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(54) **METHODS AND MATERIALS TO ENHANCE HIGH TEMPERATURE RHEOLOGY IN INVERT EMULSIONS**

VERFAHREN UND MATERIALIEN ZUR VERBESSERUNG DER HOCHTEMPERATUR-RHEOLOGIE BEI INVERTEMULSIONEN

PROCÉDÉS ET MATÉRIELS POUR AMÉLIORER LA RHÉOLOGIE À TEMPÉRATURES ÉLEVÉES DANS LES ÉMULSIONS INVERSES

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Description**BACKGROUND OF THE INVENTION****1. Field of the Invention**

[0001] The present invention relates to compositions and methods for drilling, cementing and casing boreholes in subterranean formations, particularly hydrocarbon bearing formations. More particularly, the present invention relates to compositions for improving the rheology of invert emulsion drilling fluids, particularly at high temperatures and pressures.

Description of Relevant Art

[0002] A drilling fluid or mud is a specially designed fluid that is circulated through a wellbore as the wellbore is being drilled to facilitate the drilling operation. The various functions of a drilling fluid include removing drill cuttings from the wellbore, cooling and lubricating the drill bit, aiding in support of the drill pipe and drill bit, and providing a hydrostatic head to maintain the integrity of the wellbore walls and prevent well blowouts.

[0003] An important property of the drilling fluid is its rheology, and specific rheological parameters are intended for drilling and circulating the fluid through the well bore. The fluid should be sufficiently viscous to suspend barite and drilled cuttings and to carry the cuttings to the well surface. However, the fluid should not be so viscous as to interfere with the drilling operation.

[0004] Specific drilling fluid systems are selected to optimize a drilling operation in accordance with the characteristics of a particular geological formation. Oil based muds are normally used to drill swelling or sloughing shales, salt, gypsum, anhydrite and other evaporate formations, hydrogen sulfide-containing formations, and hot (greater than about 300°F (149°C) holes, but may be used in other holes penetrating a subterranean formation as well.

[0005] An oil-based invert emulsion-based drilling fluid may commonly comprise between about 50:50 to about 95:5 by volume oil phase to water phase. Such oil-based muds used in drilling typically comprise: a base oil comprising the external phase of an invert emulsion; a saline, aqueous solution (typically a solution comprising about 30% calcium chloride) comprising the internal phase of the invert emulsion; emulsifiers at the interface of the internal and external phases; and other agents or additives for suspension, weight or density, oil-wetting, fluid loss or filtration control, and rheology control. In the past, such additives commonly included organophilic clays and organophilic lignites. See H.C.H. Darley and George R. Gray, Composition and Properties of Drilling and Completion Fluids 66-67, 561-562 (5th ed. 1988). However, recent technology as described for example in U.S. Patent Nos. 7,462,580 and 7,488,704 to Kirsner, et al., introduced "clay-free" invert emulsion-based drilling fluids, which offer significant advantages over drilling fluids containing organophilic clays.

[0006] As used herein and for the purposes of the present invention, the term "clay-free" (or "clayless") means a drilling fluid made without addition of any organophilic clays or organophilic lignites to the drilling fluid composition. During drilling, such "clay-free" drilling fluids may acquire clays and/or lignites from the formation or from mixing with recycled fluids containing clays and/or lignites. However, such contamination of "clay-free" drilling fluids is preferably avoided and organophilic clays and organophilic lignites should not be deliberately added to "clay-free" drilling fluids during drilling.

[0007] Invert emulsion-based muds or drilling fluids (also called invert drilling muds or invert muds or fluids) comprise a key segment of the drilling fluids industry, and "clay-free" invert emulsion-based muds, particularly those capable of "fragile gel" behavior as described in U.S. Patent Nos. 7,462,580 and 7,488,704 to Kirsner, et al., are becoming increasingly popular.

[0008] Clay-free invert emulsion drilling fluids, like INNOVERT® drilling fluid available from Halliburton Energy Services, Inc., in Duncan, Oklahoma and Houston, Texas, for example, have been shown to yield high performance in drilling, with "fragile gel" strengths and rheology leading to lower equivalent circulating density (ECDs) and improved rate of penetration ROP.

[0009] A limiting factor in drilling a particular portion of a well is the mud weight (density of the drilling fluid) that can be used. If too high a mud weight is used, fractures are created in the formation with resulting loss of drilling fluid and other operating problems. If too low a mud weight is used, formation fluids can encroach into the well, borehole collapse may occur due to insufficient hydrostatic support, and in extreme cases safety can be compromised due to the possibility of a well blowout. Many times, wells are drilled through weak or lost-circulation-prone zones prior to reaching a potential producing zone, requiring use of a low mud weight and installation of sequential casing strings to protect weaker zones above the potential producing zone. A particularly critical drilling scenario is one that combines deepwater and shallow overburden, as is typical of ultra deepwater fields in Brazil. This scenario is characterized by high pore fluid pressure, low effective stresses, low fracturing gradients and narrow mud weight windows.

[0010] Commercially available clay-free invert emulsion drilling fluids may have less than preferred rheology at low

mud weights, that is, mud weights ranging from about 9 ppg (1078 kg/m³) to about 12 ppg (1438 kg/m³), with temperatures up to about 375°F (191°C) or higher. Addition of inert solids may improve the rheology, but result in a decreased rate of penetration during drilling and loss of or decline in other benefits seen with a clay free system. Such inert solids include for example, fine sized calcium carbonate, and the term as used herein is not meant to be understood to include or refer to drill cuttings. Low mud weight or reduced density clay-free oil based invert emulsion drilling fluids also may show a decline in the desired "fragile gel" strength characteristic of clay-free invert emulsion drilling fluids. "Fragile gel" strength generally refers to the ability of the drilling fluid to both suspend drill cuttings at rest and show a lack of a pressure spike upon resumption of drilling. Solids added to an invert emulsion drilling fluid may be difficult to remove later in the drilling process and can ultimately lead to poor control over the rheology of the drilling fluid system and decreased rate of penetration (ROP).

[0011] Also, commonly used mineral oils often used in formulating invert emulsion fluids include n-paraffins, mixtures of n-paraffins, iso-paraffins, and cyclic and branched chain alkanes. These base oils have low viscosities and invert emulsion fluids prepared with them typically need additives to impart adequate rheology.

[0012] Increasingly, invert emulsion-based drilling fluids are being subjected to ever greater performance and cost demands as well as environmental restrictions. Consequently, there is a continuing need and industry-wide interest in new drilling fluids that provide improved performance while still affording environmental and economical acceptance.

[0013] US 3,125,517 discloses an invert emulsion drilling fluid comprising an ethoxylated amine and one or more of a monocarboxyl alkanolic, alkenolic or alkynolic fatty acid containing 2-22 carbon atoms, adipic acid, aromatic sulfonic acid and boric acid.

[0014] US 2011/0214864 and US 2012/0097403 disclose invert emulsion drilling fluids comprising a dimer diamine.

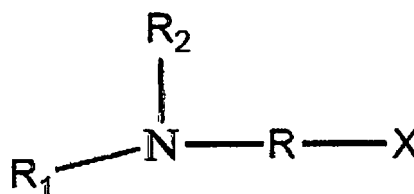
[0015] WO 2009/138383 teaches a drilling fluid comprising an oil and a polyamide emulsifier which is derived from a fatty acid and an alkoxyated polyamine.

SUMMARY OF THE INVENTION

[0016] An aspect of the present invention is an invert emulsion drilling fluid comprising:

- (a) an oleaginous continuous phase;
- (b) a non-oleaginous discontinuous phase; and
- (c) a rheology additive comprising:

- (i) a hydrophobic amine, and
 - (ii) an organic acid, an ester thereof, or a combination thereof;
- wherein the hydrophobic amine is represented by the following formula:



wherein:

R is a C₁₆₋₅₄ straight-chain or branched aliphatic, cycloaliphatic or aryl aliphatic group;
 R₁ and R₂ are each independently a hydrogen atom, an alkyl group, a cyanoalkyl group, an aminoalkyl group, an aryl group, an aminoaryl group, a hydroxyalkyl group, a carbonyl group, a carbonate group, an alkoxy group, or a hydroxyl group; and

X is an amine group optionally substituted with an alkyl group, a cyanoalkyl group, an aminoalkyl group, an aminoaryl group, or a hydroxyalkyl group, or X is an amide group, an amine oxide group, a betaine group, an ester group, a carboxylic acid group, an ether group, a hydroxyl group, a phosphate group, a phosphonate group, a pyrrolidone group, a haloformyl group, a nitrate group, a nitrite group, a sulfate group, a sulfonate group, an imidazoline group, a pyridine group, a sugar group, or any combination thereof; and wherein the organic acid has at least one COOH group and has a solubility in water of at least 0.1% w/w at 20°C.

[0017] The additive improves the rheology of the fluid without the need for the addition of inert solids, across a broad

range of temperatures and pressures. The invention is particularly advantageous at high temperatures and pressures, such as temperatures ranging from about 100°F (38°C) to about 375°F (191°C) or higher and pressures ranging from about 14 psi (97 kPa) to about 30,000 psi (207 MPa) or higher.

[0018] An example commercially available dimer diamine suitable for use in the additive of the invention is a C36 dimer diamine containing C18 fatty monoamine and C54 fatty trimer triamine which are obtained during the commercial production of the dimer diamine. Generally, quantities of such hydrophobic amine ranging from about 0.1 ppb (0.3 kg/m³) to about 20 ppb (57 kg/m³) are preferred and are effective even when the mud weight is low, that is, is in the range of about 9 ppg (1078 kg/m³) to about 12 ppg (1438 kg/m³). With this amine, generally quantities of 0.1 ppb (0.3 kg/m³) to about 20 ppb (57 kg/m³) of the organic acid or ester corresponding to the organic acid are used. Examples of commercially available organic acids suitable for use in the additive of the invention include lactic acid, formic acid, acrylic acid, acetic acid, chloroacetic acid, dichloroacetic acid, trichloroacetic acid, trifluoroacetic acid, propanoic acid, butyric acid, pentanoic acid, hexanoic acid, heptanoic acid, oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, maleic acid, fumaric acid, aspartic acid, citric acid, isocitric acid, aconitic acid, tartaric acid, benzoic acid, p-amino benzoic acid, phthalic acid, terephthalic acid, trimesic acid, without limitation

[0019] The additive of the present invention is also particularly useful in formulating oil-based invert emulsion fluids with improved rheology, even when the fluids have a low mud weight. Thus, the present invention provides improved oil-based invert emulsion drilling fluids and improved methods of drilling wellbores in subterranean formations employing such oil-based invert emulsion drilling fluids or muds, even when the muds have low mud weight. As used herein, the term "drilling" or "drilling wellbores" shall be understood in the broader sense of drilling operations, which include running casing and cementing as well as drilling, unless specifically indicated otherwise.

[0020] The invert emulsion drilling fluid of the present invention has an oil:water ratio preferably in the range of 50:50 to 95:5 and preferably employs a natural oil, such as diesel oil or mineral oil, or a synthetic base, as the oil phase, and water comprising a salt such as calcium chloride for example as the aqueous phase. The rheology additive of the invention is included for rheology stability. Clay and/or inert solids are preferably not added to provide weight or rheology control to the fluid. Invert emulsion drilling fluids of the invention may also demonstrate "fragile gel" behavior when the drilling fluid is "clay-free."

[0021] Addition of the rheology additive of the invention to the invert emulsion drilling fluid increases the Low Shear Yield Point (LSYP), Yield Point (YP), and the 10 minute Gel Strength but limits the increase in the Plastic Viscosity (PV) to about 50 % or less, relative to the drilling fluid not having the additive, when measured at 120°F (49°C). At High Pressure High Temperature (HPHT) conditions, the invert emulsion drilling fluid of the present invention comprising the rheology additive of the invention has increased LSYP, YP, and 10 minute Gel Strength but similar or lower PV, relative to the drilling fluid without the rheology additive of the invention. Such a similar or lower PV seen with the invert emulsion drilling fluid of the invention is believed to help minimize the amount of density increase caused by pumping of the fluid.

BRIEF DESCRIPTION OF THE DRAWINGS

[0022]

Figure 1 is a graph comparing the plastic viscosity, yield point and low shear yield point of an example 12 ppg (1438 kg/m³) drilling fluid formulation of the invention comprising a dimethyl adipate-fatty dimer diamine rheology additive of the invention, under various temperature and pressure conditions.

Figure 2 is a graph comparing the plastic viscosity, yield point and low shear yield point of a different example 12 ppg (1438 kg/m³) drilling fluid formulation of the invention comprising an adipic acid-fatty dimer diamine rheology additive of the invention, under various temperature and pressure conditions.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

[0023] The present invention provides an invert emulsion drilling fluid with improved performance in the field at mud weights in the range of about 9 ppg (1078 kg/m³) to about 20 ppg (2397 kg/m³). The present invention also provides a method of drilling employing an invert emulsion drilling fluid of the invention.

[0024] The oil base of invert emulsion drilling fluids of the present invention may be a natural oil such as for example mineral oil or diesel oil, or a synthetic base such as, for example, BAROID ALKANE® base comprising available from Halliburton Energy Services, Inc., in Houston, Texas and Duncan, Oklahoma, and EDC 99DW base available from TOTAL. A mineral oil may be successfully used as the oil base in the present invention, even though in the prior art some difficulties have been experienced in obtaining desirable rheological properties with mineral oils under certain conditions such as low mud weights, that is, mud weights ranging from about 9 ppg (1078 kg/m³) to about 12 ppg (1438 kg/m³), and particularly at high temperatures (greater than 225°F (107°C)). Mineral oils particularly suitable for use in the invention are selected from the group consisting of n-paraffins, iso-paraffins cyclic alkanes, branched alkanes, and

mixtures thereof.

[0025] An aqueous solution containing a water activity lowering compound, composition or material, comprises the internal phase of the invert emulsion. Such solution is preferably a saline solution comprising calcium chloride (typically about 25% to about 30%, depending on the subterranean formation water salinity or activity), although other salts or water activity lowering materials such as for example sugar known in the art may alternatively or additionally be used. Such other salts may include for example sodium chloride, sodium bromide, calcium bromide and formate salts. Water preferably comprises less than 50%, or as much as about 50%, of the drilling fluid and the oil:water ratio preferably ranges from about 50:50 to about 95:5.

[0026] Drilling fluids of the present invention uniquely include the additive of the present invention as a rheology modifier, as will be discussed further below. Further, the drilling fluids of, or for use in, the present invention, have added to them or mixed with their invert emulsion oil base, other fluids or materials needed to comprise complete drilling fluids. Such other materials optionally may include, for example: additives to reduce or control low temperature rheology or to provide thinning, for example, additives having the trade names COLDTROL®, ATC®, and OMC2™; additives for enhancing viscosity, for example, an additive having the trade name RHEMOD L™ (modified fatty acid); additives for providing temporary increased viscosity for shipping (transport to the well site) and for use in sweeps, for example, an additive having the trade name TEMPERUS™ (modified fatty acid); additives for filtration control, for example, additives having the trade names ADAPTA® and BDF-366; an emulsifier activator, such as, for example, lime; additives for high temperature high pressure control (HTHP) and emulsion stability, for example, an additive having the trade name FACTANT™ (highly concentrated tall oil derivative); and additives for emulsification, for example, an additive having the trade name EZ MUL® NT (polyaminated fatty acid). All of the aforementioned trademarked products are available from Halliburton Energy Services, Inc. in Houston, Texas, and Duncan, Oklahoma, U.S.A. As with all drilling fluids, the exact formulations of the fluids of the invention vary with the particular requirements of the subterranean formation.

[0027] A preferred commercially available drilling fluid system for use in the invention is the INNOVERT® drilling fluid system, having a paraffin/mineral oil base, available from Baroid, a Halliburton Company, in Houston, Texas and Duncan, Oklahoma. The INNOVERT® drilling fluid system typically comprises the following additives, in addition to the paraffin/mineral oil base and brine, for use as an invert emulsion drilling fluid: RHEMOD™ L modified fatty acid suspension and viscosifying agent, BDF-366™ or ADAPTA™ copolymer for HPHT filtration control, particularly for use at high temperatures, and EZ MUL® NT polyaminated fatty acid emulsifier/oil wetting agent, also particularly for use at high temperatures. Commercially available INNOVERT® drilling fluid systems also typically include TAU-MOD™ amorphous/fibrous material as a viscosifier and suspension agent. However, with the present invention, where the drilling fluid system has uniquely added thereto a hydrophobic amine and organic acid additive as a rheology modifier, TAU-MOD™ material is optional.

[0028] Invert emulsion drilling fluids of the present invention, comprising the additives of the invention, maintain acceptable and even preferred rheology measurements at low mud weights and do not experience a decreased rate of penetration (and with clay-free invert emulsion drilling fluids, also do not experience a decline in desired fragile gel strength) when in use in drilling even at high temperatures and pressures. At HPHT conditions, the invert emulsion drilling fluids of the present invention, comprising the hydrophobic amine and organic acid additive of the invention, has increased LSYP, YP, and 10 minute Gel Strength but similar PV relative to the drilling fluid without the additive of the invention. These advantages of the present invention are believed to be due to the addition of the additive of the invention to the drilling fluid. The advantages of the present invention are especially appreciated where the drilling fluid does not also contain organophilic clay or lignite.

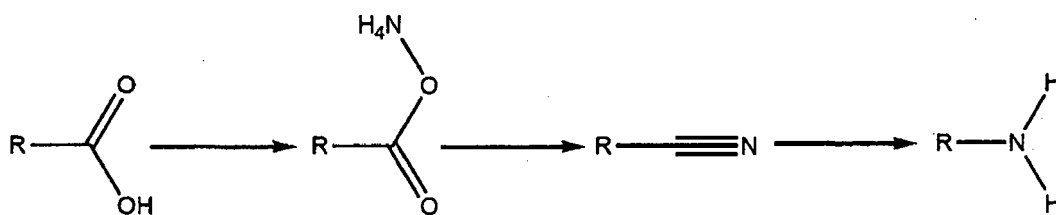
[0029] The additive of the invention comprises a dimer diamine or fatty dimer diamine and an organic acid or an ester corresponding to the organic acid, where the organic acid is a carboxylic acid with at least one COOH group and has a solubility of at least 0.1% w/w in water at 20°C.. Examples of commercially available organic acids suitable for use in the additive of the invention include lactic acid, formic acid, acrylic acid, acetic acid, chloroacetic acid, dichloroacetic acid, trichloroacetic acid, trifluoroacetic acid, propanoic acid, butyric acid, pentanoic acid, hexanoic acid, heptanoic acid, oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, maleic acid, fumaric acid, aspartic acid, citric acid, isocitric acid, aconitic acid, tartaric acid, benzoic acid, p-amino benzoic acid, phthalic acid, terephthalic acid, and trimesic acid.

[0030] Preferred commercially available fatty dimer diamines that may be used in the additive of the invention include for non-limiting example VERSAMINE® 552 hydrogenated fatty C36 dimer diamine, and VERSAMINE® 551 fatty C36 dimer diamine, both available from Cognis Corporation (functional products) of Monheim, Germany and Cincinnati, Ohio and PRIAMINE™ 1073 and PRIAMINE™ 1074 fatty C36 dimer diamine, both available from Croda Internationale Plc of Goole, East Yorkshire, United Kingdom and New Castle, Delaware. Typically, an amount of such dimer diamine in the range of about 0.1 ppb (0.3 kg/m³) to about 20 ppb (57 kg/m³) is sufficient for purposes of the invention. These fatty dimer diamines are prepared commercially from fatty dimer diacids which have been produced from dimerisation of vegetable oleic acid or tall oil fatty acid by thermal or acid catalyzed methods.

[0031] The dimerisation of C18 tall oil fatty acids produces the material leading to the C36 dimer acids. This material

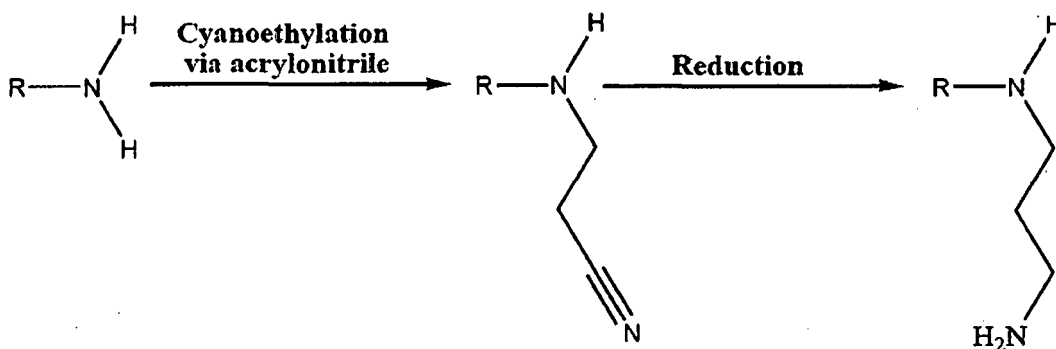
is a mixture of monocyclic dicarboxylic acid, acyclic dicarboxylic acid and bicyclic dicarboxylic acid along with small quantities of trimeric triacids. These diacids are converted into diamines via the reaction scheme given below:

Reaction Scheme I

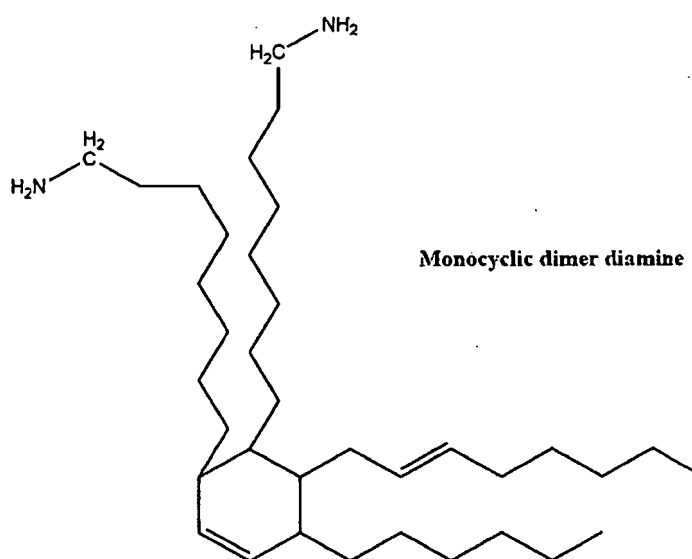


These diamines are further converted into compounds that fall under the scope of hydrophobic amine additives. These diamines are converted into cyanoethyl derivatives via cyanoethylation with acrylonitrile; these cyanoethyl derivatives are further reduced into aminopropyl amines via reduction as shown in the reaction scheme II below, as taught in United States Patent No. 4,250,045, issued February 10, 1981 to Coupland, et al.

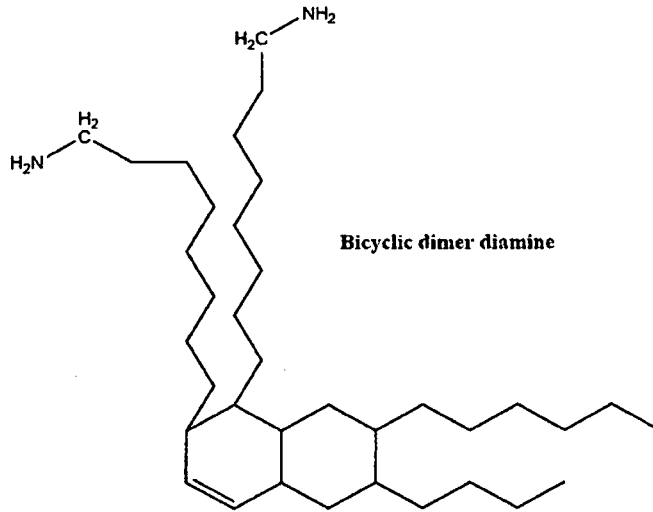
Reaction Scheme II



Dicyanoethylated dimer diamine is available commercially as Kemamine DC 3680 and 3695 and di N-aminopropylated dimer diamine is available commercially as Kemamine DD 3680 and 3695 from Chemtura Corporation USA. Different structures of the dimeric hydrophobic amine additives are given below:



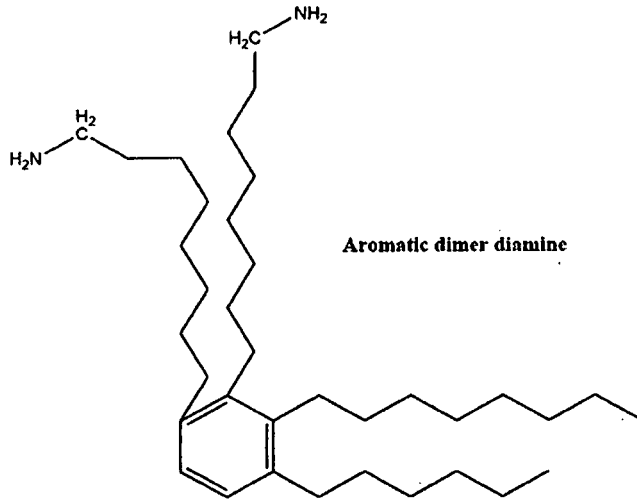
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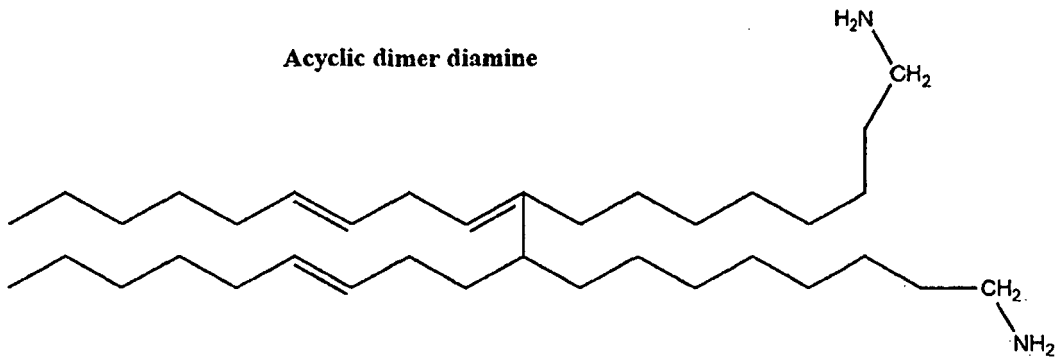


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[0032] Laboratory tests demonstrate the effectiveness of the present invention. Referring to experiments whose results are graphed in Figure 1, a 12 ppg (1438 kg/m³) INNOVERT® invert emulsion drilling fluid was prepared using paraffin/mineral oil base in a 70:30 oil to water ratio with calcium chloride brine having a water phase salinity of 250,000 parts per million (ppm). To this, additives were mixed in for a drilling fluid having the components as indicated in Table 1 below:

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TABLE 1

Effect of Adipic Acid/Fatty Dimer Diamine and Dimethyl Adipate/Fatty Dimer Diamine Additives on Rheology of 12 ppg (1438 kg/m ³) Invert Emulsion Drilling Fluids					
OWR 70:30	Time, min	1 (Base)	2	3	4
EDC 99-DW®, ppb		150.6	147.0	149.7	148.8
EZ MUL NT ®, ppb	5	11	11	11	11
Lime, ppb	5	1.3	1.3	1.3	1.3
ADAPTA®, ppb	5	2	2	2	2
Dimethyl Adipate, ppb	5	-	5	-	-
Adipic acid, ppb	5	-	-	2.5	5
CaCl ₂ soln(250K ppm), ppb	5	113.7	113.8	113.1	112.5
Revdust, ppb	5	20	20	20	20
BDF 570™ Fatty Dimer Diamine, ppb	5	2	2	2	2
BAROID® Barite, ppb	5	203.3	201.9	202.4	201.4
Hot rolled at 250F, 16 hrs					
600rpm	@120F	60	89	128	152
300 rpm	@120F	32	60	90	120
200 rpm	@120F	24	49	74	108
100 rpm	@120F	15	35	57	97
6 rpm	@120F	5	16	28	93
3 rpm	@120F	4	15	26	80
PV	@120F	28	29	38	32
YP	@120F	4	31	52	88
LSYP	@120F	3	14	24	67
10sec gel	@120F	6	21	30	82
10min gel	@120F	9	26	40	85

All trademarked products above and in other tables below are available from Halliburton Energy Services, Inc., in Houston, Texas and Duncan, Oklahoma, except that REV DUST is an artificial drill solid available from Milwhite Inc, in Houston Texas. These compositions set forth in Table 1 were hot rolled at 250°F (121°C) for 16 hours. The fluids were then further mixed for 5 minutes and evaluated on a FANN 35 rheometer at 120°F (49°C), testing Plastic Viscosity (PV), Yield Point (YP), yield stress (Tau zero) and Low Shear Yield Point (LSYP).

[0033] The Plastic Viscosity (PV), Yield Point (YP), Yield Stress (Tau zero) and Low Shear Yield Point (LSYP) of the invert emulsion drilling fluid were determined on a direct-indicating rheometer, a FANN 35 rheometer, powered by an electric motor. The rheometer consists of two concentric cylinders, the inner cylinder is called a bob, while the outer cylinder is called a rotor sleeve. The drilling fluid sample is placed in a thermostatically controlled cup and the temperature of the fluid is adjusted to 120 (± 2) °F. The drilling fluid in the thermostatically controlled cup is then placed in the annular space between the two concentric cylinders of the FANN 35. The outer cylinder or rotor sleeve is driven at a constant rotational velocity. The rotation of the rotor sleeve in the fluid produces a torque on the inner cylinder or bob. A torsion spring restrains the movement of the bob, and a dial attached to the bob indicates displacement of the bob. The dial readings are measured at different rotor sleeve speeds of 3, 6, 100, 200, 300 and 600 revolutions per minute (rpm).

[0034] Generally, Yield Point (YP) is defined as the value obtained from the Bingham-Plastic rheological model when extrapolated to a shear rate of zero. It may be calculated using 300 rpm and 600 rpm shear rate readings as noted above on a standard oilfield rheometer, such as a FANN 35 or a FANN 75 rheometer. Similarly, Yield Stress or Tau zero is the stress that must be applied to a material to make it begin to flow (or yield), and may commonly be calculated from

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rheometer readings measured at rates of 3, 6, 100, 200, 300 and 600 rpm. The extrapolation may be performed by applying a least-squares fit or curve fit to the Herchel-Bulkley rheological model. A more convenient means of estimating the Yield Stress is by calculating the Low-Shear Yield Point (LSYP) by the formula shown below in Equation 2 except with the 6 rpm and 3 rpm readings substituted for the 600 rpm and 300 rpm readings, respectively. Plastic Viscosity (PV) is obtained from the Bingham-Plastic rheological model and represents the viscosity of a fluid when extrapolated to infinite shear rate. The PV is obtained from the 600 rpm and the 300 rpm readings as given below in Equation 1. A low PV may indicate that a fluid is capable of being used in rapid drilling because, among other things, the fluid has low viscosity upon exiting the drill bit and has an increased flow rate. A high PV may be caused by a viscous base fluid, excess colloidal solids, or both. The PV and YP are calculated by the following set of equations:

$$PV = (600 \text{ rpm reading}) - (300 \text{ rpm reading}) \quad \text{(Equation 1)}$$

$$YP = (300 \text{ rpm reading}) - PV \quad \text{(Equation 2)}$$

More particularly, each of these tests were conducted in accordance with standard procedures set forth in Recommended Practice 13B-2, Recommended Practice for Field Testing of Oil-based Drilling Fluids, Fourth Edition, American Petroleum Institute, March 1, 2005.

[0035] The results of the tests reported in Table 1 demonstrate that the base formulation of the invert emulsion drilling fluid had a Yield Point (YP) of 4 and a Low-Shear Yield Point (LSYP) of 3. However, on addition of the adipic acid (HOOC-(CH₂)₄-COOH) at 2.5 ppb (7 kg/m³) concentration to the base formulation of the invert emulsion drilling fluid, the fluid's YP and LSYP improved significantly: by 1200% for the YP and 700% for the LSYP. On adding a higher concentration of adipic acid—5 ppb (14 kg/m³) concentration—to the base fluid, the fluid's YP and LSYP improved even more: by 2100% for the YP and 675% for the LSYP. Addition of dimethyl adipate (H₃COOC-(CH₂)₄-COOCH₃) at 5 ppb (14 kg/m³) concentration to the base formulation of the invert emulsion drilling fluid, also improved the fluid's YP and LSYP—by 675% for the YP and 370% for the LSYP. The PV increased only marginally, with the highest rise being 35% for 2.5 ppb (7 kg/m³) adipic acid.

[0036] Samples of formulations 2 and 3 of the invert emulsion drilling fluid set forth in Table 1, containing respectively 5 ppb (14 kg/m³) dimethyl adipate and 2 ppb (6 kg/m³) fatty dimer diamine (formulation 2) and 2.5 ppb (7 kg/m³) adipic acid and 2 ppb (6 kg/m³) fatty dimer diamine (formulation 3), were evaluated further with a FANN 75 rheometer using simulated down hole conditions, and particularly testing high temperature and high pressure rheology. The FANN 75 rheometer measures similarly as the FANN 35 rheometer but can measure rheology under high temperature and pressure. The results of these tests are set forth in Tables 2 and 3 (showing rheological data) and in Figures 1 and 2 (showing PV, YP and LSYP).

TABLE 2

Fann75 Rheology of 12 ppg (1438 kg/m ³) Invert Emulsion Drilling Fluid with Dimethyl Adipate/Fatty Dimer Diamine Additive Under High Temperature and High Pressure Conditions								
	120 F	150 F	175 F	200 F	225 F	250 F	275 F	300 F
RPM	0 psi	3000 psi	4500 psi	6000 psi	7500 psi	9000 psi	10500 psi	12000 psi
600	82	85	83	81	84	85	85	83
300	56	58	57	56	61	62	62	60
200	47	48	47	48	53	54	53	51
100	36	37	37	38	43	43	42	40
6	19	23	23	22	24	24	21	20
3	19	23	22	21	22	22	19	19
PV	26	27	26	25	23	23	23	23
YP	30	31	31	31	38	39	39	37
LSYP	19	23	21	20	20	20	17	18

TABLE 3

Fann75 Rheology of 12ppg (1438 kg/m ³) Invert Emulsion Drilling Fluid with Adipic Acid/Fatty Dimer Diamine Additive Under High Temperature and High Pressure Conditions					
	120 F	150 F	175 F	200 F	225 F
RPM	0 psi	3000 psi	4500 psi	6000 psi	7500 psi
600	127	131	127	114	110
300	87	90	87	79	78
200	73	74	71	67	67
100	56	58	56	54	54
6	30	37	38	35	33
3	30	36	36	33	32
PV	40	41	40	35	32
YP	47	49	47	44	46
LSYP	30	35	34	31	31

[0037] The rheology additive of the invention was also tested in 12 ppg invert emulsion drilling fluids prepared with different mineral oil bases and in the absence of any externally added low gravity solids (LGS). The compositions and results of these tests are shown in Table 4. Again, the additive of the invention resulted in the drilling fluids showing significant improvements in YP and LSYP, with the respective rise being 2100% for YP and 4260% for LSYP for the ESCAID 110 oil based invert emulsion fluid, and 300% for YP and 510% for LSYP for the Baroid Alkane oil based invert emulsion fluid. The PV changed only marginally in comparison to the YP and the LSYP for these mineral oils.

TABLE 4

Performance of Adipic Acid/ Fatty Dimer Diamine and Dimethyl Adipate/Fatty Dimer Diamine Additives in 12 ppg (1438 kg/m ³) Invert Emulsion Drilling Fluids Having Different Mineral Oil Bases							
OWR 70:30	Time, min	1 (BASE)	2	3 (BASE)	4	5 (BASE)	6
EDC 99-DW®, ppb		150.6	148.8	-	-	-	-
Escaid 110, ppb		-	-	146.29	144.54	-	-
Baroid Alkane, ppb		-	-	-	-	144.16	143.29
EZ MUL NT®, ppb	2	11	11	11	11	11	11
Lime, ppb	5	1.3	1.3	1.3	1.3	1.3	1.3
ADAPTA®, ppb	5	2	2	2	2	2	2
Adipic acid, ppb	5	-	5	-	5	-	2.5
CaCl ₂ soln(250K ppm), ppb	5	113.7	112.5	113.27	112.01	113.06	112.43
Revdust, ppb	5	20	20	20	20	20	20
BAROID®, ppb	5	203.3	201.4	208.1	206.1	210.5	209.5
Fatty Dimer Diamine, ppb	5	2	2	2	2	2	2
Hot rolled at 250F, 16 hrs							
600 rpm	@120F	60	152	45	161	55	132

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(continued)

Hot rolled at 250F, 16 hrs							
300 rpm	@120F	32	120	25	137	37	103
200 rpm	@120F	24	108	17	125	30	87
100 rpm	@120F	15	97	11	114	21	73
6 rpm	@120F	5	93	3	104	8	45
3 rpm	@120F	4	80	3	102	7	41
PV	@120F	28	32	20	24	18	29
YP	@120F	4	88	5	113	19	74
LSYP	@120F	3	67	3	101	6	37
10sec gel	@120F	6	82	3	101	7	40
10min gel	@120F	9	85	6	106	10	46

[0038] The rheology additive of the present invention has the further advantage of being biodegradable with low ecotoxicity. Specifically, testing in a bioassay lab at Houston, Texas indicated that fatty dimer diamines are biodegradable (66.5% and 82.1% in 28 days and 35 days respectively, marine BODIS method) and have low ecotoxicity (48-hr LC50 of >10g/L, 96-hr LC50 of >10g/L and a 96-hr NOEC of 10g/L to the marine juvenile fish *Cyprinodont variegatus*; 24-hr LC50, 48-hr LC50, 48-hr LC90 of >10g/L and a 48-hr NOEC of 10g/L for marine copepod *Acartia Tonsa*, a 10 day LC50 value of greater than 12469.47 mg/kg (via dried sediment) to the marine amphipod *Corophium volutator* in the sediment phase). The test methods for *Cyprinodon variegatus* fish are consistent with OECD 203 guideline for marine testing of offshore chemicals. The test methods for copepods *Acartia Tonsa* are consistent with ISO 14669:1999(E) guideline for marine testing of offshore chemicals while the test methods for algae *Skeletonema costatum* were consistent with ISO 10253:2006, OECD guideline as adapted for marine testing of offshore chemicals. Also, dimethyl adipate is reported as readily biodegradable (see <http://www.dow.com/custproc/products/dma.htm>) and adipic acid is reported as readily biodegradable (see <http://fscimage.fishersci.com/msds/00390.htm>). An 84% conversion of adipic acid's carbon content to carbon dioxide was observed after 30 days aerobic incubation in soil biometer flasks at an initial adipic acid concentration of 1 mg/g soil. Also, an estimated BCF value of 0.68 for adipic acid, from a measured log Kow, suggests that bioconcentration in aquatic organisms is low. Eco-toxicity study of adipic acid shows a LC50 value of 97-330 mg/L (24-96 Hr.; Static conditions, 18-22°C) for Bluegill/Sunfish. In the case of dimethyl adipate too, the bioconcentration potential is low (BCF < 100 or Log Pow < 3). An eco-toxicity study of Dimethyl Adipate indicated a LC50 value of 72mg/l and EC50 value of >100mg/lit for water flea *Daphnia magna*, (static, 48 hr.) and green alga *Selenastrum capricornutum* (Growth rate inhibition, 72 h) respectively. Due to the high biodegradability, low bioaccumulation potential and low ecotoxicity, the additives dimethyl adipate and adipic acid are believed likely to pass even stringent North Sea regulations.

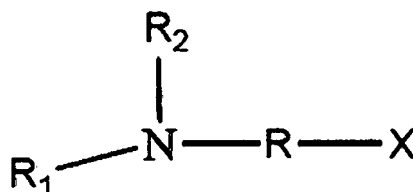
[0039] The advantages of the methods of the invention may be obtained by employing a drilling fluid of the invention in drilling operations. The drilling operations—whether drilling a vertical or directional or horizontal borehole, conducting a sweep, or running casing and cementing—may be conducted as known to those skilled in the art with other drilling fluids. That is, a drilling fluid of the invention is prepared or obtained and circulated through a wellbore as the wellbore is being drilled (or swept or cemented and cased) to facilitate the drilling operation. The drilling fluid removes drill cuttings from the wellbore, cools and lubricates the drill bit, aids in support of the drill pipe and drill bit, and provides a hydrostatic head to maintain the integrity of the wellbore walls and prevent well blowouts. The specific formulation of the drilling fluid in accordance with the present invention is optimized for the particular drilling operation and for the particular subterranean formation characteristics and conditions (such as temperatures). For example, the fluid is weighted as appropriate for the formation pressures and thinned as appropriate for the formation temperatures. The fluids of the invention afford real-time monitoring and rapid adjustment of the fluid to accommodate changes in such subterranean formation conditions. Further, the fluids of the invention may be recycled during a drilling operation such that fluids circulated in a wellbore may be recirculated in the wellbore after returning to the surface for removal of drill cuttings for example. The drilling fluid of the invention may even be selected for use in a drilling operation to reduce loss of drilling mud during the drilling operation and/or to comply with environmental regulations governing drilling operations in a particular subterranean formation.

Claims

1. An invert emulsion drilling fluid comprising:

- (a) an oleaginous continuous phase;
 (b) a non-oleaginous discontinuous phase; and
 (c) a rheology additive comprising:

- (i) a hydrophobic amine, and
 (ii) an organic acid, an ester thereof, or a combination thereof;
 wherein the hydrophobic amine is represented by the following formula:



wherein:

R is a C₁₆₋₅₄ straight-chain or branched aliphatic, cycloaliphatic or aryl aliphatic group;
 R₁ and R₂ are each independently a hydrogen atom, an alkyl group, a cyanoalkyl group, an aminoalkyl group, an aryl group, an aminoaryl group, a hydroxyalkyl group, a carbonyl group, a carbonate group, an alkoxy group, or a hydroxyl group; and
 X is an amine group optionally substituted with an alkyl group, a cyanoalkyl group, an aminoalkyl group, an aminoaryl group, or a hydroxyalkyl group, or X is an amide group, an amine oxide group, a betaine group, an ester group, a carboxylic acid group, an ether group, a hydroxyl group, a phosphate group, a phosphonate group, a pyrrolidone group, a haloformyl group, a nitrate group, a nitrite group, a sulfate group, a sulfonate group, an imidazoline group, a pyridine group, a sugar group, or any combination thereof; and
 wherein the organic acid has at least one COOH group and has a solubility in water of at least 0.1 % w/w at 20°C.

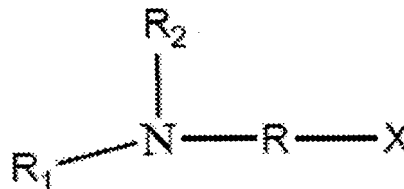
2. An invert emulsion drilling fluid according to Claim 1, wherein the organic acid is selected from lactic acid, formic acid, acrylic acid, acetic acid, chloroacetic acid, dichloroacetic acid, trichloroacetic acid, trifluoroacetic acid, propanoic acid, butyric acid, pentanoic acid, hexanoic acid, heptanoic acid, oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, maleic acid, fumaric acid, aspartic acid, citric acid, isocitric acid, aconitic acid, tartaric acid, benzoic acid, p-amino benzoic acid, phthalic acid, and trimesic acid.
3. An invert emulsion drilling fluid according to Claim 1, which is organoclay-free.
4. An invert emulsion drilling fluid according to Claim 1, wherein the oleaginous phase comprises a synthetic oil comprising an ester or olefin; a diesel oil; or a mineral oil selected from an n-paraffin, an iso-paraffin, a cyclic alkane, a branched alkane, and any mixture thereof.
5. An invert emulsion drilling fluid according to Claim 1, which has a density in the range of 1078 to 2397 kg/m³ (9 to 20 ppg).
6. An invert emulsion drilling fluid according to Claim 1, which comprises 0.24 to 96 kg/m³ (0.1 to 40 ppb) of the rheology additive.
7. An invert emulsion drilling fluid according to Claim 1, which comprises 0.24 to 48 kg/m³ (0.1 to 20 ppb) of the hydrophobic amine and 0.24 to 48 kg/m³ (0.1 to 20 ppb) of the organic acid and/or ester thereof.
8. An invert emulsion drilling fluid according to Claim 1, which has an oil:water ratio in the range of 50:50 to 95:5.

9. An invert emulsion drilling fluid according to Claim 1, wherein the non-oleaginous phase is an aqueous solution containing a water activity lowering material selected from a sugar, calcium chloride, calcium bromide, sodium chloride, sodium bromide, a formate, and any combination thereof.
10. An invert emulsion drilling fluid according to Claim 1, which comprises at least one additive selected from a weighting agent, an inert solid, a fluid loss control agent, an emulsifier, a salt, a dispersion aid, a corrosion inhibitor, an emulsion thinner, an emulsion thickener, a viscosifier, a high-pressure high-temperature (HPHT) emulsifier-filtration control agent, and any combination thereof.
11. An invert emulsion drilling fluid according to Claim 1, wherein the hydrophobic amine is a dimer diamine.
12. An invert emulsion drilling fluid according to Claim 11, wherein the hydrophobic amine is a C₃₆ dimer diamine containing a C₁₈ fatty monoamine.
13. A method of drilling in a subterranean formation, the method employing an invert emulsion drilling fluid as defined in any preceding claim.

Patentansprüche

1. Invert-Emulsionsbohrflüssigkeit, umfassend:

- (a) eine ölige kontinuierliche Phase,
- (b) eine nicht-ölige diskontinuierliche Phase und
- (c) ein Rheologieadditiv umfassend:
 - (i) ein hydrophobes Amin und
 - (ii) eine organische Säure, ein Ester davon oder eine Kombination davon, worin das hydrophobe Amin durch die folgende Formel dargestellt ist:



worin:

R eine geradkettige oder verzweigte aliphatische, cycloaliphatische oder Aryl-aliphatische C₁₆₋₅₄-Gruppe ist,
 R₁ und R₂ jeweils unabhängig voneinander ein Wasserstoffatom, eine Alkylgruppe, eine Cyanoalkylgruppe, eine Aminoalkylgruppe, eine Arylgruppe, eine Aminoarylgruppe, eine Hydroxyalkylgruppe, eine Carbonylgruppe, eine Carbonatgruppe, eine Alkoxygruppe oder eine Hydroxylgruppe sind und
 X eine Amingruppe ist, gegebenenfalls substituiert mit einer Alkylgruppe, einer Cyanoalkylgruppe, einer Aminoalkylgruppe, einer Aminoarylgruppe oder einer Hydroxyalkylgruppe ist, oder X eine Amidgruppe, eine Aminoxidgruppe, eine Betaingruppe, eine Estergruppe, eine Carbonsäuregruppe, eine Ethergruppe, eine Hydroxylgruppe, eine Phosphatgruppe, eine Phosphonatgruppe, eine Pyrrolidongruppe, eine Haloformylgruppe, eine Nitratgruppe, eine Nitritgruppe, eine Sulfatgruppe, eine Sulfonatgruppe, eine Imidazolgruppe, eine Pyridingruppe, eine Zuckergruppe oder eine Kombination davon ist und
 wobei die organische Säure mindestens eine COOH-Gruppe aufweist und eine Löslichkeit in Wasser von mindestens 0,1 Gew. % bei 20°C aufweist.

2. Invert-Emulsionsbohrflüssigkeit gemäß Anspruch 1, worin die organische Säure aus Milchsäure, Ameisensäure, Acrylsäure, Essigsäure, Chloressigsäure, Dichloressigsäure, Trichloressigsäure, Trifluoressigsäure, Propansäure, Buttersäure, Pentansäure, Hexansäure, Heptansäure, Oxalsäure, Malonsäure, Bernsteinsäure, Glutarsäure, Adipinsäure, Pimelinsäure, Korksäure, Azelainsäure, Maleinsäure, Fumarsäure, Asparaginsäure, Zitronensäure, Iso-

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zitronensäure, Aconitsäure, Weinsäure, Benzoesäure, p-Aminobenzoesäure, Phthalsäure und Trimesinsäure ausgewählt ist.

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3. Invert-Emulsionsbohrflüssigkeit gemäß Anspruch 1, die Organoton-frei ist.
4. Invert-Emulsionsbohrflüssigkeit gemäß Anspruch 1, worin die ölige Phase ein synthetisches Öl, umfassend einen Ester oder ein Olefin, ein Dieselöl oder ein Mineralöl, ausgewählt aus einem n-Paraffin, einem Iso-paraffin, einem cyclischen Alkan, einem verzweigten Alkan und einer Mischung davon, umfasst.
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5. Invert-Emulsionsbohrflüssigkeit gemäß Anspruch 1, die eine Dichte im Bereich von 1078 bis 2397 kg/m³ (9 bis 20 ppg) aufweist.
6. Invert-Emulsionsbohrflüssigkeit gemäß Anspruch 1, die 0,24 bis 96 kg/m³ (0,1 bis 40 ppb) des Rheologieadditiv umfasst.
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7. Invert-Emulsionsbohrflüssigkeit gemäß Anspruch 1, die 0,24 bis 48 kg/m³ (0,1 bis 20 ppb) des hydrophoben Amins und 0,24 bis 48 kg/m³ (0,1 bis 20 ppb) der organischen Säure und/oder des Esters davon umfasst.
8. Invert-Emulsionsbohrflüssigkeit gemäß Anspruch 1, die ein Öl:Wasser-Verhältnis im Bereich von 50:50 bis 95:5 aufweist.
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9. Invert-Emulsionsbohrflüssigkeit gemäß Anspruch 1, worin die nicht-ölige Phase eine wässrige Lösung ist, die ein wasseraktivitätserniedrigendes Material, ausgewählt aus einem Zucker, Calciumchlorid, Calciumbromid, Natriumchlorid, Natriumbromid, einem Formiat und jeglicher Kombination davon, enthält.
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10. Invert-Emulsionsbohrflüssigkeit gemäß Anspruch 1, die wenigstens ein Additiv, ausgewählt aus einem Beschwemmungsmittel, einem inerten Feststoff, einem Flüssigkeitsverlustregulierungsmittel, einem Emulgator, einem Salz, einem Dispergierhilfsmittel, einem Korrosionshemmer, einem Emulsionsverdünner, einem Emulsionsverdicker, einem Eindickmittel, einem Hochdruck-Hochtemperatur (HPHT)-Emulgator-Filtrationsregulierungsmittel und jeglicher Kombination davon, umfasst.
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11. Invert-Emulsionsbohrflüssigkeit gemäß Anspruch 1, worin das hydrophobe Amin ein dimeres Diamin ist.
12. Invert-Emulsionsbohrflüssigkeit gemäß Anspruch 11, worin das hydrophobe Amin ein dimeres C₃₆-Diamin ist, das ein C₁₋₈-Fettmonoamin enthält.
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13. Verfahren zum Bohren in eine unterirdische Formation, wobei das Verfahren eine Invert-Emulsionsbohrflüssigkeit wie in irgendeinem vorhergehenden Anspruch definiert einsetzt.

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Revendications

1. Fluide de forage en émulsion inverse comprenant :

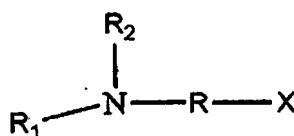
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- (a) une phase oléagineuse continue ;
- (b) une phase non oléagineuse discontinue ; et
- (c) un additif de rhéologie comprenant :

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- (i) une amine hydrophobe, et
 - (ii) un acide organique, un ester de celui-ci ou une combinaison de ceux-ci ;
- dans lequel l'amine hydrophobe est représentée par la formule suivante :

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dans laquelle :

R est un groupe aliphatique, cycloaliphatique ou aryle aliphatique, en C₁₆-C₅₄, à chaîne droite ou ramifié ;

R₁ et R₂ sont chacun indépendamment un atome d'hydrogène, un groupe alkyle, un groupe cyanoalkyle, un groupe aminoalkyle, un groupe aryle, un groupe aminoaryle, un groupe hydroxyalkyle, un groupe carbonyle, un groupe carbonate, un groupe alcoxy ou un groupe hydroxyle ; et

X est un groupe amine éventuellement substitué par un groupe alkyle, un groupe cyanoalkyle, un groupe aminoalkyle, un groupe aminoaryle ou un groupe hydroxyalkyle, ou X est un groupe amide, un groupe oxyde d'amine, un groupe bétaïne, un groupe ester, un groupe acide carboxylique, un groupe éther, un groupe hydroxyle, un groupe phosphate, un groupe phosphonate, un groupe pyrrolidone, un groupe halogénoformyle, un groupe nitrate, un groupe nitrite, un groupe sulfate, un groupe sulfonate, un groupe imidazoline, un groupe pyridine, un groupe sucre ou une quelconque combinaison de ceux-ci ; et

dans lequel l'acide organique comprend au moins un groupe COOH et a une solubilité dans l'eau d'au moins 0,1 % en poids à 20 °C.

2. Fluide de forage en émulsion inverse selon la revendication 1, dans lequel l'acide organique est choisi parmi l'acide lactique, l'acide formique, l'acide acrylique, l'acide acétique, l'acide chloroacétique, l'acide dichloroacétique, l'acide trichloroacétique, l'acide trifluoroacétique, l'acide propanoïque, l'acide butyrique, l'acide pentanoïque, l'acide hexanoïque, l'acide heptanoïque, l'acide oxalique, l'acide malonique, l'acide succinique, l'acide glutarique, l'acide adipique, l'acide pimélique, l'acide subérique, l'acide azélaïque, l'acide maléique, l'acide fumarique, l'acide aspartique, l'acide citrique, l'acide isocitrique, l'acide aconitique, l'acide tartrique, l'acide benzoïque, l'acide p-aminobenzoïque, l'acide phtalique et l'acide trimésique.
3. Fluide de forage en émulsion inverse selon la revendication 1, qui est dépourvu d'argile organique.
4. Fluide de forage en émulsion inverse selon la revendication 1, dans lequel la phase oléagineuse comprend une huile synthétique comprenant un ester ou une oléfine ; une huile de diesel ; ou une huile minérale choisie parmi une n-paraffine, une iso-paraffine, un alcane cyclique, un alcane ramifié ou un quelconque mélange de ceux-ci.
5. Fluide de forage en émulsion inverse selon la revendication 1, qui a une masse volumique dans la plage de 1078 à 2397 kg/m³ (9 à 20 ppg).
6. Fluide de forage en émulsion inverse selon la revendication 1, qui comprend 0,24 à 96 kg/m³ (0,1 à 40 ppb) de l'additif de rhéologie.
7. Fluide de forage en émulsion inverse selon la revendication 1, qui comprend 0,24 à 48 kg/m³ (0,1 à 20 ppb) de l'amine hydrophobe et 0,24 à 48 kg/m³ (0,1 à 20 ppb) de l'acide organique et/ou ester de celui-ci.
8. Fluide de forage en émulsion inverse selon la revendication 1, qui a un rapport huile:eau dans la plage de 50:50 à 95:5.
9. Fluide de forage en émulsion inverse selon la revendication 1, dans lequel la phase non oléagineuse est une solution aqueuse contenant un matériau abaissant l'activité de l'eau choisi parmi un sucre, le chlorure de calcium, le bromure de calcium, le chlorure de sodium, le bromure de sodium, un formiate et une quelconque combinaison de ceux-ci.
10. Fluide de forage en émulsion inverse selon la revendication 1, qui comprend au moins un additif choisi parmi un produit alourdissant, un solide inerte, un additif de perte de circulation, un émulsifiant, un sel, un auxiliaire de dispersion, un inhibiteur de corrosion, un diluant d'émulsion, un épaississant d'émulsion, un améliorant de viscosité, un agent de contrôle de l'émulsification-filtration à haute pression à haute température (HPHT), et une quelconque combinaison de ceux-ci.
11. Fluide de forage en émulsion inverse selon la revendication 1, dans lequel l'amine hydrophobe est une diamine dimère.
12. Fluide de forage en émulsion inverse selon la revendication 11, dans lequel l'amine hydrophobe est une diamine dimère en C₃₆ contenant une monoamine grasse en C₁₈.

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13. Procédé de forage d'une formation souterraine, le procédé employant un fluide de forage en émulsion inverse tel que défini dans une quelconque revendication précédente.

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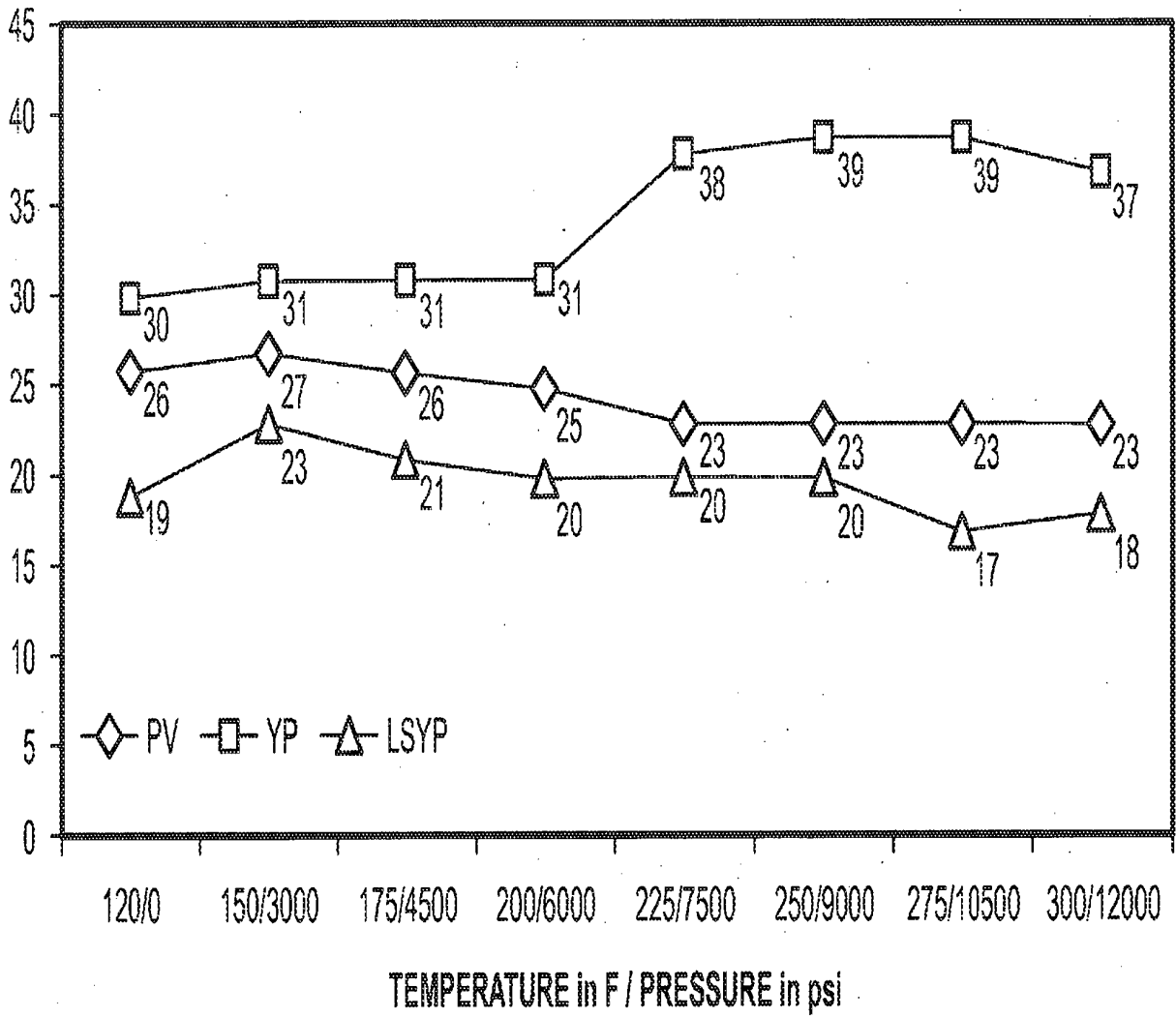


FIG. 1

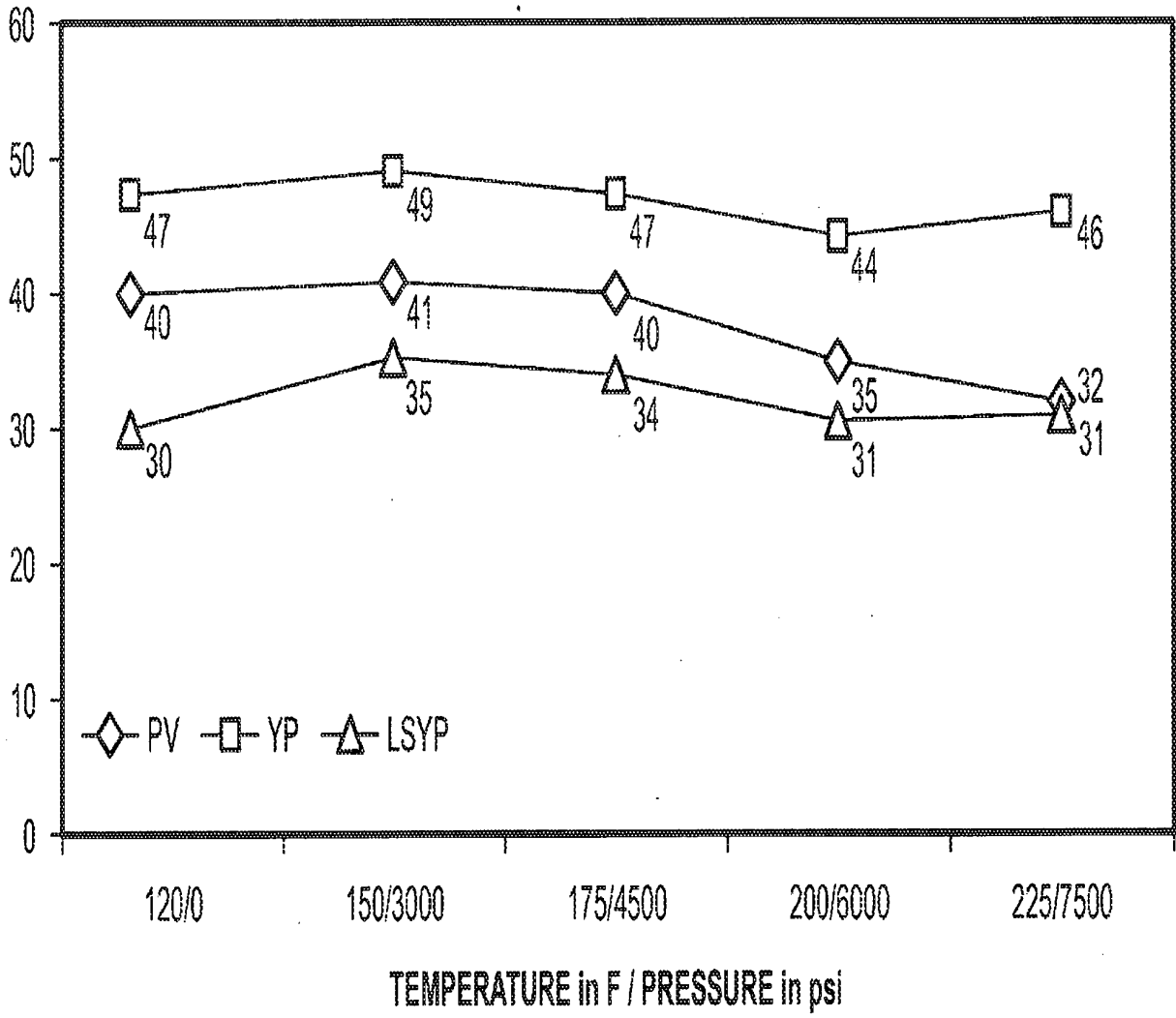


FIG. 2

REFERENCES CITED IN THE DESCRIPTION

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