8, l. 30-32: Method according to claim 1, wherein the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.</p> |
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|  | <p>high viscosities under the temperature and shear conditions existing in fractures can be prepared with kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine or an acid solution containing a suitable surface active agent or emulsifier.</p> <p>P. 14, l. 31- 15, l. 4: [I]t will be understood with benefit of this disclosure that the disclosed particulate materials may be used with any method in which a pack of particulate material is formed within a wellbore that it is permeable to fluids produced from a wellbore (<i>e.g.</i>, oil, gas, water, etc.) but that substantially prevents or reduces production of formation materials (<i>e.g.</i>, formation sand, etc.) from the formation into the wellbore.</p> | <p>be encapsulated by spray coating a variety of materials thereon. Such coating materials include, but are not limited to, waxes, drying oils such as tung oil and linseed oil, polyurethanes and cross-linked partially hydrolyzed polyacrylics.</p> <p>[0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.</p> <p>Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

|                                                                                                                                                                                                                    |                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid into the subterranean formation.                                                                                                                                                                       |                                                     |
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| <b>Claim 21 of U.S. Patent 8,236,738</b>                                                                                                                                                                           | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                           | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                               | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 21. The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates. | <p>P. 13, l. 3-5: Additional information on materials and methods for use thereof may be found in U.S. Pat. No. 6,330,916 (Rickards), which is incorporated herein by reference. Further information on materials and methods may also be found in U.S. Patent No. 6,364,018.</p> <p>U.S. Pat. No 6,330,916 (Rickards), Abstract: A subterranean formation is treated by injecting a blend of fracture proppant material and deformable particulate material</p> | [0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., |                                                     |



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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>into the formation. The deformable particulate material may combine with the fracture proppant material to increase fracture conductivity, reduce fines generation, and/or reduce proppant flowback. The fracture proppant material may be a material such as sand . . .</p> | <p>glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

|                                                                     |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.                                                                                                                                                                                                                                    |                                                     |
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| <b>Claim 22 of U.S. Patent 8,236,738</b>                            | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                   | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 22. The composition of claim 1, wherein the particulates are sands. | <p>P. 13, l. 3-5: Additional information on materials and methods for use thereof may be found in U.S. Pat. No. 6,330,916 (Rickards), which is incorporated herein by reference. Further information on materials and methods may also be found in U.S. Patent No. 6,364,018.</p> <p>U.S. Pat. No 6,330,916 (Rickards), Abstract: A subterranean formation is treated by injecting a blend of fracture proppant material and deformable particulate material into the formation. The deformable particulate material</p> | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent</p> |                                                     |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                             | may combine with the fracture proppant material to increase fracture conductivity, reduce fines generation, and/or reduce proppant flowback. The fracture proppant material may be a material such as sand . . .                                                                                                                                                                                                                                                              | may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.                                                                                                                                                           |                                                     |
| <b>Claim 32 of U.S. Patent 8,236,738</b>                                                                    | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                                        | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                            | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 32. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: | P. 2, l. 4-8: This invention relates generally to methods and compositions useful for subterranean formation treatments, such as hydraulic fracturing treatments and sand control. In particular, this invention relates to the use of relatively lightweight and/or substantially neutrally buoyant particles as proppant materials in hydraulic fracturing treatment and as particulate material in sand control methods such as gravel packing, frac pack treatments, etc. | [0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.<br><br>[0004] One problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation |                                                     |

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|                                                                                  | <p>P. 13, l. 22-25: Such other treatments may be near wellbore in nature (affecting near wellbore regions) and may be directed towards improving wellbore productivity and/or controlling the production of fracture proppant or formation sand. Particular examples include gravel packing and “frac-packs.”</p> <p>P. 3, l. 13-16: In the disclosed method, the application of relatively lightweight and/or substantially neutrally buoyant particulate material as a fracture proppant particulate advantageously may provide for substantially improved overall system performance in hydraulic fracturing applications, or in other well treating applications such as sand control.</p> | <p>finer, proppant particulates, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation or equipment placed in the formation.</p> <p>[0014] Thus, one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, preventing build-up of filter cake and/or gel residue in conductive channels (e.g., fractures) within a subterranean formation, and preventing the embedment of particulates into the surfaces of a subterranean formation.</p> |                                                                             |
| mixing water, particulates, a chemical compound for rendering the surface of the | P. 13, l. 6-13: The particles disclosed herein ( <i>e.g.</i> , relatively lightweight and/or                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   | Abstract: Provided are methods of modifying the surface stress-activated                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | Abstract: Foam fracturing fluids and methods for fracturing well formations |

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| <p>particulates hydrophobic and an oil; and</p> | <p>substantially neutrally buoyant proppant or sand control particulates, and/or particulates of naturally-occurring material that have been exposed to a modifying agent) may be mixed and pumped during any desired portion/s of well treatment (<i>e.g.</i>, hydraulic fracturing treatment, sand control treatment, <i>etc.</i>) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water, liquid hydrocarbons, and/or nitrogen or other gases.</p> <p>P. 2, l. 5-8: In particular, this invention relates to use of relatively lightweight and/or substantially neutrally buoyant particles as proppant material in hydraulic fracturing</p> | <p>reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates, at least one of which comprises a mineral surface; providing a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; and allowing the surface-treating reagent modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate.</p> <p>[0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (<i>e.g.</i>, water containing one or more salts dissolved therein), brine (<i>e.g.</i>, saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely</p> | <p>using foam as the carrier for the propping agents are disclosed.</p> <p>Col. 4, l. 28-33: Oil may be added to the foam-water slurry as a lubricant and to prevent clay swelling; oil may replace all of the water in the sand slurry, in which case a chemical suitable for foaming oil is used in place of the surfactant. Other friction reducing materials may be added to the fracturing fluid.</p> <p>Col. 7, l. 16-20: To summarize, the invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a liquid carrier comprising one or more liquids selecting of the group consisting of water and oil, and a solid proppant . . .</p> <p>Col. 8, l. 30-32: Method according to claim 1, wherein the liquid component of said foam comprises at least one</p> |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>treatments and as particulate material in sand control methods such as gravel packing, frac pack treatments, <i>etc.</i></p> <p>P. 10, l. 1-3: In another respect, disclosed herein is a method of fracturing a subterranean formation, including introducing a particulate material suspended in a carrier fluid in the subterranean formation at a pressure above a fracturing pressure of the subterranean formation...</p> <p>P. 48, claim 13: A method of fracturing a subterranean formation, comprising: introducing a particulate material suspended in a carrier fluid into said subterranean formation at a pressure above a fracturing pressure of said subterranean formation; . . .</p> <p>P. 7, l. 3-23: In a further embodiment, individual particles . . . may be optionally</p> | <p>affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprised of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> | <p>liquid selected from the group consisting of water and oil.</p> |
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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material . . . Specific examples of suitable modifying agents include, but are not limited to, agents including polyisocyanates, silanes, siloxanes, and combinations thereof. Selected modifying agent/s may be advantageously used to increase the elastic modulus of a given naturally occurring material, for example, to make particles of a given naturally-occurring material more suitable . . . for use as a relatively lightweight and/or substantially neutrally buoyant fracture proppant or sand control particulate material having increased effectiveness when exposed to higher closure stresses or other mechanical stresses that may be encountered downhole during a well treatment such as hydraulic fracturing or sand control treatment.</p> | <p>[0056] This may be accomplished in treatments performed prior to transporting the particulates to a job site, or in a treatment performed “on-the-fly.” The term “on-the-fly” is used herein to mean that one flowing stream comprising particulates is continuously introduced into another flowing stream comprising the surface-treating reagent so that the streams are combined and mixed while continuing to flow as a single stream as part of the on-going treatment at the job site. Such mixing can also be described as “real-time” mixing.</p> <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>P. 21, l. 8-14: In a further embodiment, one or more of the carbon chains R and/or R' of formula (I) may be optionally and independently derivatized, e.g., the R carbon chain and/or one or more of the R' carbon chains may each contain one or more amino functional groups, one or more halogen groups (e.g., tetrachlorosilane, methyltrichlorosilane, etc.), two or more isocyanate functional groups, two or more epoxy groups, etc. Where halogen groups (e.g., such as chlorine groups) are present, it may be desirable to rapidly neutralize any acidic by-products that may be created during a modification reaction.</p> <p>P. 21, l. 23- P. 22, l. 23: Examples of commercially available silane-based products that may be employed as modifying agents in the practice of the disclose methods and compositions include, but are not limited to, silane based products" (Ex.</p> | <p>cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground</p> |  |
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|  | <p>1006, Dawson at p. 21, l. 19-21) and teaches the specific examples of “commercially available silane-based products available from CROMPTON include CoatOSil® 1706 Silane (vinyl silane monomer), CoatOSil® 1757 Silane, CoatOSil® 1770 Silane, EURENOR® 5020 (liquid isobutylisopropylmethoxysilane), EURENOR® 5021 (liquid diisopropylmethoxysilane), EURENOR® 5022 (liquid diisobutylmethoxysilane), EURENOR® 5023 (liquid dicyclopentylmethoxysilane), Silcat® R Silane (vinylsilane with grafting and cross linking catalysts), Sikat® RHE Silane (cross linking system of silane, peroxide and catalyst), Silcat® RHS Silane (crosslinking system of silane, peroxide catalyst and antioxidants), Sikat® VS-735/1 Silane (crosslinking system of silane, peroxide, antioxidants and metal deactivator), Silcat® VS-758/0 Silane (stabilized cross linking system of silane,</p> | <p>cellulose fiber, and combinations thereof.</p> <p>[0015] A mineral surface whose reactivity has been modified according to this definition is herein referred to as “a modified mineral surface.” In certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic or hydrophilic in nature.</p> <p>[0049] In some embodiments, the surface-treating reagent may comprise other</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | peroxide and catalyst), Sikat® VS-870 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat® VS-928 Silane (stabilized crosslinking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Sikat® VS-963 Silane (stabilized cross linking system of silane, peroxide, catalyst, antioxidants and metal deactivator), Silox® VS- 911 Silane (liquid silane system), Silquest® A-1100 Silane (gammaaminopropyltriethoxysilane), Silquest® A-1101 Silane (technical grade amino silane), Silquest® A-1102 Silane (technical grade amino silane), Silquest® A-1110 Silane (primary amino silane), Silquest® A-1120 Silane, Silquest® A-1126 Silane (amino silane), Silquest® A-1128 Silane (amino silane), Silquest® A-1160 Silane (ureido silane), Silquest® A-1170 Silane (secondary amino functional silane), Silquest® A-1289 Silane, Silquest® A- | compounds that are capable of hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include, but are not limited to, vinyl monomers, dienes, keto esters, amines, substituted amine hydrochlorides, amides, alcohols, organosilanes, organotitanates, organozirconates, trivalent metal cations, tetravalent metal cations, ammonium halides, quaternary ammonium halides, ammonium salts of inorganic acids, ammonium salts of carboxylic acids, oligomeric materials, monomeric materials, oil-wetting compounds, and derivatives thereof. In some embodiments, the surface-treating reagent may comprise lecithin. In some embodiments, the surface-treating reagent may comprise a chlorosilyl group containing compound and an alkylsilane, or the reaction products thereof. In some embodiments, the surface-treating reagent may comprise an |  |
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|  | 1310 Silane, Silquest® A-137<br>Silane (monomeric<br>alkylalkoxysilane), Silquest®<br>A-151 Silane, Silquest® A-<br>1524 Silane (100-percent<br>active ureidosilane), Silquest®<br>A-1589 Silane (disulfide<br>silane), Silquest® A-171<br>Silane, Silquest® A-172 Silane<br>(vinylfunctional coupling<br>agent), Silquest® A-174<br>Silane, Silquest® A-186 Si<br>lane (epoxy functional silanes),<br>Silquest® A-187 Silane (epoxy<br>functional silanes), Silquest®<br>A-189 Silane, Silquest® A-<br>2171 Silane (silane compound<br>having vinyl and silane<br>functionality), Silquest® Alink<br>15 Silane (secondary amino<br>silane endcapper), Silquest®<br>RC-1 Silane (organosilicone<br>coupling agent), Silquest®<br>RC-2 Silane, Silquest®<br>Wetlink 78 Silane, Silquest®<br>Y-11637 Silane (primary<br>aminosilane), Silquest® Y-<br>11683 Silane (di-, tri-<br>functional ethoxy silane),<br>Silquest® Y-5997 Silane, XL-<br>PEarl® 31 Blend, XL-PEarl®<br>50 Blend, XLPEarl® 51 Blend, | organofunctional silane and an<br>aryl acid halide, or the reaction<br>products thereof. Examples of<br>suitable organofunctional<br>silanes for these embodiments<br>include, but are not limited to,<br>aminofunctional silanes,<br>ureidofunctional silanes, and<br>epoxyfunctional silanes. |  |
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|  | <p>XL-PEarl® 52 Blend, XL-PEarl® 60 Blend, XL-PEarl® 70 Blend, <i>etc.</i></p> <p>P. 13, l. 6-13: The particles disclosed herein . . . may be mixed and pumped during any desired portion/s of a well treatment (e.g., hydraulic fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water, liquid hydrocarbons, and/or nitrogen or other gases.</p> <p>P. 33, l. 6-8: Incorporates by reference Graham (U.S. Patent 3,659,651)</p> <p>Graham, Col. 5, l. 9-16: In placing the propping agent in</p> |  |  |
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|  | <p>the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or <b><i>water-in-oil emulsion</i></b> prepared in accordance with the teachings of U.S. Pat. No. 3,378,074 (Kiel)</p> <ul style="list-style-type: none"> <li>• Kiel, Col. 5, l. 66-73: High viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either water-in-oil or oil-in-water emulsions. Emulsions which have high viscosities under the temperature and shear conditions existing in fractures can be prepared with kerosene, diesel fuel, heating oil, crude oil or</li> </ul> |  |  |
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U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                         | a similar hydrocarbon oil and water, brine or an acid solution containing a suitable surface active agent or emulsifier.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |  |
| pumping the mixture into the formation. | <p>P. 13, l. 6-10: The particles disclosed herein (<i>e.g.</i>, relatively lightweight and/or substantially neutrally buoyant proppant or sand control particulates, and/or particulates of naturally-occurring material that have been exposed to a modifying agent) may be mixed and pumped during any desired portion/s of well treatment (<i>e.g.</i>, hydraulic fracturing treatment, sand control treatment, <i>etc.</i>) and may be mixed in any desired concentration with a carrier fluid.</p> <p>P. 29, l. 27-28: In another exemplary embodiment, modified particulate material may be formulated as described herein and introduced or pumped into a well...</p> | <p>Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid into the subterranean formation.</p> <p>Claim 23: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises</p> |  |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                                                                      |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | a base fluid, and a plurality of particulates, at least one of which comprises a modified material surface; and introducing the treatment fluid into the subterranean formation.                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                                                     |
| <b>Claim 40 of U.S. Patent 8,236,738</b>                                                                                                                                             | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 40. The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates. | <p>P. 13, l. 3-5: Additional information on materials and methods for use thereof may be found in U.S. Pat. No. 6,330,916 (Rickards), which is incorporated herein by reference. Further information on materials and methods may also be found in U.S. Patent No. 6,364,018.</p> <p>U.S. Pat. No 6,330,916 (Rickards), Abstract: A subterranean formation is treated by injecting a blend of fracture proppant material and deformable particulate material into the formation. The deformable particulate material may combine with the fracture proppant material to increase fracture conductivity, reduce</p> | [0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut |                                                     |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>fines generation, and/or reduce proppant flowback. The fracture proppant material may be a material such as sand . . .</p> | <p>shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate,</p> |  |
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U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                 |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                                                     |
| <b>Claim 41 of U.S. Patent 8,236,738</b>                        | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 41. The method of claim 32, wherein the particulates are sands. | <p>P. 13, l. 3-5: Additional information on materials and methods for use thereof may be found in U.S. Pat. No. 6,330,916 (Rickards), which is incorporated herein by reference. Further information on materials and methods may also be found in U.S. Patent No. 6,364,018.</p> <p>U.S. Pat. No 6,330,916 (Rickards), Abstract: A subterranean formation is treated by injecting a blend of fracture proppant material and deformable particulate material into the formation. The deformable particulate material may combine with the fracture proppant material to increase fracture conductivity, reduce fines generation, and/or reduce proppant flowback. The</p> | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid</p> |                                                     |

U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                             | fracture proppant material may be a material such as sand . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |                                                     |
| <b>Claim 51 of U.S. Patent 8,236,738</b>                                                                    | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 51. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: | <p>P. 2, l. 4-8: This invention relates generally to methods and compositions useful for subterranean formation treatments, such as hydraulic fracturing treatments and sand control. In particular, this invention relates to the use of relatively lightweight and/or substantially neutrally buoyant particles as proppant materials in hydraulic fracturing treatment and as particulate material in sand control methods such as gravel packing, frac pack treatments, etc.</p> <p>P. 13, l. 22-25: Such other treatments may be near wellbore in nature (affecting near wellbore regions) and may be directed towards</p> | <p>[0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.</p> <p>[0004] One problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, proppant particulates, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the</p> |                                                     |

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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                        | <p>improving wellbore productivity and/or controlling the production of fracture proppant or formation sand. Particular examples include gravel packing and “frac-packs.”</p> <p>P. 3, l. 13-16: In the disclosed method, the application of relatively lightweight and/or substantially neutrally buoyant particulate material as a fracture proppant particulate advantageously may provide for substantially improved overall system performance in hydraulic fracturing applications, or in other well treating applications such as sand control.</p> | <p>conductive channels and/or damage the interior of the formation or equipment placed in the formation.</p> <p>[0014] Thus, one object of the present invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, preventing build-up of filter cake and/or gel residue in conductive channels (e.g., fractures) within subterranean formation, and preventing the embedment of particulates into the surfaces of a subterranean formation.</p> |  |
| <p>Contacting particulates with a medium containing a chemical compound for rendering the surface of the particulates hydrophobic,</p> | <p>P. 7, l. 3-8: In a further embodiment, individual particles . . . may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material . . .</p>                                                                                                                                                                                                                                                                                                                                | <p>[0057] Other suitable methods for allow the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate include, but are not limited to, methods utilizing a drive mixer and solvent extraction</p>                                                                                                                                                                                                                                                                                                                                                                                                      |  |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>P. 27, l. 1-2: Modifying agents may be diluted as so desired in a suitable solvent prior to treating particles of a naturally-occurring material.</p> <p>P. 24, l. 17-26: Specific examples of suitable exposure methods include, but are not limited to, by soaking the naturally-occurring material in a modifying agent . . . by dipping the naturally-occurring material in a modifying agent, by spraying a modifying agent on to the naturally-occurring material . . . by mixing naturally-occurring particulate material with liquid, atomized and/or gaseous modifying agent in a vertical mixer . . . by flowing modifying agent through a permeable bed or pack of the naturally-occurring particles in a pressurized container. Drying of particles after exposure may be accomplished, for example, by continuously dropping through heated air...</p> | <p>methods. . . The solution comprising the surface-treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced into a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface...</p> |  |
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U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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| Separating the particulate from the medium; | <p>P. 7, l. 3-8: In a further embodiment, individual particles . . . may be optionally treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material . . .</p> <p>P. 27, l. 1-2: Modifying agents may be diluted as so desired in a suitable solvent prior to treating particles of a naturally-occurring material.</p> <p>P. 24, l. 17-26: Specific examples of suitable exposure methods include, but are not limited to, by soaking the naturally-occurring material in a modifying agent . . . by dipping the naturally-occurring material in a modifying agent, by spraying a modifying agent on to the naturally-occurring material . . . by mixing naturally-occurring particulate material with liquid, atomized and/or gaseous modifying agent in a vertical mixer . . . by flowing modifying agent</p> | <p>[0057] Other suitable methods for allow the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate include, but are not limited to, methods utilizing a drive mixer and solvent extraction methods. . . . The solution comprising the surface-treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced into a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface...</p> |  |
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U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                   | through a permeable bed or pack of the naturally-occurring particles in a pressurized container. Drying of particles after exposure may be accomplished, for example, by continuously dropping through heated air...                                                                                                                                                                                                                                                                                                                                                                            |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |
| And blending the hydrophobic particulate with water and an oil to form a mixture; | P. 13, l. 6-13: The particles disclosed herein . . . may be mixed and pumped during any desired portion/s of a well treatment (e.g., hydraulic fracturing treatment, sand control treatment, etc.) and may be mixed in any desired concentration with a carrier fluid. In this regard, any carrier fluid suitable for transporting the disclosed particles into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids comprising salt water, fresh water, liquid hydrocarbons, and/or nitrogen or other gases. | [0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the | Abstract: Foam fracturing fluids and methods for fracturing well formations using foam as the carrier for the propping agents are disclosed.<br><br>Col. 4, l. 28-33: Oil may be added to the foam-water slurry as a lubricant and to prevent clay swelling; oil may replace all of the water in the sand slurry, in which case a chemical suitable for foaming oil is used in place of the surfactant. Other friction reducing materials may be added to the fracturing fluid.<br><br>Col. 7, l. 16-20: To summarize, the invention provides a formation fracturing fluid, comprising a pressured stable |

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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>P. 33, l. 6-8: Incorporates by reference Graham (U.S. Patent 3,659,651)</p> <p>Graham, Col. 5, l. 9-16: In placing the propping agent in the fracture, the reinforced plastic particles can be blended into the carrier fluid by conventional techniques and equipment. Useful carrier fluids include water-base fluids, moderate-to-high viscosity oils, and other fluids capable of suspending the particles. Preferred fluids include high viscosity oil or <b>water-in-oil emulsion</b> prepared in accordance with the teachings of U.S. Pat. No. 3,378,074 (Kiel)</p> <ul style="list-style-type: none"> <li>Kiel, Col. 5, l. 66-73: High viscosity emulsions suitable for use as viscous fracturing fluids in carrying out the invention may be either water-in-oil or oil-in-water emulsions. Emulsions which have</li> </ul> | <p>like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprised of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> | <p>foam comprising a liquid carrier comprising one or more liquids selecting of the group consisting of water and oil, and a solid proppant . . .</p> <p>Col. 8, l. 30-32: Method according to claim 1, wherein the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.</p> |
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U.S. Patent 8,236,738 (Zhang) v. Canadian Patent 2,423,031 (Dawson) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                             | high viscosities under the temperature and shear conditions existing in fractures can be prepared with kerosene, diesel fuel, heating oil, crude oil or a similar hydrocarbon oil and water, brine or an acid solution containing a suitable surface active agent or emulsifier.                                                                                                                                                                                                                      |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |  |
| And pumping the mixture into the formation. | P. 13, l. 6-10: The particles disclosed herein ( <i>e.g.</i> , relatively lightweight and/or substantially neutrally buoyant proppant or sand control particulates, and/or particulates of naturally-occurring material that have been exposed to a modifying agent) may be mixed and pumped during any desired portion/s of well treatment ( <i>e.g.</i> , hydraulic fracturing treatment, sand control treatment, <i>etc.</i> ) and may be mixed in any desired concentration with a carrier fluid. | Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid |  |



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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                      | P. 29, l. 27-28: In another exemplary embodiment, modified particulate material may be formulated as described herein and introduced or pumped into a well...                                                                                                                                                                                   | into the subterranean formation.<br><br>Claim 23: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified material surface; and introducing the treatment fluid into the subterranean formation.                                                                        |                                                     |
| <b>Claim 57 of U.S. Patent 8,236,738</b>                             | <b>Reference to Canadian Patent 2,423,031 (Dawson)</b>                                                                                                                                                                                                                                                                                          | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                     | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 57. The composition of claim 51, wherein the particulates are sands. | P. 13, l. 3-5: Additional information on materials and methods for use thereof may be found in U.S. Pat. No. 6,330,916, which is incorporated herein by reference. Further information on materials and methods may also be found in U.S. Patent No. 6,364,018.<br><br>U.S. Pat. No 6,330,916 (Rickards), Abstract: A subterranean formation is | [0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term "particulate," as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates |                                                     |

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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>treated by injecting a blend of fracture proppant material and deformable particulate material into the formation. The deformable particulate material may combine with the fracture proppant material to increase fracture conductivity, reduce fines generation, and/or reduce proppant flowback. The fracture proppant material may be a material such as sand . . .</p> | <p>include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p> |  |
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# EXHIBIT J

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2005/0244641 (Vincent) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

| Claim 1 of U.S. Patent 8,236,738             | Reference to U.S. Publication 2005/0244641 (Vincent)                                                                                                                                                                                                  | Reference to U.S. Publication 2006/0260808 (Weaver)                                                                                                                                                                                                                                                                                                                                                                                                        | Reference to U.S. Patent 3,980,136 (Plummer) |
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| 1. An aqueous slurry composition comprising: | [0087] The fracturing fluid may be . . . water base . . . [P]articles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended. | [0058] In certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof.                                                                                                                    |                                              |
| water;                                       | [0087] The fracturing fluid may be an oil base, water base, acid, emulsion, foam, or any other fluid.                                                                                                                                                 | [0058] In certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts |                                              |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|               |                                                                                                                                                                                                           | <p>dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprise of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> |  |
| particulates; | [0002] The present invention relates to oil and gas well proppants and, more particularly, to processes for physically or chemically modifying surface characteristics of hydraulic fracturing proppants. | <p>Abstract: Provided are methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                      |  |

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|                                                                                           | <p>[0087] The fracturing fluid may be . . . water base . . .</p> <p>[P]articles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.</p>                                                                                                                                                                                                                                                                                                                                | <p>comprise: providing a plurality of particulates. . .</p> <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface.</p>                                                                                                                                                                                                                                                                                                                                                                                                           |  |
| <p>a chemical compound for rendering the surface of the particulates hydrophobic; and</p> | <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin-coated sand, and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability, alter the chemical reactivity, alter the surface topography, impart lubricity, and control relative permeability to flow of fluids such as proppants. Sands, resin coated sands, or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, the modify</p> | <p>[0015] A mineral surface whose reactivity has been modified according to this definition is herein referred to as “a modified mineral surface.” In certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, by hindering or preventing the water-wetting of that mineral surface. Such</p> |  |

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|  | <p>their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.</p> <p>[0009] These benefits can be achieved by a variety of techniques, including coating the proppants with a hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes...</p> <p>[0043] Coated samples of a sintered bauxite proppant commercially available from CARBO Ceramics Inc. . . . were prepared by coating the proppant with a product that is commercially available from SOPUS Products under the tradename "Rain-X®". Rain-X® is a glass surface treatment material that includes polyalkyl hydrogen siloxane, ethanol and isopropanol.</p> <p>[0082] First, in both gel-contaminated and</p> | <p>compounds may be hydrophobic or hydrophilic in nature.</p> <p>[0049] In some embodiments, the surface-treating reagent may comprise other compounds that are capable of hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include, but are not limited to, vinyl monomers, dienes, keto esters, amines, substituted amine hydrochlorides, amides, alcohols, organosilanes, organotitanates, organozirconates, trivalent metal cations, tetravalent metal cations, ammonium halides, quaternary ammonium halides, ammonium salts of inorganic acids, ammonium salts of carboxylic acids, oligomeric materials, monomeric materials, oil-wetting compounds, and derivatives thereof. In some embodiments, the surface-treating reagent may comprise lecithin. In some embodiments, the surface-</p> |  |
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|  | <p>uncontaminated packs, the Rain-X® treated proppant initially allowed reduced flowtimes. Secondly, both samples treated with Rain-X® showed significantly increasing flowtimes to water with subsequent flushes. It was visually observed that the Rain-X® coated proppant trapped air bubbles that accumulated over time. It was clear that the proppant was hydrophobic.</p> <p>Claim 1: A gas and oil well proppant comprising: a plurality of essentially spherical particles, wherein the particles are coated with a hydrophobic material.</p> <p>Claim 6: The proppant of claim 2, wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and polydimethyl siloxane.</p> <p>Claims 3-5, 9-10, and 15-17 also meet this limitation.</p> | <p>treating reagent may comprise a chlorosilyl group containing compound and an alkylsilane, or the reaction products thereof. In some embodiments, the surface-treating reagent may comprise an organofunctional silane and an aryl acid halide, or the reaction products thereof. Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.</p> |  |
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| an oil. | <p>[0087] The fracturing fluid may be an oil base, water base, acid, emulsion, foam, or any other fluid.</p> <p>[0009] These benefits can be achieved by a variety of techniques, including coating the proppants with a hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes, polytetrafluoroethylene (commonly known as Teflon®), plant oils, such as linseed oil, soybean oil, corn oil, cottonseed oil, vegetable oil (widely commercially available such as Crisco®), and canola oil, and hydrocarbons such as kerosene, diesel, and crude oil, petroleum distillates such as hydrocarbon liquids comprising a mixture of C<sub>7</sub> - C<sub>12</sub> aliphatic and alicyclic hydrocarbons and aromatic hydrocarbons (C<sub>7</sub>-C<sub>12</sub>), commonly known as Stoddard Solvent, aliphatic solvents,</p> | <p>[0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> | <p>Abstract: Foam fracturing fluids and methods for fracturing well formations using foam as the carrier for the propping agents are disclosed.</p> <p>Col. 4, l. 28-33: Oil may be added to the foam-water slurry as a lubricant and to prevent clay swelling; oil may replace all of the water in the sand slurry, in which case a chemical suitable for foaming oil is used in place of the surfactant. Other friction reducing materials may be added to the fracturing fluid.</p> <p>Col. 7, l. 16-20: To summarize, the invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a liquid carrier comprising one or more liquids selecting of the group consisting of water and oil, and a solid proppant . . .</p> |
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|  | <p>solvent naphtha (medium aliphatic and light aromatic), and paraffin, such as solvent dewaxed heavy paraffinic petroleum distillate.</p> <p>Claim 1: A gas and oil well proppant comprising: a plurality of essentially spherical particles, wherein the particles are coated with a hydrophobic material.</p> <p>Claim 2: The proppant of claim 1, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, hydrocarbons, copolymerized polyvinylidene chloride, glass frit and enamel.</p> <p>Claim 10: The method of claim 9, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones,</p> | <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> <p>[0054] In certain embodiments of the present invention, the surface-treating reagent may be encapsulated with various materials, which, among other things, delays its reaction with the mineral surface and/or other substances present. Solid surface-treating reagents can be encapsulated by spray coating a variety of materials thereon. Such coating materials include, but are not limited to, waxes, drying oils such as tung oil and linseed oil, polyurethanes and cross-linked partially hydrolyzed polyacrylics.</p> | <p>Col. 8, l. 30-32: Method according to claim 1, wherein the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.</p> |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>siloxanes, polytetrafluoroethylene, plant oil, hydrocarbons, copolymerized, polyvinylidene chloride, glass frit and enamel.</p> <p>Claim 21: The method of claim 20, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, hydrocarbons, copolymerized polyvinylidene chloride, glass frit and enamel.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin-coated sand and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability . . .</p> <p>[0012], [0014] Thus, various embodiments of the present</p> | <p>[0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.</p> <p>Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                                                                                                    | invention relate to concepts and techniques to treat fracturing sand and/or proppant to: . . . 2) alter or modify proppant wettability to control the relative permeability to flow of the fluids which may be encountered in the reservoir (such as oil, water, gas, chemical treatments, and fracturing fluids).                                                                                                                             | into the subterranean formation.                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |                                                     |
| <b>Claim 21 of U.S. Patent 8,236,738</b>                                                                                                                                                                           | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                                                    | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                               | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 21. The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric beads, ceramic, glass spheres, carbonate and bauxite particulates. | <p>[0007] The present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are natural sands or ceramic granules used in the hydraulic fracturing of oil and gas wells.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin coated sand and manufactured proppants used in oil and gas recovery to achieve one or more of the</p> | [0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., |                                                     |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>following desirable effects:<br/> alter the wettability . . . Sands,<br/> resin coated sands or<br/> manufactured proppants are<br/> treated, such as by coating, so<br/> as to provide a smoother<br/> surface to the<br/> particles/proppants, to modify<br/> their wettability or fluid<br/> affinity, to modify their<br/> chemical reactivity, or to<br/> reduce particle-to-particle<br/> friction properties.</p> <p>[0087] Thereafter, particles as<br/> hereinbefore described are<br/> placed in the fracture by<br/> injecting into the fracture a<br/> fluid or “slurry” into which the<br/> particles have previously been<br/> introduced and suspended.</p> | <p>glass beads), polymer<br/> materials, Teflon® materials,<br/> nut shell pieces, seed shell<br/> pieces, cured resinous<br/> particulates comprising nut<br/> shell pieces, cured resinous<br/> particulates comprising seed<br/> shell pieces, fruit pit pieces,<br/> cured resinous particulates<br/> comprising fruit pit pieces,<br/> wood, composite particulates<br/> and combinations thereof.<br/> Composite particulates may<br/> also be suitable, suitable<br/> composite materials may<br/> comprise a binder and a filler<br/> material wherein suitable filler<br/> materials include silica,<br/> alumina, filmed carbon, carbon<br/> black, graphite, mica, titanium<br/> dioxide, meta-silicate, calcium<br/> silicate, kaolin, talc, zirconia,<br/> boron, fly ash, hollow glass<br/> microspheres, solid glass,<br/> ground nut/seed shells or<br/> husks, saw dust, ground<br/> cellulose fiber, and<br/> combinations thereof.</p> <p>[0057] In some embodiments,<br/> the surface-treating reagent<br/> may be allowed to modify the</p> |  |
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|                                                                     |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.                                                                                                                                                                                                                                    |                                                     |
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| <b>Claim 22 of U.S. Patent 8,236,738</b>                            | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                    | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 22. The composition of claim 1, wherein the particulates are sands. | <p>[0007] The present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are natural sands or ceramic granules used in the hydraulic fracturing of oil and gas wells.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin coated sand and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability . . . Sands,</p> | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent</p> |                                                     |

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|                                                                                                             | <p>resin coated sands or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.</p> <p>[0087] Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.</p> | <p>may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p> |                                                     |
| <b>Claim 32 of U.S. Patent 8,236,738</b>                                                                    | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                         | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                         | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 32. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: | [0012], [0014], [0015] Thus, various embodiments of the present invention relate to concepts and techniques to treat fracturing sand and/or proppant to: . . . 2) alter or modify proppant wettability, to control the relative permeability to flow of the fluids which may be                                                                                                                                                                                                                                     | [0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing                                                             |                                                     |

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|  | <p>encountered in the reservoir (such as oil, water, gas, chemical treatments, and fracturing fluids), 3) “lubricate” the proppant to allow more efficient proppant arrangement when the fracture closes, effectively increasing packing efficiency . . .</p> <p>[0028] The “lubrication” concept reduces proppant friction, allowing superior proppant redistribution during fracture closing. This redistribution allows more efficient packing of proppant, thereby increasing grain-to-grain contact and effectively increasing proppant pack strength and reducing proppant crush.</p> | <p>operations, perforation operations, and the like.</p> <p>[0004] One problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, proppant particulates, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation or equipment placed in the formation.</p> <p>[0014] Thus, one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, preventing build-up of filter cake and/or gel residue in conductive channels (e.g.,</p> |  |
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|                                                                                                                           |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | fractures) within a subterranean formation, and preventing the embedment of particulates into the surfaces of a subterranean formation.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |
| mixing water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic and an oil; and | <p>[0087] The fracturing fluid may be an oil base, water base, acid, emulsion, foam, or any other fluid. . . . Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which particles have previously been introduced and suspended.</p> <p>[0002] The present invention relates to oil and gas well proppants and, more particularly, to processes for physically or chemically modifying the surface characteristics of hydraulic fracturing proppants.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin-coated sand, and</p> | <p>Abstract: Provided are methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates, at least one of which comprises a mineral surface; providing a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; and allowing the surface-treating reagent modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate.</p> <p>[0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is</p> | <p>Abstract: Foam fracturing fluids and methods for fracturing well formations using foam as the carrier for the propping agents are disclosed.</p> <p>Col. 4, l. 28-33: Oil may be added to the foam-water slurry as a lubricant and to prevent clay swelling; oil may replace all of the water in the sand slurry, in which case a chemical suitable for foaming oil is used in place of the surfactant. Other friction reducing materials may be added to the fracturing fluid.</p> <p>Col. 7, l. 16-20: To summarize, the invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a liquid carrier comprising one or more liquids selecting of the group</p> |

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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability, alter the chemical reactivity, alter the surface topography, impart lubricity, and control relative permeability to flow of fluids such as proppants. Sands, resin coated sands, or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, the modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties</p> <p>[0009] These benefits can be achieved by a variety of techniques, including coating the proppants with a hydrophobic material such as silicon containing compounds, including silicone materials and siloxanes, polytetrafluoroethylene (commonly known as</p> | <p>aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are</p> | <p>consisting of water and oil, and a solid proppant . . .</p> <p>Col. 8, l. 30-32: Method according to claim 1, wherein the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.</p> |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>Teflon®), plant oils, such as linseed oil, soybean oil, corn oil, cottonseed oil, vegetable oil (widely commercially available such as Crisco®), and canola oil, and hydrocarbons such as kerosene, diesel, and crude oil, petroleum distillates such as hydrocarbon liquids comprising a mixture of C<sub>7</sub> - C<sub>12</sub> aliphatic and alicyclic hydrocarbons and aromatic hydrocarbons (C<sub>7</sub>-C<sub>12</sub>), commonly known as Stoddard Solvent, aliphatic solvents, solvent naphtha (medium aliphatic and light aromatic), and paraffin, such as solvent dewaxed heavy paraffinic petroleum distillate.</p> <p>[0029] According to embodiments of the present invention, coatings affect wettability and provide significant flow benefits under multiphase flow as evidenced by the trapped gas saturation, the altered surface tension/contact angles, and the</p> | <p>generally comprised of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> <p>[0056] This may be accomplished in treatments performed prior to transporting the particulates to a job site, or in a treatment performed “on-the-fly.” The term “on-the-fly” is used herein to mean that one flowing stream comprising particulates is continuously introduced into another flowing stream comprising the surface-treating reagent so that the streams are combined and mixed while continuing to flow as a single stream as part of the on-going treatment at the job site. Such mixing can also be described as “real-time” mixing.</p> <p>[0018] The particulates suitable for use in the present invention comprise any</p> |  |
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|  | <p>electrostatic charges on the coated proppant.</p> <p>[0043] Coated samples of a sintered bauxite proppant commercially available from CARBO Ceramics Inc. . . . were prepared by coating the proppant with a product that is commercially available from SOPUS Products under the tradename “Rain-X®”. Rain-X® is a glass surface treatment material that includes polyalkyl hydrogen siloxane, ethanol and isopropanol.</p> <p>[0082] First, in both gel-contaminated and uncontaminated packs, the Rain-X® treated proppant initially allowed reduced flowtimes. Secondly, both samples treated with Rain-X® showed significantly increasing flowtimes to water with subsequent flushes. It was visually observed that the Rain-X® coated proppant trapped air bubbles that accumulated over time. It was</p> | <p>particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica,</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>clear that the proppant was hydrophobic.</p> <p>Claim 2: The proppant of claim 1, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, hydrocarbons, copolymerized polyvinylidene chloride, glass frit and enamel.</p> <p>Claim 6: The proppant of claim 2, wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and polydimethyl siloxane.</p> <p>Claim 10: The method of claim 9, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant</p> | <p>alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.</p> <p>[0015] A mineral surface whose reactivity has been modified according to this definition is herein referred to as “a modified mineral surface.” In certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, by hindering or preventing the water-wetting of that mineral surface. Such</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>oils, hydrocarbons, copolymerized, polyvinylidene chloride, glass frit and enamel.</p> <p>Claim 21: The method of claim 20, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, hydrocarbons, copolymerized polyvinylidene chloride, glass frit and enamel.</p> <p>Claims 1-6, 9-10, and 15-17 also meet this limitation.</p> | <p>compounds may be hydrophobic or hydrophilic in nature.</p> <p>[0049] In some embodiments, the surface-treating reagent may comprise other compounds that are capable of hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include, but are not limited to, vinyl monomers, dienes, keto esters, amines, substituted amine hydrochlorides, amides, alcohols, organosilanes, organotitanates, organozirconates, trivalent metal cations, tetravalent metal cations, ammonium halides, quaternary ammonium halides, ammonium salts of inorganic acids, ammonium salts of carboxylic acids, oligomeric materials, monomeric materials, oil-wetting compounds, and derivatives thereof. In some embodiments, the surface-treating reagent may comprise lecithin. In some embodiments, the surface-</p> |  |
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|  |  | <p>treating reagent may comprise a chlorosilyl group containing compound and an alkylsilane, or the reaction products thereof. In some embodiments, the surface-treating reagent may comprise an organofunctional silane and an aryl acid halide, or the reaction products thereof. Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.</p> <p>[0054] In certain embodiments of the present invention, the surface-treating reagent may be encapsulated with various materials, which, among other things, delays its reaction with the mineral surface and/or other substances present. Solid surface-treating reagents can be encapsulated by spray coating a variety of materials thereon. Such coating materials include, but are not limited to, waxes, drying oils such as tung oil and linseed oil,</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                         |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | polyurethanes and cross-linked partially hydrolyzed polyacrylics.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |  |
| pumping the mixture into the formation. | Claim 9: A method of fracturing a subterranean formation, comprising: injecting a hydraulic fluid into a subterranean formation at a rate and pressure sufficient to open a fracture therein; and injecting into the fracture a fluid containing a plurality of essentially spherical particles, wherein at least some of the particles are modified to alter the surface characteristics thereof, wherein the particles are modified by coating the particles with a hydrophobic material. | Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid into the subterranean formation.<br><br>Claim 23: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified |  |



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|                                                                                                                                                                                      |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | material surface; and introducing the treatment fluid into the subterranean formation.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |                                                     |
| <b>Claim 40 of U.S. Patent 8,236,738</b>                                                                                                                                             | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 40. The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates. | <p>[0007] The present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are natural sands or ceramic granules used in the hydraulic fracturing of oil and gas wells.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin coated sand and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability . . . Sands, resin coated sands or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify</p> | [0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, |                                                     |

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|  | <p>their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.</p> <p>[0087] Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.</p> | <p>cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

|                                                                 |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | and/or fluid into a subterranean formation or well bore.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                                                     |
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| <b>Claim 41 of U.S. Patent 8,236,738</b>                        | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 41. The method of claim 32, wherein the particulates are sands. | <p>[0007] The present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are natural sands or ceramic granules used in the hydraulic fracturing of oil and gas wells.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin coated sand and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability . . . Sands, resin coated sands or manufactured proppants are treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their</p> | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to</p> |                                                     |

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|                                                                                                             | chemical reactivity, or to reduce particle-to-particle friction properties.<br><br>[0087] Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.                                                                                                                    | introducing that solution and/or fluid into a subterranean formation or well bore.                                                                                                                                                                                                                                                                                                           |                                                     |
| <b>Claim 51 of U.S. Patent 8,236,738</b>                                                                    | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                        | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                   | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 51. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: | [0012], [0014], [0015] Thus, various embodiments of the present invention relate to concepts and techniques to treat fracturing sand and/or proppant to: . . . 2) alter or modify proppant wettability, to control the relative permeability to flow of the fluids which may be encountered in the reservoir (such as oil, water, gas, chemical treatments, and fracturing fluids), 3) “lubricate” the proppant to | [0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.<br><br>[0004] One problem that may affect fluid conductivity in the |                                                     |

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|  | <p>allow more efficient proppant arrangement when the fracture closes, effectively increasing packing efficiency . . .</p> <p>[0028] The “lubrication” concept reduces proppant friction, allowing superior proppant redistribution during fracture closing. This redistribution allows more efficient packing of proppant, thereby increasing grain-to-grain contact and effectively increasing proppant pack strength and reducing proppant crush.</p> | <p>formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, proppant particulates, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation or equipment placed in the formation.</p> <p>[0014] Thus, one object of the present invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, preventing build-up of filter cake and/or gel residue in conductive channels (e.g., fractures) within subterranean formation, and preventing the embedment of particulates into the surfaces of a subterranean formation.</p> |  |
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| <p>Contacting particulates with a medium containing a chemical compound for rendering the surface of the particulates hydrophobic,</p> | <p>[0023], [0024] Thus, exemplary techniques for treating proppant include but are not limited to: 1) pretreating proppant prior to the fracturing treatment,</p> <p>[0036] In each case, the coating was applied by mixing the proppant and the coating in a beater for approximately 30 minutes, then drying it for approximately 15 to 18 hours in an oven.</p> <p>[0037] The coating materials were added as follows. Polymethylhydrogen siloxane was added as either a 2 or 5 weight percent emulsion of siloxane in water, polydimethyl siloxane was added as a 5 weight percent emulsion of siloxane in water and Stoddard Solvent was added without water dilution. All samples were dried at 113° C. for approximately 15 to 18 hours.</p> | <p>[0057] Other suitable methods for allow the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate include, but are not limited to, methods utilizing a drive mixer and solvent extraction methods. . . . The solution comprising the surface-treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced into a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface...</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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| Separating the particulate from the medium;                                       | <p>[0036] In each case, the coating was applied by mixing the proppant and the coating in a beaker for approximately 30 minutes, then drying it for approximately 15 to 18 hours in an oven.</p> <p>[0037] The coating materials were added as follows. Polymethylhydrogen siloxane was added as either a 2 or 5 weight percent emulsion of siloxane in water, polydimethyl siloxane was added as a 5 weight percent emulsion of siloxane in water and Stoddard Solvent was added without water dilution. All samples were dried at 113° C. for approximately 15 to 18 hours.</p> | <p>[0057] Other suitable methods for allow the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate include, but are not limited to, methods utilizing a drive mixer and solvent extraction methods. . . . The solution comprising the surface-treating reagent then may be coated onto the mineral surface of the particulates by adding the solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced into a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface...</p> |                                                                                                                  |
| And blending the hydrophobic particulate with water and an oil to form a mixture; | <p>[0087] The fracturing fluid may be an oil base, water base, acid, emulsion, foam, or any other fluid. . . . Thereafter, particles as hereinbefore</p>                                                                                                                                                                                                                                                                                                                                                                                                                          | <p>[0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | <p>Abstract: Foam fracturing fluids and methods for fracturing well formations using foam as the carrier for</p> |

U.S. Patent 8,236,738 (Zhang) v. U.S. Publication 2005/0244641 (Vincent) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.</p> | <p>aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are</p> | <p>the propping agents are disclosed.</p> <p>Col. 4, l. 28-33: Oil may be added to the foam-water slurry as a lubricant and to prevent clay swelling; oil may replace all of the water in the sand slurry, in which case a chemical suitable for foaming oil is used in place of the surfactant. Other friction reducing materials may be added to the fracturing fluid.</p> <p>Col. 7, l. 16-20: To summarize, the invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a liquid carrier comprising one or more liquids selecting of the group consisting of water and oil, and a solid proppant . . .</p> <p>Col. 8, l. 30-32: Method according to claim 1, wherein the liquid component of said foam comprises at least one</p> |
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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                             |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | generally comprised of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | liquid selected from the group consisting of water and oil. |
| And pumping the mixture into the formation. | Claim 9: A method of fracturing a subterranean formation, comprising: injecting a hydraulic fluid into a subterranean formation at a rate and pressure sufficient to open a fracture therein; and injecting into the fracture a fluid containing a plurality of essentially spherical particles, wherein at least some of the particles are modified to alter the surface characteristics thereof, wherein the particles are modified by coating the particles with a hydrophobic material. | Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid into the subterranean formation.<br><br>Claim 23: A method of treating a subterranean formation comprising: providing a |                                                             |

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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                     |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified material surface; and introducing the treatment fluid into the subterranean formation.                                                                                                                                                                                                                                                                                                                                                                               |                                                     |
| <b>Claim 57 of U.S. Patent 8,236,738</b>                            | <b>Reference to U.S. Publication 2005/0244641 (Vincent)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 57. The composition of claim 51, wherein the particulates are sands | <p>[0007] The present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are natural sands or ceramic granules used in the hydraulic fracturing of oil and gas wells.</p> <p>[0008] Embodiments of the present invention relate to modifying the surface properties of natural sand, resin coated sand and manufactured proppants used in oil and gas recovery to achieve one or more of the following desirable effects: alter the wettability . . . Sands, resin coated sands or manufactured proppants are</p> | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of</p> |                                                     |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>treated, such as by coating, so as to provide a smoother surface to the particles/proppants, to modify their wettability or fluid affinity, to modify their chemical reactivity, or to reduce particle-to-particle friction properties.</p> <p>[0087] Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or “slurry” into which the particles have previously been introduced and suspended.</p> | <p>the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p> |  |
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# EXHIBIT K

U.S. Patent 8,236,738 (Zhang) v. U.S. Patent 4,231,428 (Needham) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

**U.S. Pat. No. 8,236,738 (Zhang)**  
Asserted Claims: 1, 16-23, 32, 33, 36, 38-42

| <b>Claim 1 of U.S. Patent 8,236,738</b>      | <b>Reference to U.S. Patent 4,231,428 (Needham)</b>                                                                                                                                                                                                                                                                                   | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                     | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
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| 1. An aqueous slurry composition comprising: | Col. 3, l. 19-23: Carrier fluid, preferably an aqueous fluid such as salt water or a hydrocarbon liquid such as diesel fuel, having the proppant coated with organo-silicon compound suspended therein is then pumped into the fracture.                                                                                              | [0058] In certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof.                                                        |                                                     |
| water;                                       | Col. 3, l. 19-23: Carrier fluid, preferably an aqueous fluid such as salt water or a hydrocarbon liquid such as diesel fuel, having the proppant coated with organo-silicon compound suspended therein is then pumped into the fracture.<br><br>Col. 4, l. 1-7: In a method of fracturing an underground zone or formation in oil and | [0058] In certain embodiments, particulates treated utilizing the methods of the present invention may be introduced into the subterranean formation as a component of a treatment fluid (e.g., a fracturing fluid). These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh |                                                     |

U.S. Patent 8,236,738 (Zhang) v. U.S. Patent 4,231,428 (Needham) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|               | gas wells, the steps of (a) injecting into a well a fracturing fluid to form a fracture in said formation, (b) injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound . . . | water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid.<br><br>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprise of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon. |  |
| particulates; | Col. 3, l. 5-12: Propping agents used in hydraulic fracturing normally range in particle size from about 8 to about 60 mesh, with the most common range being between 20 and 40                                                                                     | Abstract: Provided are methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |  |

U.S. Patent 8,236,738 (Zhang) v. U.S. Patent 4,231,428 (Needham) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                           | <p>mesh. Sand is by far the most common propping agent currently used because of its low cost and great abundance. However, under certain conditions, light strength glass beads, as well as bauxite (aluminum oxide), or other propping agents can be used.</p>                                                                                                                                                                                                                                                                                                                                                                       | <p>one embodiment, the methods comprise: providing a plurality of particulates. . .</p> <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface.</p>                                                                                                                                                                                                                                                                                                                                                                                                |  |
| <p>a chemical compound for rendering the surface of the particulates hydrophobic; and</p> | <p>Col. 1, l. 44-52: Accordingly, an object of this invention is to provide improved propping agents for use in hydraulic fracturing of subterranean formations. Another object of this invention is to provide particulate material coated with a water repellent useful in hydraulic fracturing of subterranean formations. A further object of this invention is to coat particulate solids useful as propping agents to increase the strength of the propping agent in the presence of water.</p> <p>Col. 1, l. 56-68: In accordance with the invention, a novel propping agent comprising an inert solid particle coated with</p> | <p>[0015] A mineral surface whose reactivity has been modified according to this definition is herein referred to as “a modified mineral surface.” In certain embodiments of the present invention, modifying the stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, by hindering or preventing the water-wetting of that mineral surface. Such compounds may be</p> |  |

U.S. Patent 8,236,738 (Zhang) v. U.S. Patent 4,231,428 (Needham) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>a water-repellent organo-silicon material is displaced down a well and into a fracture in a subterranean hydrocarbon-bearing formation. In accordance with a specific embodiment, sand coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.</p> <p>Col. 2, l. 25-41: The water-repellent material employed in the method of the present invention to coat inert particulate materials for use as propping agents is an oil-soluble organo-silicon compound. Representative organo-silicon compounds that can be used include polyalkylsiloxanes such as polymethylsiloxanes,</p> | <p>hydrophobic or hydrophilic in nature.</p> <p>[0049] In some embodiments, the surface-treating reagent may comprise other compounds that are capable of hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include, but are not limited to, vinyl monomers, dienes, keto esters, amines, substituted amine hydrochlorides, amides, alcohols, organosilanes, organotitanates, organozirconates, trivalent metal cations, tetravalent metal cations, ammonium halides, quaternary ammonium halides, ammonium salts of inorganic acids, ammonium salts of carboxylic acids, oligomeric materials, monomeric materials, oil-wetting compounds, and derivatives thereof. In some embodiments, the surface-treating reagent may comprise lecithin. In some embodiments, the surface-treating reagent may comprise</p> |  |
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U.S. Patent 8,236,738 (Zhang) v. U.S. Patent 4,231,428 (Needham) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|         | <p>polyethylsiloxanes, and the like. Additionally, polyalkylarylsiloxanes such as polymethylphenylsiloxane are also usable as a coating material of the invention. Chlorosilanes such as chlorotrimethylsilane and other silyl donors can also be used, as well as various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes, and the like, as the water-repellent coating of the propping agent. Other organo-silicon oil-soluble compounds which can be used include the ethyl silicates, methyl sodium silanolate, and other silicon resins such as mixtures of silane esters, silyl amines, and the like.</p> | <p>a chlorosilyl group containing compound and an alkylsilane, or the reaction products thereof. In some embodiments, the surface-treating reagent may comprise an organofunctional silane and an aryl acid halide, or the reaction products thereof. Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.</p> |                                                                                                                                                                                                                                                                           |
| an oil. | <p>Col. 4, l. 1-2: In a method of fracturing an underground zone or formation in oil and gas wells . . .</p> <p>Col. 1, l. 4-5: This invention relates to the treatment of subterranean hydrocarbon-bearing formations.</p>                                                                                                                                                                                                                                                                                                                                                                                                      | <p>[0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g.,</p>                                                                                                                                                           | <p>Col. 4, l. 28-33: Oil may be added to the foam-water slurry as a lubricant and to prevent clay swelling; oil may replace all of the water in the sand slurry, in which case a chemical suitable for foaming oil is used in place of the surfactant. Other friction</p> |

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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>Col. 1, l. 56-60: In accordance with the invention, a novel propping agent comprising an inert solid particle coated with a water-repellent organo-silicon material is displaced down a well and into a fracture in a subterranean hydrocarbon-bearing formation.</p> | <p>saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. . . . Suitable emulsions can be comprised of two immiscible liquids such as an aqueous</p> | <p>reducing materials may be added to the fracturing fluid.</p> <p>Col. 7, l. 16-20: To summarize, the invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a liquid carrier comprising one or more liquids selecting of the group consisting of water and oil, and a solid proppant . . .</p> <p>Col. 8, l. 30-32: Method according to claim 1, wherein the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.</p> |
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U.S. Patent 8,236,738 (Zhang) v. U.S. Patent 4,231,428 (Needham) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  |  | <p>liquid or gelled liquid and a hydrocarbon.</p> <p>[0054] In certain embodiments of the present invention, the surface-treating reagent may be encapsulated with various materials, which, among other things, delays its reaction with the mineral surface and/or other substances present. Solid surface-treating reagents can be encapsulated by spray coating a variety of materials thereon. Such coating materials include, but are not limited to, waxes, drying oils such as tung oil and linseed oil, polyurethanes and cross-linked partially hydrolyzed polyacrylics.</p> <p>[0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing</p> |  |
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U.S. Patent 8,236,738 (Zhang) v. U.S. Patent 4,231,428 (Needham) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

|                                                                                                                                                 |                                                                                                                                                  | operations, frac-packing operations, perforation operations, and the like.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                                                     |
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|                                                                                                                                                 |                                                                                                                                                  | Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid into the subterranean formation. |                                                     |
| <b>Claim 21 of U.S. Patent 8,236,738</b>                                                                                                        | <b>Reference to U.S. Patent 4,231,428 (Needham)</b>                                                                                              | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 21. The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, synthetic polymeric | Col. 1, l. 61-68: In accordance with a specific embodiment, sand coated with a water-repellent material is prepared by dissolving an oil-soluble | [0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be                                                                                                                                                                                                                                                                                                                                                                                                                              |                                                     |

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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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| beads, ceramic, glass spheres, carbonate and bauxite particulates. | <p>organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.</p> <p>Col. 4:1-7: In a method of fracturing an underground zone or formation in oil and gas wells, the steps of (a) injecting into a well a fracturing fluid to form a fracture in said formation, (b) injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound . . .</p> <p>Col. 4, l. 16-17: A method according to claim 1 wherein said particulate material comprises sand . . .</p> <p>Col. 4, l. 48-49: A method according to claim 5 wherein</p> | <p>understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium</p> |  |
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U.S. Patent 8,236,738 (Zhang) v. U.S. Patent 4,231,428 (Needham) in view of  
U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                     | said particles comprise sand . .<br>.                                                                                                                                                                        | dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.<br><br>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore. |                                                     |
| <b>Claim 22 of U.S. Patent 8,236,738</b>                            | <b>Reference to U.S. Patent 4,231,428 (Needham)</b>                                                                                                                                                          | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 22. The composition of claim 1, wherein the particulates are sands. | Col. 1, l. 61-68: In accordance with a specific embodiment, sand coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the | [0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this                                                                                                                                                                                                                                                                                                                                                                                                                  |                                                     |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>resultant solution with sand, and thereafter evaporating the solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.</p> <p>Col. 4:1-7: In a method of fracturing an underground zone or formation in oil and gas wells, the steps of (a) injecting into a well a fracturing fluid to form a fracture in said formation, (b) injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound . . .</p> <p>Col. 4, l. 16-17: A method according to claim 1 wherein said particulate material comprises sand . . .</p> <p>Col. 4, l. 48-49: A method according to claim 5 wherein said particles comprise sand . . .</p> | <p>disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

| Claim 32 of U.S. Patent 8,236,738                                                                           | Reference to U.S. Patent 4,231,428 (Needham) | Reference to U.S. Publication 2006/0260808 (Weaver)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | Reference to U.S. Patent 3,980,136 (Plummer) |
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| 32. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: |                                              | <p>[0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.</p> <p>[0004] One problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, proppant particulates, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation or equipment placed in the formation.</p> |                                              |



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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                           |                                                                                                                                                                                                                                                                                                                                                               | [0014] Thus, one object of the invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, preventing build-up of filter cake and/or gel residue in conductive channels (e.g., fractures) within a subterranean formation, and preventing the embedment of particulates into the surfaces of a subterranean formation. |                                                                                                                                                                                                                                                                                                                             |
| mixing water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic and an oil; and | Abstract: In a method of propping a fracture in subterranean formations, the invention comprises placing in the fractures particulate solids such as sand coated with a water-repellent coating composition such as an oil-soluble organo-silicon compound. The water-repellent coated solid particulate propping agent can be prepared by dissolving an oil- | Abstract: Provided are methods of modifying the surface stress-activated reactivity of proppant particulates used in subterranean operations. In one embodiment, the methods comprise: providing a plurality of particulates, at least one of which comprises a mineral surface; providing a surface-treating reagent capable of modifying the stress-activated                                                                                                                                                                  | Col. 4, l. 28-33: Oil may be added to the foam-water slurry as a lubricant and to prevent clay swelling; oil may replace all of the water in the sand slurry, in which case a chemical suitable for foaming oil is used in place of the surfactant. Other friction reducing materials may be added to the fracturing fluid. |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with particulate material, and thereafter evaporating the solvent to form a coating of organo-silicon compound on the particulate material.</p> <p>Col. 3, l. 19-23: Carrier fluid, preferably an aqueous fluid such as salt water or a hydrocarbon liquid such as diesel fuel, having the proppant coated with organo-silicon compound suspended therein is then pumped into the fracture.</p> <p>Col. 4:1-7: In a method of fracturing an underground zone or formation in oil and gas wells, the steps of (a) injecting into a well a fracturing fluid to form a fracture in said formation, (b) injecting into said formation a carrier fluid having suspended therein a proppant composed</p> | <p>reactivity of a mineral surface of a particulate; and allowing the surface-treating reagent modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate.</p> <p>[0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in</p> | <p>Col. 7, l. 16-20: To summarize, the invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a liquid carrier comprising one or more liquids selecting of the group consisting of water and oil, and a solid proppant . . .</p> <p>Col. 8, l. 30-32: Method according to claim 1, wherein the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.</p> |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>of particles coated with an organo-silicon compound . . .</p> <p>Col. 3, l. 5-12: Propping agents used in hydraulic fracturing normally range in particle size from about 8 to about 60 mesh, with the most common range being between 20 and 40 mesh. Sand is by far the most common propping agent currently used because of its low cost and great abundance. However, under certain conditions, light strength glass beads, as well as bauxite (aluminum oxide), or other propping agents can be used.</p> <p>Col. 4, l. 29-42: A method of treating fractured earthen formations in oil and gas wells comprising the steps of (a) mixing together a quantity of particulate solids suitable for use as propping agents and a quantity of an oil-soluble organo-silicon compound dissolved in a solvent and heating the resulting mixture to a temperature sufficient to</p> | <p>which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprised of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> <p>[0056] This may be accomplished in treatments performed prior to transporting the particulates to a job site, or in a treatment performed “on-the-fly.” The term “on-the-fly” is used herein to mean that one flowing stream comprising particulates is continuously introduced into another</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>evaporate said solvent and leave said solids coated with an oil-wettable coating of said organo-silicon compound, (b) dispersing said coated particulate solids into a carrying fluid, (c) introducing said carrying fluid and dispersed coated solids into a well penetrating a subterranean formation containing fractures...</p> <p>Col. 1, l. 44-52: Accordingly, an object of this invention is to provide improved propping agents for use in hydraulic fracturing of subterranean formations. Another object of this invention is to provide particulate material coated with a water repellent useful in hydraulic fracturing of subterranean formations. A further object of this invention is to coat particulate solids useful as propping agents to increase the strength of the propping agent in the presence of water.</p> | <p>flowing stream comprising the surface-treating reagent so that the streams are combined and mixed while continuing to flow as a single stream as part of the on-going treatment at the job site. Such mixing can also be described as “real-time” mixing.</p> <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut</p> |  |
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|  | <p>Col. 1, l. 56-68: In accordance with the invention, a novel propping agent comprising an inert solid particle coated with a water-repellent organo-silicon material is displaced down a well and into a fracture in a subterranean hydrocarbon-bearing formation. In accordance with a specific embodiment, sand coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.</p> <p>Col. 2, l. 25-41: The water-repellent material employed in the method of the present invention to coat inert particulate materials for use as propping agents is an oil-soluble organo-silicon compound. Representative</p> | <p>shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia, boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.</p> <p>[0015] A mineral surface whose reactivity has been modified according to this definition is herein referred to as “a modified mineral surface.” In certain embodiments of the present invention, modifying the</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>organo-silicon compounds that can be used include polyalkylsiloxanes such as polymethylsiloxanes, polyethylsiloxanes, and the like. Additionally, polyalkylarylsiloxanes such as polymethylphenylsiloxane are also usable as a coating material of the invention. Chlorosilanes such as chlorotrimethylsilane and other silyl donors can also be used, as well as various alkoxysilanes, aroxysilanes, alkoxysiloxanes, and aroxysiloxanes, and the like, as the water-repellent coating of the propping agent. Other organo-silicon oil-soluble compounds which can be used include the ethyl silicates, methyl sodium silanolate, and other silicon resins such as mixtures of silane esters, silyl amines, and the like.</p> | <p>stress-activated reactivity of a mineral surface may comprise depositing one or more compounds on the mineral surface of a particulate that are capable of affecting the ability of the mineral surface to interact with any aqueous fluid that may be present in a subterranean formation, for example, by hindering or preventing the water-wetting of that mineral surface. Such compounds may be hydrophobic or hydrophilic in nature.</p> <p>[0049] In some embodiments, the surface-treating reagent may comprise other compounds that are capable of hindering or preventing the water-wetting of a mineral surface. Examples of suitable compounds include, but are not limited to, vinyl monomers, dienes, keto esters, amines, substituted amine hydrochlorides, amides, alcohols, organosilanes, organotitanates, organozirconates, trivalent</p> |  |
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|  |  | <p>metal cations, tetravalent metal cations, ammonium halides, quaternary ammonium halides, ammonium salts of inorganic acids, ammonium salts of carboxylic acids, oligomeric materials, monomeric materials, oil-wetting compounds, and derivatives thereof. In some embodiments, the surface-treating reagent may comprise lecithin. In some embodiments, the surface-treating reagent may comprise a chlorosilyl group containing compound and an alkylsilane, or the reaction products thereof. In some embodiments, the surface-treating reagent may comprise an organofunctional silane and an aryl acid halide, or the reaction products thereof. Examples of suitable organofunctional silanes for these embodiments include, but are not limited to, aminofunctional silanes, ureidofunctional silanes, and epoxyfunctional silanes.</p> <p>[0054] In certain embodiments of the present invention, the</p> |  |
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|                                         |                                                                                                                                                                                                                                                                                                                                                                                                                            | surface-treating reagent may be encapsulated with various materials, which, among other things, delays its reaction with the mineral surface and/or other substances present. Solid surface-treating reagents can be encapsulated by spray coating a variety of materials thereon. Such coating materials include, but are not limited to, waxes, drying oils such as tung oil and linseed oil, polyurethanes and cross-linked partially hydrolyzed polyacrylics. |  |
| pumping the mixture into the formation. | Col. 4, l. 1-15: In a method of fracturing an underground zone or formation in oil and gas wells, the steps of<br>(a) injecting into a well a fracturing fluid to form a fracture in said formation,<br>(b) injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound prepared by dissolving the organo-silicon compound in a solvent | Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion                     |  |



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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|                                                                                                                                                                                      | <p>therefor, admixing the resultant solution with particulate material, and thereafter evaporating the solvent to form a silicon-coated particulate material, and</p> <p>(c) closing the well and maintaining the injection pressure on the formation until the fracture approaches the normal formation pressure.</p> <p>Col. 3, l. 19-23: Carrier fluid, preferably an aqueous fluid such as salt water or a hydrocarbon liquid such as diesel fuel, having the proppant coated with organo-silicon compound suspended therein is then pumped into the fracture.</p> | <p>of the mineral surface of at least one particulate; and introducing the treatment fluid into the subterranean formation.</p> <p>Claim 23: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified material surface; and introducing the treatment fluid into the subterranean formation.</p> |                                                     |
| <b>Claim 40 of U.S. Patent 8,236,738</b>                                                                                                                                             | <b>Reference to U.S. Patent 4,231,428 (Needham)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                             | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 40. The method of claim 32, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, synthetic polymeric beads and bauxite particulates. | Col. 1, l. 61-68: In accordance with a specific embodiment, sand coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the                                                                                                                                                                                                                                                                                                                                                           | [0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term "particulate," as used in this                                                                                                                                                                                                                                      |                                                     |

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|  | <p>resultant solution with sand, and thereafter evaporating the solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.</p> <p>Col. 4:1-7: In a method of fracturing an underground zone or formation in oil and gas wells, the steps of (a) injecting into a well a fracturing fluid to form a fracture in said formation, (b) injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound . . .</p> <p>Col. 4, l. 16-17: A method according to claim 1 wherein said particulate material comprises sand . . .</p> <p>Col. 4, l. 48-49: A method according to claim 5 wherein said particles comprise sand . . .</p> | <p>disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand, bauxite, ceramic materials, glass materials (e.g., glass beads), polymer materials, Teflon® materials, nut shell pieces, seed shell pieces, cured resinous particulates comprising nut shell pieces, cured resinous particulates comprising seed shell pieces, fruit pit pieces, cured resinous particulates comprising fruit pit pieces, wood, composite particulates and combinations thereof. Composite particulates may also be suitable, suitable composite materials may comprise a binder and a filler material wherein suitable filler materials include silica, alumina, filmed carbon, carbon black, graphite, mica, titanium dioxide, meta-silicate, calcium silicate, kaolin, talc, zirconia,</p> |  |
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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

|                                                                 |                                                                                                                                                                                                                                                                           | <p>boron, fly ash, hollow glass microspheres, solid glass, ground nut/seed shells or husks, saw dust, ground cellulose fiber, and combinations thereof.</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p> |                                              |
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| Claim 41 of U.S. Patent 8,236,738                               | Reference to U.S. Patent 4,231,428 (Needham)                                                                                                                                                                                                                              | Reference to U.S. Publication 2006/0260808 (Weaver)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | Reference to U.S. Patent 3,980,136 (Plummer) |
| 41. The method of claim 32, wherein the particulates are sands. | Col. 1, l. 61-68: In accordance with a specific embodiment, sand coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the | [0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term "particulate," as used in this disclosure, includes all known shapes of materials including                                                                                                                                                                                                                                                                                          |                                              |

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Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

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|  | <p>solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.</p> <p>Col. 4:1-7: In a method of fracturing an underground zone or formation in oil and gas wells, the steps of (a) injecting into a well a fracturing fluid to form a fracture in said formation, (b) injecting into said formation a carrier fluid having suspended therein a proppant composed of particles coated with an organo-silicon compound . . .</p> <p>Col. 4, l. 16-17: A method according to claim 1 wherein said particulate material comprises sand . . .</p> <p>Col. 4, l. 48-49: A method according to claim 5 wherein said particles comprise sand . . .</p> | <p>substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to introducing that solution and/or fluid into a subterranean formation or well bore.</p> |  |
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U.S. Publication 2006/0260808 (Weaver) or U.S. Patent 3,980,136 (Plummer)  
Challenged Claims: 1, 21, 22, 32, 40, 41, 51, 57

| Claim 51 of U.S. Patent 8,236,738                                                                           | Reference to U.S. Patent 4,231,428 (Needham) | Reference to U.S. Publication 2006/0260808 (Weaver)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | Reference to U.S. Patent 3,980,136 (Plummer) |
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| 51. A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of: |                                              | <p>[0058] The methods of the present invention may be utilized in a variety of subterranean operations known in the art. Suitable subterranean operations include drilling operations, pre-pad treatments, fracturing operations, gravel-packing operations, frac-packing operations, perforation operations, and the like.</p> <p>[0004] One problem that may affect fluid conductivity in the formation after a fracturing treatment is the tendency for particulates (e.g., formation fines, proppant particulates, etc.) to flow back through the conductive channels in the subterranean formation, which can, for example, clog the conductive channels and/or damage the interior of the formation or equipment placed in the formation.</p> |                                              |

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|                                                                                                                                 |                                                                                                                                                                                                                                                                                                                                                                                       | [0014] Thus, one object of the present invention is to preserve and enhance conductivity within a subterranean formation so that the maximum value of a fracturing treatment with respect to increased conductivity may be realized. Other objects of the present invention include, but are not limited to, preventing build-up of filter cake and/or gel residue in conductive channels (e.g., fractures) within subterranean formation, and preventing the embedment of particulates into the surfaces of a subterranean formation. |  |
| Contacting particulates with a medium containing a chemical compound for rendering the surface of the particulates hydrophobic, | Col. 4, l. 29-47: A method of treating fractured earthen formations in oil and gas wells comprising the steps of (a) mixing together a quantity of particulate solids suitable for use as propping agents and a quantity of an oil-soluble organo-silicon compound dissolved in a solvent and heating the resulting mixture to a temperature sufficient to evaporate said solvent and | [0057] Other suitable methods for allow the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate include, but are not limited to, methods utilizing a drive mixer and solvent extraction methods. . . . The solution comprising the surface-treating reagent then may be coated onto the mineral surface of the particulates by adding the                                                                                                                                       |  |

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|                                             | leave said solids coated with an oil-wettable coating of said organo-silicon compound, (b) dispersing said coated particulate solids into a carrying fluid, (c) introducing said carrying fluid and dispersed coated solids into a well penetrating a subterranean formation containing fractures, and (d) applying pressure to the fluid in the well thereby causing the fluid to contact the earthen formations and to be injected into fractures and openings extending into the formation whereby said coated particles are deposited therein. | solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced into a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface...          |  |
| Separating the particulate from the medium; | Col. 4, l. 29-47: A method of treating fractured earthen formations in oil and gas wells comprising the steps of (a) mixing together a quantity of particulate solids suitable for use as propping agents and a quantity of an oil-soluble organo-silicon compound dissolved in a solvent and heating the resulting mixture to a temperature sufficient to evaporate said solvent and                                                                                                                                                              | [0057] Other suitable methods for allow the surface-treating reagent to modify the stress-activated reactivity of the mineral surface of a particulate include, but are not limited to, methods utilizing a drive mixer and solvent extraction methods. . . . The solution comprising the surface-treating reagent then may be coated onto the mineral surface of the particulates by adding the |  |

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|                                                                                   | leave said solids coated with an oil-wettable coating of said organo-silicon compound, (b) dispersing said coated particulate solids into a carrying fluid, (c) introducing said carrying fluid and dispersed coated solids into a well penetrating a subterranean formation containing fractures, and (d) applying pressure to the fluid in the well thereby causing the fluid to contact the earthen formations and to be injected into fractures and openings extending into the formation whereby said coated particles are deposited therein. | solution to the particulates and stirring (or mulling) until the mixture is uniform. The treated particulates then may be introduced into a water-based fluid that extracts the solvent from the treated particulates, leaving behind an insoluble material that, among other things, may enhance the modification of the stress-activated reactivity of the treated mineral surface...            |                                                                                                                                                                                                                                                                                                                                   |
| And blending the hydrophobic particulate with water and an oil to form a mixture; | Col. 4, l. 29-39: A method of treating fractured earthen formations in oil and gas wells comprising the steps of . . . (b) dispersing said coated particulate solids into a carrying fluid . . .                                                                                                                                                                                                                                                                                                                                                   | [0058] These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof. Where the base fluid is aqueous, it may comprise fresh water, salt water (e.g., water containing one or more salts dissolved therein), brine (e.g., saturated salt water), or seawater. The water can be from any source, provided that it does not contain an excess of | Abstract: Foam fracturing fluids and methods for fracturing well formations using foam as the carrier for the propping agents are disclosed.<br><br>Col. 4, l. 28-33: Oil may be added to the foam-water slurry as a lubricant and to prevent clay swelling; oil may replace all of the water in the sand slurry, in which case a |



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|  |  | <p>compounds that may adversely affect other components in the treatment fluid. Where the base fluid is non-aqueous, the base fluid may comprise any number of organic liquids. Examples of suitable organic liquids include, but are not limited to, mineral oils, synthetic oils, esters, and the like. Any organic liquid in which a water solution of salts can be emulsified also may be suitable for use as a base fluid in the methods of the present invention.</p> <p>[0059] Suitable treatment fluids may take on a variety of physical forms, including aqueous gels, viscoelastic surfactant gels, oil gels, foamed gels, and emulsions. Suitable aqueous gels are generally comprised of water and one or more gelling agents. Suitable emulsions can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and a hydrocarbon.</p> | <p>chemical suitable for foaming oil is used in place of the surfactant. Other friction reducing materials may be added to the fracturing fluid.</p> <p>Col. 7, l. 16-20: To summarize, the invention provides a formation fracturing fluid, comprising a pressured stable foam comprising a liquid carrier comprising one or more liquids selecting of the group consisting of water and oil, and a solid proppant . . .</p> <p>Col. 8, l. 30-32: Method according to claim 1, wherein the liquid component of said foam comprises at least one liquid selected from the group consisting of water and oil.</p> |
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| And pumping the mixture into the formation. | Col. 4, l. 29-47: A method of treating fractured earthen formations in oil and gas wells comprising the steps of (a) mixing together a quantity of particulate solids suitable for use as propping agents and a quantity of an oil-soluble organo-silicon compound dissolved in a solvent and heating the resulting mixture to a temperature sufficient to evaporate said solvent and leave said solids coated with an oil-wettable coating of said organo-silicon compound, (b) dispersing said coated particulate solids into a carrying fluid, (c) introducing said carrying fluid and dispersed coated solids into a well penetrating a subterranean formation containing fractures, and (d) applying pressure to the fluid in the well thereby causing the fluid to contact the earthen formations and to be injected into fractures and openings extending into the formation whereby said coated particles are deposited therein. | Claim 11: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, a plurality of particulates, at least one of which comprises a mineral surface, and a surface-treating reagent capable of modifying the stress-activated reactivity of a mineral surface of a particulate; allowing the surface-treating reagent to modify the stress-activated reactivity of at least a portion of the mineral surface of at least one particulate; and introducing the treatment fluid into the subterranean formation.<br><br>Claim 23: A method of treating a subterranean formation comprising: providing a treatment fluid that comprises a base fluid, and a plurality of particulates, at least one of which comprises a modified material surface; and introducing the treatment fluid |  |
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|                                                                     |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | into the subterranean formation.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                                                     |
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| <b>Claim 57 of U.S. Patent 8,236,738</b>                            | <b>Reference to U.S. Patent 4,231,428 (Needham)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | <b>Reference to U.S. Publication 2006/0260808 (Weaver)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | <b>Reference to U.S. Patent 3,980,136 (Plummer)</b> |
| 57. The composition of claim 51, wherein the particulates are sands | <p>Col. 1, l. 61-68: In accordance with a specific embodiment, sand coated with a water-repellent material is prepared by dissolving an oil-soluble organo-silicon compound in a solvent therefor, admixing the resultant solution with sand, and thereafter evaporating the solvent to form a silicon-coated sand which can then be used as a propping medium in fractured formations containing hydrocarbons.</p> <p>Col. 4:1-7: In a method of fracturing an underground zone or formation in oil and gas wells, the steps of (a) injecting into a well a fracturing fluid to form a fracture in said formation, (b) injecting into said formation a carrier fluid having suspended therein a proppant composed</p> | <p>[0018] The particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface. It should be understood that the term “particulate,” as used in this disclosure, includes all known shapes of materials including substantially spherical materials, fibrous materials, polygonal materials (such as cubic materials) and mixtures thereof. Suitable particulates include, but are not limited to, sand . . .</p> <p>[0057] In some embodiments, the surface-treating reagent may be allowed to modify the stress-activated reactivity of the mineral surface of a particulate by placing it in a solution and/or treatment fluid that comprises the particulate, which may be done prior to, during, or subsequent to</p> |                                                     |

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|  | <p>of particles coated with an organo-silicon compound . . .</p> <p>Col. 4, l. 16-17: A method according to claim 1 wherein said particulate material comprises sand . . .</p> <p>Col. 4, l. 48-49: A method according to claim 5 wherein said particles comprise sand . . .</p> | <p>introducing that solution and/or fluid into a subterranean formation or well bore.</p> |  |
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