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(71) Applicant (for CA only): **SCHLUMBERGER CANADA LIMITED** [CA/CA]; 7th floor 525-3rd, Avenue S.W., Eau Claire Place 1, Alberta T2P OG4, Calgary (CA).

(71) Applicant (for FR only): **SERVICES PETROLIERS SCHLUMBERGER** [FR/FR]; 42, rue Saint-Dominique, F-75007 Paris (FR).

(71) Applicant (for GB, JP, NL only): **SCHLUMBERGER HOLDINGS LIMITED** [GB/GB]; P.O. Box 71, Craigmur Chambers, Road Town, Tortola (VG).

(71) Applicant (for AL, AU, AZ, BG, CO, CZ, DE, DK, GQ, GR, HU, ID, IE, IL, IT, KR, KZ, LT, MX, MY, NO, NZ, OM, PL, RO, RU, SI, SK, TD, TM, TN, TR, TT, UZ, ZA only): **SCHLUMBERGER TECHNOLOGY B.V.** [FR/FR]; Parkstraat 83-89, The Hague, 2514 JG (FR).

(71) Applicant (for all designated States except AL, AU, AZ, BG, CA, CO, CZ, DE, DK, FR, GB, GQ, GR, HU, ID, IE, IL, IT, JP, KR, KZ, LT, MX, MY, NO, NZ, OM, PL, RO, RU, SI, SK, TD, TM, TN, TR, TT, US, UZ, ZA): **PRAD RESEARCH AND DEVELOPMENT N.V.** [NL/NL]; De Ruyterkade 62, Willemstad, Curacao (AN).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **BARMATOV, Evgeny Borisovich** [RU/RU]; ul. Tsentralnaya, 17, Sipachi vill., Smolensky region, 528136 (RU). **ES-KIN, Dmitry Isaakovich** [RU/RU]; ul. Demakova, 17-128, Novosibirsk, 630117 (RU). **WILLBERG, Dean** [CA/US]; 10548 East Cerulean Way, Tucson, AZ 85747 (US). **MILLER, J., Matthew** [GB/GB]; Schlumberger Cambridge Research, High Cross, Madingley Rd., Cambridge, CB3 0EL (GB).

(74) Agent: **PROKOFIEVA, Zhanna Sergeevna**; LLC "Technology Company Schlumberger", per. Ogorodnaya Sloboda, 5A, Moscow, 101000 (RU).

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(54) Title: PROPPANT FLOWBACK CONTROL USING ENCAPSULATED ADHESIVE MATERIALS

(57) Abstract: The present Invention relates generally to the field of petroleum production. More specifically, the present Invention relates to a method for stimulation of reservoirs for fluid production. A method of treating a subterranean formation includes the initial stage, wherein a fracture is propped with a conventional proppant mixed with an encapsulated tackifying (adhesive) compound. The amount of encapsulated tackifying compound preferably varies from about 0.01 to about 20 weight per cent relative to the proppant. An encapsulated adhesive compound and proppant may be pre-mixed or a mixture may be prepared on the fly at the well site and then introduced into the subterranean formation. A second stage includes fracture closure that leads to capsule breakage and release of the adhesive compound.

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PROPPANT FLOWBACK CONTROL USING ENCAPSULATED ADHESIVE MATERIALS

The present Invention relates generally to the field of petroleum production. More specifically, the present Invention relates to a method for stimulation of reservoirs for fluid production.

In one common stimulation technique, proppant is pumped into a fracture created by hydraulic fracturing in the near-wellbore zone. Wide application of this approach is often hindered by proppant flowback, that is, a process of proppant removal from the proppant pack into the well bore. Proppant can be removed as both intact particles and/or as particle debris generated due to proppant crush. Experience shows that up to about 20% of the pumped proppant is usually removed from a fracture during fracture cleanup and fluid production. Proppant flowback is a serious problem occurring during completion and production.

One of the major problems caused by proppant flowback is a change in the fracture geometry. The fracture sections from which proppant is removed may become narrower. This may lead to reduction of the fracture conductivity, which in turn causes decreases in fluid production. Another problem that can be caused by high proppant flowback is a failure of downhole equipment, such as electrical submersible pumps (ESP's). Proppant crush is also an important problem. The closure pressure leads to partial proppant fragmentation, causing fines generation. Packs of polydispersed granular material are characterized by low porosity and conductivity. Proppant debris removed from a fracture may significantly

contribute to ESP erosion as well. Elimination of the proppant flowback leads to both a significant increase in proppant pack conductivity and an increase in ESP lifetime.

There are many known methods for reducing proppant flowback.

The most widely accepted method is the use of proppant material having a hardenable resin coating (see, for example, US3492147, US3929191, US5218038, and US5639806) which is pumped into the fracture at the end of the fracturing treatment. However, the use of this type of proppant has serious limitations caused by unwanted chemical reactions of the resin with the fracturing fluid. Firstly, these reactions induce partial degradation of the coating and reduce the strength of binding between proppant particles. This process results in proppant pack loosening. Secondly, degradation components of the coating cause an unpredictable change in the rheology of the fracturing fluid.

A second method of reducing proppant flowback is certain uses of "tackifying compounds". The "tackifying compound" is a liquid compound, or a solution of such a compound, capable of forming at least a partial coating upon the substrate material (proppant) with which it is mixed prior to or subsequent to placement in the subterranean formation. Compounds suitable for use as a tackifying compound include almost any compound that, when in liquid form or in a solvent solution, will form a non-hardening coating by themselves. The words "material" or "agent" may be used interchangeably here with "compound".

A liquid form or solution of a tackifying compound has been incorporated into an intimate mixture with a particulate material such as

conventional proppants or gravel packing materials and introduced into subterranean formations. (When the word proppant is used, it is to be understood that both proppant and gravel are meant; proppants and gravels are included in “particulates”.) The liquid or solution of tackifying compound interacts mechanically with the particles of particulate introduced into the subterranean formation and the adhered fines to limit or prevent the flowback of fines to the wellbore.

In another method, a tackifying compound has been incorporated in an intimate mixture with a particulate material such as conventional proppants or gravel packing materials together with a hardenable resin. Deposition of proppants coated with the tackifying compound and resin material causes particulate adjacent to the coated material to adhere to the coated material, thereby creating proppant agglomerates which bridge against other particles in the formation to minimize initial particulate flowback, and the hardenable resin subsequently consolidates the particulate before and during flowback.

The technical result of the present Invention is the development of (a) a new material for more efficient hydraulic fracturing of subterranean formations and for control of proppant flowback during fluid production, and (b) a method of using this material. This new material is nonsoluble capsules with a tackifying agent inside. This material provides no adhesion as prepared, unlike the tackifying additives known previously. The adhesion is activated *in situ* and this activation process may be enhanced by use of a pressure pulse generator.

The advantages of the compositions and methods of the Invention include the following.

- 1) The proppant-laden fracturing fluid is injected without the negative effects of having adhesive materials in the wellbore. This avoids proppant agglomeration during treating and reduces the probability of perforation plugging during hydraulic fracturing. This also reduces proppant sedimentation during treatment and fracture closure.
- 2) The protective shells prevent chemical reaction between the tackifying agent and the fracturing fluid. This avoids damage to the fracturing fluid and preserves the high adhesion activity of the encapsulated agent.
- 3) The capsules make delivery of the tackifying agent to the fracture much simpler; conventional equipment for proppant handling and mixing may be used.
- 4) The composition and method reduces the consumption of tackifying agent (and so reduces the total amount of tackifying agent needed).

Summary of the Invention

A method of treating a subterranean formation is given. According to the Invention, the method includes several stages. In the initial stage, a fracture is propped with a conventional proppant mixed with an encapsulated tackifying (adhesive) compound. The amount of encapsulated tackifying compound preferably varies from about 0.01 to about 20 weight per cent relative to the proppant. An encapsulated adhesive compound and proppant may be pre-mixed or a mixture may be prepared on the fly at the well site and then introduced into the subterranean formation. A second stage

includes fracture closure that leads to capsule breakage and release of the adhesive compound.

In an optional final stage, a pressure pulse generator is employed for intensifying the adhesive compound release and for uniform dispersion in the inter-pore medium. As a result, the adhesive compound forms a non-hardening coating on the proppant and reinforces the proppant pack in the fracture. New encapsulated adhesive compounds are also given; these and the method of treating of subterranean formations lead to reduced proppant flowback and to maintaining the initial conductivity of the proppant pack.

A composition of the Invention is a capsule consisting of at least a protective shell containing a tackifying compound. The tackifying compound may be a material selected, for example, from at least one of polyamides; quaternized polyamides; polyesters; polycarbonates; polycarbamates; natural resins; acrylates; silylated polyamides; and mixtures of these compounds. The tackifying compound may be non-hardening. The capsule size may range from about 3.36 mm (about 6 mesh) to about 0.25 mm (about 60 mesh), and the protective shell may have a thickness in the range of about 0.01 to about 1 mm.

The protective shell may be formed from a water-soluble polymer selected, for example, from at least one of a polysaccharide; a polylactide; a polyglycolide; a polyorthoester; a polyaminoacid, a polyactioacid, a polyglycolacid, a polyacrylamide, poly(ϵ -caprolactone); a chitosan; and mixtures of these polymers. The protective shell may also be formed from an oil-soluble polymer, selected, for example, from at least one of a polyester; a polyolefins; a low density polyethylene; a high density

polyethylene; a polypropylene; and mixtures of these materials. The protective shell may also be formed from an insoluble polymer compound selected, for example, from at least one of polyesters; polyacrylates; polyimides; phenol-aldehyde resins; fluoroplasts; polymethacrylates; polyvinylidene chlorides; polyvinylchlorides; and mixtures of these materials.

The capsule may also contain at least one of a deformable material; a surfactant; a multifunctional material; a degradable material; a filler material; an inert filler material; and mixtures of these materials.

Detailed Description of the Invention

It should be understood that throughout this specification, when a concentration or amount or other parameter range is described as being useful, or suitable, or the like, it is intended that any and every concentration or amount or other parameter within the range, including the end points, is to be considered as having been stated. Furthermore, each numerical value should be read once as modified by the term “about” (unless already expressly so modified) and then read again as not to be so modified unless otherwise stated in context. For example, “a range of from 1 to 10” is to be read as indicating each and every possible number along the continuum between about 1 and about 10. In other words, when a certain range is expressed, even if only a few specific data points are explicitly identified or referred to within the range, or even when no data points are referred to within the range, it is to be understood that the inventors appreciate and understand that any and all data points within the range are to be considered

to have been specified, and that the inventors have possession of the entire range and all points within the range.

Although the following discussion emphasizes fracturing, the composition and method of the Invention may be used in fracturing, gravel packing, and combined fracturing and gravel packing in a single operation ("frac and pack", "frac-pack", "frac-n-pack", stimpack, etc.).

The method of proppant flowback control and permeability retention uses new encapsulated tackifying (adhesive) compounds. One method of well treatment includes the steps of:

- a) injection of a slurry including a proppant material and an encapsulated adhesive compound; and
- b) allowing fracture closure that leads to capsule breakage and release of the adhesive compound into the proppant pack.

In another embodiment, the method of well treatment includes the steps of:

- a) injection of a slurry including a proppant material and an encapsulated adhesive compound;
- b) allowing fracture closure that leads to capsule breakage and release of the adhesive compound into the proppant matrix; and
- c) employing a pressure pulse generator to intensify the adhesive compound release and to affect more uniform dispersion of the adhesive compound into the inter-pore medium.

The encapsulated adhesive compound (capsules or microspheres) has a "core-shell" structure: a non-hardening adhesive compound ("core") encapsulated in an impermeable coating ("shell"). The chemical

composition of the protecting shell is one or more of a water or oil-soluble polymeric material, a degradable compound, or a cross-linked polymer.

The tackifying substance is a liquid, or a solution of a liquid compound or a solution of a solid compound that transforms into the liquid state at the subterranean formation temperature.

A particularly preferred group of tackifying compounds is those polyamides that are liquids at the temperature of the subterranean formation to be treated, or that are in solvent solution, and that are, by themselves, non-hardening when present on the proppant introduced into the subterranean formation. A particularly preferred material is a condensation reaction product made from (a) and (b) polyamines. Such commercially available polyacids include mixtures of C₃₆ dibasic acids containing some trimer and higher oligomers and also small amounts of monomer acids. Other polyacids include trimer acids, synthetic acids produced from fatty acids, maleic anhydride, acrylic acid, and the like. Such acid compounds are available from companies such as Witco Corporation (Chemtura Corp., Greenwich, CT, U. S. A.), Union Camp Corporation (International Paper, Memphis, TN, U. S. A.), Chemtall (Riceboro, GA, U. S. A.), and Emery Industries (Cincinnati, OH, U. S. A.).

The polyamides thus made may be converted to quaternary compounds by reaction with methylene chloride, dimethyl sulfate, benzyl chloride, diethyl sulfate, and the like. The quaternization reaction may be employed to improve the chemical compatibility of the tackifying compound with the other chemicals utilized in the treatment fluids. Quaternization of

the tackifying compound may reduce such effects as breaking of fracturing fluids.

Additional compounds that may be utilized as tackifying compounds include liquid forms or solutions of, for example, polyesters, polycarbonates and polycarbamates, natural resins such as shellac, and the like.

Compositions suitable for use as tackifying agents in the present Invention comprise any compound that may form a non-hardening coating upon a particulate. Additional examples of suitable tackifying agents include non-aqueous tackifying agents, aqueous tackifying agents, and silyl modified polyamides.

Non-aqueous tackifying agents generally comprise polyamides (condensation reaction product of a polyacid and a polyamine) that are liquids or in solution at the temperature of the subterranean formation, and are, by themselves, non-hardening.

Non-aqueous tackifying agents may be combined with multifunctional materials capable of reacting with the tackifying compounds to form hardened coatings. A "hardened coating" as used herein means that the reaction of the tackifying compound with the multifunctional material results in a substantially non-flowable reaction product that exhibits a higher compressive strength in a consolidated agglomerate than the tackifying compound alone with the particulates (for example proppant).

The "aqueous tackifying agents" are defined as those tackifying materials that are soluble in aqueous fluids. Aqueous tackifying agents are capable of forming a partial coating upon the surface of proppants. Aqueous tackifying agents are not significantly tacky when placed onto a particulate,

but are capable of being "activated" by an activating agent (or compound or material) to transform the compound into a sticky, tackifying compound at a desirable time. Such activating agents are well-known in the art.

Suitable aqueous tackifying compounds include derivatives of acrylic acid polymers, for example, acrylic acid ester polymers, acrylic acid derivative polymers, acrylic acid homopolymers, acrylic acid ester homopolymers (such as poly(methyl acrylate), poly(butyl acrylate), and poly(2-ethylhexyl acrylate)), acrylic acid ester co-polymers, methacrylic acid derivative polymers, methacrylic acid homopolymers, methacrylic acid ester homopolymers (such as poly(methyl methacrylate), poly(butyl methacrylate), and poly(2-ethylhexyl methacrylate)), acrylamido-methyl-propane sulfonate polymers, acrylamido-methyl-propane sulfonate derivative polymers, acrylamido-methyl-propane sulfonate co-polymers, and acrylic acid/acrylamido-methyl-propane sulfonate co-polymers, and combinations of such materials. These and other suitable aqueous tackifying agents are described in U. S. Patent Application Ser. No. 10/864,061, filed on Jun. 9, 2004, and U.S. Patent Application Ser. No. 10/864,618, filed on Jun. 9, 2004.

US20050059558 and WO2006032833 disclose an extended list of suitable tackifying agents.

Silyl-modified polyamide compounds suitable for use as tackifying agents are substantially self-hardening compositions. Substantially self-hardening or self-cross-linking compositions, such as the silyl-modified polyamide compounds disclosed in US6439309, may also be used as the tackifying compounds of the present Invention. Silyl-modified polyamides

may be based, for example, on the reaction product of a silylating agent with a polyamide or a mixture of polyamides. When these silyl-modified polyamide compounds are introduced in the unhardened state into a subterranean formation, these compounds are capable of at least partially adhering to naturally-occurring particulates or proppant and then are further capable of self-hardening into a substantially non-tacky state without the need for the presence of separate hardening components or reactive components.

Addition of certain surfactants (such as those described in US5787986) to the tackifying compound may improve or facilitate the coating of the tackifying compound upon the particulate.

The particle containing a tackifying compound may also contain a multifunctional material, such as those described in US6047772. The multifunctional material reacts with the tackifying compound on the proppant to consolidate at least a portion of the proppant within the formation. Preferred multifunctional materials include at least one of aldehydes, dialdehydes, diacid halides, dihalides, polyacid anhydrides, epoxides and hemiacetals.

The compositions and methods of the present Invention may also be used in conjunction with deformable particles, such as those described in US6742590, EP1398460, and MX 03008020. Preferably, deformable particles or solid materials of a larger size than the proppant particles are added and uniformly suspended in the fracturing fluid along with the proppant particles and encapsulated tackifying compound so that the smaller proppant particles stick to the larger deformable particles. The preferred

deformable particle or solid material is glass, ceramic, rubber, silicon, plastic, polymer, resin or metal, in the shape of fibers, shavings, platelets, and irregular shaped pieces. The deformable particles are preferably formed of rubber-coated proppant, resin beads, soft metal particulates, resin coated metal particulates and the like.

The methods and compositions of the Invention may also be used with a degradable material, such as those described in US20040261995 and WO2005000993, capable of undergoing an irreversible degradation downhole. The term "irreversible" means that the degradable material, once degraded downhole, should not recrystallize or reconsolidate while downhole. The terms "degradation" or "degradable" refer to both of the two relatively extreme cases of hydrolytic degradation that the degradable material may undergo, i.e., heterogeneous (or bulk erosion) and homogeneous (or surface erosion), and any stage of degradation in between these two. This degradation can be a result of a chemical or thermal reaction. The proppant, encapsulated tackifying material, and degradable material may be pre-blended or may be mixed on-the-fly at the well site. The weight concentration of the degradable material in the total composition preferably ranges from about 0.1% to about 30%. A concentration of degradable material between about 1% and about 5% by weight is most preferable.

Preferably, the degradable material is selected to have a size, and shape similar to the size and shape of the curable proppant particulates to help maintain substantial uniformity within the mixture. It is preferable if the proppant particulates and the degradable material do not segregate within the proppant composition.

Whichever degradable material is utilized, the degradable materials may have any shape, depending on the desired characteristics of the resultant voids in the proppant matrix including but not limited to particles having the physical shape of platelets, shavings, flakes, ribbons, rods, strips, spheroids, toroids, pellets, tablets, or any other physical shape.

Mixtures of proppant and encapsulated tackifying agent may also include composite particles comprising a degradable material and a filler material, for example as disclosed in U. S. Patent Applications US20050126780, and US20050130848. Such composite particles generally are more resistant to crushing forces within a fracture (as compared to degradable materials by themselves) and may help support the fracture and maintain the integrity of the proppant matrix. Also, when the composite particle degrades in the proppant pack, voids that have a desirable degree of integrity are formed, at least in part due to the high crush strength of the composite particles and the consolidation of the proppant matrix. Such voids increase the proppant pack permeability.

Suitable composite particles include one or more than one of a polysaccharide; a chitin; a chitosan; a protein; an aliphatic polyester; a poly(lactide); a poly(glycolide); a poly(ϵ -caprolactone); a poly(hydroxy ester ether); a poly(hydroxybutyrate); a polyanhydride; a polycarbonate; a poly(orthoester); a poly(acetal); a poly(acrylate); a poly(alkylacrylate); a poly(amino acid); a poly(ethylene oxide); a poly ether ester; a polyester amide; a polyamide; a polyphosphazene; and copolymers and blends of such materials.

Inert fillers may also be included in mixtures of proppant and encapsulated tackifying agent. Suitable inert fillers are materials that enhance the compressive strength of a proppant pack. Suitable fillers include calcium carbonate, talc, mica, glass, silica, silica flour, other similar mineral fillers, and mixtures of such materials.

The concentration of composite particles and/or fillers in the mixtures of proppant and encapsulated tackifying agent composition preferably range from about 0.1% to about 30% by weight of the total.

Any portion of the proppant particulates may be coated with a curable resin. A tackifying agent may be used rather than a curable resin. The tackifying agent helps distribute the degradable material within the proppant composition and keep it in place within the proppant matrix.

Suitable coating (encapsulating) materials include a water-soluble polymer taken from the classes of a polysaccharide; a polylactide; a polyglycolide; a polyorthoester; a polyaminoacid; a polylactoacid; a polyglycolacid; a polyacrylamide; a poly(ϵ -caprolactone); a chitosan; or a mixture of such materials.

Other suitable materials for the shells of encapsulated tackifying compounds include oil-soluble substances such as polyesters, polyolefins, high density polyethylene, low density polyethylene, and polypropylene. The shell may also be made from an insoluble polymer compound selected from the polyesters, polyacrylates, polyimides, phenol-aldehyde resins and mixtures of such materials. The shell may also be made from fluoroplasts, polymethacrylates, polyvinylidene chlorides, polyvinylchlorides, and mixtures of such materials.

Suitable shell thicknesses of capsules are in the range of from about 0.01 mm to about 1 mm. Suitable sizes of capsules containing adhesive compounds range from about 3.36 mm (about 6 mesh) to about 0.25 mm (about 60 mesh).

The encapsulated adhesive compound and proppant may be pre-mixed on or off location or may be mixed on the fly at the well site and then introduced into a subterranean formation. The blend preferably includes an amount of an encapsulated adhesive compound of between about 0.01% and about 20% by weight of proppant. Most preferably, the blend contains the tackifying compound in an amount of from about 0.05 to about 3.0 percent active material by weight of proppant. It is to be understood that larger quantities may be used; however, the larger quantities generally do not significantly increase performance and could undesirably reduce the permeability of the particulate pack.

The mechanism of the adhesive compound release may be one or more than one of shell breakage due to crushing, dissolution of the shell in the fracturing fluid or in subterranean water or crude oil, or diffusion of fluid into the capsule causing breakage, or diffusion of the tackifying compound out of the capsule. The adhesive compound (tackifying agent) is then dispersed in the inter-pore medium in the proppant in the fracture.

The shell may be partially destroyed during a fracture treatment at high pressure due to multiple collisions with proppant particles in the flow of the slurry. Shell thickness may influence a hydrostatic (diffusion) release mechanism. Aqueous or oil soluble polymer coatings may be employed.

Breakage of the capsule shells during the fracture closure is the major mechanism providing release of the adhesive compound. For efficient capsule breakage, capsule size should be larger than the pore sizes of the proppant pack.

To provide efficient coating of the particles (proppant) with the adhesive compound, the adhesive compound should be dispersed in the porous medium. If there is insufficient fluid motion relative to the particles, then the principle mechanism of dispersion is molecular diffusion, which is very slow.

The dispersion problem may be solved as follows. Pressure pulse generators provide high-frequency pressure pulses, causing fluid medium oscillations, the amplitudes of which are comparable to the proppant pack pore size. Micro-vortexes arising in the inter-pore medium provide efficient dispersion.

Any known pressure pulse generator may be employed; examples include cavitation generators, electro-hydraulic generators, ultrasonic generators, hydrodynamic generators, inducing water hammers at the end of the treatment, etc. An optimal depth of treatment by a pressure pulse generator is 5-10 meters.

An additional manner of using the methods and compositions of the present Invention is to use proppant mixed with encapsulated tackifying material in the far wellbore area of the fracture and then place an agent capable of controlling particulate flowback in the near wellbore area. The agent capable of controlling particulate flowback may be proppant coated with curable resin, may be fibers, or may be a screen sized to control the

flowback of the proppant. Placing proppant mixed with encapsulated tackifying material in the far wellbore area acts to help control the migration of formation sands and fines. Placing proppant coated with curable resin and/or fibers in the fracture near the well bore, or placing a screen in the well bore, acts to keep the proppant in place instead of producing it along with the produced fluids.

One of the major advantages of the introduction of encapsulated adhesive compounds is the ability to pump a proppant slurry containing only particles that do not have tacky properties. Thus the particles do not agglomerate. Compared to the composition and method of pumping a slurry containing an unprotected, available adhesive tackifying agent, the method and composition of the present Invention provides: (i) reduction in the probability of a screen-out, and (ii) lower proppant settling velocity during pumping and fracture closing. Furthermore, the Invention increases proppant pack strength even if closure stress cycling is employed, and encapsulated adhesive compounds do not require any shut-in time for activation.

Some tackifying compounds (such as those described in US5853048) may be used both to control fines migration and to control corrosion of ferrous metals. For example, suitable polyamide materials form a very thin film on the ferrous metal surfaces, protecting them from contact with aqueous fluids.

Encapsulated adhesive compounds are most effective if a forced closure procedure is applied.

The compositions and methods of the Invention may also be used for reduction of coal fines production from subterranean coal formations, including subterranean formations penetrated by gravel packed wellbores. The compositions and methods of the Invention may be used with treatment chemicals such as inhibitors, biocides, breakers, buffers, paraffin inhibitor and corrosion inhibitors. The compositions and methods of the Invention may be used when at least a portion of the proppant is resin-coated, resulting in tackified resin coated proppant.

Suitable proppant materials include any proppant or gravel used in the industry, for example ceramic particulate, sand of different shapes, proppant or sand with cured resin coating, expanded haydite, vermiculite, agloporite, or proppants with curable resin coating, and mixtures of such materials.

The Invention may be used in wells of any orientation, in open or cased holes, and with or without screens. The Invention may be used for wells for production, injection, or storage of any fluids, such as water, hydrocarbons or carbon dioxide.

Experimental:

Experiments on proppant flowback and proppant pack strength were carried out with apparatus including a pressure cell, built of Hastelloy and having a size 12 x12 mm and a slot width of 10 mm. The tested sample was put into the cell and water was pumped through the slot. The water pumping system was a closed circuit including a 100 l/min water pump having a flow rate control, a flowmeter linked to a computer, a reduction valve, and a sedimentation tank. The flow rate was operator-controlled according to the readings of the flowmeter.

Experiments were carried out with the axial force impact produced by a hydraulic press. The system had a device for collecting any proppant carried out of the cell by flowback. This device included a gravitation filter with a multicell assembly for sampling. The entire system was automated. Data acquisition and processing were computer-controlled. The apparatus provided data on the proppant pack strength through a gradual increase in the water flow rate when the critical pack-destruction rate was reached.

Example 1:

The proppant used was 0.42 to 0.84 mm (20/40 mesh) sand and the encapsulated tackifying compound was used at a concentration of 1 weight per cent of the proppant. The particulate was mixed with the fracture fluid. The size of the capsules of tackifying compound was 0.42 to 0.84 mm (20/40 mesh), and the shell thickness was about 50 microns. The reference sample was pure 0.42 to 0.84 mm (20/40 mesh) sand. The sample was loaded between two Ohio sandstone slabs in the apparatus for testing of the proppant pack strength. A standard procedure was used: a 20.6 MPa closure pressure was applied to the cell, then the cell was heated up to 95°C and kept at this temperature for 2 hours at a constant water flow rate of 50 ml/min until complete degradation of the fracture fluid had occurred. The pack strength testing was carried out with a stream of hot water (95°C) containing 2% KCl. The solution flow rate was increased gradually until complete destruction of the pack had occurred. Pack destruction was indicated by a drastic pressure drop (as shown by readings from differential pressure gauges) and by visual observation of the proppant particulate on the gravitation filters. Measurements demonstrated a 19-fold increase in the

strength of the pack from sand initially mixed with encapsulated tackifying agent in comparison to the reference case of pure proppant.

Example 2:

The proppant used was 0.42 to 0.84 mm (20/40 mesh) sand and the encapsulated tackifying compound was used at a concentration of 1 weight per cent of the proppant. The particulate was mixed with the fracture fluid. The size of the capsules of tackifying compound was 0.42 to 0.84 mm (20/40 mesh), and the shell thickness was about 50 microns. The same apparatus as example 1 was used. The sample was loaded between two Ohio sandstone slabs in the apparatus for testing of the proppant pack strength. The proppant bed contained the operating element of an ultrasound piezoelectric generator. This element was a metal cylinder having a diameter of 5 mm and a length of 10 mm. This cell was compressed with a closure pressure of 20.6 MPa. Then the cell was heated up to 95°C and kept at this temperature for 2 hours at a constant water flow rate of 50 ml/min until complete degradation of the fracture fluid had occurred. After that, the tackifying agent was further activated with ultrasound emissions of the piezoelectric generator (frequency of 10-15 kHz; power of 20W) for 10 minutes. The pack strength testing was carried out with a stream of hot water (95°C) containing 2% KCl. The solution flow rate was increased gradually until complete destruction of the pack had occurred. Pack destruction was indicated by a drastic pressure drop (as shown by readings from differential pressure gauges) and by visual observation of the proppant particulate on the gravitation filters. Measurements demonstrated a 48-fold increase in the strength of the pack initially mixed with encapsulated tackifying agent and

further treated with pressure pulses (ultrasound) in comparison to a reference case of pure proppant.

We claim:

1. A method for creating a fracture in a subterranean formation comprising injecting a mixture of a proppant with an encapsulated tackifying compound, and allowing said tackifying compound to be released during or after fracture closure.
2. The method of claim 1 further wherein the tackifying compound release process is enhanced with pressure pulses.
3. The method of a claim 1 or 2 wherein the amount of encapsulated tackifying compound varies from 0.01 to 20 weight per cent relative to the proppant.
4. A capsule comprising a protective shell containing a tackifying compound.
5. The capsule of claim 4 wherein the tackifying compound comprises a material selected from at least one of polyamides; quaternized polyamides; polyesters; polycarbonates; polycarbamates; natural resins; acrylates; silylated polyamides; and mixtures thereof.
6. The capsule of claim 4 wherein the tackifying compound is non-hardening.
7. The capsule of claim 4 wherein the capsule size ranges from 0.25 mm to 3.36 mm, and the protective shell has a thickness in the range of 0.01 to 1 mm.
8. The capsule of claim 4 wherein the protective shell comprises a water-soluble polymer.

9. The capsule of claim 8 wherein the protective shell comprises a water-soluble polymer selected from at least one of a polysaccharide; a polylactide; a polyglycolide; a polyorthoester; a polyaminoacid, a polyactoid, a polyglycolacid, a polyacrylamide, poly(ϵ -caprolactone); a chitosan; and mixtures thereof.

10. The capsule of claim 4 wherein the protective shell comprises an oil-soluble polymer.

11. The capsule of claim 10 wherein the protective shell comprises an oil-soluble polymer selected from at least one of a polyester; a polyolefins; a low density polyethylene; a high density polyethylene; a polypropylene; and mixtures thereof.

12. The capsule of claim 4 wherein the protective shell comprises an insoluble polymer compound selected from at least one of polyesters; polyacrylates; polyimides; phenol-aldehyde resins; fluoroplasts; polymethacrylates; polyvinylidene chlorides; polyvinylchlorides; and mixtures thereof.

13. The capsule of claim 4 further comprising at least one of a deformable material; a surfactant; a multifunctional material; a degradable material; a filler material; an inert filler material; and mixtures thereof.

INTERNATIONAL SEARCH REPORT

International application No.
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A. CLASSIFICATION OF SUBJECT MATTER

E21B 43/267 (2006.01)

C09K 8/92 (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)

C09K 8/00, 8/60, 8/80, 8/92, E21B 43/00, 43/25-43/267

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

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

RUPAT, RUPAT_OLD, RUABRU, USPTO, EAPATIS, PAJ, PCT, Esp@cenet

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	US 2005/0194136 A1 (PHILIP D. NGUYEN et al.) 08.09.2005, claim 1, 9-17, 51, paragraph [0011] - [0013], [0024] - [0028], [0030] - [0032]	1, 4-11, 13 3, 12
Y	US 6209643 B1 (HALLIBURTON ENERGY SERVICES, INC) 03.04.2001, claims, col. 9, lines 9-12	3
Y	US 2004/0043906 A1 (STEPHEN MARK HEATH et al.) 04.03.2004, claims, paragraph [006]	12

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance
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 "L" 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)
 "O" document referring to an oral disclosure, use, exhibition or other means
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"T" 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
 "X" 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
 "Y" 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
 "&" document member of the same patent family

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Name and mailing address of the ISA/RU FIPS
Russia, 123995, Moscow, G-59, GSP-5,
Berezhkovskaya nab., 30-1
Facsimile No. 243-3337

Authorized officer

A. Ereemeeva

Telephone No. (499) 240-2591