Filed: September 30, 2017

UNITED STATES PATENT AND TRADEMARK OFFICE

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

U.S. Patent No. 8,236,738 to Zhang

Inter Partes Review IPR2017-02100

Petition for *Inter Partes* Review of U.S. Patent No. 8,236,738

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1002	Declaration of Dr. Gary Wooley, Ph.D. • Ex. A: Resume of Gary Wooley • Ex. B: Recent Case Listing of Gary Wooley • Ex. C: '738 Patent v. Nguyen Claim Chart • Ex. D: '738 Patent v. Dawson Claim Chart • Ex. E: '738 Patent v. Vincent Claim Chart • Ex. F: '738 Patent v. Weaver Claim Chart • Ex. G: '738 Patent v. Needham Claim Chart • Ex. H: '738 Patent v. Nguyen in view of Weaver or Plummer Claim Chart • Ex. I: '738 Patent v. Dawson in view of Weaver or Plummer Claim Chart • Ex. J: '738 Patent v. Vincent in view of Weaver or Plummer Claim Chart • Ex. K: '738 Patent v. Needham in view of Weaver or Plummer Claim Chart
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," as of the filing date of this Petition ("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")

Exhibit #	Description
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")
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")
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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

No.	Limitation
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; and
51C	blending the hydrophobic particulate with water and an oil to form a mixture; and
51D	pumping the mixture into the formation.
57	57. The composition of claim 51, wherein the particulates are sands.

I. INTRODUCTION

Preferred Proppants, LLC, Preferred Pipeline, LLC, Preferred Resin Sales, LLC, Preferred Resin of Genoa, LLC, and Preferred Sands of Wisconsin, LLC ("Petitioners") petition for *Inter Partes* Review of claims 1, 21, 22, 32, 40, 41, 51, and 57 of U.S. Patent 8,236,738 to Zhang ("the '738 patent") (Ex. 1001), which is assigned to Trican Well Service Ltd. ("Trican"), under 35 U.S.C. §§ 311–319 and 37 C.F.R. Part 42 and seek cancelation of claims 1, 21, 22, 32, 40, 41, 51 and 57 of the '738 patent ("the challenged claims") as unpatentable.

This Petition is filed in accordance with 37 C.F.R. § 42.106(a). Also filed is a power of attorney and an exhibit list per § 42.10(b) and § 42.63(e), respectively. Pursuant to 37 C.F.R. § 42.103, the appropriate fee per § 42.15(a) accompanies this Petition.

II. MANDATORY NOTICES

A. Real Parties-In-Interest Under 37 C.F.R. § 42.8(b)(1)

The real parties-in-interest for Petitioners are Preferred Proppants, LLC, Preferred Pipeline, LLC, Preferred Resin Sales, LLC, Preferred Resin of Genoa, LLC, and Preferred Sands of Wisconsin, LLC.

B. Related Matters Under 37 C.F.R. § 42.8(b)(2)

The following litigations or instituted *inter partes* reviews related to the '738 patent are pending: *Trican Well Service, Ltd. v. Preferred Proppants, LLC, Preferred Pipeline, LLC, Preferred Resin Sales, LLC, Preferred Resin of Genoa,*

LLC, and Preferred Sands of Wisconsin, LLC, Case No. 4:17-cv-01340 (S.D. Tex. May 1, 2017) ("Texas litigation"). The Petitioners are concurrently filing a second petition for *inter partes* review related to the '738 patent, IPR2017-02099. Moreover, Petitioners have already filed petitions for *inter partes* review of U.S. Patent 7,723,724 (the "'274 patent") issued to Zhang and assigned to Trican, IPR2017-01807 and IPR2017-01815. While the '274 patent is not related to the '738 patent, they are patentably similar, and Trican has asserted both patents against the same products in the Texas litigation.

C. Lead And Back-Up Counsel Under 37 C.F.R. § 42.8(b)(3)

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D. Service Information Under 37 C.F.R. § 42.8(b)(4)

Please direct all correspondence to counsel at the contact information above. Petitioners consent to electronic service by e-mail at the above listed email addresses.

III. GROUNDS FOR STANDING (37 C.F.R. §§ 42.101 and 42.104)

As required by 37 C.F.R. § 42.104(a), Petitioners certify that the '738 patent is available for *inter partes* review and that Petitioners are not barred or estopped from requesting *inter partes* review on grounds identified herein.

IV. IDENTIFICATION OF CHALLENGE AND STATEMENT OF THE PRECISE RELIEF REQUESTED (37 C.F.R. § 42.22(a) and 37 C.F.R. § 42.104(b))

Petitioners respectfully request *inter partes* review and cancellation of the challenged claims based on the following grounds:

Ground 1: Claims 1, 21, 22, 32, 40, 41, 51, and 57 are obvious under 35 U.S.C. § 103 over Nguyen in view of Weaver or Plummer.

Ground 2: Claims 1, 21, 22, 32, 40, 41, 51, and 57 are obvious under 35 U.S.C. § 103 over Dawson in view of Weaver or Plummer.

Ground 3: Claims 1, 21, 22, 32, 40, 41, 51, and 57 are obvious under 35 U.S.C. § 103 over Vincent in view of Weaver or Plummer.

Ground 4: Claims 1, 21, 22, 32, 40, 41, 51, and 57 are obvious under 35 U.S.C. § 103 over Needham in view of Weaver or Plummer.

In support of these grounds for unpatentability, Petitioners submit the expert

declaration of Gary Wooley, Ph.D. (Ex. 1002). Petitioners also rely on exhibits referenced in the Listing of Exhibits.

V. THRESHOLD REQUIREMENT FOR INTER PARTES REVIEW

A petition for *inter partes* review must demonstrate "a reasonable likelihood that the petitioner would prevail with respect to at least 1 of the claims challenged in the petition." 35 U.S.C. § 314(a). This Petition meets this threshold.

VI. STATEMENT OF REASONS FOR THE RELIEF REQUESTED

A. Summary of the Argument

The purported invention of the '738 patent is directed to an aqueous slurry composition for use in petroleum and pipeline industries. Ex. 1001, Abstract.[1] The '738 patent indicates that in a hydraulic fracturing operation, "a fracturing fluid is injected through a wellbore…[and] particulates, called proppants, are suspended in the fracturing fluid and transported into the fractures as a slurry." *Id.*, 1:28-34. The cited prior art taught, among other things, the use of particulates coated with organo-silicons, siloxanes, silanes, and other hydrophobic rendering compounds, entraining said particulates into an aqueous slurry, adding oil to the slurry composition, and using that composition in oil and gas implementations. Ex. 1002, ¶¶35-169. It was known before the priority date of the '738 patent that coating particulates with

[1] References to U.S. patents are made in the following convention: X:Y, where X refers to the ***column***, and Y refers to the line(s). References to Canadian patents are made in the following convention: X:Y, where X refers to the ***page***, and Y refers to the line(s).

hydrophobic rendering compounds for use in hydraulic fracturing and gravel packing would increase hydrocarbon production. *Id.* All elements of the challenged claims are met by the prior art.

B. Level of Ordinary Skill in the Art

A person of ordinary skill in the art ("PHOSITA") is presumed to be aware of all pertinent art, think along conventional wisdom in the art, and is a person of ordinary creativity. The relevant scientific field is fluids related to oil and gas wellbores, which includes drilling, completion, hydraulic fracturing, and gravel packing fluids, as well as pipeline fluids. *Id.*, ¶2. A PHOSITA would have (a) at least a bachelor's degree in engineering or chemistry, or equivalent experience and education, and (b) 3-5 years of experience in the petroleum or other industry working with wellbore and/or pipeline fluids. Wellbore fluids include drilling, completion, hydraulic fracturing, and gravel packing fluids. *Id.*, ¶40. Additional experience could substitute for the degree. *Id.*

C. U.S. Patent 8,236,738

1. Specification of the '738 Patent

The '738 patent states that it "relates to [a]n aqueous slurry composition having water, particulates, a chemical compound for rendering the surface of the particulates hydrophobic and an oil" (Ex. 1001, 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*., 3:10-15). The abstract states the slurry composition is used in petroleum and pipeline industries, and that the composition includes an aqueous carrier fluid and "a small amount of an oil." *Id*., Abstract. Per the specification, the purported invention is useful in controlling proppant flowback in hydraulic fracturing and in reducing formation sand production during well production. *Id.*, 4:33-35.

The '738 patent specifies a wide variety of organosiloxanes and organosilanes that may be used as hydrophobic rendering compounds. *Id.*, 5:34-45 (organosiloxanes); *id.*, 6:1-43 (organosilanes).

Moreover, the '738 patent discloses that "when a small amount of an oil … is mixed into the aqueous slurry containing the hydrophobized sands, the hydrophobic aggregation is enhanced significantly." *Id*., 3:65-4:1. The '738 specification provides no meaningful disclosure of 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. Ex. 1002, ¶61.

2. File History of the '738 Patent

The '738 patent was filed as U.S. Patent Application No. 12/597,370 (the "'370 application") on October 23, 2009. The '370 application claims priority to Provisional Application 60/924,006 filed April 26, 2007.

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

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.

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.

Claim 26 as filed is noteworthy because the mixture did not require oil.

On July 8, 2011, the Examiner issued the first Office Action rejecting 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. Ex. 1003, 885. The Examiner found that

> Zhang teaches a 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).

In response, the Applicant amended the rejected claims. *Id*., 907-12. Claim 1 was amended to include "[a]n aqueous slurry composition on the ground surface suitable for use as a well service slurry composition." *Id*., 914. 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." *Id*., 915.

The Applicant argued that Zhang was not prior art because it published less than one year before the priority date of the '370 application. *Id*., 916. Additionally, the Applicant stated that he "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… are not anticipated by Zhang." *Id*., 916-17. By amending claim 26 by adding 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. 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 (i.e., "oil" was the purportedly novel feature). *Id*., 916.

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. *Id*., 977. The Examiner withdrew the anticipation rejection because Zhang was not § 102(b) art but rejected claims 1-25 and 46-52 under 35 U.S.C. § 112 because the phrase "on the ground surface" finds no basis in the original disclosure. *Id*., 979-80.

The Applicant filed a Request for Continued Examination in response to the Office Action and amended claim 1 to remove the limitation "on the ground surface suitable for use as a well service operation." *Id.*, 1006. The Applicant removed the limitation intended to exclude formation oil from the composition of claim 1.

The Patent Office issued a Notice of Allowance and the '738 patent issued thereafter.

D. Claim Construction (37 C.F.R. §§ 42.100(b), 42.104(b)(3))

Pursuant to 37 C.F.R. § 42.100(b), a claim is given its broadest reasonable

interpretation in light of the specification. *Cuozzo Speed Techs., LLC v. Lee*, 136 S. Ct. 2131(2016).

The following term in the claims of the '738 patent should be construed for purposes of this petition:

- The phrase "a chemical compound for rendering the surface of the particulates hydrophobic" should be construed to mean: "a chemical compound capable of making particulates hydrophobic."

Ex. 1002, ¶¶170-174. Petitioners submit for purposes of this petition only that the remaining terms in the claims of the '738 patent do not have any special meanings and are presumed to take on their broadest reasonable meaning consistent with the understanding of a PHOSITA when read in light of the '738 patent's specification.

E. Grounds for Unpatentability Under 35 U.S.C. § 103

1. Claims 1, 21, 22, 32, 40, 41, 51, and 57 Are Obvious over Nguyen in view of Weaver or Plummer

A PHOSITA would have been motivated to combine Nguyen with Weaver because both teach methods for modifying sand particulates with a water-repellent organosilane for use in hydraulic fracturing. It would have been obvious to a PHOSITA to substitute either the mixed aqueous/non-aqueous fluid or the emulsion treatment fluid of Weaver for the aqueous carrier fluid of Nguyen, since both were known in the art and there was a high likelihood of success. A PHOSITA would also be motivated to substitute Plummer's oil and water liquid carrier for Nguyen's

carrier fluid or to add Plummer's oil to Nguyen's slurry as a lubricant friction-reducer, or to reduce clay swelling, as taught by Plummer, to improve the compositions and methods of Nguyen. Ex. 1002, ¶561.

a. Independent Claim 1 is Obvious

(i) Preamble for Claim 1: "An aqueous slurry composition comprising"

Nguyen teaches an aqueous slurry composition by reciting "mixing particles in a carrier fluid comprising a water-based polymer to form a mixture." Ex. 1005, claim 63. Weaver teaches 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." Ex. 1008, [0058]. Nguyen and Weaver teach this preamble. Ex. 1002, ¶¶563-566.[2]

(ii) Limitation 1A: "water"

According to Nguyen, "[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, [0012]. Nguyen also teaches that "[f]racturing or gravel packing fluids which can be utilized in accordance with the present invention include

[2] The preambles found within the challenged claims are not limiting, but out of abundance of caution, Preferred notes that the preambles of the challenged claims are nevertheless met by the prior art. *E.g.*, *Catalina Mktg. Int'l v. Coolsavings.com, Inc.*, 289 F.3d 801, 808-09, (Fed. Cir. 2002); MPEP § 2111.

gelled water or oil base liquids, foams and ***emulsions***." *Id*., [0011]. A PHOSITA would recognize that emulsions used in oil and gas well service operations are typically comprised of a mixture of oil and water. Ex. 1002, ¶568. Weaver discloses that particulates may be introduced in treatment fluid. "These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, ***or a mixture thereof***." Ex. 1008, [0058] (listing oils). "Suitable treatment fluids may take on a variety of physical forms, including…emulsions…***Suitable emulsions*** can be comprised of two immiscible liquids such as an ***aqueous liquid*** or gelled liquid and a hydrocarbon." *Id*., [0059]. Nguyen and Weaver meet this limitation. Ex. 1002, ¶¶567-571.

(iii) Limitation 1B: "particulates"

Nguyen meets this limitation: "The preferred particles are sand." Ex. 1005, [0016]. Nguyen also discloses a "method…comprising mixing particles in a carrier fluid" to "form a mixture." *Id*., claim 63; Ex. 1002, ¶573. Weaver also meets this limitation: "particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface." Ex. 1008, [0018]; Ex. 1002, ¶574.

(iv) Limitation 1C: "a chemical compound for rendering the surface of the particulates hydrophobic"

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, 4:62-67. The '738 patent recites examples of organosilanes include $\boldsymbol{(CH_3)_2SiCl_2}$ [dichlorodimethylsilane], $\boldsymbol{(C_6H_5)_2SiCl_2}$ [dichlorodiphenylsilane], $\boldsymbol{(CH_3)_3SiCl}$ [chlorotrimethylsilane], and $\boldsymbol{(C_6H_5)_2Si(OCH_3)_2}$ [dimethoxydiphenylsilane]." *Id.*, 5:66-6:7. Nguyen teaches that "particles [that] are dry coated with a water-repellent organo-silicon material." Ex. 1005, [0005]. Nguyen further teaches that "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." *Id*., [0006]; Ex. 1002, ¶¶576-580. Because Nguyen discloses the same chemicals for rendering particles hydrophobic listed in the '738 patent, Nguyen meets this limitation.

Moreover, Weaver discloses coating particulates with a surface-treating reagent wherein "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…***organosilanes***…" Ex. 1008, [0049]. This limitation is met. Ex. 1002, ¶¶576-583.

(v) Limitation 1D: "oil"

Nguyen teaches inclusion of oil in the carrier fluid: "[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, [0011]. A PHOSITA knows that emulsions include mixtures of oil and water. Ex. 1002, ¶585. Additionally, Nguyen teaches an on-the-fly preparation of the slurry: 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*., [0008].

Weaver's treatment fluids "…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, [0058]. "***Suitable emulsions*** can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and ***a hydrocarbon***."

Id., [0059].

Moreover, both Nguyen and Weaver discuss the presence of formation oil, as both recite methods of treating hydrocarbon-producing wells or subterranean formations. Ex. 1005, Abstract; Ex. 1008, [0058], claim 11. Nguyen and Weaver disclose the inclusion of oil to the composition. Ex. 1002, ¶¶590-593.

Plummer teaches "a liquid carrier comprising ***one or more*** liquids selected from the group consisting of water and oil." Ex. 1010, 7:18-20. Plummer also teaches "[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.*, 4:28-33. Plummer also teaches this limitation. Ex. 1002, ¶¶594-596.

b. Independent Claim 32 Is Obvious

(i) Preamble for Claim 32: "A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of"

Nguyen recites that "particles are dry coated with a water-repellent organo-silicon material" and "then pumped downhole…to the wellbore-screen annulus to function as a gravel pack." Ex. 1005, [0005]. A PHOSITA knows that gravel packs are used as methods of controlling proppant flowback. Ex. 1002, ¶600. 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…***frac-packing operations***." Ex. 1008, [0058]. A PHOSITA recognizes that

frac-packing operations are a type of gravel pack especially designed to control proppant flowback. Ex. 1002, ¶602.

Additionally, because Nguyen in view of Weaver and Plummer discloses the use of all the claimed ingredients, including hydrophobic particles and oil, it necessarily controls proppant flowback, consistent with the '738 specification. Ex. 1001, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which controls proppant flowback).

(ii) Limitation 32A: "mixing water"

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, [0012]. 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, [0011]. A PHOSITA would recognize that emulsions used in oil and gas well operations are typically comprised of a mixture of oil and water. Ex. 1002, ¶608. Nguyen discloses "mixing particles in a carrier fluid…to form a mixture." Ex. 1005, claim 63. Weaver teaches that particulates may be introduced in treatment fluid. "These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof." Ex. 1008, [0058]. "Suitable treatment fluids may take on a variety of physical forms, including…emulsions….***Suitable emulsions*** can be comprised of

two immiscible liquids such as an ***aqueous liquid*** or gelled liquid and a hydrocarbon." *Id*., [0059]. Weaver describes that the hydrophobic particulates are mixed before transport to a site or on-the-fly. *Id*., [0056]. Nguyen and Weaver meet this element. Ex. 1002, ¶¶607-612.

(iii) Limitation 32B: "particulates"

Nguyen and Weaver meet this limitation (Ex. 1002, ¶¶613-616): "The preferred particles are sand." Ex. 1005, [0016]. Nguyen also discloses a "method…comprising mixing particles in a carrier fluid" to "form a mixture." *Id*., claim 63. Weaver teaches that "particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface." Ex. 1008, [0018]. Weaver describes that the hydrophobic particulates are mixed before transport to a site or on-the-fly. *Id*., [0056].

(iv) Limitation 32C: "a chemical compound for rendering the surface of the particulates hydrophobic"

Nguyen teaches "particles [that] are dry coated with a water-repellent organo-silicon material." Ex. 1005, [0005]. As shown above in §VI.E.1.a.iv, Nguyen discloses the exact same chemicals for rendering the surface of the particulates hydrophobic as those found in the '738 patent. *Compare id.*, [0006] (referencing chlorotrimethylsilane, dimethoxydiphenylsilane, dichlorodimethylsilane, dichlorodiphenylsilane, among many other hydrophobizing chemicals) *with* Ex.

1001, 5:66-6:7. Ex. 1002, ¶¶618-621.

Weaver also teaches that "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***." Ex. 1008, [0049]. This limitation is met. Ex. 1002, ¶¶617-624.

(v) Limitation 32D: "oil"

Nguyen teaches inclusion of oil in the carrier fluid: "[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, [0011]. A PHOSITA knows that emulsions include mixtures of oil and water. Ex. 1002, ¶626. Additionally, Nguyen teaches an on-the-fly preparation of the slurry: 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*. [0008].

Weaver discloses "[t]hese treatment fluids 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…***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, [0058]. "***Suitable emulsions*** can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and ***a hydrocarbon***." *Id*., [0059].

Plummer teaches "a liquid carrier comprising ***one or more*** liquids selected from the group consisting of water and oil." Ex. 1010, 7:18-20. Plummer also teaches "[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*., 4:28-33. This limitation is met. Ex. 1002, ¶¶625-633.

(vi) Limitation 32E: "pumping the mixture into the formation"

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 the carrier fluid." *Id*., claim 75. 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, claim 23. This element is met. Ex. 1002, ¶¶634-638.

c. Independent Claim 51 is Obvious

(i) Preamble of Claim 51: "A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of"

Nguyen recites that "particles are dry coated with a water-repellent organo-silicon material" and "then pumped downhole…to the wellbore-screen annulus to function as a gravel pack." Ex. 1005, [0005]. A PHOSITA knows that gravel packs are methods of controlling proppant flowback. Ex. 1002, ¶640. 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…***frac-packing operations***." Ex. 1008, [0058]. A PHOSITA recognizes that frac-packing operations are a type of gravel pack especially designed to control proppant flowback. Ex. 1002, ¶642.

Additionally, because Nguyen in view of Weaver and Plummer discloses the use of all the claimed ingredients, including oil and hydrophobic particulates, it necessarily controls proppant flowback, consistent with the '738 specification. Ex. 1001, 3:30-59, 3:65-4:4 (explaining that adding oil increases hydrophobic aggregation, which controls proppant flowback); Ex. 1002, ¶645.

(ii) Limitation 51A: "contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic"

As previously indicated in §VI.E.1.a.iv, Nguyen and Weaver disclose modifying agents that render the surface of the particulates hydrophobic. 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." Ex. 1005, [0007]; claim 4. 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 include[s]…solvent extraction methods." Ex. 1008, [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***." *Id*. This limitation is met. Ex. 1002, ¶¶647-650.

(iii) Limitation 51B: "separating the particulate from the medium"

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, [0007]. Weaver teaches "[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.***" Ex. 1008, [0057]. A PHOSITA would understand that evaporating the solvent by drying and extracting the solvent from the particles are methods of separating the particulates. Ex. 1002, ¶¶651-654.

(iv) Limitation 51C: "blending the hydrophobic particulate with water and an oil to form a mixture"

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, [0011]. A PHOSITA would recognize that an emulsion may be comprised of oil and water. Ex. 1002, ¶656. Nguyen teaches that the particulates coated with the water repellent (*i.e.*, hydrophobic) composition are mixed with the carrier fluid to form a mixture (*id.*, claim 63,

abstract), which, as disclosed above, may be an emulsion (*id*., [0011]). 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***." Ex. 1008, [0058]. Weaver also discloses that "[s]uitable treatment fluids may take on a variety of physical forms, including…emulsions….***Suitable emulsions*** can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid ***and a hydrocarbon***." *Id*., [0059]. A PHOSITA would recognize, and the '738 patent confirms, that oil is a "hydrocarbon." Ex. 1002, ¶660. Plummer teaches "a liquid carrier comprising ***one or more*** liquids selected from the group consisting of water and oil." Ex. 1010, 7:18-20. Plummer also teaches "[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling…" *Id*., 4:28-33. This limitation is met. Ex. 1002, ¶¶655-664.

(v) Limitation 51D: "pumping the mixture into the formation

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***…" Ex. 1005, claim 63. 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, claim 23; claim 11. This limitation is met. Ex.

1002, ¶¶665-669.

Nguyen in view of Weaver or Plummer renders claim 51 obvious. *Id*., ¶670.

d. Dependent Claims 21, 22, 40, 41, and 57 Are Obvious

In comparison to their respective independent claims, the only additional limitation recited in claims 21, 22, 40, 41, and 57[3] is the type of particulate.[4] Nguyen teaches "[v]arious kinds of particulates can be utilized as a proppant including sand….The preferred particles are sand." Ex. 1005, [0016], claim 8. Weaver teaches "[s]uitable particulates include, but are not limited to, sand." Ex. 1008, [0018]. These dependent claims are met. Ex. 1002, ¶¶671-677

Nguyen in view of Weaver or Plummer renders challenged claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent obvious. *Id*., ¶678.

2. Claims 1, 21, 22, 32, 40, 41, 51, and 57 Are Obvious over Dawson in view of Weaver or Plummer

A PHOSITA would be motivated to combine Dawson with Weaver because both teach methods for modifying particulates with a silane for use in hydraulic fracturing. It would have been obvious to a PHOSITA to substitute either the

[3] Claim 57 is also unpatentable and invalid under 35 U.S.C. § 112. Claim 57 covers "the ***composition*** of claim 51," yet, claim 51 covers "a ***method*** for controlling proppant flowback." Ex. 1001, 13:28-34; 14:24-25. Because there is no "composition" in claim 51, claim 57 is rendered indefinite and lacks enablement.

[4] With respect to Markush group claim elements such as those found in Claims 21, 22, 40, 41, and 57, "the entire element is disclosed by the prior art if one alternative in the Markush group is in the prior art." *Fresenius USA, Inc. v. Baxter Int'l, Inc.*, 582 F.3d 1288, 1298 (Fed. Cir. 2009).

aqueous/non-aqueous mixture or the emulsion treatment fluid of Weaver for the aqueous carrier fluid of Dawson, since both were known in the art and there was a high likelihood of success. A PHOSITA would also be motivated to substitute Plummer's oil and water liquid carrier for Dawson's carrier fluid or to add Plummer's oil to Dawson's slurry as a lubricant friction-reducer, or to reduce clay swelling, as taught by Plummer, to improve the compositions and methods of Dawson. Ex. 1002, ¶680.

a. Independent Claim 1 is Obvious

(i) Preamble for Claim 1: "An aqueous slurry composition comprising"

Dawson recites "[a] slurry including the disclosed particulate material…and a carrier fluid." Ex. 1006, 14:12-15. The carrier fluid may include salt water or fresh water. *Id*., 13:10-13. Weaver teaches 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." Ex. 1008, [0058]. This preamble is met. Ex. 1002, ¶¶682-685.

(ii) Limitation 1A: "water"

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*., 13:10-13. Weaver discloses that particulates may be introduced in treatment fluid. "These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or a mixture thereof." Ex. 1008, [0058]. "Suitable treatment fluids may take on a variety of physical forms, including…emulsions….***Suitable emulsions*** can be comprised of two immiscible liquids such as an ***aqueous liquid*** or gelled liquid and a hydrocarbon." *Id*., [0059]. This limitation is met. Ex. 1002, 686-690.

(iii) Limitation 1B: "particulates"

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, 2:6-7. Dawson further discloses that the "particulate material [is] suspended in a carrier fluid." *Id*., 10:1-3; 48, claim 13. Weaver also meets this limitation: "particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface." Ex. 1008, [0018]. This element is met. Ex. 1002, ¶¶691-694.

(iv) Limitation 1C: "a chemical compound for rendering the surface of the particulates hydrophobic"

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, 4:62-67. The '738 states that "examples of organosilanes include: ***CH_3SiCl_3*** [i.e., methyltrichlorosilane]." *Id.*, 5:66-6:1.

Dawson teaches that particles may be "treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material." Ex. 1006, 7:3-8. Specifically, Dawson discloses that "examples of suitable modifying agents include, but are not limited to, agents including polyisocyanates, ***silanes, siloxanes***, and combinations thereof." *Id.*, 7:14-15. Specifically, Dawson teaches that the modifying agent may contain "one or more halogen groups, ([] ***methyltrichlorosilane***, *etc.*)." *Id.*, 21:10-11. Dawson teaches using silanes and siloxanes and a specific hydrophobizing chemical listed by the '738.

Weaver teaches coating particulates with a surface-treating reagent wherein "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…***organosilanes*** …" Ex. 1008, [0049]. This limitation is met. Ex. 1002, ¶¶695-704.

(v) Limitation 1D: "oil"

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, 13:6-13. A PHOSITA would recognize that liquid hydrocarbons can be hydrocarbon oils. Ex. 1002, ¶707. Dawson teaches the limitation of oil included in the carrier fluid comprising both oil and water. *Id*.

Further, Dawson incorporates by reference[5] U.S. Patent 3,659,651 to Graham ("Graham"). Ex. 1006, 33:6-8. 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,

[5] *Callaway Golf v. Acushnet Co.*, 576 F.3d 1331, 1346 (Fed. Cir. 2009) ("Material not explicitly contained in the single, prior art document may still be considered for purposes of anticipation if that material is incorporated by reference into the document."). This applies to all prior art, discussed herein, which incorporates material by reference.

5:9-16 (citing Ex. 1013 to Kiel, 5:65-72 (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.")).

Weaver discloses using treatment 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 non-aqueous, the base fluid may comprise … mineral oils, synthetic oils***, esters, and the like." Ex. 1008, [0058]. Weaver discloses using emulsions as base fluids: "***Suitable emulsions*** can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and ***a hydrocarbon***." *Id*., [0059].

Moreover, both Dawson and Weaver discuss the presence of formation oil, as both recite methods of treating a wellbore producing oil or subterranean formations. Ex. 1006, 14:31-15:4; Ex. 1008, [0058], claim 11. Formation oil would be present in Dawson's and Weaver's compositions. Ex. 1002, ¶¶713-716.

Plummer teaches "a liquid carrier comprising ***one or more*** liquids selected from the group consisting of water and oil." Ex. 1010, 7:18-20. Plummer further

teaches "[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.*, 4:28-33. Plummer also teaches this limitation.

b. Independent Claim 32 Is Obvious

(i) Preamble for Claim 32: "A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of"

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***.'" Ex. 1006, 13:23-25. Weaver similarly 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, [0058]. A PHOSITA knows that a frac-pack is a type of gravel pack especially designed to control proppant flowback. Ex. 1002, ¶¶723, 725. Moreover, because both Weaver and Dawson teach the use of hydrophobic proppants as well as oil in the hydraulic fracturing slurry, they both necessarily control proppant flowback, consistent with the specification of the '738 patent. Ex. 1001, Abstract, 1:60-2:34, 3:30-59, 3:65-4:4. This preamble is met. Ex. 1002, ¶¶722-729.

(ii) Limitation 32A: "mixing water"

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, 13:6-10. Dawson teaches "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*., 13:10-13. Weaver teaches that particulates may be introduced in treatment fluid. "These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, or ***a mixture thereof***." Ex. 1008, [0058]. "Suitable treatment fluids may take on a variety of physical forms, including…emulsions….***Suitable emulsions*** can be comprised of two immiscible liquids such as an ***aqueous liquid*** or gelled liquid and a hydrocarbon." *Id*., [0059]. Weaver describes that the hydrophobic particulates are mixed before transport to a site or on-the-fly. *Id*., [0056]. This element is met. Ex. 1002, ¶¶730-735.

(iii) Limitation 32B: "particulates"

Dawson teaches the "use of relatively lightweight and/or substantially neutrally buoyant ***particles as proppant material*** in hydraulic fracturing treatments." Ex. 1006, 2:6-7. Dawson further discloses that the "particulate material [is] suspended in a carrier fluid." *Id*., 10:1-3; 48, claim 13. A PHOSITA would understand that the particulates are suspended when mixed with the carrier fluid.

Ex. 1002, ¶738. Weaver teaches that "particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface." Ex. 1008, [0018]. Weaver describes that the hydrophobic particulates are mixed before transport to a site or on-the-fly. *Id*., [0056]. This element is disclosed. Ex. 1002, ¶¶736-740.

(iv) Limitation 32C: "a chemical compound for rendering the surface of the particulates hydrophobic"

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, 7:3-8. Specifically, Dawson discloses that "examples of suitable modifying agents include, but are not limited to, agents including polyisocyanates, ***silanes, siloxanes***, and combinations thereof." *Id.*, 7:14-15. Dawson teaches that the modifying agent may be ***methyltrichlorosilane***. *Id*., 21:10-11. Dawson teaches using silanes and siloxanes and a specific hydrophobizing chemical listed by the '738, so this element is met. Ex. 1002, ¶¶742-747.

Weaver also teaches that "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***." Ex. 1008, [0049]. This limitation is met. Ex. 1002, ¶¶741-750.

(v) Limitation 32D: "oil"

As shown above, Dawson discloses that "[t]he particles…may be mixed in any desired concentration with a carrier fluid….Carrier fluids comprising salt water, fresh water, ***liquid hydrocarbons***, and/or nitrogen or other gases" may be used. Ex. 1006, 13:6-13. A PHOSITA would understand that Dawson's carrier fluid could comprise both water and hydrocarbons and that liquid hydrocarbons encompass various hydrocarbon oils. Ex. 1001, 11:31-33 ("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. 1002, ¶¶752-753. Further, as demonstrated in §VI.E.2.a.v, Dawson teaches use of oil in the carrier fluid by incorporating by reference the water and oil emulsions disclosed in the Graham and Kiel patents.

Weaver discloses "[t]hese treatment fluids 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 …***mineral oils, synthetic oils***, esters, and the like." Ex. 1008, [0058]. Weaver also discloses that emulsion can be used as the base fluid, including emulsions "comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and ***a hydrocarbon***." *Id*., [0059].

Plummer teaches "a liquid carrier comprising ***one or more*** liquids selected from the group consisting of water and oil." Ex. 1010, 7:18-20. Plummer also

teaches "[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling." *Id*., 4:28-33.

This limitation is met. Ex. 1002, ¶¶751-761.

(vi) Limitation 32E: "pumping the mixture into the formation"

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, 13:6-10. Dawson also teaches that "modified particulate material may be formulated as described herein and introduced or pumped into a well." *Id*., 29:27-28. A PHOSITA would know that the treatment fluid for both hydraulic fracturing and sand control treatments are pumped into the formation. Ex. 1002, ¶763.

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, claim 23, claim 11. This element is met. Ex. 1002, ¶¶762-766.

c. Independent Claim 51 is Obvious

(i) Preamble of Claim 51: "A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of"

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***.'" Ex. 1006, 13:23-25. Weaver similarly 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, [0058]. A PHOSITA knows that a frac-pack is a type of gravel pack especially designed to control proppant flowback. Ex. 1002, ¶¶768, 770. Moreover, because both Weaver and Dawson teach the use of hydrophobic proppants as well as oil in the hydraulic fracturing slurry, they both necessarily control proppant flowback, consistent with the specification of the '738 patent. Ex. 1001, Abstract, 1:60-2:34, 3:30-59, 3:65-4:4. This preamble is satisfied. Ex. 1002, ¶¶768-774.

(ii) Limitation 51A: "contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic"

As previously indicated in §VI.E.2.a.iv, Dawson and Weaver disclose chemical compound coatings that render the surface of the particulates hydrophobic.

Dawson teaches that particles may be "treated by exposure to a modifying agent that is capable of interacting with compounds present in or on a natural material." Ex. 1006, 7:3-8. 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*., 27:1-2. Dawson further discloses that "[s]pecific examples of ***suitable exposure methods*** include, but are not limited to, by ***soaking…dipping…mixing…[and] by flowing*** modifying agent through a permeable bed or pack of the naturally-occurring particles in a pressurized container. " *Id*., 24:17-26. 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 include[s]…solvent extraction methods." Ex. 1008, [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***." *Id*. This limitation is met. Ex. 1002, ¶¶775-778.

(iii) Limitation 51B: "separating the particulate from the medium"

Dawson discloses that "***[d]rying of particles after exposure*** may be accomplished, for example, by continuously dropping through heated air." Ex. 1006, 24:17-26. The particulates are separated from the medium, according to Dawson, and this limitation is met. Ex. 1002, ¶780. Weaver teaches that after being

coated, "***[t]he 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.***" Ex. 1008, [0057]. A PHOSITA would understand that drying the particles and extracting the solvent from the particles are methods of separating the particulates. Ex. 1002, ¶781.

(iv) Limitation 51C: "blending the hydrophobic particulate with water and an oil to form a mixture"

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, 13:6-13. Dawson teaches the limitation of oil included in the composition and discloses that the carrier fluid may comprise both water ***and*** a liquid hydrocarbon. *Id*.; Ex. 1002, ¶784. A PHOSITA would recognize, and the '738 patent confirms, that liquid hydrocarbons encompass various hydrocarbon oils. Ex. 1001, 11:31-33 (identifying hydrocarbon oil as one type of

oil). Ex. 1002, ¶785. As shown above in §VI.E.2.a.v, Dawson teaches use of water and oil emulsions by incorporating by reference the Graham and Kiel patents.

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*., [0059]. A PHOSITA would recognize that "hydrocarbon" is another term for oil. Ex. 1002, ¶789. Plummer teaches "a liquid carrier comprising ***one or more*** liquids selected from the group consisting of water and oil." Ex. 1010, 7:18-20. Plummer also teaches "[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling …." *Id*., 4:28-33.

This limitation is met. Ex. 1002, ¶783-792.

(v) Limitation 51D: "pumping the mixture into the formation

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, 13:6-10. 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, claim 23; claim 11. This limitation is met. Ex. 1002, ¶793-797.

d. Dependent Claims 21, 22, 40, 41, and 57 Are Obvious

The only additional limitation recited in claims 21, 22, 40, 41, and 57 is the type of particulate. 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, 13:3-5. Rickards discloses that the "fracture proppant material may be a material such as sand." Ex. 1011, Abstract. Weaver teaches "[s]uitable particulates include, but are not limited to, sand." Ex. 1008, [0018]. These dependent claims are met. Ex. 1002, ¶¶798-804.

Dawson in view of Weaver or Plummer renders challenged claims 1, 21, 22, 32, 40, 41, 51, and 57 of the '738 patent obvious. *Id*., ¶805.

3. Claims 1, 21, 22, 32, 40, 41, 51, and 57 Are Obvious over Vincent in view of Weaver or Plummer

A PHOSITA would be motivated to combine Vincent with Weaver because both teach methods for modifying sand particulates with a water-repellent compound for use in hydraulic fracturing. It would have been obvious to a PHOSITA to substitute the either the aqueous/non-aqueous mixture or the emulsion treatment fluid of Weaver for the aqueous fracture fluid of Vincent, since the use both were known in the art and there was a high likelihood of success. A PHOSITA would

also be motivated to substitute Plummer's oil and water liquid carrier for Vincent's fracture fluid or add Plummer's oil to Vincent's slurry as a lubricant friction-reducer, or to reduce clay swelling, as taught by Plummer, to improve the compositions and methods of Vincent. Ex. 1002, ¶807.

a. Independent Claim 1 is Obvious

(i) Preamble for Claim 1: "An aqueous slurry composition comprising"

Vincent teaches the "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, [0087]. Vincent discloses that the slurry can be water based. *Id.* Weaver teaches "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, [0058]. This preamble is taught. Ex. 1002, ¶¶809-812.

(ii) Limitation 1A: "water"

Likewise, Vincent and Weaver meet this limitation. "The fracturing fluid may be an oil base, ***water base***, acid, ***emulsion***, foam, or any other fluid." Ex. 1007, [0087]. A PHOSITA would understand that an emulsion may contain oil and water. Ex. 1002, ¶814. Weaver discloses that particulates may be introduced in treatment fluid. "These treatment fluids generally comprise a base fluid, which may be

aqueous or non-aqueous, or a mixture thereof." Ex. 1008, [0058]. "Suitable treatment fluids may take on a variety of physical forms, including…emulsions….***Suitable emulsions*** can be comprised of two immiscible liquids such as an ***aqueous liquid*** or gelled liquid and a hydrocarbon." *Id*., [0059]. This limitation is met. Ex. 1002, ¶¶813-817.

(iii) Limitation 1B: "particulates"

Vincent's disclosure "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, [0002]. Vincent further teaches that the particulates would be mixed into the aqueous carrier fluid to form a slurry. *Id.*, [0087]. Weaver discloses "particulates suitable for use in the present invention comprise any particulate that comprises a mineral surface." Ex. 1008, [0018]. This element is disclosed. Ex. 1002, ¶¶818-821.

(iv) Limitation 1C: "a chemical compound for rendering the surface of the particulates hydrophobic"

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, 4:62-67. The '738 patent further states

"[a]mong different organosiloxane compounds which are useful for the present invention, polysiloxanes modified with…organic quaternary siloxanes are examples. *Id*., 6:44-48. The '738 patent explains that "[t]he simplest polysiloxanes are ***polydimethylsiloxanes***." *Id*., 5:8-9.

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*** …" Ex. 1007, [0008]. Vincent further teaches that these benefits can be achieved by "coating the proppants with a ***hydrophobic material such as silicon containing compounds***, ***including silicone materials and siloxanes***." *Id*., [0009]. Vincent also discloses a coated proppant "wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and ***polydimethyl siloxane***." *Id*., claim 6.

Moreover, Weaver teaches that "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*** …" *Id*., [0049]. This limitation is met. Ex. 1002, ¶¶822-830.

(v) Limitation 1D: "oil"

Vincent teaches the use of an oil in the composition: the desired effects "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, [0009]. Additionally, Vincent describes the hydrophobic material as comprising ***"one or more hydrophobic materials selected from the group consisting of silicones***, siloxanes, polytetrafluoroethylene, ***plant oils***, [and] ***hydrocarbons***." *Id*., claim 2. A PHOSITA would understand the term "hydrocarbons" to include hydrocarbon oil. Ex. 1002, ¶834. 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). 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, [0087]. A PHOSITA would understand that an emulsion can be comprised of oil and water. Ex. 1002, ¶832.

Weaver discloses using treatment 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 non-aqueous, the base fluid may comprise …***

mineral oils, synthetic oils, esters, and the like." Ex. 1008, [0058]. Suitable emulsions for use as a base fluid "can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and ***a hydrocarbon***." *Id*., [0059].

Moreover, both Vincent and Weaver discuss the presence of formation oil, as both recite methods of treating a reservoir producing oil or subterranean formations. Ex. 1007, [0008], [0012], [0014]; Ex. 1008, [0058], claim 11.

Plummer teaches "a liquid carrier comprising ***one or more*** liquids selected from the group consisting of water and oil." Ex. 1010, 7:18-20. Plummer also teaches "[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*., 4:28-33. This limitation is met. Ex. 1002, ¶¶838-845.

b. Independent Claim 32 Is Obvious

(i) Preamble for Claim 32: "A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of"

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, [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*., [0028]. A PHOSITA would understand that "controlling relatively permeability to flow of fluids," and "increasing proppant pack strength" would control and reduce proppant flowback. Ex. 1002, ¶850.

Weaver discloses that his invention may be utilized in a variety of subterranean operations known in the art, including fracturing operations, gravel-packing operations, ***frac-packing operations***. Ex. 1008, [0058]. A PHOSITA knows that a frac-pack is a type of gravel pack especially designed to control proppant flowback. Ex. 1002, ¶851.

Moreover, because both Vincent and Weaver teach the use of hydrophobic proppants as well as oil in the hydraulic fracturing slurry, they both necessarily control proppant flowback, consistent with the specification of the '738 patent. Ex. 1001, Abstract, 1:60-2:34, 3:30-59, 3:65-4:4. This preamble is met. Ex. 1002, ¶¶847-855.

(ii) Limitation 32A: "mixing water"

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, [0087]. A PHOSITA would understand that an emulsion may

comprise oil and water. Ex. 1002, ¶857. To be suspended, the particles of Vincent would have been mixed with the carrier fluid. *Id*.

Weaver teaches that particulates may be introduced in treatment fluid. "These treatment fluids generally comprise a base fluid, which may be ***aqueous*** or non-aqueous, or a mixture thereof." Ex. 1008, [0058]. "Suitable treatment fluids may take on a variety of physical forms, including…emulsions….***Suitable emulsions*** can be comprised of two immiscible liquids such as an ***aqueous liquid*** or gelled liquid and a hydrocarbon." *Id*., [0059]. Weaver describes that the hydrophobic particulates are mixed before transport to a site or on-the-fly. *Id*., [0056]. This limitation is met. Ex. 1002, ¶¶856-861.

(iii) Limitation 32B: "particulates"

Vincent's disclosure "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, [0002]. Vincent further teaches that the particulates would be mixed into the aqueous carrier fluid to form a slurry. *Id.*, [0087]. Weaver teaches that "particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface." Ex. 1008, [0018]. Weaver describes that the hydrophobic particulates are mixed before transport to a site or on-the-fly. *Id*., [0056]. This element is met. Ex. 1002, ¶¶862-865.

(iv) Limitation 32C: "a chemical compound for rendering the surface of the particulates hydrophobic"

The '738 patent discloses the use of ***polydimethylsiloxanes*** as a chemical compound for rendering the surface of the particulates hydrophobic. Ex. 1001, 5:8-9. Likewise, Vincent discloses coated proppant "wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and ***polydimethyl siloxane***." *Id*., claim 6.

Weaver also teaches that "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***." Ex. 1008, [0049]. This limitation is met. Ex. 1002, ¶¶866-873.

(v) Limitation 32D: "oil"

Vincent teaches the use of an oil: the desired effects "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,

[0009]. Additionally, Vincent teaches, "the hydrophobic material [may] ***comprise[] one or more hydrophobic materials selected from the group consisting of silicones***, siloxanes, polytetrafluoroethylene, ***plant oils***, [and] ***hydrocarbons***." *Id*., claim 2. A PHOSITA would understand the term "hydrocarbon" to include hydrocarbon oil. Ex. 1002, ¶877. 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). 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, [0087]. A PHOSITA would understand that an emulsion can be comprised of oil and water. Ex. 1002, ¶878.

Weaver discloses using treatment fluid: "These treatment fluids generally comprise a base fluid, which may be aqueous or non-aqueous, ***or a mixture thereof***." Ex. 1008, [0058]. Weaver discloses using synthetic and mineral oils as non-aqueous fluids, as well as base fluids comprising an emulsion of an aqueous liquid and a hydrocarbon. *Id*., [0058-0059].

Plummer teaches "a liquid carrier comprising ***one or more*** liquids selected from the group consisting of water and oil." Ex. 1010, 7:18-20. Plummer further teaches "[o]il may be added to the foam-water slurry as a lubricant and to prevent

clay swelling …" *Id*., 4:28-33. This limitation is met. Ex. 1002, ¶¶874-884.

(vi) Limitation 32E: "pumping the mixture into the formation"

Vincent teaches "[a] method of fracturing a subterranean formation, comprising:… injecting into the fracture a fluid containing a plurality of essentially spherical particles…wherein the particles are modified by coating the particles with a hydrophobic material." Ex. 1007, claim 9.

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, claim 23, claim 11. Vincent and Weaver teach this exact element. Ex. 1002, ¶¶885-889.

c. Independent Claim 51 is Obvious

(i) Preamble of Claim 51: "A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of"

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, [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*., [0028]. A PHOSITA would understand that "controlling relatively permeability to flow of fluids," and "increasing proppant pack strength" would control and reduce proppant flowback. Ex. 1002, ¶894. Weaver discloses that his invention may be utilized in a variety of subterranean operations known in the art, including "fracturing operations, gravel-packing operations, ***frac-packing operations***, perforation operations, and the like." Ex. 1008, [0058]. A PHOSITA knows that a frac-pack is a type of gravel pack especially designed to control proppant flowback. Ex. 1002, ¶896.

Moreover, because both Weaver and Vincent teach the use of hydrophobic proppants as well as oil in the hydraulic fracturing slurry, they both necessarily control proppant flowback, consistent with the specification of the '738 patent. Ex. 1001, Abstract, 1:60-2:34, 3:30-59, 3:65-4:4. This preamble is disclosed. Ex. 1002, ¶¶891-899.

(ii) Limitation 51A: "contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic"

As previously indicated in §§VI.E.3.a.iv, Vincent and Weaver disclose modifying agents that render the surface of the particulates hydrophobic. Vincent discloses "pretreating proppant prior to the fracturing treatment." Ex. 1007, Ex.

1007, [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*., [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*., [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." *Id*., [0037]. 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 include[s]…solvent extraction methods." Ex. 1008, [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***." *Id*. This limitation is met. Ex. 1002, ¶¶900-902.

(iii) Limitation 51B: "separating the particulate from the medium"

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." Ex. 1007, [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 113º C. for approximately 15 to 18 hours." *Id*., [0037]. Weaver teaches "***[t]he 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.***" Ex. 1008, [0057]. A PHOSITA would understand that drying the particles and extracting the solvent from the particles are methods of separating the particulates. Ex. 1002, ¶¶904-905.

(iv) Limitation 51C: "blending the hydrophobic particulate with water and an oil to form a mixture"

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, [0087]. A PHOSITA would understand that an emulsion can be comprised of oil and water. Ex. 1002, ¶908. 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, claim 2. Vincent teaches the use of two hydrophobic rendering compounds: for example, polydimethyl siloxane, satisfies a compound for rendering the surface of the particulates hydrophobic, and a plant oil or hydrocarbons satisfies the oil. Ex. 1002, ¶908.

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*., [0059]. A PHOSITA would recognize, and the '738 patent confirms, that "hydrocarbon" is another term for oil. Ex. 1002, ¶911. Plummer teaches "a liquid carrier comprising ***one or more*** liquids selected from the group consisting of water and oil." Ex. 1010, 7:18-20. Plummer also teaches "[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling …" *Id*., 4:28-33. This limitation is satisfied. Ex. 1002, ¶¶907-914.

(v) Limitation 51D: "pumping the mixture into the formation

Vincent teaches "[a] method of fracturing a subterranean formation, comprising:…injecting into the fracture a fluid containing a plurality of essentially spherical particles…wherein the particles are modified by coating the particles with a hydrophobic material." Ex. 1007, claim 9. 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, claim 23; claim 11. This limitation is met. Ex. 1002, ¶¶915-918.

d. Dependent Claims 21, 22, 40, 41, and 57 Are Obvious

Claims 21, 22, 40, 41, and 57 specify the type of particulate in the claimed composition. Vincent teaches this limitation: "[t]he present process is one for modifying the surface properties of hydraulic fracturing proppants. Proppants are ***natural sands***." Ex. 1007, [0007]. 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*., [0087].

Weaver teaches "[s]uitable particulates include, but are not limited to, ***sand***." Ex. 1008, [0018]. 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*., [0057]. The sand particulates are mixed with the treatment fluid before being pumped down the formation. This limitation is met. Ex. 1002, ¶¶920-925.

Vincent in view of Weaver or Plummer renders challenged claims 1, 21, 22, 32, 40, 41, 51, and 57 of the ’738 patent obvious. Ex. 1002, ¶926.

4. Claims 1, 21, 22, 32, 40, 41, 51, and 57 Are Obvious over Needham in view of Weaver or Plummer

A PHOSITA would have been motivated to combine Needham with Weaver because both teach methods for modifying sand particulates with water-repellent organosilanes for use in hydraulic fracturing. It would have been obvious to a PHOSITA to substitute either the mixture of aqueous/non-aqueous fluid or the emulsion treatment fluid of Weaver for the aqueous carrier fluid of Needham, since the use of both were known in the art and there was a high likelihood of success. A PHOSITA would also be motivated to substitute Plummer’s oil and water liquid carrier for Needham’s carrier fluid or add Plummer’s oil to Needham’s slurry as a lubricant friction-reducer, or to reduce clay swelling, as taught by Plummer, to improve the compositions and methods of Needham. Ex. 1002, ¶928.

a. Independent Claim 1 is Obvious

(i) Preamble for Claim 1: “An aqueous slurry composition comprising”

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 therein is then pumped into the fracture.” Ex. 1009, 3:19-23. The combination of the aqueous carrier fluid with the coated proppant yields the claimed slurry

composition. Ex. 1002, ¶931. Weaver teaches 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." Ex. 1008, [0058]. This preamble is taught. Ex. 1002, ¶¶930-933.

(ii) Limitation 1A: "water"

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. 1009, 3:19-23. Weaver teaches the use of treatment fluids 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, [0058]. This element is met. Ex. 1002, ¶¶934-939.

(iii) Limitation 1B: "particulates"

Needham discloses that an "object of this invention is to coat particulate solids useful as propping agents." Ex. 1009, 1:50-51. Needham meets this limitation. Ex. 1002, ¶941. So does Weaver who teaches that "particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface." *Id*., [0018]. Ex. 1002, ¶943.

(iv) Limitation 1C: "a chemical compound for rendering the surface of the particulates hydrophobic"

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, 4:62-67. The '738 patent continues "[e]xamples of organosilanes include: …***$(CH_3)_3SiCl$***." *Id.*, 5:66-6:3.

Needham teaches that an "object of this invention is to provide particulate material coated with a water repellent useful in hydraulic fracturing of subterranean formations." Ex. 1009, 1:44-46. Further, "[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 a coating material of the invention. ***Chlorosilanes such as chlorotrimethylsilane [$(CH_3)_3SiCl$]*** and other silyl donors can also be used." *Id.*, 2:28-34.

Moreover, Weaver teaches that "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***…" *Id.*, [0049]. This limitation is disclosed. Ex. 1002, ¶944-

952.

(v) Limitation 1D: "oil"

Needham teaches "a method of fracturing an underground zone or formation in ***oil and gas wells***, …" Ex. 1008, 4:1-2. A PHOSITA would understand that hydrocarbon-bearing formations contain oil, as is evidenced by claim 1 of Needham. Ex. 1002, ¶¶958-959, 961. Because the formation contains oil, the composition of Needham must comprise oil. *Id*., ¶961.

Weaver discloses using treatment fluids that "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 … mineral oils, synthetic oils***, esters, and the like." Ex. 1008, [0058]. Suitable emulsions for use as base fluids in Weaver "can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and ***a hydrocarbon***." *Id*., [0059].

Moreover, Weaver also discusses the presence of formation oil, reciting methods of treating subterranean formations. Ex. 1008, [0058], claim 11. Consistent with the specification and prosecution history of the '738 patent, Needham and Weaver disclose the inclusion of oil to the composition. Ex. 1002, ¶¶957-961.

Plummer teaches "a liquid carrier comprising ***one or more*** liquids selected from the group consisting of water and oil." Ex. 1010, 7:18-20. Plummer teaches "[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.*, 4:28-33. Plummer also teaches this limitation. Ex. 1002, ¶¶957-66.

b. Independent Claim 32 is Obvious

(i) Preamble for Claim 32: "A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of"

Weaver discloses that his invention may be utilized in a variety of subterranean operations including ***frac-packing operations***. Ex. 1008, [0058]. A PHOSITA knows frac-packs are a type of gravel pack operation especially designed to control proppant flowback. Ex. 1002, ¶969. Moreover, because both Weaver and Needham teach the use of hydrophobic proppants as well as oil in the slurry composition, they both necessarily control proppant flowback, consistent with the specification of the '738 patent. Ex. 1001, Abstract, 1:60-2:34, 3:30-59, 3:65-4:4. This preamble is met. Ex. 1002, ¶¶968-973.

(ii) Limitation 32A: "mixing water"

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. 1009, 3:19-23. A PHOSITA understands Needham's disclosure to mean that the water is mixed with the proppant, and then pumped into the fracture. Ex. 1002, ¶975-976.

Additionally, Weaver discloses using either an aqueous fluid, non-aqueous

fluid or mixture of the two, or an emulsion as a treatment fluid where "***[s]uitable emulsions*** can be comprised of two immiscible liquids such as an ***aqueous liquid*** or gelled liquid and a hydrocarbon." *Id*., [0058-0059]. This element is met. Ex. 1002, ¶¶974-980.

(iii) Limitation 32B: "particulates"

Needham discloses that an "object of this invention is to coat particulate solids useful as propping agents." Ex. 1009, 1:50-51. Needham teaches that the coated particulates are "dispersed into a carrying fluid." *Id*., 4:30-42. Weaver teaches that "particulate suitable for use in the present invention comprise any particulate that comprises a mineral surface." *Id*., [0018]. Weaver describes that the hydrophobic particulates are mixed before transport to a site or on-the-fly. *Id*., [0056]. This limitation is met. Ex. 1002, ¶¶981-985.

(iv) Limitation 32C: "a chemical compound for rendering the surface of the particulates hydrophobic"

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….***Chlorosilanes such as chlorotrimethylsilane*** and other silyl donors can also be used." Ex. 1009, 2:25-41.

Needham teaches the same organosilane as the '738 patent: chlorotrimethylsilane ($(CH_3)_3SiCl$). *Compare id*., *with* Ex. 1001, 6:2.

Weaver also teaches that "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***." Ex. 1008, [0049]. This limitation is met. Ex. 1002, ¶¶986-994.

(v) Limitation 32D: "oil"

Weaver discloses using treatment fluid: "These treatment fluids 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, [0058]. "***Suitable emulsions*** can be comprised of two immiscible liquids such as an aqueous liquid or gelled liquid and ***a hydrocarbon***." *Id*., [0059].

Plummer teaches "a liquid carrier comprising ***one or more*** liquids selected from the group consisting of water and oil." Ex. 1010, 7:18-20. Plummer also teaches "[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling …" *Id*., 4:28-33. This limitation is met. Ex. 1002, ¶¶995-1001. It would have been obvious to a PHOSITA to add the oil of Weaver or Plummer to the aqueous carrier fluid of Needham. *Id*., ¶1001.

(vi) Limitation 32E: "pumping the mixture into the formation"

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 the fracture***." Ex. 1009, 3:19-23.

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, claim 23. This limitation is met. Ex. 1002, ¶¶1002-1006.

c. Independent Claim 51 is Obvious

(i) Preamble of Claim 51: "A method of controlling proppant flowback in a hydrocarbon producing formation comprising the steps of"

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, [0058]. A PHOSITA knows that a frac-pack is a type of gravel pack especially designed to control proppant flowback. Ex. 1002, ¶1008. Moreover, because Needham in view of Weaver teaches the use of hydrophobic proppants as well as oil in the slurry

composition, they both necessarily control proppant flowback, consistent with the specification of the '738 patent. Ex. 1001, Abstract, 1:60-2:34, 3:30-59, 3:65-4:4. This preamble is disclosed. Ex. 1002, ¶¶1008-1012.

(ii) Limitation 51A: "contacting particulates with a medium containing a chemical compound to render the surface of the particulates hydrophobic"

As previously indicated in §§VI.E.4.a.iv, Needham and Weaver disclose modifying agents that render the surface of the particulates hydrophobic. 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…" Ex. 1009, 4:29-37.

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 include[s]…solvent extraction methods." Ex. 1008, [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***." *Id*. This limitation is met. Ex. 1002, ¶¶1013-1016.

(iii) Limitation 51B: "separating the particulate from the medium"

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***…" Ex. 1009, 29-37. Weaver teaches "***[t]he 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.***" Ex. 1008, [0057]. A PHOSITA would understand that evaporating the solvent and extracting the solvent from the particles are methods of separating the particulates. Ex. 1002, ¶¶1018-1019. This limitation is met. *Id*., ¶¶1017-1020.

(iv) Limitation 51C: "blending the hydrophobic particulate with water and an oil to form a mixture"

Needham discloses that a preferred carrier fluid is aqueous and "dispersing said coated particulate solids into a carrying fluid." Ex. 1009, 4:38-39; 3:19-20. Weaver discloses the use of a mixture of aqueous and non-aqueous fluids as a carrier or base fluid wherein the non-aqueous fluids include oils. Ex. 1008, [0058]. 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.*, [0059]. A PHOSITA would recognize, and the '738 patent confirms, that "hydrocarbon" is another term for oil. Ex. 1002, ¶1025. Plummer teaches "a liquid carrier comprising ***one or more*** liquids selected from the group consisting of water and oil." Ex. 1010, 7:18-20. Plummer teaches "[o]il may be added to the foam-water slurry as a lubricant and to prevent clay swelling …" *Id.*, 4:28-33. It would have been obvious to substitute the carrier fluid taught by Weaver (through the aqueous/non-aqueous mixture or emulsion) or Plummer (oil and water liquid carrier) for the aqueous carrier fluid taught by Needham or to add the oil taught by Plummer (by adding oil as a friction reducer) to the aqueous carrier fluid of Needham. Ex. 1002, ¶¶1021-1028.

(v) Limitation 51D: "pumping the mixture into the formation

Needham teaches "introducing said carrying fluid and dispersed coated solids into a well penetrating a subterranean formation containing fractures." Ex. 1009, 4:40-42. 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, claim

23. This limitation is met. ¶¶1029-1033.

d. Dependent Claims 21, 22, 40, 41, and 57 Are Obvious

Needham discloses "***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*., 1:61-68. Weaver discloses "[s]uitable particulates include, but are not limited to, ***sand***." Ex. 1008, [0018]. Weaver also discloses the sand can be mixed with the treatment fluid prior to, during, or subsequent to introducing the slurry into the wellbore." *Id*., [0057]. Weaver and Needham meet these claims. Ex. 1002, ¶¶1034-1038.

Needham in view of Weaver or Plummer renders claims 1, 21, 22, 40, 41, 51, and 57 obvious. Ex. 1002, ¶1039.

F. Conclusion

For the foregoing reasons, Petitioners request that the Board institute *inter partes* review and determine that all challenged claims be canceled as obvious.

Respectfully submitted,

Dated: September 30, 2017

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CERTIFICATE OF COMPLIANCE WITH TYPE-VOLUME LIMITATION

This Petition complies with the type-volume limitation of 37 C.F.R. § 42.24(a)(1) because this Petition contains 13,953 words, as determined by the word-count function of Microsoft Word, excluding the parts of the Petition exempted by Rule (i.e., a table of contents, a table of authorities, mandatory notices under 37 C.F.R. § 42.8, a certificate of service or word count, or appendix of exhibits or claim listing).

Date: September 30, 2017

/s/ *John R. Keville*
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Lead Counsel for Petitioners

CERTIFICATE OF SERVICE

Pursuant to 37 C.F.R. §§ 42.6(e) and 42.105, I certify that I caused to be served a true and correct copy of: Petition for *Inter Partes* Review of U.S. Patent No. 8,236,738 and Exhibits 1001-1019 by Express Mail on this day on the Patent Owner's correspondence address of record for the subject patent as follows:

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