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addition, the intentional alteration of the wettability of a proppant will significantly change the fluid flow characteristics within the pore structure of the proppant. Since formation fines are typically transported only by the water phase (the fines are water-wet), such coated proppants may be significantly less damaged by migrating fines than conventional non-treated proppants.

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Example 7

According to this Example, a multiphase flow test was conducted. The multiphase flow test was conducted with respect to uncoated and polydimethyl siloxane coated CARBOHSPTM and a slurry of the proppant was top loaded between two saturated Ohio Sandstone cores. In this Example, the proppant samples were evaluated at 4000 psi closure stress and 150°F. In this test, saturated gas was flowed through the cells at a constant rate (26 1/min) while increasing rates of water were simultaneously pumped through. The differential pressure was measured as the liquid flow was increased; and it was desired that the differential pressure or "dP" be as low as possible. The results from the multiphase flow test are shown in Table 12.

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Table 12

Liquid Flow Rate (g/min)	Differential Pressure (bar)	
	Uncoated	Polydimethyl Siloxane
0	0.061	0.039
46.1	0.143	0.120
63.7	0.233	0.176
99.0	0.401	0.296
152.3	0.479	0.437
0	0.079	0.055

As set forth in Table 12, the polydimethyl siloxane coating showed improved (lower) pressure differential at all liquid flow rates compared to the uncoated control. Also, the beta factor for the polydimethyl siloxane sample was improved: 0.205 atm·s²/kg compared to 0.262 atm·s²/kg for the uncoated control. The multiphase flow test results in terms of a lower beta and improved multiphase flow for the polydimethyl siloxane coated CARBOHSPTM compared to the control indicated that the polydimethyl siloxane coating created a much smoother surface and covered some

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of the surface porosity of the CARBOHSPTM. It was confirmed by scanning electron microscopy ("SEM") at a power of 500x that the polydimethyl siloxane coating had indeed created a much smoother surface and appeared to have covered substantially all of the surface porosity of the CARBOHSPTM.

The chemically coated and/or treated particles of the present invention are useful as a propping agent in methods of fracturing subterranean formations to increase the permeability thereof.

When used as a propping agent, the particles of the present invention may be handled in the same manner as other propping agents. The particles may be delivered to the well site in bags or in bulk form along with the other materials used in fracturing treatment. Conventional equipment and techniques may be used to place the particles as a propping agent.

A viscous fluid, frequently referred to as a "pad", is injected into the well at a rate and pressure to initiate and propagate a fracture in the subterranean formation. The fracturing fluid may be an oil base, water base, acid, emulsion, foam, or any other fluid. Injection of the fracturing fluid is continued until a fracture of sufficient geometry is obtained to permit placement of the propping pellets. Thereafter, particles as hereinbefore described are placed in the fracture by injecting into the fracture a fluid or "slurry" into which the particles have previously been introduced and suspended. Following placement of the particles, the well is shut-in for a time sufficient to permit the pressure in the fracture to bleed off into the formation. This causes the fracture to close and apply pressure on the propping particles which resist further closure of the fracture. The resulting proppant distribution is usually, but not necessarily, a multi-layer pack.

The foregoing description and embodiments are intended to illustrate the invention without limiting it thereby. It will be understood that various modifications can be made in the invention without departing from the spirit or scope thereof.

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What is claimed is:

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1. A gas and oil well proppant comprising:

a plurality of essentially spherical particles, wherein the particles are coated with a hydrophobic material.

- 2. The proppant of claim 1, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, hydrocarbons, copolymerized polyvinylidene chloride, glass frit and enamel.
- The proppant of claim 1, wherein the proppant particles are coated with the hydrophobic material by one or more of spraying, dipping or soaking the proppant particles in a liquid solution of the hydrophobic material, applying a sheet of film to the proppant particles, fusing material to the proppant particles, electroplating, plasma spraying, sputtering, fluidizing and powder coating.
 - 4. The proppant of claim 2, wherein the hydrophobic material comprises a siloxane based on the structural unit R₂SiO, wherein R is an alkyl group.
 - 5. The proppant of claim 2, wherein a hydrophobic material comprises a nonvolatile linear siloxane of the composition:

$$(R_2) \\ | \\ 20 \qquad (R_1)_3 - Si - O - ((R_1) - Si - O)_n - Si - (R_3)_3$$

where (R_1) is an alkyl group having from one to three carbon atoms, (R_2) is hydrogen or an alkyl group having from one to three carbon atoms, (R_3) is an alkyl group having from one to four carbon atoms and n is a number between 50 and 200.

- 25 6. The proppant of claim 2, wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and polydimethyl siloxane.
 - 7. The proppant of claim 2, wherein the plant oils comprise at least one member selected from the group consisting of linseed oil, soybean oil, corn oil, cottonseed oil, vegetable oil and canola oil.
- 8. The proppant of claim 2, wherein the hydrocarbons comprise at least one member selected 30 from the group consisting of kerosene, diesel, crude oil, petroleum distillates, aliphatic solvents, solvent naphtha and paraffin.

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9. A method of fracturing a subterranean formation, comprising:

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injecting a hydraulic fluid into a subterranean formation at a rate and pressure sufficient to open a fracture therein; and

injecting into the fracture a fluid containing a plurality of essentially spherical particles, wherein at least some of the particles are modified to alter the surface characteristics thereof, wherein the particles are modified by coating the particles with a hydrophobic material.

- 10. The method of claim 9, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, hydrocarbons, copolymerized polyvinylidene chloride, glass frit and enamel.
- 11. The method of claim 9, wherein the proppant particles are coated with the hydrophobic material by one or more of spraying, dipping or soaking the proppant particles in a liquid solution of the hydrophobic material, applying a sheet of film to the proppant particles, fusing material to the proppant particles, electroplating, plasma spraying, sputtering, fluidizing and powder coating.
- 15 12. The method of claim 9, wherein the modification of the particles alters at least one of the chemical reactivity of the particles, the surface topography of the particles, the wettability of the particles and the lubricity of the particles.
 - 13. The method of claim 10, wherein the particles are coated with a silicone prior to injection into the fracture.
- 20 14. The method of claim 10, wherein the particles are coated with a silicone during injection into the fracture.
 - 15. The method of claim 10, wherein the hydrophobic material comprises a siloxane based on the structural unit R₂SiO, wherein R is an alkyl group.
- 16. The method of claim 10, wherein the hydrophobic material comprises a nonvolatile linear25 siloxane of the composition:

$$(R_2)$$

 $|$
 $(R_1)_3$ -Si-O- $((R_1)$ -Si-O)_n-Si- $(R_3)_3$

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where (R_1) is an alkyl group having from one to three carbon atoms, (R_2) is hydrogen or an alkyl group having from one to three carbon atoms, (R_3) is an alkyl group having from one to four carbon atoms and n is a number between 50 and 200.

- 17. The method of claim 10, wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and polydimethyl siloxane.
 - 18. The method of claim 10, wherein the plant oils comprise at least one member selected from the group consisting of linseed oil, soybean oil, corn oil, cottonseed oil, vegetable oil and canola oil.
 - 19. The method of claim 10, wherein the hydrocarbons comprise at least one member selected from the group consisting of kerosene, diesel, crude oil, petroleum distillates, aliphatic solvents, solvent naphtha and paraffin.
 - 20. A method of modifying the surface properties of hydraulic fracturing proppant particles, comprising:

coating the particles with a hydrophobic material.

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- 21. The method of claim 20, wherein the hydrophobic material comprises one or more hydrophobic materials selected from the group consisting of silicones, siloxanes, polytetrafluoroethylene, plant oils, hydrocarbons, copolymerized polyvinylidene chloride, glass frit and enamel.
 - 22. The method of claim 20, wherein the proppant particles are coated with the hydrophobic material by one or more of spraying, dipping or soaking the proppant particles in a liquid solution of the hydrophobic material, applying a sheet of film to the proppant particles, fusing material to the proppant particles, electroplating, plasma spraying, sputtering, fluidizing and powder coating.
 - 23. The method of claim 20, wherein the modification of the particles alters at least one of the chemical reactivity of the particles, the surface topography of the particles, the wettability of the particles and the lubricity of the particles.
- 25 24. The method of claim 20, wherein the hydrophobic material comprises a siloxane based on the structural unit R₂SiO, wherein R is an alkyl group.
 - 25. The method of claim 20, wherein the hydrophobic material comprises a nonvolatile linear siloxane of the composition:

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$$(R_2)$$

 $|$
 $(R_1)_3$ -Si-O- $((R_1)$ -Si-O)_n-Si- $(R_3)_3$

where (R₁) is an alkyl group having from one to three carbon atoms, (R₂) is hydrogen or an alkyl group having from one to three carbon atoms, (R₃) is an alkyl group having from one to four carbon atoms and n is a number between 50 and 200.

- 26. The method of claim 20, wherein the hydrophobic material is selected from the group consisting of polymethylhydrogen siloxane and polydimethyl siloxane.
- 10 27. The method of claim 20, wherein the plant oils comprise at least one member selected from the group consisting of linseed oil, soybean oil, corn oil, cottonseed oil, vegetable oil and canola oil.
 - 28. The method of claim 20, wherein the hydrocarbons comprise at least one member selected from the group consisting of kerosene, diesel, crude oil, petroleum distillates, aliphatic solvents, solvent naphtha and paraffin.

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(54) Title: METHOD FOR MAKING TRANSPORTABLE AQUEOUS SLURRIES BY PARTICULATE HYDROPHOBICIZA-TION

(57) Abstract: An aqueous slurry composition for use in industries such as the petroleum and pipeline industries includes a particulate, an aqueous liquid and a chemical compound that renders the particulate surface extremely hydrophobic. The slurry is produced by rendering the surface of the particulate extremely hydrophobic during or before making the slurry.



Method for Making Transportable Aqueous Slurries by Particulate hydrophobicization

BACKGROUND OF THE INVENTION

Field of the Invention

This invention relates to an aqueous slurry composition and to a method of making such a composition.

Discussion of the Prior Art

Aqueous particulate slurries are commonly used or encountered in many industries including the petroleum, pipeline, construction and cleaning industries. Slurries are mixtures normally consisting of particulates and an aqueous liquid and play an essential role in many industrial operations. For example, slurries are used when transporting particulates above ground, from the surface to a subterranean formation or from a subterranean formation to the surface. The most commonly used particulates include sand, ceramic particles, carbonate particles, glass spheres, bauxite (aluminum oxide), resin coated particulates and coal particulates The particulate sizes normally range from about 10 to about 100 US mesh, and the particles have densities significantly higher than the density of water. For example, the density of sand is at about 2.6 g/cm³ while the density of water is 1 g/cm³. Sand is by far the most commonly used particulate.

To make relatively stable slurry, particulates must be suspended in a liquid medium for a lengthy period at static or/and dynamic conditions. Convention wisdom tells us that the viscosity or viscoelasticity of the liquid medium must be sufficiently high to be able to suspend particulates. The most commonly used method for increasing viscosity or viscoelasticity of a liquid medium is by adding

a viscosifier, for example, a natural or synthetic polymer or a viscoelastic surfactant to the liquid medium. It is not unusual that a polymer is used with a foaming agent in order to take advantage of both viscoelastic and foaming properties. However, the use of polymers in slurries increases cost and results in operational difficulties. In particular applications, for example, hydraulic fracturing of subterranean formations, the use of polymers in slurry impedes oil and gas production due to large amounts of residue left in the formation. As for viscoelastic surfactants, although they have fewer residues compared to normal polymers, their cost is usually much higher. In many other applications such as gravel-pack, well completion and sand transportation through pipelines, it is highly desirable to make stable particulate slurry without using a viscosifier.

Hydraulic fracturing operations are used extensively in the petroleum industry to enhance oil and gas production. In hydraulic fracturing, a fracturing fluid is injected through a wellbore into a subterranean formation at a pressure sufficient to initiate fracturing, which increases oil and gas production. Frequently, particulates, called proppants, are suspended in a fracturing fluid and transported into a fracture as slurry. Proppants include sands, ceramic particulates, glass spheres, bauxite particulates, resin coated sands and other particulates known in the industry. Among them sand is by far the most commonly used proppant. Fracturing fluids in common use include water-based as well as hydrocarbonbased fluids. In water-based fracturing fluids, a polymer or viscoelastic surfactant is normally employed to increase the viscoelasticity of the fluid. In most case the viscoelastic property of the fluids is essential for transporting proppants deep into

a formation. At the last stage of the fracturing treatment, fracturing fluid flows back to the surface and the proppants are left in the fracture forming a proppant pack to prevent the fracture from closing after pressure is released. A proppantfilled fracture provides a highly conductive channel that allows oil and/or gas to seep through more efficiently to the wellbore. The conductivity of the proppant pack plays a dominant role in production enhancement. Polymer residues from fracturing fluids are known to greatly reduce the conductivity of the propognit pack. Compared to polymeric viscosifiers, viscoelastic surfactants cause less damage to formations and proppant packs. However, they are much more expensive. Accordingly, a need exists for a composition for efficiently transporting proppants deep into a formation at low cost while at the same time causing little damage to the formation and proppant pack. Grain size, concentration, and the packing pattern of proppants are also important factors in determining the conductivity. Despite extensive research in recent years, limited progress has been achieved to maximize the conductivity of a proppant pack in a fracture. Therefore, a need exists for making a composition for use in a proppant pack with improved conductivity.

Proppant flowback after fracturing treatments has long been plaguing the petroleum industry. Flowback reduces the amount of proppants in the formation leading to a less conductive fracture. As disclosed, for example in US Patent 6,047,772, various methods have been tried to solve the flowback problem. In one method, resins are used to coat the proppant and make them very tacky. In doing so, the proppant grains tend to agglomerate reducing flowback. This

method is not only expensive, but the tacky resins introduced in the proppant pack tend to reduce its conductivity. Therefore, there is a need for a composition and method for making slurry, which can form a stable proppant pack, which resists proppant flowback while at the same time has a high conductivity.

When drilling subterranean formations for oil and gas, aqueous-based drilling fluids are normally used. During drilling large amounts of particles, called cuttings are generated. Cuttings have different sizes ranging from fines to pebbles. The drilling fluid is circulated through the wellbore to make slurry with the cuttings in situ and transports them out of wellbore. In most cases, polymers as well as clays are added to the drilling fluids to increase their viscosity/ viscoelasticity in order to transport the cuttings efficiently. However, polymers as well as clay fines, can easily penetrate pores or thin fractures in a formation and reduce formation permeability significantly, especially near a wellbore. Reduced formation permeability impedes oil and/or gas production. Therefore it is highly desirable to provide a drilling fluid that can make stable slurry in situ with the cuttings and transport them out of the wellbore, while causing little formation damage.

The escalating price of oil and its alarming depletion rate have caused people to consider using coal as an oil substitute. Several factors have slowed the substitution of coal for oil. One factor is the difficulty in transporting coal costeffectively over long distance through pipelines. It is therefore highly desirable to provide a composition for making coal slurry which is stable, highly fluid and costeffective to transport.

In oil sand operation massive amount of sands are left after oil is stripped from the sand surface. Finding a more cost effective way to transport sands efficiently over distance through pipelines has long been required in the industry. Thus, a composition and a method for making stable and highly fluid sand slurries at low cost would be quite useful.

The object of the present invention is to meet the above defined needs by providing an aqueous slurry composition, which can be used in a stable, highly conductive proppant pack, for efficiently transporting proppants into a subterranean formation, and for use in transporting cuttings, coal and sand.

SUMMARY OF THE INVENTION

Accordingly, the invention relates to an aqueous slurry composition comprising particulates, an aqueous liquid and a chemical compound that renders the surface of the particulates extremely hydrophobic.

The invention also relates to a method of producing an agueous slurry composition comprising the steps of mixing particulates with an aqueous liquid, and rendering the particulate surface extremely hydrophobic during or before mixing the particulates with the agueous liquid.

The present invention is based on the discovery that when the surface of the particulates becomes extremely hydrophobic, the slurry has several novel properties. For example, particulates tend to move cohesively instead as individual grains; the bulk volume of settled particulates tend to be significantly greater than in a slurry formed by convention methods under the same conditions; the particulate pack formed tends to have high conductivity and be

easily dewatered, and the slurry tends to be fluid and stable without using a viscosifier. The larger bulk volume of the particulate pack indicates a larger porosity and therefore higher conductivity. This is particularly beneficial for improving fracturing treatment, since, as mentioned above, the conductivity of the proppant pack is the dominant property affecting fracturing treatment. The extremely hydrophobic surface of the particulate further reduces the dragging force exerted by the fluid and makes it more difficult for proppants to be carried away by the fluid. This is particularly beneficial for minimizing proppant flowback after fracturing treatments, leading to increased proppant conductivity. In conventional slurry, viscosity or viscoelasticity of the liquid plays the dominant role while the interfacial interactions between the particulate surface and the liquid play negligible role. However it is discovered in the present invention that when the surface of the particulate becomes extremely hydrophobic, the interfacial interactions between the surface and the aqueous liquid become increasingly important, and even can play a dominant role.

In general, the interfacial interactions between a solid substrate and a liquid mainly depend on the surface properties and the surface tension of the liquid. Normally the macroscopic properties of a surface can be characterized by observing the shape of a liquid droplet on the solid substrate, which is the result of free energy of the surface, as well as the free energy of the liquid. When a liquid does not completely wet a surface, it forms an angle θ , which is known as the contact angle. The contact angle is the angle formed between a solid substrate and the tangent line at the point of contact between a liquid droplet and

the solid substrate. Contact angle can be measured directly on macroscopic, smooth, nonporous, planar solid substrates by merely placing a droplet of the liquid or solution on the solid substrate and determining the contact angle by any of number of techniques. It is known that majority of natural occurred minerals are water-wet. It is also known that certain hydrocarbon compounds, for example, some conventional quaternary surfactants, amine surfactants and cationic polyacrylamides can be used to reduce the surface energy of certain particulates and make the particulate surface more hydrophobic. However, the "hydrophobicity" imparted by such compounds is not high enough to be included in the term of "extremely high hydrophobicity" as in the case of the present invention. In the present invention, by "extremely hydrophobic" it means that the contact angle of water on the solid substrate is greater than about 90°. The chemical compounds that can render the particulate surface extremely hydrophobic are referred as "extremely hydrophobic rendering compounds" (EHRC), EHRC normally are those compounds that contain organosilane or organosilloxane groups or fluoro-organic groups. Because of such groups, EHRC are able to impart hydrophobicity to solid surface to a level that conventional hydrocarbon surfactants or polymers are not able to achieve.

The slurry can be made on the ground or in situ in a subterranean formation. The slurry finds numerous applications in many industries, including

(a) transporting particulates over various distances, either on the surface of the ground, from the surface to a subterranean formation or from a subterranean formation to the surface, and

(b) well service operations including stimulation, drilling, completion, gravel-pack, controlling sand production and the like.

DETAILED DESCRIPTION OF THE INVENTION

A gas can be added to the slurry. Suitable gases for use in the slurry include air, carbon dioxide, nitrogen, methane and mixtures thereof. The gas can be introduced into the slurry during preparation thereof. For example, when the slurry is pumped through a pipe at a sufficient rate, gas such as air can be introduced into the slurry. In the present case, by "aqueous liquids" is meant water, salt solutions, water containing an alcohol or other organic solvents, mixtures of water with carbon dioxide and the like. It will be appreciated that the additives other than water in the aqueous liquid are used in amounts or in a manner that does not adversely affect the present invention. The aqueous fluid can also contain polymers which can be linear or cross-linked. For example, in so-called slick-water fracturing, a small amount of polymer is normally added to reduce friction during pumping. The size of particulates in the composition is about 10-100 US mesh, which is about 150 to 1400 µm. It should be understood that the size distribution of particulates can be narrow or wide. Suitable particulates include sand, ceramic, glass beads, bauxite, resin coated sand, carbonates and coal particulates.

There are several approaches to make particulate surfaces extremely hydrophobic. One method is to use certain organosilicon compounds to render the surface of particulates such as sands, ceramic particles, glass spheres and bauxite extremely hydrophobic. The organosilicon compounds include

organosiloxane, organosilane, fluoro-organosiloxane and fluoro-organosilane compounds. The organosiloxane compounds include quaternary siloxane compounds including quaternary polydimethyl siloxane or diquaternary polydimethyl siloxane and siloxane amines. The organosilane compounds include alkylchlorosilane, for example methyltrichlorosilane, dimethyldichlorosilane, trimethylchlorosilane, octadecyltrichlorosilane, alkylalkoxysilane compounds, for example methyl-, propyl-, isobutyl- and octyltrialkoxysilanes. The fluoro-organosilane compounds include 2-(n-perfluorooctyl)-ethyltriethoxysilane, and perfluoro-octyldimethyl chlorosilane. Other types of chemical compounds which can be used to render particulate surface extremely hydrophobic are certain fluoro-substituted compounds, for example certain fluoro-organic compounds. Examples are described in US Patents Nos. 4,564,456; 4,689,085; 5,098,979; 5,209,775; 5,240,760; 5,359,104; 6,132,638 and 6,830,811 and Canadian Patent No. 2,213,168. In some cases, when using the composition described herein, a catalyst might be preferred to speed up the interaction between an EHRC and the particulate surface. For different particulates, certain EHRC may be preferred over others.

There are many types of organosilicon compounds which can be used to impart extreme hydrophobicity to particulate surfaces. One example is an organosilane which can be represented by the formula

R_nSiX_(4-n) (1)

wherein R is an organic radical containing 1-50 carbon atoms, X is a halogen, alkoxy, acyloxy or amine containing 1-50 carbon atoms and n has a value of 1 -

3. Examples of suitable organosilanes include:

CH₃SiCl₃, CH₃CH₂SiCl₃, (CH₃)₂SiCl₂, (CH₃CH₂)₂SiCl₂, (C₆H₅)₂SiCl₂, (C₆H₅)SiCl₃, (CH₃)₃SiCl, CH₃HSiCl₂, (CH₃)₂HSiCl, CH₃SiBr₃, (C₆H₅)SiBr, (CH₃)₂SiBr₂, (CH₃CH₂)₂SiBr₂, (C₆H₅)SiBr₂, (CH₃)₃SiBr, CH₃HSiBr₂, (CH₃)₂HSiBr, Si(OCH₃)₄, CH₃Si(OCH₃)₃, CH₃Si(OCH₂CH₃)₃, CH₃Si(OCH₂CH₂CH₃)₃, CH₃Si[O(CH₂)₃CH₃]₃, CH₃CH₂Si(OCH₂CH₃)₃, C₆H₅Si(OCH₃)₃, C₆H₅CH₂Si(OCH₃)₃, C₆H₅Si(OCH₂CH₃)₃, CH₂=CHCH₂Si(OCH₃)₃, (CH₃)₂Si(OCH₃)₂, (CH₃)₂Si(OCH₂CH₃)₂, (CH₃)₂Si(OCH₂CH₂CH₃)₂, $(CH_3)_2Si[O(CH_2)_3CH_3]_2$, $(CH_3CH_2)_2Si(OCH_2CH_3)_2$, $(C_6H_5)_2Si(OCH_3)_2$, $(C_6H_5CH_2)_2Si(OCH_3)_2$, $(C_6H_5)_2Si(OCH_2CH_3)_2$, $(CH_2=CH_2)Si(OCH_3)_2$, (CH₂=CHCH₂)₂Si(OCH₃)₂, (CH₃)₃SiOCH₃, CH₃HSi(OCH₃)₂, (CH₃)₂HSi(OCH₃), CH₃Si(OCH₂CH₂CH₃)₃, CH₂=CHCH₂Si(OCH₂CH₂OCH₃)₂, $(C_6H_5)_2Si(OCH_2CH_2OCH_3)_2$, $(CH_3)_2Si(OCH_2CH_2OCH_3)_2$, (CH₂=CH₂)₂Si(OCH₂CH₂OCH₃)₂, (CH₂=CHCH₂)₂Si(OCH₂CH₂OCH₃)₂, (C₆H₅)₂Si(OCH₂CH₂OCH₃)₂, CH₃Si(CH₃COO)₃, methyldiethylchlorosilane, butyltrichlorosilane diphenyldichlorosilane, vinyltrichlorosilane, methyltrimethoxysilane, vinyltriethoxysilane, vinyltris(methoxyethoxy)silane, methacryloxypropyltrimethoxysilane, glycidoxypropyltrimethoxysilane, aminopropyltriethoxysilane, divinyldi-2-methoxysilane, ethyltributoxysilane, isobutyltrimethoxysilane, hexyltrimethoxysilane, n-octyltriethoxysilane,

dihexyldimethoxysilane: trichloro-octadecylsilane and quaternary ammonium

silane including 3-(trimethoxysilyl)propyldimethyloctadecyl ammonium chloride. 3-(trimethylethoxysilylpropyl)didecylmethyl ammonium chloride and triethoxysilyl soyapropyl dimonium chloride.

Different polysiloxane compounds can also be useful for the present invention. Polysiloxanes modified with organic cationic or amphoteric groups including organic betaine polysiloxanes and organic quaternary polysiloxanes are examples. One type of betaine polysiloxane or quaternary polysiloxane is represented by the formula

$$R_{1} \longrightarrow S_{1} \longrightarrow C \longrightarrow S_{1} \longrightarrow$$

wherein each of the groups R₁ to R₆, and R₈ to R₁₀ represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R₇ represents an organic betaine group for betaine polysiloxane, for example betaine polysiloxane copolyol, or an organic quaternary group for quaternary polysiloxane, and may contain a hydroxyl group or other functional groups containing N, P or S, and have different numbers of carbon atoms, and m and n are from 1 to 200. For example when R₇ is an organic quaternary group it can be represented by the group

$$\begin{bmatrix} -Z & N^{+} & R^{2} \\ -Z & N^{+} & R^{2} \end{bmatrix} X^{-} \text{ or } \begin{bmatrix} R^{4} & O \\ -Z & N^{+} & (CH_{2})_{x}R^{6} & CR^{7} \\ R^{5} & R^{5} \end{bmatrix} X^{-}$$

wherein R¹, R², R³ are alkyl groups with 1 to 22 carbon atoms or alkenyl groups with 2 to 22 carbon atoms. R⁴, R⁵, R⁶ are alkyl groups with 1 to 22 carbon atoms or alkenvl groups with 2 to 22 carbon atoms; R⁶ is -O- or the NR⁸ group, R⁸ being an alkyl or hydroxyalkyl group with 1 to 4 carbon atoms or a hydrogen group; Z is a bivalent hydrocarbon group with at least 4 carbon atoms, which may have a hydroxyl group and may be interrupted by an oxygen atom, an amino group or an amide group; x is 2 to 4; The R¹, R², R³, R⁴, R⁵, R⁶ may be the same or the different, and X is an inorganic or organic anion. Such compounds are commercial available from Degussa Corporation and Dow Corning Corporation.

Other example of organo-modified polysiloxanes include di-betaine polysiloxanes and di-quaternary polysiloxanes, which can be represented by the formula

wherein the groups R₁₂ to R₁₇ each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, both R₁₁ and R₁₈ group represent an organic betaine group for di-betaine polysiloxanes or an organic quaternary group for di-quaternary, for example Quaternium 80 (INCI), and may contain a hydroxyl group or other functional groups containing N, P or S, and have different numbers of carbon atoms, and m is from 1 to 200. For example when R₁₁ and R₁₈ is an organic quaternary group it can be represented by the group

$$\begin{bmatrix} -Z & R^1 \\ -Z & N^+ & R^2 \end{bmatrix} X^- \text{ or } \begin{bmatrix} R^4 \\ -Z & N^+ & (CH_2)_x R^6 & CR^7 \end{bmatrix} X^-$$

wherein R¹, R², R³, R⁴, R⁵, R⁶, Z, X⁻ and x are defined above. Such compounds are commercially available from Degussa Corporation or Dow Corning Corporation. It should be apparent to those skilled in the art that there are different mono- and di-quaternary polysiloxanes, mono- and di-betaine polysiloxanes and other organo-modified polysiloxane compounds which can be useful in the present invention. These compounds are widely used in personal care products, for example in US patents 4,054,161; 4,891,166; 5,235,082; 5,306,434; 5,474,835; 5,616,758; 6,277,361 and 6,482,969.

Another example of organosilicon compounds which can be used in the composition of the present invention are fluoro-organosilane or fluoroorganosiloxane compounds in which at least part of the organic radicals in the silane or siloxane compounds are fluorinated. Suitable examples are fluorinated chlorosilanes or fluorinated alkoxysilanes including 2-(n-perfluorooctyl)ethyltriethoxysilane, perfluoro-octyldimethyl chlorosilane, (CF₃CH₂CH₂)₂Si(OCH₃)₂, CF₃CH₂CH₂Si(OCH₃)₃. (CF₃CH₂CH₂)₂Si(OCH₂CH₂OCH₃)₂ and CF₃CH₂CH₂Si(OCH₂CH₂OCH₃)₃. Other compounds which can be used are fluoro-substituted compounds, for example, certain fluoro-organic compounds.

The slurry according to the present invention can be prepared, for example, by mixing water with the particulates and an EHRC. Normally sufficient shear is needed. Alternatively, the particulates can be first treated by contacting a fluid medium containing an EHRC to cause the particulates to become extremely hydrophobic and then separating the particulates from the medium. The fluid medium can be a liquid or a gas. The hydrophobic particulates can later be used to make slurry. Water is the most preferred aqueous liquid for making the slurry. Certain salts, some conventional hydrocarbon surfactants or polymers can be added to the slurry at concentrations and in a manner which would not adversely affect the slurry. For example, when conventional surfactants, polymers or other additives are added to the slurries, one should try to avoid forming insoluble precipitates with the EHRC, or making large changes to the surface energy of the particulate surface, or greatly reducing the surface tension of the aqueous liquid. In some cases, a very low surface tension of the aqueous liquid is not desirable. When the surface tension of the liquid is too low, more water can be added or some of the aqueous liquid can be replaced with new water.

The slurries can be prepared on surface (above ground) or in a subterranean formation where the particulates, an aqueous fluid, and an EHRC. for example a di-quaternary polysiloxane, are mixed in situ. Examples of situations where in situ mixing is used include drilling and wellbore cleanout operations. Alternatively, the particulates can be first mixed with a liquid in which an EHRC is dispersed or dissolved and then the particulates separated from the

liquid or dried. The thus treated particulates can subsequently be used to make the slurry. Various proppants including sands, ceramic particulates or resin coated sands can be treated according to the present invention during manufacturing process. The thus prepared hydrophobic particulates can be used as proppants in fracturing operations. Depending on the amount and size of the particulates in the slurry, a wide range of EHRC concentration can be used to render the particulate surface extremely hydrophobic. Usually the amount of EHRC added is very small and has no apparent effect on the viscosity of the liquid to which it is added. For example, the concentration of EHRC in the slurry can be as low as a few ppm to hundreds of ppm. In most applications, it is unnecessary to add EHRC in an amount larger than 1 percent of the total liquid.

The following examples serve to illustrate the concepts of the present invention.

Example 1

50 ml of water and 50 grams of 20/40 mesh fracturing sands were added into each of two glass bottles (200ml). 0.5 ml of Tegopren 6923, a di-quaternary ploysiloxane from Degussa Corp., was added into one of the bottles and the other bottle was used as control. The bottles were vigorously shaken and then let stand to allow sands settle down. The volumes of the settled sands in the two bottles were compared. In the bottle containing Tegopren 6923, the volume of the settled sands was about 40 percent greater than the one without. When the bottles were tilted, the settled sands in the bottle with straight water tended to

move as individual sand grains, while the settled sands containing Tegopren 6923 tended to move as cohesive masses.

Example 2

50 ml water, 50 grams of 20/40 mesh fracturing sands, 0.5 ml of Tegopren 6923 and 0.1 ml of Aquard 18-50, a hydrocarbon quaternary ammonium salt from Akzo Nobel Corp., was mixed into a glass bottle (200ml). The bottles were vigorously shaken and then let stand to allow sands settle down. The sand grains immediately following agitation were fully distributed in water making stable slurry. After one hour, about half amount of sands settled down to the bottom while the other half was floating on the top.

Example 3

100 ml of water and 50 grams of 20/40 mesh ceramic proppants were added into each of two glass bottles (200ml). 0.5 ml of TEGO Betaine 810, a capryl/capramidopropyl betaine from Degussa Corp., and 1 ml of a solution containing 20% Tegopren 6924, a di-quaternary ploysiloxane from Degussa Corp., and 80% of ethylene glycol mono-butyl ether were added into one of the bottles, and the other bottle was used as control. The bottles were vigorously shaken and then let stand to allow proppants settle down. In the one containing Tegopren 6924 about 25% of proppants was floating on the top and the remaining 75% settled on the bottom. The volume of the 75% settled proppants was still significantly larger than the control one. When the bottles were tilted, the settled proppants in the bottle with straight water tended to move as individual

grains, while the settled proppants containing Tegopren 6924 tended to move as cohesive masses.

Example 4

100 ml of water and 50 grams of 40/70 mesh fracturing sand were added into each of two glass bottles (200ml). 0.1 ml of Tegopren 6924 and 0.1 ml of TEGO Betaine 810, were added and further added 2 wt% KCl. The other bottle was used as control. The bottles were vigorously shaken and then let stand to allow sands settle down. The volumes of the settled sands in the two bottles were compared. In the bottle containing Tegopren 6924, about 15% of sands was floating on the top and the remaining 85% settled on the bottom. The volume of the 85% settled sand was still significantly larger than the control one. When the bottles were tilted, the settled sands in the bottle with straight water tended to move as individual grains, while the settled sands containing Tegopren 6924 tended to move as cohesive masses.

Example 5

100 ml of water and 50 grams of 40/70 mesh fracturing sand were added into each of two glass bottles (200ml). 0.5 ml of TEGO Betaine 810 and 1 ml of a solution containing 20% Tegopren 6924 and 80% of ethylene glycol mono-butyl ether were added into one of the bottles. After thoroughly mixing the sands were separated from the liquid and dried at room temperature. The prehydrophobonized sands were mixed with 100ml water and shaken vigorously. In the bottle containing Tegopren 6924, about 40% of sands was floating on the top and the remaining 60% settled on the bottom. The volume of the 60% settled

sand was still significantly larger than the control one. When the bottles were tilted, the settled sands in the bottle with straight water tended to move as individual grains, while the settled sands containing Tegopren 6924 tended to move as cohesive masses.

Example 6

100 ml of water and 50 grams of coal particulates were added into each of two glass bottles (200ml). 0.5 ml of TEGO Betaine 810 and 1 ml of a solution containing 20% Tegopren 6924 and 80% of ethylene glycol mono-butyl ether were added into one of the bottles. The other bottle was used as control. In the bottle containing Tegopren 6924, about 45% of coal particulates was floating on the top and the remaining 55% settled on the bottom. The volume of the 55% settled coal particulates was about 15% smaller than the control one.

Example 7

100 ml of water and 50 grams of 40/70 mesh fracturing sand were added into each of two glass bottles (200ml). 0.03 ml of Maquat QSX, a quaternary silane compound characterized as triethoxysilyl soyapropyl dimonium chloride in butylene glycol, was added into one of the bottles. The other bottle was used as control. After being thoroughly mixed the liquid above the settled sand was discarded and replaced with same amount of water. The bottles were vigorously shaken and then let stand to allow sands settle down. The volumes of the settled sands in the two bottles were compared. In the bottle containing Maquat QSX, about 5% of sands was floating on the top and remain 95% settled on the bottom. The volume of the 95% settled sand was still significantly larger than the

control one. When the bottles were tilted, the settled sands in the bottle with straight water tended to move as individual grains, while the settled sands containing Maquat QSX tended to move as cohesive masses.

Example 8

100 ml of water and 50 grams of 20/40 mesh resin coated sands were added into each of two glass bottles (200ml). 0.5 ml of TEGO Betaine 810 and 1 ml of a solution containing 20% Tegopren 6924 and 80% of ethylene glycol mono-butyl ether were added into one of the bottles, and the other bottle was used as control. The bottles were vigorously shaken and then let stand to allow resin coated sands settle down. The volume of settled sands containing Tegopren 6924 is about twice of that in the control one.

As mentioned above, the present invention is particularly useful in many applications in the petroleum industry as well as in other industries. Examples include various well service operations including hydraulic fracturing, gravel pack, wellbore cleanout and drilling, particulate transportation through pipe lines and sand blasting.

When used in a hydraulic fracturing operation, a large amount of proppants can effectively be transported into subterranean formation without using a viscosifier. It is not only cost-effective but also eliminates damage to the formation and proppant pack caused by polymer residues. An EHRC, for example a di-quaternary polysiloxane can be added to a water-based fracturing fluid containing proppants to make the slurry and then pumped into the formation during the proppant stage. Various aqueous fracturing fluids including water.

brine, linear polymer fluid, cross-linked polymer fluid and viscoelastic surfactant fluid can be used. It is particularly beneficial to use the slurry in so-called slickwater fracturing treatment. In conventional slick-water fracturing operations, due to the low viscosity of the fluid, only low concentration of proppants can be effectively pumped deep into a formation, and moreover the proppants tend to settle down on the bottom of the fracture, resulting in lower conductivity. With the composition of the present invention, high concentration of proppants can easily be pumped deep into a formation and the proppants are more evenly distributed in the fracture, leading to improved conductivity of the proppant pack. During the fracturing operation, the EHRC can be added on-the-fly. Optionally, one can use proppants already rendered extremely hydrophobic in the fracturing operation. Another benefit of the slurry of the present invention is that the fluid is readily reuseable after flow back from a well. This has great significance considering there is limited water supply in a number of places.

The present invention also provides a new method for preventing proppant flowback after a fracturing treatment. In field operations, proppants can be pumped into a formation using the composition of the present invention. Various aqueous fracturing fluid, for example water, brine, a linear polymer fluid, a crosslinked polymer fluid or a viscoelastic surfactant fluid can be used. Alternatively, a fluid medium containing an EHRC can be pumped into the formation following the proppant stage to mix with particulates already in the formation. The particulates in the slurry tend to move cohesively in contrast to conventional slurries under the same conditions. It is worth noting that the cohesiveness

among the proppant grains in the present slurry originates from hydrophobic interactions, instead of tackiness as described, for example in US Patent 6,047,772.

The slurry of the present invention is particularly useful in gravel-pack operations where sand slurry is normally pumped into a wellbore to prevent excessive amount of sands from flowing into the wellbore from the formation. The present method is cost effective and the sand pack formed has a high conductivity. Similarly, the slurry can also be used in so-called formation consolidation operations. In such an operation, a fluid containing an EHRC is injected into a formation to increase cohesiveness among sand grains to consolidate the formation and to reduce sand production.

In drilling operations, an EHRC can be added directly to a water-based drilling fluid. It is particularly useful when the EHRC is added to water or brine for use as a drilling fluid. During a drilling operation, the fluid forms slurry in situ with cuttings and transports the cuttings out of the wellbore. A gas such as nitrogen or carbon dioxide can be mixed with the slurry during drilling. Since it is not necessary to use polymers or clays to viscosify the fluid, there is much less formation damage. Moreover, the cuttings can be easily removed on the surface and the fluid becomes readily re-useable. Different formations including sandstone, carbonate, shale and coal seams can be drilled using the slurry of the present invention.

Similarly in wellbore cleanout operations, water or brine containing an EHRC can circulate in the wellbore and form slurry with debris in situ. The debris

is subsequently transported out of the wellbore as slurry. The fluid is readily reuseable after separation from the debris.

For transporting particulates through pipelines slurry can be prepared by mixing the ingredients and then pumping the slurry through the pipeline.

CLAIMS:

1. An aqueous slurry composition comprising:

- (a) water;
- (b) particulates; and
- a chemical compound for rendering the surface of the particulates (c) extremely hydrophobic.
- 2. The composition of claim 1, wherein the particulates are selected from the group consisting of sand, resin coated sand, ceramic, carbonate, bauxite, shale and coal particulates.
- 3. The composition of claim 1, wherein the particulates are subterranean formation particulates.
- 4. The composition of claim 1, wherein the chemical compound is selected from the group consisting of an organosilane, an organosiloxane, a fluoroorganosilane, a fluorro-organosiloxane and a fluoro-organic compound.
- 5. The composition of claim 1, wherein the chemical compound is an organosilane having the formula

R_nSiX_(4-n)

wherein R is an organic radical having 1-50 carbon atoms, X is a halogen, alkoxy, acyloxy or amine and n has a value of 1-3.

The composition of claim 5, wherein the organosilane is selected from the 6. group consisting of:

CH₃SiCl₃, CH₃CH₂SiCl₃, (CH₃)₂SiCl₂, (CH₃CH₂)₂SiCl₂, (C₆H₅)₂SiCl₂, (C₆H₅)SiCl₃, (CH₃)₃SiCl, CH₃HSiCl₂, (CH₃)₂HSiCl, CH₃SiBr₃,

(C₆H₅)SiBr, (CH₃)₂SiBr₂, (CH₃CH₂)₂SiBr₂, (C₆H₅)SiBr₂, (CH₃)₃SiBr, CH₃HSiBr₂, (CH₃)₂HSiBr, Si(OCH₃)₄, CH₃Si(OCH₃)₃, CH₃Si(OCH₂CH₃)₃, CH₃Si(OCH₂CH₂CH₃)₃, CH₃Si[O(CH₂)₃CH₃]₃, CH₃CH₂Si(OCH₂CH₃)₃, $C_6H_5Si(OCH_3)_3$, $C_6H_5CH_2Si(OCH_3)_3$, $C_6H_5Si(OCH_2CH_3)_3$, $CH_2=CHCH_2Si(OCH_3)_3$, (CH₃)₂Si(OCH₃)₂, (CH₃)₂Si(OCH₂CH₃)₂, (CH₃)₂Si(OCH₂CH₂CH₃)₂, $(CH_3)_2Si[O(CH_2)_3CH_3]_2$, $(CH_3CH_2)_2Si(OCH_2CH_3)_2$, $(C_6H_5)_2Si(OCH_3)_2$, $(C_6H_5CH_2)_2Si(OCH_3)_2$, $(C_6H_5)_2Si(OCH_2CH_3)_2$, $(CH_2=CH_2)Si(OCH_3)_2$, (CH₂=CHCH₂)₂Si(OCH₃)₂, (CH₃)₃SiOCH₃, CH₃HSi(OCH₃)₂, (CH₃)₂HSi(OCH₃), CH₃Si(OCH₂CH₂CH₃)₃, CH₂=CHCH₂Si(OCH₂CH₂OCH₃)₂, $(C_6H_5)_2Si(OCH_2CH_2OCH_3)_2$, $(CH_3)_2Si(OCH_2CH_2OCH_3)_2$, $(CH_2=CH_2)_2Si(OCH_2CH_2OCH_3)_2$, $(CH_2=CHCH_2)_2Si(OCH_2CH_2OCH_3)_2$, (C₆H₅)₂Si(OCH₂CH₂OCH₃)₂, CH₃Si(CH₃COO)₃, methyldiethylchlorosilane, butyltrichlorosilane diphenyldichlorosilane, vinyltrichlorosilane, methyltrimethoxysilane, vinyltriethoxysilane, vinyltris(methoxyethoxy)silane, methacryloxypropyltrimethoxysilane, glycidoxypropyltrimethoxysilane, aminopropyltriethoxysilane, divinyldi-2-methoxysilane, ethyltributoxysilane, isobutyltrimethoxysilane, hexyltrimethoxysilane, n-octyltriethoxysilane, dihexyldimethoxysilane; trichloro-octadecylsilane, 3-(trimethoxysilyl)propyldimethyloctadecyl ammonium chloride, 3-(trimethylethoxysilylpropyl)didecylmethyl ammonium chloride and triethoxysilyl soyapropyl dimonium chloride.

The composition of claim 1, wherein the chemical compound is an organo-7. siloxane having the formula

wherein each of R₁ to R₆ and R₈ to R₁₀, represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R₇ represents an organic betaine group for an organic betaine polysiloxane or an organic quaternary group for an organic quaternary polysiloxane, and m and n are from 1 to 200.

- 8. The composition of claim 7, wherein the chemical compound is an organic betaine polysiloxane.
- 9. The composition of claim 8, wherein the chemical compound is an organic betaine polysiloxane, and the values of m and n are from 1 to 100.
- 10. The composition of claim 9, wherein the chemical compound is an organic betaine polysiloxane, wherein each of R₁ to R₆ and R₈ to R₁₀ is a methyl group and the values of m and n are from 1 to 100.
- 11. The composition of claim 7, wherein the chemical compound is an organic quaternary polysiloxane.
- 12. The composition of claim 11, wherein the chemical compound is an organic quaternary polysiloxane, and the values of m and n are from 1 to 100.
- 13. The composition of claim 12, wherein the chemical compound is an organic quaternary polysiloxane, and each of R1 to R6 and R8 to R10 is a methyl group and the values of m and n are from 1 to 100.
- 14. The composition of claim 1, wherein the chemical compound is an organosiloxane having the formula

where R₁₂ to R₁₇ each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R₁₁ and R₁₈ each represents an organic betaine group for a di-betaine polysiloxane or an organic quaternary group for a diquaternary polysiloxane, and m is 1 to 200.

- 15. The composition of claim 14, wherein the chemical compound is a di-betaine polysiloxane.
- 16. The composition of claim 15, wherein the chemical compound is a di-betaine polysiloxane and the values of m are from 1 to 100.
- 17. The composition of claim 16, wherein each of R₁₂ to R₁₇ represents a methyl group and the m is from 1 to 100.
- The composition of claim 14, wherein the chemical compound is a di-18. quaternary polysiloxane.
- The composition of claim 18, wherein the chemical compound is a di-19. quaternary polysiloxane, and the values of m are from 1 to 100.
- The composition of claim 19, wherein each of R₁₂ to R₁₇ represents a 20. methyl group and the m is from 10 to 100.
- The composition of any one of claims 1 to 20 includes a gas. 21.
- The composition of claim 21, wherein the gas is selected from the group 22. consisting of air, nitrogen, carbon dioxide, methane and mixtures thereof.

23. A method of producing an aqueous slurry composition comprising the step of mixing water, particulates and a chemical compound for rendering the surface of the particulates extremely hydrophobic.

- The method of claim 23, wherein the particulates are selected from the 24. group consisting of sand, resin coated sand, ceramic, carbonate, bauxite, shale and coal particulates.
- The method of claim 23, wherein the chemical compound is selected from 25. the group consisting of an organosilane, an organosiloxane, a fluoroorganosilane, a fluoro-organosiloxane and a fluoro-organic compound.
- 26. The composition of claim 25, wherein the chemical compound is an organosilane having the formula

wherein R is an organic radical having 1-50 carbon atoms, X is a halogen, alkoxy, acyloxy or amine and n has a value of 1-3.

The composition of claim 25, wherein the chemical compound is an 27. organo-polysiloxane having the formula

wherein each of R₁ to R₆ and R₈ to R₁₀, represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R₇ represents an organic betaine group for an organic betaine polysiloxane or an organic quaternary group for an organic quaternary polysiloxane, and m and n are from 1 to 200.

- 28. The composition of claim 27, wherein the particulates are sands.
- The composition of claim 25, wherein the chemical compound is an organo-siloxane having the formula

where R_{12} to R_{17} each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R_{11} and R_{18} each represents an organic betaine group for a di-betaine polysiloxane or an organic quaternary group for a di-quaternary polysiloxane, and m is 1 to 200.

- 30. The composition of claim 29, wherein the particulates are sands.
- 31. The method of claim 23, including the step of subjecting the slurry composition to shear in the presence of a gas.
- 32. The method of claim 31, wherein the gas is selected from the group consisting of air, nitrogen, carbon dioxide, methane and mixtures thereof.
- 33. The method of claim 23, including the steps of contacting the particulate with a medium containing the chemical compound to render the particulate hydrophobic, separating the particulate from the medium; and blending the hydrophobic particulate with water.
- 34. The method of claim 33, including the step of subjecting the slurry composition to shear in the presence of a gas.
- 35. The method of claim 33, wherein the gas is selected from the group consisting of air, nitrogen, carbon dioxide, methane and mixtures thereof.

36. The composition of claim 33, wherein the chemical compound is an organo-polysiloxane having the formula

wherein each of R₁ to R₆ and R₈ to R₁₀, represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R₇ represents an organic betaine group for an organic betaine polysiloxane or an organic quaternary group for an organic quaternary polysiloxane, and m and n are from 1 to 200.

- 37. The composition of claim 36, wherein the particulates are sands.
- 38. The composition of claim 33, wherein the chemical compound is an organosiloxane having the formula

where R₁₂ to R₁₇ each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R₁₁ and R₁₈ each represents an organic betaine group for a di-betaine polysiloxane or an organic quaternary group for a diquaternary polysiloxane, and m is 1 to 200.

- 39. The composition of claim 38, wherein the particulates are sands.
- 40. A method of producing an aqueous slurry composition for a well service operation in various formations, comprising the step of mixing water, particulates

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and a chemical compound for rendering the surface of the particulates extremely hydrophobic.

- The well service operation in claim 40 is selected from the group 41. consisting of hydraulic fracturing, drilling, completion and sand pack operation.
- 42. The method of claim 40, including the step of subjecting the slurry composition to shear in the presence of a gas.
- 43. The fluid in Claim 40 can be re-used after separated from the particulates.
- 44. The composition of claim 40, wherein the chemical compound is an organo-polysiloxane having the formula

wherein each of R₁ to R₆ and R₈ to R₁₀, represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R₇ represents an organic betaine group for an organic betaine polysiloxane or an organic quaternary group for an organic quaternary polysiloxane, and m and n are from 1 to 200.

45. The composition of claim 40, wherein the chemical compound is an organo-siloxane having the formula

$$R_{11} - S_{i} = \begin{bmatrix} R_{12} & R_{14} \\ 0 & S_{i} \end{bmatrix}_{0} - \begin{bmatrix} R_{16} \\ S_{i} \end{bmatrix}_{m} - \begin{bmatrix} R_{16} \\ S_{i} \end{bmatrix}_{R_{17}}$$

where R₁₂ to R₁₇ each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R₁₁ and R₁₈ each represents an organic betaine WO 2006/116868 PCT/CA2006/000705

group for a di-betaine polysiloxane or an organic quaternary group for a diquaternary polysiloxane, and m is 1 to 200.

- A method of producing an aqueous slurry composition for transporting 46. particulates through a pipeline, comprising the step of mixing water, particulates and a chemical compound for rendering the surface of the particulates extremely hydrophobic, and pumping the slurry through the pipeline.
- 47. The composition of claim 46, wherein the particulates are sands.
- 48. The composition of claim 46, wherein the particulates are coal particulates.

International application No. PCT/CA2006/000705

CLASSIFICATION OF SUBJECT MATTER

PC: C08L 83/04 (2006.01), C09K8/68 (2006.01), E21B 43/267 (2006.01) and B01J2/30 (2006.01) According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC C09K8/00 (version 8.0)

IPC C09K7/00 (versions 7.0 and earlier)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic database(s) consulted during the international search (name of database(s) and, where practicable, search terms used) Canadian Patent Databases, WEST, Espacenet, Delphion/DWPI, SCOPUS, ACS Journals, Google Scholar

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
x	CA2423031 A1 (BJ SERVICES COMPANY) 01 January 2003 (01-01-2003) abstract; page 12, lines 21-29; page 20, line 15 to page 23, line 21; page 29, lines 20-23; examples 3-5	1, 2, 4, 5, 23-26
х	CA1071076 A (DRESSER INDUSTRIES) 01 October 1975 (01-10-1975) examples 3 and 4	1, 2, 4, 5, 23-26
x	CA1104804 A (UNION CARBIDE) 28 December 1978 (28-12-1978) page 4; example 1	1, 2, 4, 5, 23-26
x	US4454056 A (DEGUSSA) 12 June 1984 (12-06-1984) : example 5	1, 2, 4, 5, 23-26
x	JP58146434 (NEOS KK) 01 September 1983 (01-09-1983) English abstract	1, 2, 4, 5, 23-26, 46, 48

[X]	Further documents are listed in the continuation of Box C.	[X]	Seé patent family annex.
* "A" "E" "L"	Special categories of cited documents: document defining the general state of the art which is not considered to be of particular relevance earlier application or patent but published on or after the international filing date document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) document referring to an oral disclosure, use, exhibition or other means	"Y"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"P"	document published prior to the international filing date but later than the priority date claimed	"&"	document member of the same patent family
	of the actual completion of the international search 14 2006 (14-07-2006)		of mailing of the international search report
Name and mailing address of the ISA/CA Canadian Intellectual Property Office Place du Portage I, C114 - 1st Floor, Box PCT 50 Victoria Street		1000	g-Huang Chen (819) 956-4113

Form PCT/ISA/210 (second sheet) (April 2005)

50 Victoria Street

Gatineau, Quebec K1A 0C9 Facsimile No.: 001(819)953-2476

Page 2 of 5

International application No. PCT/CA2006/000705

tegory*	Citation of document, with indication, where appropriate, of the	es Relevant to claim No.		
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Page 3 of 5

Information on patent family members

International application No. PCT/CA2006/000705

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International application No. PCT/CA2006/000705

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Form PCT/ISA/210 (extra sheet) (April 2005)

Page 5 of 5

Electronic Patent Application Fee Transmittal						
Application Number:	12	759469				
Filing Date:	13-	Apr-2010				
Title of Invention:	METHOD FOR MAKING PARTICULATE SLURRIES AND PARTICULATE SLURRY COMPOSITIONS					
First Named Inventor/Applicant Name:	Kewei Zhang					
Filer:	Michael A. Schade/Amy Conrad					
Attorney Docket Number:	28	35.005				
Filed as Large Entity						
Filing Fees for Utility under 35 USC 111(a)						
Description		Fee Code	Quantity	Amount	Sub-Total in USD(\$)	
Basic Filing:						
Pages:						
Claims:						
Miscellaneous-Filing:						
Petition:						
Patent-Appeals-and-Interference:						
Post-Allowance-and-Post-Issuance:						
Extension-of-Time:						

Description	Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Miscellaneous:				
Submission-Information Disclosure Stmt	1806	1	180	180
	Tot	al in USD	(\$)	180

Electronic Acknowledgement Receipt				
EFS ID:	27332064			
Application Number:	12759469			
International Application Number:				
Confirmation Number:	7030			
Title of Invention:	METHOD FOR MAKING PARTICULATE SLURRIES AND PARTICULATE SLURRY COMPOSITIONS			
First Named Inventor/Applicant Name:	Kewei Zhang			
Customer Number:	30589			
Filer:	Michael A. Schade/Amy Conrad			
Filer Authorized By:	Michael A. Schade			
Attorney Docket Number:	2885.005			
Receipt Date:	26-OCT-2016			
Filing Date:	13-APR-2010			
Time Stamp:	16:55:09			
Application Type:	Utility under 35 USC 111(a)			

Payment information:

Submitted with Payment	yes
Payment Type	CARD
Payment was successfully received in RAM	\$180
RAM confirmation Number	102716INTEFSW16572300
Deposit Account	
Authorized User	

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

Preferred Proppants, LLC, Preferred Pipeline, LLC, Preferred Resin Sales, LLC, Ex. 1004, p. 545 of 669 Preferred Resin of Genoa, LLC, Preferred Sands of Wisconsin, LLC

File Listin	g:				
Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.
			56989		
1	Transmittal Letter	2885005_SB21_Transmittal_Re sponse_SuppIDS.pdf	4fc9cc905cc20b956870301ba0bcfcdb9340 81e9	no	1
Warnings:	-				
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2		2885005_Response_to_OA072 616.pdf	8722a4c94d582109ac05240fb10c030d0d3 a8e92	yes	14
	Multip	art Description/PDF files in .	zip description		
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	Amendment/Req. Reconsideration	Amendment/Req. Reconsideration-After Non-Final Reject			
	Claims	Claims			
	Applicant Arguments/Remarks	Made in an Amendment	11	14	
Warnings:			*	3	
Information:			,		
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3	Transmittal Letter	2885005_Supp_IDS_Letter.pdf	813a0975603c9366769496c66cc8906753e 24821	no	2
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4 :	Information Disclosure Statement (IDS) Form (SB08)	2885005_Supp_IDS.pdf	64f62a2badab2e4336d2cc4de9c4695038c 7dce5	no	1
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6	Foreign Reference	C2_GB2387191A.pdf	e3f1563dca8543cfd445f7afc3b8ce88ca8d0 82f	no	92
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9	Fee Worksheet (SB06)	fee-info.pdf	35f10651b46652f84f47eced7a621f2a34a2 dSee	no	2
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New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

Doc Code: TRAN.LET

Document Description: Transmittal Letter

PTO/SB/21 (07-09) Approved for use through 07/31/2012. OMB 0651-0031

Under the Paperwork Reduction Act of 1995.				U.S. DEPARTMENT OF COMMERCE displays a valid OMB control number.	
	Application Number	12/759,469			
TRANSMITTAL	Filing Date	April 13, 201	0	P. T. MARTINESIA MARKAN	
FORM	First Named Inventor	Kewei Zhang	Kewei Zhang		
	Art Unit	1768 C	onf. No.: 7030		
(to be used for all correspondence after initial t	Examiner Name	Bland, Alicia			
	87 Attorney Docket Number	2885.005			
Total Number of Fages III This Submission		Market No. (1988)	1-1864-1-1		
	ENCLOSURES (Check a	that apply)			
Fee Transmittal Form	Drawing(s)] [After A	Allowance Communication to TC	
Fee Attached	Licensing-related Papers] [l Communication to Board eals and Interferences	
	Petition Petition to Convert to a Provisional Application Power of Attorney, Revocatic Change of Correspondence Terminal Disclaimer Request for Refund CD, Number of CD(s) Landscape Table on C Remarks ELECTRONICALLY TRANSMITTED 1. SB21 Transmittal Form (1 pg); 2. Response (14 pgs); 3. Information Disclosure Statement L 4. Information Disclosure Statement 5 5. Cited Foreign Patent Documents (1)	Address L	Propri		
	URE OF APPLICANT, ATTO	RNEY, OR	AGENT		
Firm Name DUNLAP CODDING, P.C.					
Signature					
Printed name Michael A. Schade	- Combine				
Date		Reg. No.			
10/26/2016		6	3,605		
CE	RTIFICATE OF TRANSMISS	ON/MAILI	NG		
I hereby certify that this correspondence is be sufficient postage as first class mail in an envithe date shown below:	ing acsimile transmitted to the USPT	O or deposited Patents, P.O	d with the Uni	ted States Postal Service with slexandria, VA 22313-1450 on	
Signature	_				
Typed or printed name Michael A. Schade			Date	10/26/2016	

This collection of information is required by 37 CFR 1.5. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to 2 hours to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

Index the Panarwork Reduction Act of 1995 no parents are required to recognific as collection of information unless it displays a valid OMS control number

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	FOR		NUM	MBER FIL	.ED	NUMBER EXTRA		RATE (\$)	F	FEE (\$)
	BASIC FEE (37 CFR 1.16(a), (b),	or (c))		N/A		N/A		N/A		
Ш	SEARCH FEE (37 CFR 1.16(k), (i), (or (m))		N/A		N/A		N/A		
	EXAMINATION FE (37 CFR 1.16(o), (p),			N/A		N/A		N/A		
	TAL CLAIMS CFR 1.16(i))			min	us 20 = *			X \$ =		
	EPENDENT CLAIM CFR 1.16(h))	S		mi	nus 3 = *			X \$ =		
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	mandal processors and a second control					V-2002-VP-2000-17880-1		TOTAL ADD'L FE	E	
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This collection of information is required by 37 CFR 1.16. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS

ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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UNITED STATES DEPARTMENT OF COMMERCE United States Patent and Trademark Office Address: COMMISSIONER FOR PATENTS P.O. Box 1450 Alexandria, Virginia 22313-1450 www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
12/759,469	04/13/2010	Kewei Zhang	2885.005	7030
30589 DUNLAP COD	7590 11/25/2016 DDING P.C.		EXAM	INER
PO BOX 16370)		BLAND,	ALICIA
OKLAHOMA	CITY, OK 73113		ART UNIT	PAPER NUMBER
			1768	
			NOTIFICATION DATE	DELIVERY MODE
			11/25/2016	ELECTRONIC

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

docketing@dunlapcodding.com

	Application No. 12/759,469	Applicant(s) ZHANG, KEV	
Office Action Summary	Examiner ALICIA BLAND	Art Unit 1768	AIA (First Inventor to File) Status No
The MAILING DATE of this communication app Period for Reply	ears on the cover sheet with the c	orrespondend	e address
A SHORTENED STATUTORY PERIOD FOR REPLY THIS COMMUNICATION. - Extensions of time may be available under the provisions of 37 CFR 1.13 after SIX (6) MONTHS from the mailing date of this communication. If NO period for reply is specified above, the maximum statutory period w - Failure to reply within the set or extended period for reply will, by statute, Any reply received by the Office later than three months after the mailing earned patent term adjustment. See 37 CFR 1.704(b).	36(a). In no event, however, may a reply be tim vill apply and will expire SIX (6) MONTHS from cause the application to become ABANDONE	nely filed the mailing date of D (35 U.S.C. § 133	this communication.
Status			
1) Responsive to communication(s) filed on 10/26 A declaration(s)/affidavit(s) under 37 CFR 1.1			
	action is non-final.		
3) An election was made by the applicant in response		set forth durin	g the interview on
the restriction requirement and election	아이어나 2000 - 아이저 "~~" - 시에 하는 아이스 " 아이스" 아이스 나이어 (~~) 아이저 아이스		
4) Since this application is in condition for allowar	nce except for formal matters, pro	secution as t	o the merits is
closed in accordance with the practice under E	x parte Quayle, 1935 C.D. 11, 45	3 O.G. 213.	
Disposition of Claims*			
5) Claim(s) <u>1-4,6-11,13,20,21,25-29,36 and 40-54.</u> 5a) Of the above claim(s) <u>See Continuation Short</u> 6) Claim(s) is/are allowed.			
7) Claim(s) 1,3,7,10,40,42,43,45 and 52 is/are rej	ected.		
8) Claim(s) is/are objected to.			
9) Claim(s) are subject to restriction and/or			
* If any claims have been determined allowable, you may be eli			way program at a
participating intellectual property office for the corresponding ap	5년 1일: 60년 1일 : 11일 1일: 12일 1일: 12일 1일: 12일 1일		
http://www.uspto.gov/patents/init_events/pph/index.jsp or send	an inquiry to PPHfeedback@uspto.c	<u>lov</u> .	
Application Papers 10) The specification is objected to by the Examine 11) The drawing(s) filed on is/are: a) acce Applicant may not request that any objection to the of Replacement drawing sheet(s) including the corrections.	epted or b) objected to by the Edrawing(s) be held in abeyance. See	37 CFR 1.85(
Priority under 35 U.S.C. § 119			
12) Acknowledgment is made of a claim for foreign	priority under 35 U.S.C. § 119(a)	-(d) or (f).	
Certified copies:	, b	- 13, - 151	
a) All b) Some** c) None of the:			
1. Certified copies of the priority document2. Certified copies of the priority document		ion No	
2. Certified copies of the priority document3. Copies of the certified copies of the priority			
application from the International Bureau	등을 맞지 않는 것이 없었다면 하는 것이라면 맛있다는 그렇다는 가 있었다. 사용하게 하는 사람은 사용하다 하나 하다 하다.	eu III IIIIS IVai	onal Stage
** See the attached detailed Office action for a list of the certifie			
Attachment(s)			
1) Notice of References Cited (PTO-892)	3) Interview Summary	(PTO-413)	
 Information Disclosure Statement(s) (PTO/SB/08a and/or PTO/S Paper No(s)/Mail Date <u>10/26/16</u>. 	Paper No(s)/Mail Da		

Continuation of Disposition of Claims: Claims withdrawn from consideration are 2,4,6,8,9,11,13,20,21,25-29,36,41,44,46-51,53 and 54.

Art Unit: 1768

1. As previously set forth: The present application is being examined under the pre-

AIA first to invent provisions.

DETAILED ACTION

Priority

2. As previously set forth: The claims have priority of the parent case (but not the provisional because cationic is not disclosed therein). Thus the effective date is 5/2/06.

Terminal Disclaimer

3. As previously set forth: A terminal disclaimer over parent case US 7723274 has been filed/approved in this application.

Information Disclosure Statement

4. The information disclosure statement (IDS) submitted on 10/26/16 has been considered by the examiner.

Election/Restrictions

5. As previously set forth: Applicant's election of (a) a fluid without gas and (b) polysiloxanes modified with a cationic group in the reply filed on 12/3/16 is acknowledged. Because applicant did not distinctly and specifically point out the supposed errors in the restriction requirement, the election has been treated as an election without traverse (MPEP § 818.01(a)).

Art Unit: 1768

Applicant's election by original presentation, as previously set forth, results in claims 47-51 being withdrawn.

Though Applicant elected the species of page 3 of the remarks dated 12/3/15, said species is not incorporated by reference into the instant specification and thus the Examiner is not searching that species (further said species is not in the instant claims).

6. Claims are pending 1-4, 6-11, 13, 20-21, 25-29, 36, 40-54; Claims 2, 4, 6, 8-9, 11, 13, 20-21, 25-29, 36, 41, 44, 46-51, 53-54 are withdrawn; and Claims 1, 3, 7, 10, 40, 42-43, 45, 52 are prosecuted below.

Response to Amendments/Arguments

7. Applicant's amendments/arguments are not sufficient to overcome the previous positions. Applicant argues the present invention is drawn to proppant suspension without the need to viscosify the slurry. Applicant argues viscosifying is essential to Nguyen. Applicant argues the purpose of Nguyen is to prevent the adsorption of the gel-polymer onto the surface of the proppant which is not the purpose of the instant application. Applicant argues the instant application is drawn to water repellent materials to increase the buoyancy thereof.

The Examiner disagrees. The new amendment of the claim is merely a property that the particle/composition must be capable of performing. It is the Examiner's position that the composition of the references must be capable of performing this property since the same coating and particle are used in the references as used by

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Applicant. The new amendment does not exclude the use of viscosifying agents in the composition of the claim (E.G. the claim is drawn to "a composition...comprising" and thus use of a viscosifying agent in the composition is embraced therein). That viscosifying may be essential to the prior art is thusly moot. That Nguyen is coating the proppant for different purposes than Applicant is also moot since the claimed composition is met. Applicant's arguments are thusly not persuasive and the rejection is as set forth below.

Response to Affidavit dated 5/10/16

8. As previously set forth: Applicant's affidavit and additional references are persuasive to show/clarify the instant invention. Therein the absorption of the cationic group to the surface charges on the particles result in a hydrophobic coating on the particle.

Claim Rejections - 35 USC § 112

The following is a quotation of 35 U.S.C. 112(b):

(b) CONCLUSION.—The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the inventor or a joint inventor regards as the invention.

The following is a quotation of 35 U.S.C. 112 (pre-AIA), second paragraph: The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

9. Claims 1 and its dependents are rejected under 35 U.S.C. 112(b) or 35 U.S.C. 112 (pre-AIA), second paragraph, as being indefinite for failing to particularly point out

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and distinctly claim the subject matter which the inventor or a joint inventor, or for pre-AIA the applicant regards as the invention.

Claim 1 recites the property "without viscosifying the slurry composition", it is unclear whether this refers to (a) the use of additional viscosifying agents and/or (b) if the particles themselves would viscosify some compositions but not others. Appropriate clarification and correction to the claims is required.

Claim Rejections - 35 USC § 103

The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

10. Claims 1, 3, 7, 10, 40, 42, 43, 45, 52 is/are rejected under pre-AIA 35 U.S.C. 103(a) as being unpatentable over Nguyen (US 2004/0023818) in view of Herzig (US 2004/0029981), or, Herzig in view of Nguyen.

Elements of this rejection are as previously set forth, reiterated below in italics. New claim 52 is added and is met by elements previously set forth over claim 45. Regarding the new property limitations: Since the same particles and hydrophobizing agent are used, it is the Examiner's position the composition is capable of performing the properties of the claim. There is no exclusion of viscosifying components from the claimed composition, thus that the prior art may include such is moot at this time.

Nguyen discloses methods for enhancing the clean-up of wells comprising pumping particles that are coated with a water-repellent composition into a well

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(abstract). The particles may be sand, ceramic, glass spheres [0001], [0018] having a size of 20/40 mesh (exemplified [0018]). The base fluid is water or brine [0013]. The coating is formed using various siloxanes of [0006]. The coating may be applied by having a composition of particles, water, siloxane and solvent [0008] meeting the basic composition requirements of the claims.

Herzig discloses aqueous compositions comprising an organosilicon compound, in its acid anion (e.g. cationic) form, water, and optional solvents (abstract). The composition may be used for hydrophobicizing surfaces such as silicate/glass/ceramic and natural rock [0204]. The organosilicon compound may have (a) the anionic amine substituent of [0019] which is attached to siloxane unit [0039] wherein a can be 1 and R can be a methyl group, and (b) siloxane units of [0032] wherein a may be 2 or 3 and R may be a methyl group, these overlap the 2 repeat units of instant claim 7 (wherein for the unsubstituted siloxane a=2) and the 2 end groups (wherein for the unsubstituted siloxane unit a=3). Thus, the organosilicon compounds of Herzig embrace the compound of instant claim 7. Herzig thusly teaches this compound to be suitable for the intended use of hydrophobizing silicate/glass/ceramic and rock surfaces.

It would have been obvious to one of ordinary skill in the art at the time of the invention to include in Nguyen the use of the compound taught by Herzig since it is recognized in the art of organosilicon compounds for rendering glass/ceramic/natural rock (which embraces sand) hydrophobic. See Sinclair & Carroll Co. v. Interchemical Corp., 325 U.S. 327, 65 USPQ 297 (1945), wherein the selection of a known material

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based on its suitability for its intended use supported a prima facie obviousness determination.

Alternatively, it would have been obvious to one of ordinary skill in the art at the time of the invention to include in Herzig the use of sand particles of size 20/40 mesh, as taught by Nguyen, since it is recognized in the art for hydrophobicizing surfaces to function equivalently to ceramic/glass surfaces taught in Herzig.

The limitations "aqueous hydraulic fracturing slurry" and "for use in" are the future intended use of the composition. A recitation of the intended use of the claimed invention must result in a structural difference between the claimed invention and the prior art in order to patentably distinguish the claimed invention from the prior art. If the prior art structure is capable of performing the intended use, then it meets the claim.

Elements above meet the requirements of claims 1, 3, 7 (wherein "at least one" of each of the units discussed above is disclosed, thus embracing the ranges of claim 7), 10, 43.

The Examiner notes that the "re-used" water of claims 40, 42, 45 has been purified of debris in the specification, meaning it is just plain water. Thus "re-used" water is merely a product by process limitation, the process of making not being pertinent unless Applicant shows a distinct product is produced. Claims 40, 42, 45 are thusly met by the references.

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Conclusion

Applicant's amendment necessitated the new ground(s) of rejection presented in this Office action. Accordingly, THIS ACTION IS MADE FINAL. See MPEP § 706.07(a). Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

A shortened statutory period for reply to this final action is set to expire THREE MONTHS from the mailing date of this action. In the event a first reply is filed within TWO MONTHS of the mailing date of this final action and the advisory action is not mailed until after the end of the THREE-MONTH shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of the advisory action. In no event, however, will the statutory period for reply expire later than SIX MONTHS from the date of this final action.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to ALICIA BLAND whose telephone number is (571)272-2451. The examiner can normally be reached on M-F 10:00 AM to 4:00 PM.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Susannah Chung can be reached on 571-272-6098. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Art Unit: 1768

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/ALICIA BLAND/ Primary Examiner, Art Unit 1768

Search Notes Application/Control No. 12759469 Examiner ATNAF ADMASU Applicant(s)/Patent Under Reexamination ZHANG, KEWEI Art Unit 1796

CPC- SEARCHED							
Symbol Date Exan							
CPC COMBINATION SETS - S							
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	US CLASSIFICATION SE	ARCHED	
Class	Subclass	Date	Examiner
507	205, 234	5/22/2010	/ASA/

SEARCH NOTES							
Search Notes	Date	Examiner					
Performed inventors search in EAST and PALM	5/22/2010	/ASA/					
Classes 507/, 175/ and 166/ searched limited to key terms	5/22/2010	/ASA/					
Reviewed US 7,723,274	5/22/2010	/ASA/					
EAST search conducted -Summary is attached	5/22/2010	/ASA/					
EAST search updated - Summary is attached	2/4/2011	/ASA/					
EAST search updated	12/21/2014	/ASA/					
inventor, EAST class (507 166 516 175 C09K8) and keyword search	7/20/2016	AB					
inventor, EAST class (507 166 516 175 C09K8) and keyword search: see attached	11/18/2016	AB					

	INTERFERENCE SEARCH	1	
US Class/ CPC Symbol	US Subclass / CPC Group	Date	Examiner

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Page 1 of 1

ELECTRONICALLY FILED ON: 10/26/2015

Doc code: IDS

Doc description: Information Disclosure Statement (IDS) Filed

Substitute for form PTO/SB/08a

SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)

Application Number	12/759,469		
Filing Date	04/13/2010		
First Named Inventor	Kewei Zhang		
Art Unit	1768		
Examiner Name	Bland, Alicia		
Attorney Docket Number	2885.005		

U. S. PATENT DOCUMENTS									
Examiner Initial*	Cite No	Patent Number	Kind Code ¹	Issue Date	Name of Patentee or Applicant of cited Document	Pgs, Col., Lns, where Relevant Psgs or Relevant Figs Appear			
	A1								

	U. S. PATENT APPLICATION PUBLICATIONS									
Examin er Initial*	Cite No	Publication Number	Kind Code ¹	Publication Date	Name of Patentee or Applicant of cited Document	Pgs, Col., Lns, where Relevant Psgs or Relevant Figs Appear				
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Exa m. Initi al*	Cite No	Foreign Document Number ³	Count ry code ²	Kind Cod e ⁴	Publication Date	Name of Patentee or Applicant of cited Document	Pgs, Col., Lns, where Relevant Pssgs or Relevant Figs Appear	T5
	C1	2002/22759	WO		03/21/2002	Thuslick, Inc.		
	C2	2,387,191	GB		10/08/2003	BJ Services Company		ia .
	C3	2005/100007	WO		10/27/2005	Carbo Ceramics, Inc.		
	C4	2006/116868	WO		11/09/2006	Trican Well Services Ltd.		

NON-PATENT LITERATURE DOCUMENTS							
Examiner Initial*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T ⁵				
	D1						

EXAMINER SIGNATURE							
Examiner Signature	/ALICIA	BLAND/	Date Considered	11/15/2016			

^{*}EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

¹ See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. ² Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). ³ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁴ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. ⁵ Applicant is to place a check mark here if English language translation is attached.

EAST Search History

EAST Search History (Prior Art)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	179115	(("507") or ("166") or ("516") or ("175")).CLAS.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	OFF	2016/11/18 13:41
L2	28007	c09k8/\$.cpc.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2016/11/18 13:41
L3	184697	L1 L2	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2016/11/18 13:41
L4	2255964	(modify modifying modified coat coated coating) with (surface particle particulate proppant)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2016/11/18 13:41
L5	6701	(polysiloxane polydiorganosiloxane silane siloxane silicone polyorganosiloxane organopolysiloxane dioragnopolysiloxane) near2 (cationic)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2016/11/18 13:41
L6	53	3 and 4 and 5	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2016/11/18 13:42

11/18/2016 1:42:35 PM

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Harbellandbadhallabbl



Courtesy Reminder for Application Serial No: 12/759,469

Attorney Docket No: 2885.005 Customer Number: 30589

Date of Electronic Notification: 11/25/2016

This is a courtesy reminder that new correspondence is available for this application. If you have not done so already, please review the correspondence. The official date of notification of the outgoing correspondence will be indicated on the form PTOL-90 accompanying the correspondence.

An email notification regarding the correspondence was sent to the following email address(es) associated with your customer number: docketing@dunlapcodding.com

To view your correspondence online or update your email addresses, please visit us anytime at https://sportal.uspto.gov/secure/myportal/privatepair. If you have any questions, please email the Electronic Business Center (EBC) at EBC@uspto.gov or call 1-866-217-9197.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.:

12/759,469

Confirmation No.:

7030

Inventor:

Kewei Zhang

Filed:

April 13, 2010

Art Unit:

1768

Examiner:

Bland, Alicia

Title:

METHOD FOR MAKING PARTICULATE SLURRIES AND

PARTICULATE SLURRY COMPOSITIONS

Docket No.:

2885.005

Customer No.:

30589

AMENDMENT AND RESPONSE

In response to the final Office Action mailed November 25, 2016, reconsideration is respectfully requested in view of the amendments and/or remarks as indicated below:

Amendments to the Claims are reflected in the listing of the claims which begins on page 2 of this paper.

Remarks begin on page 11 of this paper.

Amendments to the Claims

This listing of claims will replace all prior versions and listings of claims in the application.

Listing of Claims:

- 1. (Currently Amended) An aqueous hydraulic fracturing slurry composition for use in hydraulic fracturing operations comprising:
 - (a) water:
 - inorganic particulates ranging in size from about 10 to about 100 US (b) mesh: and
 - a chemical compound for rendering the surface of the particulates extremely hydrophobic,
 - wherein the chemical compound is selected from the group consisting of polysiloxanes modified with one or more organic cationic groups, polysiloxanes modified with one or more organic amphoteric groups, quaternary silanes, and combinations thereof,
 - wherein the hydraulic fracturing slurry composition can suspend the particulates under static and dynamic conditions without vicosifying the slurry composition and further wherein the aqueous hydraulic fracturing slurry composition is substantially free of a viscosifier such that the aqueous hydraulic fracturing slurry composition suspends the particulates under static and dynamic conditions without viscosifying the aqueous hydraulic fracturing slurry composition.

- 2. (Withdrawn) The composition of claim 1, wherein the chemical compound is a polysiloxane modified with one or more organic amphoteric groups.
- 3. (Previously Presented) The composition of claim 1, wherein the chemical compound is a polysiloxane modified with one or more organic cationic groups.
- 4. (Withdrawn) The composition of claim 1, wherein the chemical compound is a quaternary silane.
 - 5. (Cancelled)
- 6. (Withdrawn) The composition of claim 1, wherein the chemical compound is a polysiloxane having the formula

wherein each of R₁ to R₆ and R₈ to R₁₀ represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R₇ represents an organic amphoteric group and m and n are from 1 to 200.

7. (Previously Presented) The composition of claim 1, wherein the chemical compound is a polysiloxane having the formula

$$R_{1} \longrightarrow Si \longrightarrow O \longrightarrow Si \longrightarrow O \longrightarrow Si \longrightarrow O \longrightarrow Si \longrightarrow R_{10}$$

$$R_{3} \longrightarrow R_{5} \longrightarrow R_{5} \longrightarrow R_{7} \longrightarrow R_{9}$$

wherein each of R₁ to R₆ and R₈ to R₁₀ represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R7 represents an organic cationic group and m and n are from 1 to 200.

8. (Withdrawn) The composition of claim 1, wherein the chemical compound is a polysiloxane having the formula

where R₁₂ to R₁₇ each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, one of R₁₁ and R₁₈ represents an organic amphoteric group and the other of R₁₁ and R₁₈ represents an organic amphoteric group or an organic radical and m is from 1 to 200.

9. (Withdrawn) The composition of claim 1, wherein the chemical compound is a polysiloxane having the formula

where R₁₂ to R₁₇ each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, one of R₁₁ and R₁₈ represents an organic cationic group and the other of R₁₁ and R₁₈ represents an organic cationic group or an organic radical and m is from 1 to 200.

- 10. (Previously Presented) The composition of any one of claims 1 to 4 and 6 to 9, wherein the particulates are sand proppants.
- 11. (Withdrawn) The composition of any one of claims 1 to 4 and 6 to 9, wherein the particulates are ceramic particulates.

12. (Cancelled)

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

14. - 19. (Cancelled)

- 20. (Withdrawn) The composition of claim 13, wherein the chemical compound is a polysiloxane modified with one or more organic amphoteric groups.
- 21. (Withdrawn) The composition of claim 13, wherein the chemical compound is a polysiloxane modified with one or more organic cationic groups.

22-24. (Cancelled)

25. (Withdrawn) The composition of claim 13, wherein the chemical compound is a polysiloxane having the formula

$$\begin{array}{c|c}
R_1 & R_2 & R_4 \\
R_1 & S_1 & S_1 & S_1 \\
R_3 & R_5 & R_7 & R_9
\end{array}$$

wherein each of R₁ to R₆ and R₈ to R₁₀ represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R7 represents an organic amphoteric group and m and n are from 1 to 200.

26. (Withdrawn) The composition of claim 13, wherein the chemical compound is a polysiloxane having the formula

wherein each of R₁ to R₆ and R₈ to R₁₀ represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R7 represents an organic cationic group and m and n are from 1 to 200.

27. (Withdrawn) The composition of claim 13, wherein the chemical compound is a polysiloxane having the formula

where R₁₂ to R₁₇ each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, one of R₁₁ and R₁₈ represents an organic amphoteric group and the other of R₁₁ and R₁₈ represents an organic amphoteric group or an organic radical and m is from 1 to 200.

28. (Withdrawn) The composition of claim 13, wherein the chemical compound is a polysiloxane having the formula

where R₁₂ to R₁₇ each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, one of R₁₁ and R₁₈ represents an organic cationic group and the other of R₁₁ and R₁₈ represents an organic cationic group or an organic radical and m is from 1 to 200.

29. (Withdrawn) The composition of claim 13, wherein the gas is selected from a group consisting of air, nitrogen, carbon dioxide, methane and mixtures thereof.

30-35. (Cancelled)

36. (Withdrawn) The composition of claim 13, wherein the particulates are sand proppants and wherein the gas is nitrogen or carbon dioxide.

37-39. (Cancelled)

- 40. (Previously Presented) The composition of claim 1, wherein the water is reused water after flow back from a well.
- 41. (Withdrawn) The composition of claim 13, wherein the water is re-used water after flow back from a well.
- 42. (Previously Presented) The composition of claim 7, wherein the water is reused water after flow back from a well.
- 43. (Previously Presented) The composition of claim 7, wherein each of R₁ to R₆ and R₈ to R₁₀ is a methyl group.
 - 44. (Withdrawn) The composition of claim 7, further comprising a gas.
- 45. (Previously Presented) The composition of claim 43, wherein the water is reused water after flow back from the well.

- 46. (Withdrawn) The composition of claim 44, wherein the water is re-used water after flow back from the well.
- 47. (Withdrawn) A method of transporting proppants into a subterranean formation, comprising pumping the composition of claim 1 into the subterranean formation.
- 48. (Withdrawn) A method of transporting proppants into a subterranean formation, comprising pumping the composition of claim 7 into the subterranean formation.
- 49. (Withdrawn) A method of transporting proppants into a subterranean formation, comprising pumping the composition of claim 13 into the subterranean formation.
- 50. (Withdrawn) A method of transporting proppants into a subterranean formation, comprising pumping the composition of claim 3 into the subterranean formation.
- 51. (Withdrawn) A method of transporting proppants into a subterranean formation, comprising pumping the composition of claim 44 into the subterranean formation.

- 52. (Previously Presented) The composition of claim 3, wherein the water is reused water after flow back from a well.
- 53. (Withdrawn) The composition of claim 13, wherein the particulates are sand proppants and wherein the gas is nitrogen or carbon dioxide.
- 54. (Withdrawn) The composition of claim 44, wherein the particulates are sand proppants and wherein the gas is nitrogen or carbon dioxide.
- The composition of claim 1, wherein the composition is free of a 55. (New) viscosifier.

REMARKS

This is intended to be a complete response to the final Office Action mailed November 25, 2016 (the "Office Action"), in connection with the above-identified application. Claims 1-4, 6-11, 13, 20-21, 25-29, 36, and 40-55 are pending in the application. Any amendments made herein should not be taken as an admission that the prior art teaches any part or element of the claims as previously presented. Claim 1 has been amended herein to recite that the aqueous hydraulic fracturing slurry composition is substantially free of a viscosifier such that the aqueous hydraulic fracturing slurry composition suspends the particles under static and dynamic conditions without viscosifying the aqueous hydraulic fracturing slurry composition. Additionally, new dependent claim 55 has been added hereto to more fully define certain aspects of the presently claimed inventive concept(s). Support for these amendments can be found, for instance, in paragraphs [0006], [0015], [0020], [0028], [0040], and [0043] of the specification of the present application. Applicant submits that the amendments and newly added claim do not add new matter.

Applicant does not acquiesce to the correctness of the rejections or objections in the Office Action, and reserves the right to present specific arguments regarding any rejected or objected-to claims not specifically addressed. Further, Applicant reserves the right to pursue the full scope of the subject matter of the claims in a subsequent patent application that claims priority to the present application.

At least in view of the reasons set forth below, Applicant respectfully submits that the claims now pending in the application are in a condition for allowance.

RESPONSE TO THE REJECTION OF CLAIM 1 UNDER PRE-AIA 35 U.S.C. § 112 (b)

In the Office Action, the Examiner has rejected Claim 1 under pre-AIA 35 U.S.C. § 112(b), second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which the inventor or a joint inventor, or for pre-AIA the applicant regards as the invention. More specifically, on page 5 of the Office Action, the Examiner states that the recitation of "without viscosifying the slurry composition" is unclear. Applicant has herein amended independent claim 1 to specifically clarify the scope of this limitation in independent claim 1, as amended. More specifically, independent claim 1 has been amended to clarify that particulates are suspended in aqueous hydraulic fracturing slurry composition despite the slurry composition being substantially free of a viscosifier and further without viscosifying the slurry composition via a viscosifier.

In view of the above, Applicant respectfully requests that the Examiner withdraw the 35 U.S.C. § 112(b) rejection of independent claim 1, as amended, and pass the presently amended claims to an expedient allowance.

RESPONSE TO THE REJECTION OF CLAIMS 1, 3, 7, 10, 40, 42-43, 45, and 52 UNDER PRE-AIA 35 U.S.C. § 103(a)

In the Office Action, the Examiner has rejected Claims 1, 3, 7, 10, 40, 42-43, 45, and 52 under pre-AIA 35 USC § 103(a) as being unpatentable over Nguyen (U.S. Publication No. 2004/0023818) in view of Herzig (U.S. Publication No. 2004/0029981), or, Herzig in view of Nguyen. Applicant respectfully traverses the rejection in view of the amendments made to the claims and the remarks which follow.

Applicant appreciates the Examiner's thorough examination of the application and respectfully submits that the amendments to claim 1 overcome the rejection under 35 U.S.C. § 103 over Nguyen in view of Herzig and Herzig in view of Nguyen. In particular, independent claim 1 has been amended to recite that the aqueous hydraulic fracturing slurry composition is substantially free of a viscosifier. That is, the slurry composition only includes, if it includes at all, trace or minimal amounts of a viscosifier. The trace or minimal amounts of the viscosifer do not adversely affect the functioning of the slurry, and their inclusion (or lack of inclusion) are inconsequential to the viscosity of the slurry composition. Accordingly, the presently claimed inventive concept(s), as recited in Applicant's independent claim 1, as amended, do not require the addition of a viscosifier to suspend the proppants within the aqueous hydraulic fracturing slurry composition. As previously stated, support for such amendment can be found, for instance, in paragraphs [0006], [0015], [0020], [0028], [0040], and [0043] of the specification of the present application.

The Examiner states that Nguyen discloses each and every element of the claimed subject matter with the exception of the organosilicon compound, which the Examiner asserts is taught by Herzig. Additionally, the Examiner states that Herzig discloses each and every element of the claimed subject matter with the exception of sand particles having a 20/40 mesh, which the Examiner asserts is taught by Nguyen. For at least the reasons stated hereinbelow, Applicant respectfully asserts that the combination of Nguyen and Herzig (or, alternatively, the combination of Herzig and Nguyen) fails to teach each and every element of the claimed subject matter, as currently amended.

The objective of the invention described in Nguyen is to prevent the adsorption of polymer from the gel-polymer on the surface of the proppant. See, e.g., Nguyen at paragraph [0003]. This adsorption is to be prevented because the gel-polymer forms a coating on the surface of the proppant which is difficult to remove and which compromises the utility and efficacy of the proppant. However, Nguyen expressly teaches that a critical component of the disclosed fracturing fluid is the inclusion of "a gelling agent for gelling the water...[that] increase[es] its viscosity, and optionally, a crosslinking agent for crosslinking the gel and further increasing the viscosity of the fluid." See, Nguyen at paragraphs [0012] and [0014]-[0015]. Moreover, as stated in paragraph [0012] of Nguyen, the use of these viscosifiers increases the "viscosity of the gelled or gelled and crosslinked fracturing fluid" such that fluid loss is mitigated and "the fracturing fluid [is allowed] to transport significant quantities of suspended fibrous bundles and proppant into the created fractures." Accordingly, the fracturing fluid disclosed in Nguyen requires the use of a viscosifier(s) in amounts that viscosify the fracturing fluid to suspend the proppant(s) present in the fracturing fluid.

As recited in Applicant's independent claim 1, as amended, the aqueous hydraulic fracturing slurry composition is "substantially free of a viscosifier such that the aqueous hydraulic fracturing slurry composition suspends the particulates under static and dynamic conditions without viscosifying the aqueous hydraulic fracturing slurry composition." Neither Nguyen nor Herzig teach or suggest that the fracturing fluid is substantially free of a viscosifier, nor that the particulates within the fracturing fluid are suspended and able to be effectively transported to a fracture in the absence of a viscosifier. As previously stated, a viscosifier(s) is critical to the functioning of the fracturing fluid disclose in Nguyen as the viscosifier(s) substantially viscosify the

fracturing fluid to allow for suspension of the proppants within the fracturing slurry.

Accordingly, Applicant respectfully submits that independent claim 1, as amended, and

all claims depending therefrom are inventive and non-obvious over a combination of

either Nguyen and Herzig or Herzig and Nguyen.

CONCLUSION

Applicant submits that this is intended to be a complete response to the Final

Office Action mailed November 25, 2016. It is respectfully submitted that this application

is in a condition for allowance. In the event the Examiner's next action is not a Notice of

Allowance, the Applicant's undersigned attorney respectfully requests that the Examiner

initiate telephonic communication in an effort to resolve any remaining issues and move

the prosecution of these claims forward in an expedient manner.

Respectfully submitted,

Michael A. Schade

Registration Number 63,605

DUNLAP CODDING, P.C.

P.O. Box 16370

Oklahoma City, Oklahoma 73113

Telephone: (405) 607-8600

Facsimile: (405) 607-8686

Email: mschade@dunlapcodding.com

Attorney for Applicant

Electronic Acknowledgement Receipt			
EFS ID:	28140492		
Application Number:	12759469		
International Application Number:			
Confirmation Number:	7030		
Title of Invention:	METHOD FOR MAKING PARTICULATE SLURRIES AND PARTICULATE SLURRY COMPOSITIONS		
First Named Inventor/Applicant Name:	Kewei Zhang		
Customer Number:	30589		
Filer:	Michael A. Schade/Candice Conrad		
Filer Authorized By:	Michael A. Schade		
Attorney Docket Number:	2885.005		
Receipt Date:	23-JAN-2017		
Filing Date:	13-APR-2010		
Time Stamp:	17:16:11		
Application Type:	Utility under 35 USC 111(a)		

Payment information:

Submitted with Payment	no
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File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
			58271		
1	Transmittal Letter	2885005_Sb21Transmittal_012 32017.pdf	27b5b4f05376f738aa61bdb2fc045dd5495 8d57a	no	1

Properties Proppants, LLC, Preferred Pipeline, LLC, Preferred Resin Sales, LLC, Preferred Resin of Genoa, LLC, Preferred Sands of Wisconsin, LLC Ex. 1004, p. 581 of 669

Information:				
		440363		
2	2885005_AmendmentandResp onse_01232017.pdf	a55c11b60ab269a6bc79f854344a465ddc5 66e2a	yes 15	
	Multipart Description/PDF files in .	zip description		
	Document Description	Start	End	
	Amendment/Req. Reconsideration-After Non-Final Reject	1	1	
	Claims		10	
	Applicant Arguments/Remarks Made in an Amendment		15	
Warnings:				
Information:				
	Total Files Size (in bytes)	498	634	

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

Doc Code: TRAN.LET

Document Description: Transmittal Letter

	•		11.5	Datent and	Approved for t	PTO/SB/21 (07-09) ise through 07/31/2012. OMB 0651-0031 e; U.S. DEPARTMENT OF COMMERCE
Under the Pa	perwork Reduction Act of 199	5. no person	s are required to respond to a			s it displays a valid OMB control number.
(Application Number	12/759,46	69	(1)
TR	RANSMITTAL		Filing Date	April 13, 2	2010	
	FORM		First Named Inventor	Kewei Zh	ang	
			Art Unit	1768 (Co	nf. No.: 7030)	
(to be used for	r all correspondence after initia	l filing)	Examiner Name	Bland, Ali	cia	
	of Pages in This Submission	16	Attorney Docket Number	2885.005		
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		T	LUSURES (Check a	all that appl		er Allowance Communication to TC
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☐ F	ee Attached		Licensing-related Papers			peal Communication to Board Appeals and Interferences
Amendm	ent/Reply	ΙП ,	Petition			peal Communication to TC peal Notice, Brief, Reply Brief)
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	fter Final		Provisional Application Power of Attorney, Revocat	tion		prietary Information
_ L A	ffidavits/declaration(s)		Change of Correspondence			tus Letter
Extension	n of Time Request		Terminal Disclaimer			er Enclosure(s) (please Identify ow):
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			CD, Number of CD(s)			
Information	on Disclosure Statement	- '				
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Reply to	Missing Parts/	1. SB21	Transmittal Form (1 pg); a	ind		
	te Application Reply to Missing Parts	2. Resp	onse to the Final Office Act	tion dated 1	1/25/2016 (15	pgs).
	nder 37 CFR 1.52 or 1.53					
	SIGNA	TURE C	F APPLICANT, ATT	ORNEY,	OR AGENT	
Firm Name	DUNLAP CODEING, P.O).				
Signature	Min	191100				
Printed name	Michael A. Schade		1			
Date	01/23/2017			Reg. No.	63,605	
		7 17 7 15				
	c	ERTIFIC	CATE OF TRANSMIS	SION/MA	ILING	109
I hereby certify the sufficient postage the date shown be	I hereby certify that this correspondence is being facsimile transmitted to the USPTO or deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on the date shown below:					
Signature	Malet	/		ST		
					Pass	
Typed or printed	name Michael A. Scha	de			Da	e 01/23/2017

This collection of information is required by 37 CFR 1.5. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to 2 hours to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

UNITED STATES DEPARTMENT OF COMMERCE United States Patent and Trademark Office Address: COMMISSIONER FOR PATENTS P.O. Box 1450 Alexandria, Virginia 22313-1450 www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.	
12/759,469	04/13/2010	Kewei Zhang	2885.005	7030	
30589 DUNLAP COD	7590 02/16/2017 DDING P.C.		EXAM	INER	
PO BOX 16370 OKLAHOMA CITY, OK 73113			BLAND, ALICIA		
			ART UNIT	PAPER NUMBER	
			1768		
			NOTIFICATION DATE	DELIVERY MODE	
			02/16/2017	ELECTRONIC	

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

docketing@dunlapcodding.com

Advisory Action Before the Filing of an Appeal Brief

Application No. 12/759,469	Applicant(s) ZHANG, KEWEI		
Examiner ALICIA BLAND	Art Unit 1768	AIA (First Inventor to File) Status	

	ALICIA BLAND	1/68	No		
The MAILING DATE of this communication a	ppears on the cover sheet with	the correspon	dence address		
THE REPLY FILED <u>23 January 2017</u> FAILS TO PLACE THIS APPLICATION IN CONDITION FOR ALLOWANCE. <u>NO NOTICE OF APPEAL FILED</u>					
 The reply was filed after a final rejection. No Notice of Appeal of the following replies: (1) an amendment, affidavit, or other e 					
(2) a Notice of Appeal (with appeal fee) in compliance with 37 CFR 1.114 if this is a utility or plant application. Note that RCI following time periods:	s are not permitted in design application				
 a) The period for reply expiresmonths from the m 	H IN NOTE (
b) The period for reply expires on: (1) the mailing date of this					
no event, however, will the statutory period for reply expir			U.74		
c) A prior Advisory Action was mailed more than 3 months a within 2 months of the mailing date of the final rejection. the prior Advisory Action or SIX MONTHS from the mailin Examiner Note: If box 1 is checked, check either be FIRST RESPONSE TO APPLICANT'S FIRST AFT REJECTION. ONLY CHECK BOX (c) IN THE LIM	The current period for reply expires g date of the final rejection, whicheven (a), (b) or (c). ONLY CHECK BOXER-FINAL REPLY WHICH WAS FIL TED SITUATION SET FORTH UND	months from the first earlier. K (b) WHEN THIS ED WITHIN TWO SER BOX (c). Se	om the mailing date of S ADVISORY ACTION IS THE D MONTHS OF THE FINAL DEMORPS 100.07(f).		
Extensions of time may be obtained under 37 CFR 1.136(a). The					
fee have been filed is the date for purposes of determining the pertension fee under 37 CFR 1.17(a) is calculated from: (1) the experimental conditions of (2) as set forth in (b) or (c) above, if checked. A final rejection, even if timely filed, may reduce any earned patent NOTICE OF APPEAL	piration date of the shortened state by reply received by the Office late	utory period for or than three mo	reply originally set in the final		
 The Notice of Appeal was filed on A brief in complement of Appeal (37 CFR 41.37(a)), or any extension there has been filed, any reply must be filed within the time period AMENDMENTS 	eof (37 CFR 41.37(e)), to avoid dis				
3. The proposed amendments filed after a final rejection, but	prior to the date of filing a brief, w	ill <u>not</u> be entere	d because		
a) They raise new issues that would require further co					
b) They raise the issue of new matter (see NOTE below)					
 c) They are not deemed to place the application in bet appeal; and/or 	r de la responsación de la companya de la companya La companya de la co		ifying the issues for		
 d) They present additional claims without canceling a continuation Sheet. (See 37 CFR 1.11 		ected claims.			
4. The amendments are not in compliance with 37 CFR 1.12	1. See attached Notice of Non-Co	mpliant Amendr	nent (PTOL-324).		
5. Applicant's reply has overcome the following rejection(s):					
 Newly proposed or amended claim(s) would be allowable claim(s). 					
 For purposes of appeal, the proposed amendment(s): (a) new or amended claims would be rejected is provided beloafFIDAVIT OR OTHER EVIDENCE 		vill be entered, a	and an explanation of how the		
8. A declaration(s)/affidavit(s) under 37 CFR 1.130(b) was/we	ore filed on				
 The affidavit or other evidence filed after final action, but be applicant failed to provide a showing of good and sufficient presented. See 37 CFR 1.116(e). 	fore or on the date of filing a Notice				
10. The affidavit or other evidence filed after the date of filing because the affidavit or other evidence failed to overcome sufficient reasons why it is necessary and was not earlier process.	all rejections under appeal and/or	appellant fails to			
11. The affidavit or other evidence is entered. An explanation REQUEST FOR RECONSIDERATION/OTHER	그리고 있었다. 그리고 이 이번 사람들은 아이는 아이들에 들었다면 하는 아이들은 그릇을 하는데 하는데 어디를 가게 되었다.	The same of the sa	ttached.		
12. ☑ The request for reconsideration has been considered but does NOT place the application in condition for allowance because: See Continuation Sheet.					
13. Note the attached Information Disclosure Statement(s). (F	TO/SB/08) Paper No(s)				
14. Other: STATUS OF CLAIMS					
15. The status of the claim(s) is (or will be) as follows:					
Claim(s) allowed:					
Claim(s) objected to: Claim(s) rejected: 1,3,7,10,40,42,43,45,52.					
Claim(s) withdrawn from consideration:					
	/ALICIA DI AND/				
	/ALICIA BLAND/ Primary Examiner, Art L	Jnit 1768			

Continuation of 3. NOTE: the proposed amendments require new search and consideration. Further "substantially free" does not seem to have support in the specification and thus the issues of new matter and 112 definiteness (e.g. it is not defined in the specification) are raised.

Continuation of 12. does NOT place the application in condition for allowance because: Applicant's arguments are drawn to new limitations that have not been entered. The new matter and 112 issues with the proposed limitations are again noted herein. Arguments drawn to the new amendments are thusly moot at this time.

DO NOT ENTER: /A.B/

02/10/2017

ELECTRONICALLY TRANSMITTED: 01/23/2017

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.:

12/759,469

Confirmation No.:

7030

Inventor:

Kewei Zhang

Filed:

April 13, 2010

Art Unit:

1768

Examiner:

Bland, Alicia

Title:

METHOD FOR MAKING PARTICULATE SLURRIES AND

PARTICULATE SLURRY COMPOSITIONS

Docket No.:

2885.005

Customer No.:

30589

AMENDMENT AND RESPONSE

In response to the final Office Action mailed November 25, 2016, reconsideration is respectfully requested in view of the amendments and/or remarks as indicated below:

Amendments to the Claims are reflected in the listing of the claims which begins on page 2 of this paper.

Remarks begin on page 11 of this paper.

UNITED STATES DEPARTMENT OF COMMERCE United States Patent and Trademark Office Address: COMMISSIONER FOR PATENTS P.O. Box 1450 Alexandria, Virginia 22313-1450 www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.	
12/759,469	04/13/2010	Kewei Zhang	2885.005	7030	
30589 DUNLAP COD	7590 03/20/2017 DDING P.C.		EXAM	INER	
PO BOX 16370 OKLAHOMA CITY, OK 73113			BLAND, ALICIA		
			ART UNIT	PAPER NUMBER	
			1768		
			NOTIFICATION DATE	DELIVERY MODE	
			03/20/2017	ELECTRONIC	

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

docketing@dunlapcodding.com

Applicant-Initiated Interview Summary	Application No.	Applicant	(s)	
	12/759,469	ZHANG, KEWEI		
	Examiner	Art Unit	AIA (First Inventor to File) Status	Page
	ALICIA BLAND	1768	No	1 of 2

All participants (applicant, applicant's representative, PTO personnel):

- 1. ALICIA BLAND (Primary Examiner); Telephonic 2. Michael Schade (Attorney of Record); Telephonic
- Susan Rancourt (Attorney); Telephonic
 Shawn Goodwin (Attorney); Telephonic

Date of Interview: 14 March 2017

Claim(s) discussed: 1

Identification of prior art discussed: Nguyen

Amendment Proposed: Applicant proposed amendments such as "consisting essentially of" language and also "without a

viscosifier"

Issues Discussed:

Proposed Amendments:

The proposed amendment such as a negative limitation to exclude a viscosifier seemed to overcome Nguyen. Applicant was concerned as to whether small amounts (e.g. non-viscosifying amounts) could then be used to sidestep the patent, but the Examiner put forth that Nguyen would then meet the composition. Applicant was also concerned over what is excluded by the term "viscosifier" since anything added to water can affect the viscosity thereof, and the Examiner put forth that it would exclude those elements of [0006], e.g. natural and synthetic polymer viscosifiers and viscoelastic surfactants. The Examiner put forth that new search would need to be conducted if the amendment successfully overcame Nguyen.

/ALICIA BLAND/	
Primary Examiner, Art Unit 1768	

Applicant recordation instructions: The formal written reply to the last Office action must include the substance of the interview. (See MPEP section 713.04). If a reply to the last Office action has already been filed, applicant is given a non-extendable time limit of the longer of one month or thirty days from this interview date, or the mailing date of this interview summary form, whichever is later, to file a statement of the substance of the interview.

Examiner recordation instructions: Examiners must summarize the substance of any interview of record. A complete and proper recordation of the substance of an interview should include the items listed in MPEP 713.04 for complete and proper recordation including the identification of the general thrust of each argument or issue discussed, a general indication of any other pertinent matters discussed regarding patentability and the general results or outcome of the interview, to include an indication as to whether or not agreement was reached on the issues raised.

Applicant is reminded that a complete written statement as to the substance of the interview must be made of record in the application file. It is the applicant's responsibility to provide the written statement, unless the interview was initiated by the Examiner and the Examiner has indicated that a written summary will be provided. See MPEP 713.04

Please further see:

MPEP 713.04

Title 37 Code of Federal Regulations (CFR) § 1.133 Interviews, paragraph (b) 37 CFR § 1.2 Business to be transacted in writing

Page 2 of 2 Application No. 12/759,469

PTOL-413/413b (Rev. 01/01/2015)

Interview Summary

Paper No. 20170314

U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Request	Application Number	12/759,469
for	Filing Date	April 13, 2010
Continued Examination (RCE) Transmittal Address to: Mail Stop RCE Commissioner for Patents P.O. Box 1450	First Named Inventor	Kewei Zhang
	Art Unit	1768
	Examiner Name	Alicia Bland
Alexandria, VA 22313-1450	Attorney Docket Number	2885.005

This is a Request for Continued Examination (RCE) under 37 CFR 1.114 of the above-identified application.

Request for Continued Examination (RCE) practice under 37 CFR 1.114 does not apply to any utility or plant application filed prior to June 8, 1995, or to any design application. See Instruction Sheet for RCEs (not to be submitted to the USPTO) on page 2.

 Submission required under 37 CFR 1.114 Note: If the RCE is proper, an amendments enclosed with the RCE will be entered in the order in which they were applicant does not wish to have any previously filed unentered amendment(s) entered amendment(s). 	filed unless applicant instructs otherwise. If							
a. Previously submitted. If a final Office action is outstanding, any amendment considered as a submission even if this box is not checked.	Previously submitted. If a final Office action is outstanding, any amendments filed after the final Office action may be considered as a submission even if this box is not checked.							
i. Consider the arguments in the Appeal Brief or Reply Brief previous	Consider the arguments in the Appeal Brief or Reply Brief previously filed on							
li. Other	Other							
b. 🗸 Enclosed								
I. ✓ Amendment/Reply iii. Info	ormation Disclosure Statement (IDS)							
ii. Affidavit(s)/ Declaration(s) iv. Oth	ner							
2. Miscellaneous								
Suspension of action on the above-identified application is requested un	der 37 CFR 1.103(c) for a							
a period of months. (Period of suspension shall not exceed 3 months)	; Fee under 37 CFR 1.17(i) required)							
b. Other								
3. Fees The RCE fee under 37 CFR 1.17(e) is required by 37 CFR 1.114 when the	the RCE is filed.							
The Director is hereby authorized to charge the following fees, any unde								
a. Deposit Account No								
i. RCE fee required under 37 CFR 1.17(e)								
ii. Extension of time fee (37 CFR 1.136 and 1.17)								
iii. Other								
b. Check in the amount of \$enc	closed							
c. Payment by credit card (Form PTO-2038 enclosed) Electronically Trans	smitted							
WARNING: Information on this form may become public. Credit card information she card information and authorization on PTO-2038.	ould not be included on this form. Provide credit							
SIGNATURE OF APPLICANT, ATTORNEY, OR AGE	ENT REQUIRED							
Signature Wolfe In	Date March 27, 2017							
Name (Print/Type) Michael A. Schade	Registration No. 63,605							
CERTIFICATE OF MAILING OR TRANSMIS	SSION							
I hereby certify that this correspondence is being deposited with the United States Postal Service with s addressed to: Mail Stop RCE, Commissioner for Patents, P. O. Box 1450, Alexandria, VA 22313-1450 of Confice on the date shown below. ELECTRONICALLY TRANSMITTED								
Signature Mathematical Signature	La. March 27 2047							
Name (Print/Type) Michael A. Schade	Date March 27, 2017							

This collection of information is required by 37 CFR 1.114. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SE ND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Mail Stop RCE, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

Approved for use through 3/31/2013. OMB 0651-0031 U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Onder the Paperwork Reduction Act of 1995,	The persons are requ	ance to respond to a concer		t Number (Optional)	
PETITION FOR EXTENSION	1.136(a) ₂₈₈	5.005			
Application Number 12/759,469 Filed April 13, 201			13, 2010		
For Method for Making Particulate Slurries and Particula				lurry Compositions	
Art Unit 1768 Examiner Alicia Bland					
This is a request under the provisions of 37 (CFR 1 136(a) to ex			dentified application	
The requested extension and fee are as follo				om semble bedate kom king som en et a et kom en alle kompeten kom en et kom i	
The requested extension and the are as follows:	Fee Fee	Small Entity Fee		ow).	
✓ One month (37 CFR 1.17(a)(1))	\$200	\$100	Micro Entity Fee \$50	s 200.00	
	240562.7070			7	
Two months (37 CFR 1.17(a)(2))	\$600	\$300	\$150	\$	
Three months (37 CFR 1.17(a)(3))	\$1,400	\$700	\$350	\$	
Four months (37 CFR 1.17(a)(4))	\$2,200	\$1,100	\$550	\$	
Five months (37 CFR 1.17(a)(5))	\$3,000	\$1,500	\$750	\$	
Applicant asserts small entity status	s. See 37 CFR 1.2	7.			
Applicant certifies micro entity statu Form PTO/SB/15A or B or equivalent mu	s. See 37 CFR 1.2	29. d or have been submitted p	reviously.		
A check in the amount of the fee is	enclosed.				
Payment by credit card. Form PTO-	2038 is attached.				
The Director has already been auth	orized to charge fe	ees in this application to	a Deposit Account.		
The Director is hereby authorized to	charge any fees	which may be required,	or credit any overpayr	nent, to	
Deposit Account Number		*			
Payment made via EFS-Web.					
WARNING: Information on this form may credit card information and authorization		Credit card information	n should not be inclu	ded on this form. Provide	
I am the					
applicant.					
attorney or agent of recor	d. Registration nu	mber 63,605			
					
attorney or agent acting u	inder 37 CFR 1.34			·	
My Sch Signature		March 2			
Michael A. Schade		405-607	Date 2-8600	.	
Typed or printed name	e		Telephone	Number	
NOTE: This form must be signed in accorda multiple forms if more than one signature is r	nce with 37 CFR				
moneyo formo il more than one signature is i	oquilou, acc belov	Troit.		9 F	

This collection of information is required by 37 CFR 1.136(a). The information is required to obtain or retain a benefit by the public, which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 6 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Mail Stop PCT, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

forms are submitted.

* Total of

Electronically Transmitted On: March 27, 2017

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.

: 12/759,469

Confirmation No.: 7030

Inventor

: Kewei Zhang

Filed

: April 13, 2010

TC/Au

: 1768

Examiner

: Bland, Alicia

Title

: METHOD FOR MAKING PARTICULATE SLURRIES

AND PARTICULATE SLURRY COMPOSITIONS

Docket No.

: 2885.005

Customer No.

: 30589

STATEMENT OF INTERVIEW SUMMARY

Sir:

In response to the Interview Summary form mailed on March 20, 2017, the Applicant's attorney submits the following:

The interview occurred on March 14, 2017.

The interview was a telephonic interview.

The participants at the interview were Examiner Alicia Bland and the Applicant's attorney's Susan Rancourt, Sean Goodwin and Michael Schade.

No exhibits were shown or discussed.

Issues discussed: Proposed Amendments such as "consisting essentially of" language and also "without a viscosifier."

Claims discussed: Claim 1

Identification of prior art discussed: Nguyen

The Applicant's attorneys and Examiner Bland disagreed regarding the pending claims and the characterization of the prior art. No agreement was reached.

The foregoing is intended to be a complete summary of the interview which occurred on March 14, 2017.

Should the Examiner have any comments or questions regarding the foregoing, Applicants' undersigned attorney would welcome a discussion regarding the same with the Examiner.

Respectfully submitted,

Michael A. Schade

Red. No. 63,605

DUNLAP CODDING, P.C.

P.O. Box 16370

Oklahoma City, Oklahoma 73113

Telephone: (405) 607-8600 Facsimile: (405) 607-8686

Attorney of Record for Applicant

Electronic Patent Application Fee Transmittal						
Application Number:	12	759469				
Filing Date:	13-	13-Apr-2010				
Title of Invention:	METHOD FOR MAKING PARTICULATE SLURRIES AND PARTICULATE SLURRY COMPOSITIONS					
First Named Inventor/Applicant Name:	Kewei Zhang					
Filer:	Mie	chael A. Schade/Car	ndice Conrad			
Attorney Docket Number:	28	35.005				
Filed as Large Entity						
Filing Fees for Utility under 35 USC 111(a)						
Description		Fee Code	Quantity	Amount	Sub-Total in USD(\$)	
Basic Filing:						
Pages:						
Claims:						
Miscellaneous-Filing:						
Petition:						
Patent-Appeals-and-Interference:						
Post-Allowance-and-Post-Issuance:						
Extension-of-Time:						

Description	Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Extension - 1 month with \$0 paid	1251	1	200	200
Miscellaneous:				
RCE- 2ND AND SUBSEQUENT REQUEST	1820	1	1700	1700
	Tot	al in USD	(\$)	1900

Electronic Acknowledgement Receipt				
EFS ID:	28751866			
Application Number:	12759469			
International Application Number:				
Confirmation Number:	7030			
Title of Invention:	METHOD FOR MAKING PARTICULATE SLURRIES AND PARTICULATE SLURRY COMPOSITIONS			
First Named Inventor/Applicant Name:	Kewei Zhang			
Customer Number:	30589			
Filer:	Michael A. Schade/Candice Conrad			
Filer Authorized By:	Michael A. Schade			
Attorney Docket Number:	2885.005			
Receipt Date:	27-MAR-2017			
Filing Date:	13-APR-2010			
Time Stamp:	19:21:24			
Application Type:	Utility under 35 USC 111(a)			

Payment information:

Submitted with Payment	yes
Payment Type	CARD
Payment was successfully received in RAM	\$1900
RAM confirmation Number	032817INTEFSW19232300
Deposit Account	
Authorized User	

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

Preferred Proppants, LLC, Preferred Pipeline, LLC, Preferred Resin Sales, LLC, Ex. 1004, p. 597 of 669 Preferred Resin of Genoa, LLC, Preferred Sands of Wisconsin, LLC

File Listing	1:					
Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)	
			61043			
1	Transmittal Letter	2885005_Sb21Transmittal_032 72017.pdf	d79055fe52e67a6aab69a1100e754cc0764 76fe3	no	1	
Warnings:						
Information:						
			442567			
2		2885005_RCEandRespstoFOA_ 03272017.pdf	yes 4e15a7ab29a650c8340e7(ca9edfcb431811 867a		15	
	Multip	part Description/PDF files in .:	zip description	(
	Document De	scription	Start	End		
	Amendment Submitted/Entere	Amendment Submitted/Entered with Filing of CPA/RCE				
	Claims	2 10		10		
	Applicant Arguments/Remarks	Made in an Amendment	11	15		
Warnings:		:	,			
Information:						
	Request for Continued Examination	2885005_Sb30RCETransmittal_	73026	400.50		
3	(RCE)	03272017.pdf	a9df181dfa4bc07bea0e5f7b78e34ed539f9 46d2	no	1	
Warnings:		2	-			
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Information:						
4	Extension of Time	2885005_PetForExtofTime_032 72017.pdf	57121 e6d916e5c004e264173716b2c94a160c682 c30a6	no	1	
Warnings:					6	
Information:						
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6	Fee Worksheet (SB06)	fee-info.pdf	32666	no	2
	Fee Worksheet (SB06)	fee-info.pdf	no 668997fe8b0c6cc7b3590f22cc85359a3d3e c7a1		2
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Information	:				
Warnings:					
5	examiner			no	2
_	Applicant summary of interview with	2885005_StmtofInterviewSum	48799		

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

Doc Code: TRAN.LET

Typed or printed name

Document Description: Transmittal Letter

PTO/SB/21 (07-09) Approved for use through 07/31/2012. OMB 0651-0031

Under the Paperwork Reduction Act of 1995, no		Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE collection of information unless it displays a valid OMB control number.
	Application Number	12/759,469
TRANSMITTAL	Filing Date	April 13, 2010
FORM	First Named Inventor	

Application Number	12/759,469		
Filing Date	April 13, 2010	2000	
First Named Inventor	Kewei Zhang		
Art Unit	1768 (Conf. No.: 7030)		
Examiner Name	Bland, Alicia		
Attorney Docket Number	2885.005		
	Filing Date First Named Inventor Art Unit Examiner Name	Filing Date April 13, 2010 First Named Inventor Kewei Zhang Art Unit 1768 (Conf. No.: 7030) Examiner Name Bland, Alicia	

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	√ F	ee Attached		Licensing-related Papers				l Communication to Board peals and Interferences	
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Certified Copy of Priority Document(s) Reply to Missing Parts/ Incomplete Application Reply to Missing Parts under 37 CFR 1.52 or 1.53			1) sb21 2) Requi 3) sb30 4) Stater 5) Petitic	ically Transmitted: Transmittal Form (1 page); est for Request for Continued Examin RCE Transmittal Form (1 page); ment of Interview Summary of Examin on for Extension of Time (1 page).	er Interview on 03	3/14/2017 (2 pages); a	N 01 NT 0150	
		SIGNA	TURE	OF APPLICANT, ATT	ORNEY, O	OR AG	ENT		
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Signat	ure	Mkherte					334545		
Printed	d name	Michael A. Schade							
Date		March 27, 2017			Reg. No.	63,60	15		
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sufficie the da	ent postage te shown b	e as first class mail in an en						e United States Postal Service Alexandria, VA 22313-1450 o	
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Typed	or printed	name Michael A. So	hade				Date	March 27, 2017	

This collection of information is required by 37 CFR 1.5. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to 2 hours to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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ELECTRONICALLY TRANSMITTED: March 27, 2017

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.:

12/759,469

Confirmation No.:

7030

Inventor:

Kewei Zhang

Filed:

April 13, 2010

Art Unit:

1768

Examiner:

Bland, Alicia

Title:

METHOD FOR MAKING PARTICULATE SLURRIES AND

PARTICULATE SLURRY COMPOSITIONS

Docket No.:

2885.005

Customer No.:

30589

REQUEST FOR CONTINUED EXAMINATION AND AMENDMENT AND RESPONSE TO FINAL OFFICE ACTION

In response to the Final Office Action mailed November 25, 2016, Applicant submits the following amendments and remarks in conjunction with the Request for Continued Examination (RCE) filed concurrently herewith. Please amend the abovereferenced application as follows:

Amendments to the Claims are reflected in the listing of the claims which begins on page 2 of this paper.

Remarks begin on page 11 of this paper.

Amendments to the Claims

This listing of claims will replace all prior versions and listings of claims in the application.

Listing of Claims:

- 1. (Currently Amended) An aqueous hydraulic fracturing slurry composition for use in hydraulic fracturing operations comprising:
 - (a) water;
 - inorganic particulates ranging in size from about 10 to about 100 US (b) mesh; and
 - a chemical compound for rendering the surface of the particulates extremely hydrophobic,
 - wherein the chemical compound is selected from the group consisting of polysiloxanes modified with one or more organic cationic groups, polysiloxanes modified with one or more organic amphoteric groups, quaternary silanes, and combinations thereof,
 - wherein the hydraulic fracturing slurry composition can suspend the particulates under static and dynamic conditions without vicosifying the slurry composition and further wherein the aqueous hydraulic fracturing slurry composition is free of a viscosifier such that the aqueous hydraulic fracturing slurry composition suspends the particulates under static and dynamic conditions.

- 2. (Withdrawn) The composition of claim 1, wherein the chemical compound is a polysiloxane modified with one or more organic amphoteric groups.
- 3. (Previously Presented) The composition of claim 1, wherein the chemical compound is a polysiloxane modified with one or more organic cationic groups.
- 4. (Withdrawn) The composition of claim 1, wherein the chemical compound is a quaternary silane.
 - 5. (Cancelled)
- 6. (Withdrawn) The composition of claim 1, wherein the chemical compound is a polysiloxane having the formula

wherein each of R₁ to R₆ and R₈ to R₁₀ represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R7 represents an organic amphoteric group and m and n are from 1 to 200.

7. (Previously Presented) The composition of claim 1, wherein the chemical compound is a polysiloxane having the formula

wherein each of R₁ to R₆ and R₈ to R₁₀ represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R7 represents an organic cationic group and m and n are from 1 to 200.

8. (Withdrawn) The composition of claim 1, wherein the chemical compound is a polysiloxane having the formula

$$R_{11} = \begin{bmatrix} R_{12} & & & R_{14} \\ & & & \\ S_{1} & & & \\ & & \\ R_{13} & & & \\$$

where R₁₂ to R₁₇ each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, one of R₁₁ and R₁₈ represents an organic amphoteric group and the other of R₁₁ and R₁₈ represents an organic amphoteric group or an organic radical and m is from 1 to 200.

9. (Withdrawn) The composition of claim 1, wherein the chemical compound is a polysiloxane having the formula

where R₁₂ to R₁₇ each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, one of R₁₁ and R₁₈ represents an organic cationic group and the other of R₁₁ and R₁₈ represents an organic cationic group or an organic radical and m is from 1 to 200.

- 10. (Previously Presented) The composition of any one of claims 1 to 4 and 6 to 9, wherein the particulates are sand proppants.
- 11. (Withdrawn) The composition of any one of claims 1 to 4 and 6 to 9, wherein the particulates are ceramic particulates.
 - 12. (Cancelled)
- 13. (Withdrawn) The aqueous hydraulic fracturing slurry composition of claim 1 further comprising a gas.
 - 14. 19. (Cancelled)
- 20. (Withdrawn) The composition of claim 13, wherein the chemical compound is a polysiloxane modified with one or more organic amphoteric groups.
- 21. (Withdrawn) The composition of claim 13, wherein the chemical compound is a polysiloxane modified with one or more organic cationic groups.

22-24. (Cancelled)

25. (Withdrawn) The composition of claim 13, wherein the chemical compound is a polysiloxane having the formula

$$R_{1} \longrightarrow Si \longrightarrow O \longrightarrow Si \longrightarrow O \longrightarrow Si \longrightarrow O \longrightarrow Si \longrightarrow R_{10}$$

$$R_{3} \longrightarrow R_{5} \longrightarrow R_{5} \longrightarrow R_{7} \longrightarrow R_{9}$$

wherein each of R₁ to R₆ and R₈ to R₁₀ represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R₇ represents an organic amphoteric group and m and n are from 1 to 200.

26. (Withdrawn) The composition of claim 13, wherein the chemical compound is a polysiloxane having the formula

wherein each of R₁ to R₆ and R₈ to R₁₀ represents an organic radical containing 1-6 carbon atoms, typically a methyl group, R₇ represents an organic cationic group and m and n are from 1 to 200.

27. (Withdrawn) The composition of claim 13, wherein the chemical compound is a polysiloxane having the formula

where R₁₂ to R₁₇ each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, one of R₁₁ and R₁₈ represents an organic amphoteric group and the other of R₁₁ and R₁₈ represents an organic amphoteric group or an organic radical and m is from 1 to 200.

28. (Withdrawn) The composition of claim 13, wherein the chemical compound is a polysiloxane having the formula

where R_{12} to R_{17} each represents an organic radical containing 1-6 carbon atoms, typically a methyl group, one of R₁₁ and R₁₈ represents an organic cationic group and the other of R₁₁ and R₁₈ represents an organic cationic group or an organic radical and m is from 1 to 200.

29. (Withdrawn) The composition of claim 13, wherein the gas is selected from a group consisting of air, nitrogen, carbon dioxide, methane and mixtures thereof.

30-35. (Cancelled)

36. (Withdrawn) The composition of claim 13, wherein the particulates are sand proppants and wherein the gas is nitrogen or carbon dioxide.

37-39. (Cancelled)

- 40. (Previously Presented) The composition of claim 1, wherein the water is reused water after flow back from a well.
- 41. (Withdrawn) The composition of claim 13, wherein the water is re-used water after flow back from a well.
- 42. (Previously Presented) The composition of claim 7, wherein the water is reused water after flow back from a well.
- 43. (Previously Presented) The composition of claim 7, wherein each of R₁ to R₆ and R_8 to R_{10} is a methyl group.
 - 44. (Withdrawn) The composition of claim 7, further comprising a gas.
- 45. (Previously Presented) The composition of claim 43, wherein the water is reused water after flow back from the well.

- 46. (Withdrawn) The composition of claim 44, wherein the water is re-used water after flow back from the well.
- 47. (Withdrawn) A method of transporting proppants into a subterranean formation, comprising pumping the composition of claim 1 into the subterranean formation
- 48. (Withdrawn) A method of transporting proppants into a subterranean formation, comprising pumping the composition of claim 7 into the subterranean formation.
- 49. (Withdrawn) A method of transporting proppants into a subterranean formation, comprising pumping the composition of claim 13 into the subterranean formation.
- 50. (Withdrawn) A method of transporting proppants into a subterranean formation, comprising pumping the composition of claim 3 into the subterranean formation.
- 51. (Withdrawn) A method of transporting proppants into a subterranean formation, comprising pumping the composition of claim 44 into the subterranean formation.

- 52. (Previously Presented) The composition of claim 3, wherein the water is reused water after flow back from a well.
- 53. (Withdrawn) The composition of claim 13, wherein the particulates are sand proppants and wherein the gas is nitrogen or carbon dioxide.
- 54. (Withdrawn) The composition of claim 44, wherein the particulates are sand proppants and wherein the gas is nitrogen or carbon dioxide.

REMARKS

This is intended to be a complete response to the Final Office Action mailed November 25, 2016 (the "Office Action"), in connection with the above-identified application. In view of the final rejection, Applicant submits herewith a Request for Continued Examination along with the Amendment and Response. Claims 1-4, 6-11, 13, 20-21, 25-29, 36, and 40-54 are pending in the application. Any amendments made herein should not be taken as an admission that the prior art teaches any part or element of the claims as previously presented. Claim 1 has been amended herein to recite that the aqueous hydraulic fracturing slurry composition is free of a viscosifier such that the aqueous hydraulic fracturing slurry composition suspends the particles under static and dynamic conditions without viscosifying the aqueous hydraulic fracturing slurry composition. Support for these amendments can be found, for instance, in paragraphs [0006], [0015], [0020], [0028], [0040], and [0043] of the specification of the present application. Applicant submits that the amendments and newly added claim do not add new matter.

The Examiner issued an Advisory Action for the present application on February 16, 2017; however, in the Advisory Action, the Examiner notes that the previously proposed amendments were not entered.

Additionally, a telephonic interview was conducted on March 14, 2017 to discuss the nature of the final rejections. A summary of the conversation is detailed in the Applicant's State of Interview Summary filed contemporaneously with this Response.

Applicant does not acquiesce to the correctness of the rejections or objections in the Office Action, and reserves the right to present specific arguments regarding any rejected or objected-to claims not specifically addressed. Further, Applicant reserves the right to pursue the full scope of the subject matter of the claims in a subsequent patent application that claims priority to the present application.

At least in view of the reasons set forth below, Applicant respectfully submits that the claims now pending in the application are in a condition for allowance.

RESPONSE TO THE REJECTION OF CLAIM 1 UNDER PRE-AIA 35 U.S.C. §

In the Office Action, the Examiner has rejected Claim 1 under pre-AIA 35 U.S.C. § 112(b), second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which the inventor or a joint inventor, or for pre-AIA the applicant regards as the invention. More specifically, on page 5 of the Office Action, the Examiner states that the recitation of "without viscosifying the slurry composition" is unclear. Applicant has herein amended independent claim 1 to specifically clarify the scope of this limitation in independent claim 1, as amended. More specifically, independent claim 1 has been amended to clarify that particulates are suspended in the aqueous hydraulic fracturing slurry composition despite the slurry composition being free of a viscosifier.

In view of the above, Applicant respectfully requests that the Examiner withdraw the 35 U.S.C. § 112(b) rejection of independent claim 1, as amended, and pass the presently amended claims to an expedient allowance.

RESPONSE TO THE REJECTION OF CLAIMS 1, 3, 7, 10, 40, 42-43, 45, and 52 UNDER PRE-AIA 35 U.S.C. § 103(a)

In the Office Action, the Examiner has rejected Claims 1, 3, 7, 10, 40, 42-43, 45, and 52 under pre-AIA 35 USC § 103(a) as being unpatentable over Nguyen (U.S. Publication No. 2004/0023818) in view of Herzig (U.S. Publication No. 2004/0029981), or, Herzig in view of Nguyen. Applicant respectfully traverses the rejection in view of the amendments made to the claims and the remarks which follow.

Applicant appreciates the Examiner's thorough examination of the application and respectfully submits that the amendments to claim 1 overcome the rejection under 35 U.S.C. § 103 over Nguyen in view of Herzig and Herzig in view of Nguyen. In particular, independent claim 1 has been amended to recite that the aqueous hydraulic fracturing slurry composition is free of a viscosifier. That is, the slurry composition explicitly does not include a viscosifier and/or a viscosifying agent. Accordingly, the presently claimed inventive concept(s), as recited in Applicant's independent claim 1, as amended, do not require the addition of a viscosifier to suspend the proppants within the aqueous hydraulic fracturing slurry composition. As previously stated, support for such amendment can be found, for instance, in paragraphs [0006], [0015], [0020], [0028], [0040], and [0043] of the specification of the present application.

The Examiner states that Nguyen discloses each and every element of the claimed subject matter with the exception of the organosilicon compound, which the Examiner asserts is taught by Herzig. Additionally, the Examiner states that Herzig discloses each and every element of the claimed subject matter with the exception of sand particles having a 20/40 mesh, which the Examiner asserts is taught by Nguyen. For at least the reasons stated hereinbelow, Applicant respectfully asserts that the

combination of Nguyen and Herzig (or, alternatively, the combination of Herzig and Nguyen) fails to teach each and every element of the claimed subject matter, as currently amended.

The objective of the invention described in Nguyen is to prevent the adsorption of polymer from the gel-polymer on the surface of the proppant. See, e.g., Nguyen at paragraph [0003]. This adsorption is to be prevented because the gel-polymer forms a coating on the surface of the proppant which is difficult to remove and which compromises the utility and efficacy of the proppant. However, Nguyen expressly teaches that a critical component of the disclosed fracturing fluid is the inclusion of "a gelling agent for gelling the water...[that] increase[es] its viscosity, and optionally, a crosslinking agent for crosslinking the gel and further increasing the viscosity of the fluid." See, Nguyen at paragraphs [0012] and [0014]-[0015]. Moreover, as stated in paragraph [0012] of Nguyen, the use of these viscosifiers increases the "viscosity of the gelled or gelled and crosslinked fracturing fluid" such that fluid loss is mitigated and "the fracturing fluid [is allowed] to transport significant quantities of suspended fibrous bundles and proppant into the created fractures." Accordingly, the fracturing fluid disclosed in Nguyen requires the use of a viscosifier(s) in amounts that viscosify the fracturing fluid to suspend the proppant(s) present in the fracturing fluid.

As recited in Applicant's independent claim 1, as amended, the aqueous hydraulic fracturing slurry composition is "free of a viscosifier such that the aqueous hydraulic fracturing slurry composition suspends the particulates under static and dynamic conditions." Neither Nguyen nor Herzig teach or suggest that the fracturing fluid is free of a viscosifier, nor that the particulates within the fracturing fluid are suspended and able to be effectively transported to a fracture in the absence of a viscosifier. As previously stated, a viscosifier(s) is critical to the functioning of the fracturing fluid disclosed in Nguyen as the viscosifier(s) substantially viscosify the fracturing fluid to allow for suspension of the proppants within the fracturing slurry. Accordingly, Applicant respectfully submits that independent claim 1, as amended, and all claims depending therefrom are inventive and non-obvious over a combination of

CONCLUSION

either Nguyen and Herzig or Herzig and Nguyen.

Applicant submits that this is intended to be a complete response to the Final Office Action mailed November 25, 2016. It is respectfully submitted that this application is in a condition for allowance. In the event the Examiner's next action is not a Notice of Allowance, the Applicant's undersigned attorney respectfully requests that the Examiner initiate telephonic communication in an effort to resolve any remaining issues and move the prosecution of these claims forward in an expedient manner.

Respectfully submitted,

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Attorney for Applicant

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P	ATENT APPL		E DETI	RMINATION		Application	n or Docket Number 1/759,469	Filing Date 04/13/2010	To be Mailed	
	ENTITY: ARGE SMALL MICRO									
	APPLICATION AS FILED – PART I (Column 1) (Column 2)									
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FOR NUMBER FILED NUMBER EXTRA BASIC FEE N/A N/A							N/A		. EE (\$)	
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<u> </u>	(37 CFR 1.16(o), (p),		N/A		N/A	_	N/A			
(37	ΓAL CLAIMS CFR 1.16(i))		mir	us 20 = *			X \$ =			
	EPENDENT CLAIM CFR 1.16(h))			nus 3 = *			X \$ =	<u> </u>		
	If the specification and drawings exceed 100 sheets of paper, the application size fee due is \$310 (\$155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).									
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		(Column 1)		APPLICAT	(Column 3)	DED – PA	ART II			
AMENDMENT	03/27/2017	CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXT	RA	RATE (\$)	ADDITIO	ADDITIONAL FEE (\$)	
OME	Total (37 CFR 1.16(i))	· 48	Minus	** 65	= 0		x \$80 =		0	
EN I	Independent (37 CFR 1.16(h))	• 1	Minus ***3		= 0		× \$420 =		0	
AM	Application Si	ze Fee (37 CFR	1.16(s))	_						
Ш	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))									
							TOTAL ADD'L FE	<u> </u>	0	
		(Column 1)		(Column 2)	(Column 3)					
		CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXT	RA	RATE (\$)	ADDITIO	ONAL FEE (\$)	
Ë	Total (37 CFR 1.16(i))	3. %)	Minus	**	=		X \$ =			
ENDME	Independent (37 CFR 1.16(h))	3. 9 3	Minus	***	=		X \$ =			
JEN	Application Size Fee (37 CFR 1.16(s))									
AM	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))									
Agor CHITA		Managara and Sandara and Sandara			NOTE THE LINE	7	TOTAL ADD'L FE			
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NOTICE OF ALLOWANCE AND FEE(S) DUE

30589 7590 04/11/2017 DUNLAP CODDING, P.C. PO BOX 16370 OKLAHOMA CITY, OK 73113 EXAMINER

BLAND, ALICIA

ART UNIT PAPER NUMBER

1768 DATE MAILED: 04/11/2017

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.	
12/759.469	04/13/2010	Kewei Zhang	2885.005	7030	

TITLE OF INVENTION: METHOD FOR MAKING PARTICULATE SLURRIES AND PARTICULATE SLURRY COMPOSITIONS

APPLN. TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	UNDISCOUNTED	\$960	\$0	\$0	\$960	07/11/2017

THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. PROSECUTION ON THE MERITS IS CLOSED. THIS NOTICE OF ALLOWANCE IS NOT A GRANT OF PATENT RIGHTS. THIS APPLICATION IS SUBJECT TO WITHDRAWAL FROM ISSUE AT THE INITIATIVE OF THE OFFICE OR UPON PETITION BY THE APPLICANT. SEE 37 CFR 1.313 AND MPEP 1308.

THE ISSUE FEE AND PUBLICATION FEE (IF REQUIRED) MUST BE PAID WITHIN THREE MONTHS FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. THIS STATUTORY PERIOD CANNOT BE EXTENDED. SEE 35 U.S.C. 151. THE ISSUE FEE DUE INDICATED ABOVE DOES NOT REFLECT A CREDIT FOR ANY PREVIOUSLY PAID ISSUE FEE IN THIS APPLICATION. IF AN ISSUE FEE HAS PREVIOUSLY BEEN PAID IN THIS APPLICATION (AS SHOWN ABOVE), THE RETURN OF PART B OF THIS FORM WILL BE CONSIDERED A REQUEST TO REAPPLY THE PREVIOUSLY PAID ISSUE FEE TOWARD THE ISSUE FEE NOW DUE.

HOW TO REPLY TO THIS NOTICE:

I. Review the ENTITY STATUS shown above. If the ENTITY STATUS is shown as SMALL or MICRO, verify whether entitlement to that entity status still applies.

If the ENTITY STATUS is the same as shown above, pay the TOTAL FEE(S) DUE shown above.

If the ENTITY STATUS is changed from that shown above, on PART B - FEE(S) TRANSMITTAL, complete section number 5 titled "Change in Entity Status (from status indicated above)".

For purposes of this notice, small entity fees are 1/2 the amount of undiscounted fees, and micro entity fees are 1/2 the amount of small entity fees.

II. PART B - FEE(S) TRANSMITTAL, or its equivalent, must be completed and returned to the United States Patent and Trademark Office (USPTO) with your ISSUE FEE and PUBLICATION FEE (if required). If you are charging the fee(s) to your deposit account, section "4b" of Part B - Fee(s) Transmittal should be completed and an extra copy of the form should be submitted. If an equivalent of Part B is filed, a request to reapply a previously paid issue fee must be clearly made, and delays in processing may occur due to the difficulty in recognizing the paper as an equivalent of Part B.

III. All communications regarding this application must give the application number. Please direct all communications prior to issuance to Mail Stop ISSUE FEE unless advised to the contrary.

IMPORTANT REMINDER: Utility patents issuing on applications filed on or after Dec. 12, 1980 may require payment of maintenance fees. It is patentee's responsibility to ensure timely payment of maintenance fees when due.

PART B - FEE(S) TRANSMITTAL

Complete and send this form, together with applicable fee(s), to: Mail Mail Stop ISSUE FEE

■ Applicant asserting small entity status. See 37 CFR 1.27

■ Applicant changing to regular undiscounted fee status.

Authorized Signature _

Commissioner for Patents P.O. Box 1450

Alexandria, Virginia 22313-1450 or Fax (571)-273-2885

INSTRUCTIONS: This form should be used for transmitting the ISSUE FEE and PUBLICATION FEE (if required). Blocks 1 through 5 should be completed where appropriate. All further correspondence including the Patent, advance orders and notification of maintenance fees will be mailed to the current correspondence address as indicated unless corrected below or directed otherwise in Block 1, by (a) specifying a new correspondence address; and/or (b) indicating a separate "FEE ADDRESS" for maintenance fee notifications.

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission. CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address) Certificate of Mailing or Transmission I hereby certify that this Fee(s) Transmittal is being deposited with the United States Postal Service with sufficient postage for first class mail in an envelope addressed to the Mail Stop ISSUE FEE address above, or being facsimile transmitted to the USPTO (571) 273-2885, on the date indicated below. 30589 7590 04/11/2017 DUNLAP CODDING, P.C. PO BOX 16370 OKLAHOMA CITY, OK 73113 (Depositor's name) (Signature (Date APPLICATION NO. FILING DATE FIRST NAMED INVENTOR ATTORNEY DOCKET NO. CONFIRMATION NO. 12/759,469 04/13/2010 2885.005 7030 Kewei Zhang TITLE OF INVENTION: METHOD FOR MAKING PARTICULATE SLURRIES AND PARTICULATE SLURRY COMPOSITIONS PUBLICATION FEE DUE PREV. PAID ISSUE FEE APPLN. TYPE **ENTITY STATUS** ISSUE FEE DUE TOTAL FEE(S) DUE DATE DUE UNDISCOUNTED \$960 \$0 \$0 \$960 07/11/2017 nonprovisional **EXAMINER** ART UNIT CLASS-SUBCLASS BLAND, ALICIA 507-234000 1768 1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363). For printing on the patent front page, list (1) The names of up to 3 registered patent attorneys ☐ Change of correspondence address (or Change of Correspondence Address form PTO/SB/122) attached. or agents OR, alternatively, (2) The name of a single firm (having as a member a Tree Address" indication (or "Fee Address" Indication form PTO/SB/47; Rev 03-02 or more recent) attached. Use of a Customer registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed. Number is required. 3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type) PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document has been filed for recordation as set forth in 37 CFR 3.11. Completion of this form is NOT a substitute for filing an assignment. (A) NAME OF ASSIGNEE (B) RESIDENCE: (CITY and STATE OR COUNTRY) Please check the appropriate assignee category or categories (will not be printed on the patent): 🔲 Individual 📮 Corporation or other private group entity 📮 Government 4a. The following fee(s) are submitted: 4b. Payment of Fee(s): (Please first reapply any previously paid issue fee shown above) ☐ Issue Fee A check is enclosed. Publication Fee (No small entity discount permitted) Payment by credit card. Form PTO-2038 is attached. The director is hereby authorized to charge the required fee(s), any deficiency, or credits any Advance Order - # of Copies overpayment, to Deposit Account Number 5. Change in Entity Status (from status indicated above) NOTE: Absent a valid certification of Micro Entity Status (see forms PTO/SB/15A and 15B), issue fee payment in the micro entity amount will not be accepted at the risk of application abandonment. ☐ Applicant certifying micro entity status. See 37 CFR 1.29

Registration No. Typed or printed name Ex. 1004, p. 618 of 669 Preferred Proppants, LLC, Preferred Pipeline, LLQ: Resin Sales, LLC,

entity status, as applicable.

NOTE: This form must be signed in accordance with 37 CFR 1.31 and 1.33. See 37 CFR 1.4 for signature requirements and certifications.

<u>NOTE:</u> If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.

NOTE: Checking this box will be taken to be a notification of loss of entitlement to small or micro

Date



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE United States Patent and Trademark Office Address: COMMISSIONER FOR PATENTS P.O. Box 1450 Alexandria, Virginia 22313-1450

ww.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.		
12/759,469	04/13/2010	Kewei Zhang	2885.005	7030		
30589 759	90 04/11/2017		EXAM	INER		
DUNLAP CODD			BLAND, ALICIA			
PO BOX 16370 OKLAHOMA CIT	Y, OK 73113		ART UNIT	PAPER NUMBER		
			1768			
			DATE MAILED: 04/11/201	7		

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b)

(Applications filed on or after May 29, 2000)

The Office has discontinued providing a Patent Term Adjustment (PTA) calculation with the Notice of Allowance.

Section 1(h)(2) of the AIA Technical Corrections Act amended 35 U.S.C. 154(b)(3)(B)(i) to eliminate the requirement that the Office provide a patent term adjustment determination with the notice of allowance. See Revisions to Patent Term Adjustment, 78 Fed. Reg. 19416, 19417 (Apr. 1, 2013). Therefore, the Office is no longer providing an initial patent term adjustment determination with the notice of allowance. The Office will continue to provide a patent term adjustment determination with the Issue Notification Letter that is mailed to applicant approximately three weeks prior to the issue date of the patent, and will include the patent term adjustment on the patent. Any request for reconsideration of the patent term adjustment determination (or reinstatement of patent term adjustment) should follow the process outlined in 37 CFR 1.705.

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at 1-(888)-786-0101 or (571)-272-4200.

OMB Clearance and PRA Burden Statement for PTOL-85 Part B

The Paperwork Reduction Act (PRA) of 1995 requires Federal agencies to obtain Office of Management and Budget approval before requesting most types of information from the public. When OMB approves an agency request to collect information from the public, OMB (i) provides a valid OMB Control Number and expiration date for the agency to display on the instrument that will be used to collect the information and (ii) requires the agency to inform the public about the OMB Control Number's legal significance in accordance with 5 CFR 1320.5(b).

The information collected by PTOL-85 Part B is required by 37 CFR 1.311. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, Virginia 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, Virginia 22313-1450. Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

- The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether disclosure of these records is required by the Freedom of Information Act.
- A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
- A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
- 4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
- A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
- A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes
 of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C.
 218(c)).
- 7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
- 8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspection or an issued patent.
- 9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency if the USPTO becomes aware of a violation or notential violation of law or regulation of 669 Preferred Resin of Genoa, LLC, Preferred Sands of Wisconsin, LLC

	Application No. 12/759,469	Applicant(s) ZHANG, KEWEI		
Notice of Allowability	Examiner ALICIA BLAND	Art Unit 1768	AIA (First Inventor to File) Status No	

The MAILING DATE of this communication appears on the All claims being allowable, PROSECUTION ON THE MERITS IS (OR REMAIL HERE), a Notice of Allowance (PTOL-85) or other NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RIGHTS. To of the Office or upon petition by the applicant. See 37 CFR 1.313 and MPR	MAINS) CLOSED in this application. If not included appropriate communication will be mailed in due course. THIS his application is subject to withdrawal from issue at the initiative
 This communication is responsive to <u>rce dated 3/27/17</u>. A declaration(s)/affidavit(s) under 37 CFR 1.130(b) was/were file 	d on
 An election was made by the applicant in response to a restriction re- requirement and election have been incorporated into this action. 	quirement set forth during the interview on; the restriction
 The allowed claim(s) is/are <u>1-4,6-11,13,20,21,25-29,36 and 40-54</u>. A from the Patent Prosecution Highway program at a participating int more information, please see http://www.uspto.gov/patents/init_event 	ellectual property office for the corresponding application. For
4. Acknowledgment is made of a claim for foreign priority under 35 U.S.	C. § 119(a)-(d) or (f).
Certified copies:	
a) All b) Some *c) None of the:	
Certified copies of the priority documents have been recommendate.	The state of the s
2. Certified copies of the priority documents have been rec	distribution by the Court of th
3. Copies of the certified copies of the priority documents	nave been received in this national stage application from the
International Bureau (PCT Rule 17.2(a)).	
* Certified copies not received:	
Applicant has THREE MONTHS FROM THE "MAILING DATE" of this conoted below. Failure to timely comply will result in ABANDONMENT of the THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.	
5. CORRECTED DRAWINGS (as "replacement sheets") must be subm	nitted.
including changes required by the attached Examiner's Amendr Paper No./Mail Date	nent / Comment or in the Office action of
Identifying indicia such as the application number (see 37 CFR 1.84(c)) she each sheet. Replacement sheet(s) should be labeled as such in the header	
 DEPOSIT OF and/or INFORMATION about the deposit of BIOLOGIC attached Examiner's comment regarding REQUIREMENT FOR THE D 	
A	
Attachment(s) 1. Notice of References Cited (PTO-892)	5. Examiner's Amendment/Comment
2. ☐ Information Disclosure Statements (PTO/SB/08),	6. ☑ Examiner's Statement of Reasons for Allowance
Paper No./Mail Date 3.	7 Char
of Biological Material	7. Other
4. ☐ Interview Summary (PTO-413), Paper No./Mail Date	

U.S. Patent and Trademark Office PTOL-37 (Rev. 08-13) 20170330

Notice of Allowability

Part of Paper No./Mail Date

Application/Control Number: 12/759,469 Page 2

Art Unit: 1768

1. The present application is being examined under the pre-AIA first to invent provisions.

DETAILED ACTION

Election/Restrictions

2. The elected Claims are allowable. The restriction requirement as set forth in the Office action mailed on 10/8/15, has been reconsidered in view of the allowability of claims to the elected invention pursuant to MPEP § 821.04(a). The restriction requirement is hereby withdrawn as to any claim that requires all the limitations of an allowable claim. Specifically, Claims 2, 4, 6, 8, 9, 11, 13, 20-21, 25-29, 36, 41, 44,45-54 are no longer withdrawn from consideration because the claim(s) requires all the limitations of an allowable claim.

In view of the above noted withdrawal of the restriction requirement, applicant is advised that if any claim presented in a continuation or divisional application is anticipated by, or includes all the limitations of, a claim that is allowable in the present application, such claim may be subject to provisional statutory and/or nonstatutory double patenting rejections over the claims of the instant application.

Once a restriction requirement is withdrawn, the provisions of 35 U.S.C. 121 are no longer applicable. See In re Ziegler, 443 F.2d 1211, 1215, 170 USPQ 129, 131-32 (CCPA 1971). See also MPEP § 804.01.

3. Claims 1-4, 6-11, 13, 20-21, 25-29, 36, 40-54 are pending and allowed. Application/Control Number: 12/759,469 Page 3

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REASONS FOR ALLOWANCE

4. The following is an examiner's statement of reasons for allowance: Applicant has amended the claims to require "wherein the...composition is free of a viscosifier". Such overcomes the prior art of record. The particles of the prior art are used in fracturing fluids and said fluids include gelling/viscosifying agents. Said gelling/viscosifying agents are required in order to suspend solids such as proppants therein. Removing the viscosifying agent is not suggested or disclosed.

Any comments considered necessary by applicant must be submitted no later than the payment of the issue fee and, to avoid processing delays, should preferably accompany the issue fee. Such submissions should be clearly labeled "Comments on Statement of Reasons for Allowance."

Any inquiry concerning this communication or earlier communications from the examiner should be directed to ALICIA BLAND whose telephone number is (571)272-2451. The examiner can normally be reached on M-F 10:00 AM to 4:00 PM.

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at http://www.uspto.gov/interviewpractice.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Susannah Chung can be reached on 571-272-6098. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Application/Control Number: 12/759,469 Page 4

Art Unit: 1768

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/ALICIA BLAND/ Primary Examiner, Art Unit 1768



UNITED STATES DEPARTMENT OF COMMERCE United States Patent and Trademark Office Address: COMMISSIONER FOR PATENTS P.O. Box 1450 Alexandria, Virginia 22313-1450 www.uspto.gov

BIB DATA SHEET

CONFIRMATION NO. 7030

SERIAL NUMBER FILING or 371(c)					CLASS	GR	OUP ART	OUP ART UNIT ATTORNEY D		
12/759,46	12/759,469 04/13/20				507	1768			2885.005	
APPLICANTS										
INVENTORS Kewei Zhang, Calgary, CANADA;										
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** FOREIGN AF	PPLICA	TIONS *****	******	*****	*					
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Foreign Priority claime		Yes No	□ Mot of		STATE OR	100000	IEETS	TOT		INDEPENDENT
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