Title: COMPOSITIONS AND METHODS FOR CLEANING A WELLBORE PRIOR TO CEMENTING

Abstract: Wellbore-cleaning compositions comprising microemulsions are useful during cementing operations. The microemulsion solvent may be selected from the group comprising aliphatic solvents and glycol ethers. The microemulsion surfactants comprise a water-wetting surfactant, a cleaning surfactant and an emulsifying surfactant. The microemulsion may further comprise a non-ionic surfactant and a non-ionic co-surfactant. The wellbore-cleaning compositions may be pumped alone, ahead of a conventional spacer fluid or scavenger slurry and behind a conventional spacer fluid or scavenger slurry, in addition, the wellbore-cleaning composition may be incorporated into a carrier fluid such as (but not limited to) a conventional spacer fluid or scavenger slurry. The resulting fluid mixture may be pumped as a spacer fluid.

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COMPOSITIONS AND METHODS FOR CLEANING A WELLBORE PRIOR TO CEMENTING

RELATED APPLICATIONS


BACKGROUND

[002] This disclosure relates generally to compositions and methods for treating a subterranean well prior to cementing. More specifically, the present disclosure relates to compositions and methods for cleaning casing and wellbore surfaces with fluids comprising a microemulsion.

[003] Some statements may merely provide background information related to the present disclosure and may not constitute prior art.

[004] The use of oil or synthetic-based drilling fluid (SBM/OBM) is widespread in many areas for a variety of reasons, including excellent shale inhibition, high rates of penetration and high lubricity. Oil- or synthetic-based drilling fluids generally comprise invert emulsion fluids, where the continuous or external phase is predominantly organic (e.g., mineral oil or synthetic oil), and the inverse or internal phase is usually aqueous (e.g., brines). The stability of invert emulsions is generally maintained by one or more additives present in the fluid, such as emulsifiers, emulsion stabilizing agents, and oil-wetting agents.

[005] When drilling is performed with SBM/OBM fluids, the wellbore becomes oil-wet. Prior to cementing, the casing also becomes oil-wet while being run into the hole. This condition commonly results in poor bonding between the set cement and the casing and wellbore surfaces. Poor cement bonding may compromise the hydraulic seal in the

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annulus, potentially resulting in fluid communication between subterranean zones. Therefore, to ensure successful cementing, two conditions are necessary: (1) the SBM/ObM is effectively removed from the borehole; and (2) the wellbore-wall and casing surfaces are water-wet. Failure to satisfy Condition 1 may cause contamination of the cement slurry, and the cement performance may suffer. Failure to satisfy Condition 2 may lead to poor bonding between the cement and the borehole-wall and casing surfaces.

[006] Despite efforts to prepare the borehole properly prior to cementing, Cement Bond Logs (CBL) commonly reveal poor or no bonding, or poor-quality cement behind casing. Current solutions to achieve mud removal and water wetting include pumping fluids that separate the cement slurry from the drilling fluid. The fluid may be a single-stage, viscous, water-base spacer that contains surfactants with strong water-wetting tendencies. Or, a two-stage spacer system may be pumped that comprises base oil, solvents, or water-base chemical washes with surfactants, in combination with a viscous water-base spacer that contains water-wetting surfactants.


**SUMMARY**

[008] The present document discloses improved wellbore-cleaning compositions and methods for optimal displacement of SBM/ObM, thus allowing superior bonding of cement in the annular space between casing and the formation face.
The compositions and methods comprise using a wellbore-cleaning composition comprising a microemulsion to clean a subterranean well borehole and casing prior to cementing. The microemulsion is formed by combining a solvent, a co-solvent, a water-wetting surfactant, a cleaning surfactant, an emulsifying surfactant, and water or brine. These additives may be mixed together to form a single-phase, optically-clear, thermodynamically-stable microemulsion. The microemulsion may further comprise a non-ionic surfactant and a non-ionic co-surfactant.

The wellbore-cleaning composition may be used alone as a spacer fluid and/or as a separate stage preceding or following a conventional spacer fluid or scavenger slurry. In addition, the composition may be incorporated within a carrier fluid, and the resulting fluid mixture may be pumped as a spacer fluid.

In an aspect, embodiments relate to wellbore-cleaning compositions that comprise a microemulsion.

In a further aspect, embodiments relate to methods for cleaning a section of a wellbore prior to a cementing operation.

In yet a further aspect, embodiments relate to methods for cementing a subterranean well having a wellbore.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 shows the mud-removal efficiency as measured during grid tests.

Figure 2 shows the results of wettability tests.

DETAILED DESCRIPTION

At the outset, it should be noted that in the development of any such actual embodiment, numerous implementation-specific decisions must be made to achieve the developer's specific goals, such as compliance with system related and business related constraints, which will vary from one implementation to another. Moreover, it will be
appreciated that such a development effort might be complex and time consuming but would nevertheless be a routine undertaking for those of ordinary skill in the art having the benefit of this disclosure. The description and examples are presented solely for the purpose of illustrating the preferred embodiments should not be construed as a limitation to the scope and applicability of the disclosed embodiments. While the compositions of the present disclosure are described herein as comprising certain materials, it should be understood that the composition could optionally comprise two or more chemically different materials. In addition, the composition can also comprise some components other than the ones already cited.

[0017] In the summary and the description, each numerical value should be read once as modified by the term "about" (unless already expressly so modified), and then read again as not so modified unless otherwise indicated in context. Also, in the summary and the detailed description, it should be understood that a concentration range listed or described as being useful, suitable, or the like, is intended that any and every concentration within the range, including the end points, is to be considered as having been stated. 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. Thus, even if specific data points within the range, or even no data points within the range, are explicitly identified or refer to only a few specific data points, it is to be understood that the Applicants appreciate and understand that any and all data points within the range are to be considered to have been specified, and that the Applicants have disclosed and enabled the entire range and all points within the range.

[0018] Microemulsions are macroscopically homogeneous mixture of oil, water and surfactant. They form upon simple mixing of the components, and do not require the high-shear conditions generally required for creating ordinary emulsions. Microemulsions are thermodynamically, not kinetically, stabilized, and may consist of one, two or three phases. They may consist of oil dispersed in water (O/W) or water dispersed in oil (W/O) emulsions. Microemulsions are generally described as Winsor Type I, II, III or IV emulsions. A system or formulation is defined as: Winsor I when it contains a microemulsion in equilibrium with an excess oil phase; Winsor II when it
contains a microemulsion in equilibrium with excess water; Winsor III when it contains a middle phase microemulsion in equilibrium with excess water and excess oil; and Winsor IV when it contains a single-phase microemulsion with no excess oil or excess water. More information about microemulsions and especially about Winsor IV can be found in S. Ezrahi, A. Aserin and N. Garti, "Chapter 7: Aggregation Behavior in One-Phase (Winsor IV) Microemulsion Systems", in P. Kumar and K. L. Mittal, ed., Handbook of Microemulsion Science and Technology, Marcel Dekker, Inc., New York, 1999, pp. 185-246.

[0019] In an aspect, embodiments relate to a wellbore-cleaning composition comprising a microemulsion. The composition may be introduced into the wellbore to displace synthetic-base and/or oil-base drilling fluid, and provide clean and water-wet casing and borehole surfaces prior to cementing. The microemulsion is formed by combining a solvent, a co-solvent, a water-wetting surfactant, a cleaning surfactant, and water or brine. The combination preferably forms a thermodynamically stable, optically-clear, single-phase Winsor Type IV microemulsion. The microemulsion may further comprise a non-ionic surfactant and a non-ionic co-surfactant.

[0020] The solvent is preferably selected from the group of aliphatic solvents including (but not limited to) hexane, diesel, gasoline, kerosene and deodorized kerosene. Of these, kerosene is preferred. The co-solvent is preferably selected from the group of glycol ethers, such as (but not limited to) ethylene glycol monomethyl ether, ethylene glycol monoethoxy ether, ethylene glycol monopropyl ether, ethylene glycol monoisopropyl ether, ethylene glycol monobutyl ether, ethylene glycol monophenyl ether, ethylene glycol monobenzyl ether, diethylene glycol monooxy ether, diethylene glycol monoethoxy ether, and diethylene glycol monoo-n-buty ether. The solvent concentration in the microemulsion is preferably between about 10% and 45% by weight, and more preferably between about 30% and 40% by weight. The co-solvent concentration in the microemulsion is preferably between about 10% and 40% by weight, and more preferably between about 30% and 40% by weight.
The water-wetting surfactant preferably comprises an alcohol ethoxylate. The most preferred of these are alkyl alcohol ethoxylates with carbon-chain lengths between about 9 and 11. The water-wetting-surfactant concentration in the microemulsion is preferably between about 1% and 10% by weight, and more preferably between about 5% and 7% by weight.

The cleaning surfactant is preferably selected from one or more salts of n-alkylbenzenesulfonic acid, such as (but not limited to) ammonium dodecylbenzene sulfonate, calcium dodecylbenzene sulfonate, magnesium isododecylbenzene sulfonate, potassium dodecylbenzene sulfonate, sodium decylbenzene sulfonate, sodium dodecylbenzene sulfonate (linear), sodium docosylbenzene sulfonate (branched) and sodium tridecylbenzene sulfonate. Of these, sodium dodecylbenzene sulfonate is preferred. The cleaning-surfactant concentration in the microemulsion is preferably between about 5% and 20% by weight, and more preferably between about 10% and 15% by weight.

The emulsifying surfactant preferably comprises a polysorbate selected from (but not limited to) polyoxyethylene (20) sorbitan monolaurate, polyoxyethylene (20) sorbitan monoplamitate, polyoxyethylene (20) sorbitan monooleate and polyoxyethylene (20) sorbitan monostearate. The preferred oil-solubilizing surfactant is polyoxyethylene (20) sorbitan monooleate. Of these, polyoxyethylene (20) sorbitan monooleate is most preferred. The emulsifying-surfactant concentration in the microemulsion is preferably between about 1% and 5% by weight, and more preferably between about 1.5% and 3.0% by weight.

Water or brine is preferably present at concentrations between about 1% and 20% by weight, preferably between about 7% and 9% by weight.

The microemulsion may further comprise a non-ionic surfactant and a non-ionic co-surfactant. The non-ionic surfactant preferably comprises an alkyl polyglycoside. The most preferred of these have carbon-chain lengths between about 8 and 10. The non-ionic co-surfactant preferably comprises an alcohol alkoxylate, most preferably containing polyethylene groups, polypropylene groups or both. The non-ionic-surfactant
concentration in the microemulsion is preferably between about 1% and 6% by weight, and preferably between about 4% and 6% by weight. The non-ionic co-surfactant concentration in the microemulsion is preferably between about 1% and 5% by weight, and more preferably between about 2% and 3% by weight.

[0026] The wellbore-cleaning composition may further comprise a carrier fluid such as (but not limited to) a conventional water-base spacer fluid or scavenger slurry. Despite being diluted, the microemulsions surprisingly remain intact, and their cleaning capabilities are undiminished. Such microemulsion/carrier fluid mixtures are particularly advantageous from a logistical point of view.

[0027] Those skilled in the art will understand that conventional water-base spacer fluids are not microemulsions, and maybe exemplified by MUDPUSH™ spacers, available from Schlumberger. The conventional water-base spacer fluid may be viscosified and weighted, or viscosified and unweighted. Those skilled in the art will also understand that scavenger slurries are aqueous suspensions of solids such as (but not limited to) Portland cement, and frequently contain customary cement additives such as (but not limited to) retarders, fluid-loss additives and dispersants. When the wellbore-cleaning composition is incorporated within the carrier fluid (e.g., water-base spacer fluid or scavenger slurry), the microemulsion concentration in the resulting fluid mixture is preferably between about 5 vol% and 20 vol%, more preferably between about 10 vol% and 15 vol%, and most preferably between about 10 vol% and 12 vol%.

[0028] The wellbore-cleaning composition, the carrier fluid or both may be viscosified. Non-limiting examples of viscosifying agents suitable for use herein include aluminum phosphate ester, alkyl quaternary ammonium bentonite, alkyl quaternary ammonium montmorillonite, xanthan gum, gelatin, pectin, cellulosic derivatives, gum arabic, guar gum, locust bean gum, tara gum, cassia gum, agar, n-octenyl succinated starch, porous starch, alginates, carrageenates, chitosan, scleroglucan, diutan, welan gum and an organophilic clay such as CLAYTONE™ (available from Southern Clay Products, Inc.; Gonzalez, TX, USA).
A trimer acid based rheology modifier such as RHEFLAT™ (available from M-I SWACO, Houston, TX, USA) may be also added to the viscosified wellbore-cleaning composition to achieve a flat rheology profile. Without wishing to be bound by any theory, this compound is believed to enhance low-end viscosity and yield point by interacting with fine solids such as organophillic clay and weighting solids such as calcium carbonate, barite, and hematite. The rheology modifier is preferably present in an amount of from about 1 to 5% by weight of the microemulsion.

The density of the wellbore-cleaning composition, the carrier fluid or both may also be adjusted by, for example, adding a suitable weighting agent or lightweight material. Suitable weighting agents include (but are not limited to) barite, an inorganic cement, calcium carbonate, hematite, ilmenite, magnesium tetraoxide and silica. Suitable lightweight materials include (but are not limited to) ceramic microspheres, glass microspheres, uintaite, uintahite, coal and nitrogen. It should be noted that weighting agents such as coarse barite, coarse calcium carbonate or coarse hematite may also be used as weighting agents in the present context, inorganic cements comprise, but are not limited to, Portland cement, calcium aluminate cement, lime/silica blends, blast furnace slag, fly ash, Sorel cements, chemically bonded phosphate ceramics and geopoymers.

In a further aspect, embodiments relate to a method for cleaning a section of a wellbore prior to a cementing operation, the wellbore having a casing suspended therein, and having been treated with an oil- or synthetic-base drilling fluid. The method comprises pumping the disclosed wellbore-cleaning compositions into the wellbore, thereby providing clean and water-wet casing and borehole surfaces. The clean surface will provide superior bonding of cement to the cleaned surfaces.

The wellbore-cleaning compositions according to the present disclosure may be pumped alone, ahead of a conventional water-base spacer fluid or scavenger slurry and behind a conventional water-base spacer fluid or scavenger slurry. In this disclosure, scavenger slurries will be considered to be a type of spacer fluid. In addition, as previously discussed, the disclosed compositions are also effective when incorporated into a carrier fluid such as (but not limited to) a conventional water-base spacer fluid or
scavenger slurry. When the disclosed composition is incorporated within the carrier fluid, the microemulsion concentration in the resulting fluid mixture is preferably between about 5 vol% and 20 vol%, more preferably between about 10 vol% and 15 vol%, and most preferably between about 10 vol% and 12 vol%.

[0033] The wellbore-cleaning composition, the carrier fluid or both may be viscosified. Non-limiting examples of viscosifying agents suitable for use herein include aluminum phosphate ester, alkyl quarternary ammonium bentonite, alkyl quaternary ammonium montmorillonite, xanthan gum, gelatin, pectin, cellulosic derivatives, gum arabic, guar gum, locust bean gum, tara gum, cassia gum, agar, n-octenyl succinylated starch, porous starch, alginates, carrageenates, chitosan, scleroglucan, diutan, weian gum and an organophilic clay such as CLAYTONE™ (available from Southern Clay Products, Inc.; Gonzalez, TX, USA).

[0034] A trimer acid based rheology modifier such as RHEFLAT™ (available from M-I SWACO, Houston, TX, USA) may be also added to the viscosified wellbore-cleaning composition to achieve a flat rheology profile. Without wishing to be bound by any theory, this compound is believed to enhance low-end viscosity and yield point by interacting with fine solids such as organophilic clay and weighting solids such as calcium carbonate, barite, and hematite. The rheology modifier is preferably present in an amount of from about 1 to about 5% by weight of the microemulsion.

[0035] The density of the wellbore-cleaning composition, the carrier fluid or both may also be adjusted by, for example, adding a suitable weighting agent or lightweight material. Suitable weighting agents include (but are not limited to) barite, an inorganic cement, calcium carbonate, hematite, ilmenite, magnesium tetraoxide and silica. Suitable lightweight materials include (but are not limited to) ceramic microspheres, glass microspheres, uintaite, uintahite, coal and nitrogen. It should be noted that weighting agents such as coarse barite, coarse calcium carbonate or coarse hematite may also be used as weighting agents in the present context, inorganic cements comprise, but are not limited to, Portland cement, calcium aluminate cement, lime/silica blends, blast furnace slag, fly ash, Sore! cements, chemically bonded phosphate ceramics and geopolymers.
In yet a further aspect, embodiments relate to methods for cementing a subterranean well having a borehole. The wellbore has a casing suspended therein and contains, or has been treated with, an oil-base or synthetic-base drilling fluid. The methods comprising (i) providing the disclosed wellbore-cleaning composition that comprises a microemulsion, (ii) pumping the composition into the region between the casing and the wellbore, (iii) providing a cement slurry, and (iv) pumping the cement slurry into the region between the casing and the wellbore. The wellbore-cleaning composition removes the drilling fluid from the region between the casing and the wellbore, thereby providing clean and water-wet casing and borehole surfaces.

The wellbore-cleaning composition according to the present disclosure may be pumped alone, ahead of a conventional water-base spacer fluid or scavenger slurry and behind a conventional water-base spacer fluid or scavenger slurry. In this disclosure, scavenger slurries will be considered to be a type of spacer fluid. In addition, as mentioned previously, the disclosed wellbore-cleaning compositions are also effective when incorporated into a carrier fluid such as (but not limited to) a conventional water-base spacer fluid or scavenger slurry. The resulting fluid mixture may be pumped as a spacer fluid. Such mixtures are particularly advantageous from a logistical point of view. When the wellbore-cleaning composition is incorporated within the carrier fluid (e.g., water-base spacer fluid or scavenger slurry), the microemulsion concentration in the resulting fluid mixture is preferably between about 5 vol% and 20 vol%, more preferably between about 10 vol% and 15 vol%, and most preferably between about 10 vol% and 12 vol%.

The wellbore-cleaning composition, the carrier fluid or both may be viscosified. Non-limiting examples of viscosifying agents suitable for use herein include aluminum phosphate ester, alkyl quaternary ammonium bentonite, alkyl quaternary ammonium montmorillonite, bentonite, an inorganic cement, xanthan gum, gelatin, pectin, cellulosic derivatives, gum arabic, guar gum, locust bean gum, tara gum, cassia gum, agar, n-octenyl succinated starch, porous starch, alginates, carrageenates, chitosan, scleroglucan, diutan, welan gum and an organophilic clay such as CLAYSTONE™ (available from Southern Clay Products, Inc., Gonzalez, TX, USA).
A trimer acid based rheology modifier such as RHEFLAT™ (available from M-I SWACO, Houston, TX, USA) may also be added to the viscosified wellbore-cleaning composition to achieve a flat rheology profile. Without wishing to be bound by any theory, this compound is believed to enhance low-end viscosity and yield point by interacting with fine solids such as organophillic clay and weighting solids such as calcium carbonate, barite, and hematite. The rheology modifier is preferably present in an amount of from about 1 to about 5% by weight of the microemulsion.

The density of the wellbore-cleaning composition, the carrier fluid or both may also be adjusted by, for example, adding a suitable weighting agent or lightweight material. Suitable weighting agents include (but are not limited to) barite, an inorganic cement, calcium carbonate, hematite, ilmenite, magnesium tetraoxide and silica. Suitable lightweight materials include (but are not limited to) ceramic microspheres, glass microspheres, untaite, uintahite, coal and nitrogen. It should be noted that weighting agents such as coarse barite, coarse calcium carbonate or coarse hematite may also be used as weighting agents in the present context. Inorganic cements comprise, but are not limited to, Portland cement, calcium aluminate cement, lime/silica blends, blast furnace slag, fly ash, Sorel cements, chemically bonded phosphate ceramics and geopolymers.

Preferably, the density of the spacer fluid is equal to or greater than the density of the SBM/OBM, and equal to or less than the density of the cement slurry.

In all embodiments, the present microemulsions may also be used as a "chemical wash" which is to say pumping an aqueous solution containing the microemulsions as disclosed herein. Said operation might be done after a drilling fluid but before a conventional spacer or scavenger or it can be done after a conventional spacer or scavenger has been pumped.

EXAMPLES

The following examples serve to illustrate the embodiments.
Spacer Fluid Preparation

A microemulsion was formulated for optimal removal of synthetic oil-base-mud ahead of primary cementing operations. Comprising a mixture of solvents, surfactants and water, the resulting microemulsion was a translucent fluid with the solvent portion as the external phase. The microemulsion composition comprised the ingredients shown in Table 1.

<table>
<thead>
<tr>
<th>Compounds</th>
<th>Concentration (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kerosene</td>
<td>35.24</td>
</tr>
<tr>
<td>Ethylene Glycol Monobutyl Ether</td>
<td>35.24</td>
</tr>
<tr>
<td>Alkylbenzene Sulfonate</td>
<td>12.63</td>
</tr>
<tr>
<td>C₉₋₁₁ Alcohol Ethoxylate</td>
<td>5.86</td>
</tr>
<tr>
<td>Sorbitan Monooleate</td>
<td>2.37</td>
</tr>
<tr>
<td>Deionized Water</td>
<td>8.66</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>100.0</strong></td>
</tr>
</tbody>
</table>

Table 1. Microemulsion composition.

For the following examples, the microemulsion of Table 1 was either added to a conventional spacer fluid—MUDPUSH™ II, available from Schlumberger—or a scavenger slurry. The microemulsion concentration was 10 vol%.

The MUDPUSH™ spacer-fluid density varied from 1.42-1.74 kg/L (11.8-14.5 lbm/gal). The base spacer composition is shown in Table 2. Barite or calcium-carbonate weighting agent was used to adjust the spacer-fluid density.

<table>
<thead>
<tr>
<th>Item</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh Water</td>
<td></td>
</tr>
<tr>
<td>MUDPUSH™ II Additive</td>
<td>14.2 g/L (5 lbm/bbl H₂O)</td>
</tr>
<tr>
<td>Polypropylene Glycol Antifoam</td>
<td>0.6 mL/L (0.1 gal/bbl H₂O)</td>
</tr>
<tr>
<td><strong>Agent</strong></td>
<td></td>
</tr>
</tbody>
</table>

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The base spacer fluid (without microemulsion or barite) was prepared in a Waring™ blender using a 1-L blender bowl. The fluid was mixed for 5 minutes at 4,000 RPM. The base fluid was transferred to a 1-L beaker, and a 5-cm diameter impeller-blade stirring device was lowered into the fluid. The mixing speed varied from 500 to 600 RPM, sufficient to create a vortex in the fluid. The barite was added to the fluid, and stirring continued for 30 minutes. The microemulsion was then added, and stirring continued for an additional 10 minutes.

### Test Methods

#### Tile Test

The tile-test procedure is used to determine mud-removal efficiency, and consists of the following steps.

1. Select strips of ceramic tile that are 2.54 cm wide and 7.62 cm long (1 in. wide and 3 in. long), providing a glazed, impermeable surface and a rough, permeable surface.

2. Stand the strip vertically in the mud sample for 30 min, allowing the mud to coat both sides. The mud should cling to both sides and form a cake on the permeable side due to absorption.

3. Secure the tile on a stirrer motor, using a clamp assembly or other available method. For the present experiments, the Applicants secured the tile on the impeller shaft.

4. Fill a beaker with the spacer fluid and immerse the coated tile. The test fluid may be heated to a desired temperature.

5. Rotate the tile at 100 RPM for at least 10 minutes.
6. After removing the tile, the mud should be removed from the glazed surface of the tile if the spacer is performing effectively. Mud removal should be apparent on the glazed side, and a gentle stream of water should remove any spacer residue. Note whether or not a water bead on the tile surface flattens, indicating a water-wet condition.

7. On the permeable side of the tile, some degree of mud removal may occur, but this may not be immediately obvious due to the painting effect of the spacer. A gentle stream of water should remove the weighted spacer from the tile, and any remaining mud will be visible.

8. The process may be repeated to find the optimal contact time.

Compatibility Test

[0049] The Compatibility Test was performed in accordance with the procedure given by American Petroleum Institute Publication API RP 10B-2, "Recommended Practice for Testing Well Cements." This document is also available from the International Organization for Standards, published as ISO 10426-2. Rheological measurements were recorded from the mud, cement and spacer fluids individually. Next, rheological measurements are performed on mixtures of cement/mud, cement/spacer and mud/spacer. The volumetric proportions of the mixtures were 95/5, 75/25, 50/50, 25/75, and 5/95. Finally, rheological measurements were performed on a 25/50/25 mixture of mud/spacer/cement. Testing was performed with a Chan 35 viscometer. The fluid temperature was 150°F (65.5°C). Each fluid was preconditioned in an atmospheric consistometer for 20 minutes at 150°F (65.5°C).

[0050] Fluid compatibility was assessed by calculating the R-Index Value—R. The calculation is as follows: 

\[ R = \frac{\text{Highest 100-RPM reading from a mixture} - \text{Highest 100-RPM reading from an individual fluid}}{\text{Highest 100-RPM reading from an individual fluid}} \]

The compatibility assessment is given in Table 3.

<table>
<thead>
<tr>
<th>If ( R )</th>
<th>Fluids</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>( R &lt; 0 )</td>
<td>are compatible</td>
<td></td>
</tr>
<tr>
<td>( 0 &lt; R &lt; 40 )</td>
<td>Fluids are compatible, BUT friction pressures should be verified to avoid fracturing the formation</td>
<td></td>
</tr>
<tr>
<td>( 41 &lt; R &lt; 70 )</td>
<td>are slightly incompatible. Additional testing is required.</td>
<td></td>
</tr>
</tbody>
</table>
Casing Water Wetting Test (CWWT)

[0051] The Casing Water Wetting Test was employed to determine the mud-removal effectiveness of the surfactant. The test was performed with 4-in. by 1-in. (10.2-cm by 2.54-cm) casing coupons. The procedure is as follows.

[0052] The spacer was preheated to 150°F (65.5°C) for 30 minutes, and transferred to a cup from a Chan 35 rotational viscometer (available from Chandler Engineering, Broken Arrow, OK, USA).

[0053] A casing coupon was statically submerged for 10 minutes in 1.52-kg/L (12.7-lbm/gal) synthetic oil-base mud, also preheated to 150°F (65.5°C).

[0054] The coupon was removed from the oil-base mud, and the convex face of the coupon was cleaned with paper.

[0055] The coupon was transferred to the viscometer cup containing the spacer. With the mud-covered face facing the inside of the cup, the coupon was fixed inside the cup such that the lower 2/3 was submerged in the spacer.

[0056] The viscometer rotor, without the bob, was lowered into the spacer fluid in the cup. The rotor was then operated at 100 RPM for 30 minutes.

[0057] The coupon was removed, and the qualitative evaluation of water wetting proceeded. A piece of Teflon tape (as it closely represents an oil-wet surface) was placed on the coupon. A 20-microliter drop of distilled water was placed on the surfaces of the Teflon tape, the untreated oil-wet area of coupon, and the 2/3 of the coupon that had been submerged in the spacer. The diameters of the droplets were measured.

[0058] The diameter ratio between the droplet on Teflon (D1) and that on the spacer-treated surface (D2) was calculated. Then, the guideline shown in Table 4 was used to determine the contact angle and the water wettability.

| R > 71 | Definitely Incompatible. An alternative formulation must be found. |

Table 3. Definition of R-Index value.
The grid test measures the capability of a surfactant to remove a gelled layer of mud with gentle erosion. A 30-mesh metallic grid was placed over a closed rotor on a Chan 35 viscometer. The grid and rotor assembly were weighed and recorded as \( W_1 \). The grid and rotor assembly were then covered for 10 minutes with 1.52-kg/L (12.7-lbm/gal) synthetic oil-based mud, preheated to 150°F (65.5°C). They were removed, weighed and recorded as \( W_2 \). The grid and rotor assembly were reattached to the viscometer and submerged in spacer fluid, also preheated to 150°F (65.5°C), and rotated at 100 RPM for 30 minutes. They were then removed, weighed and recorded as \( W_3 \). The mud removal percentage (%) was calculated by the following equation.

\[
\text{Mud Removal (\%) } = \left[ 1 - \frac{(W_3 - W_1)}{(W_2 - W_1)} \right] \times 100
\]

Wettability Test (Reverse Emulsion Test)

The spacer and mud were conditioned at 150°F (65.5°C) for 20 minutes in atmospheric consistometers. Then, the spacer was placed in a heated Waring-blender bowl, equipped with a conductivity probe and meter. Sufficient spacer was added to cover the probe and reset the conductivity meter to 3.0 mA. The spacer was removed from the blender bowl, and 250 mL of synthetic oil-based mud (SBM) were placed in the preheated Waring blender, and agitated for 2 minutes at a speed sufficient to observe a visible vortex. The spacer was slowly added to the SBM in 10- to 20-mL increments. The

<table>
<thead>
<tr>
<th>Diameter ratio, D2/D1</th>
<th>Contact Angle (degrees)</th>
<th>Water Wettability</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>150</td>
<td>Poor Wetting &gt; 90°</td>
</tr>
<tr>
<td>1.3</td>
<td>90</td>
<td>90°&lt; Fair Wetting &gt;30°</td>
</tr>
<tr>
<td>2.1</td>
<td>30</td>
<td>Good Wetting &lt;30°</td>
</tr>
<tr>
<td>2.9</td>
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<tr>
<td>6.5</td>
<td>0</td>
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</tbody>
</table>

Table 4. Guideline for the Contact Angle and Water Wettability
conductivity value of the mixture was observed and recorded after each increment. When the total volume of mud and spacer reached 500 mL, 250 mL of the SBM-spacer mixture were removed. The remaining 250 mL of SBM-spacer mixture were agitated for 2 minutes at 150°F (65.5°C), again at a speed sufficient to observe a vortex. Then, additional spacer was added in 10- to 20 mL increments until the 3.0 mA reading was attained.

Example 1—Tile Test

[0061] A 1.68 kg/L (14.0 lbm/gal) MUDPUSH™ spacer fluid (of Table 2), weighted with barite was combined with the microemulsion of Table 1. The microemulsion concentration in the spacer-fluid/microemulsion mixture was 10 vol%. The mud was a 1.42 kg/L (11.8 lbm/gal) NOVAPLUS™ mud (available from MI-SWACO, Houston, TX, USA).

[0062] The results indicated that, after 10 minutes of stirring, the mud was effectively removed from the tile surface, and the surface was water wet.

Example 2—Compatibility Tests

[0063] Fluid-compability tests were performed between spacer fluids containing the microemulsion of Table 1 and various synthetic-base muds.

[0064] Table 5 shows the compatibility-test result for the following fluid combination — 1.32 kg/L RHELIAN™ mud (available from MI-SWACO, Houston, TX, USA); 1.41 kg/L spacer fluid: 53.5 wt% microemulsion of Table 1, 1.2 wt% CLAYTONE™ AF, 0.5 wt% RHEFLAT™ (available from MI-SWACO), 44.8 wt% calcium carbonate. The test temperature was 48.3°C (119°F). The R-Index was -6, indicating fluid compatibility.

<table>
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<tr>
<th>Fluid</th>
<th>100-RPM Reading</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.32 kg/L (11.0 lbm/gal) RHELIAN™ Mud</td>
<td>35</td>
</tr>
</tbody>
</table>
Table 5. Microemulsion Spacer/Synthetic-base mud compatibility evaluation.

Table 6 shows the compatibility-test result for the following fluid combination—
1.32 kg/L RHELIANT™ mud (available from MI-SWACO, Houston, TX, USA); 1.56 kg/L spacer fluid: 52.0 wt% microemulsion of Table 1, 1.0 wt% CLAYTONE™ AF, 0.5 wt% RHEFLAT™ (available from MI-SWACO), 46.5 wt% barite. The test temperature was 48.3°C (119°F). The R-Index was -3, indicating fluid compatibility.
Table 7 shows the compatibility-test results for the following fluid combination — 1.48 kg/L ACCOLADE™ mud (available from Baroid Fluid Services, Houston, TX, USA); 1.62 kg/L spacer fluid: 48.6 wt% microemulsion of Table 1, 1.0 wt% CLAYTON E™ AF, 0.5 wt% RHEFLAT™, 49.9 wt% barite. The test temperature was 65.6°C (150°F). The R-Index was -4, indicating fluid compatibility.

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<th>Fluid</th>
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<tr>
<td>1.48 kg/L (12.4 lbm/gal) ACCOLADE™ Mud</td>
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<tr>
<td>1.62 kg/L (13.5 lbm/gal) Microemulsion Spacer Fluid</td>
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</tr>
<tr>
<td>5:95 Spacer:Mud</td>
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<tr>
<td>25:75 Spacer:Mud</td>
<td>30</td>
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<tr>
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<tr>
<td>75:25 Spacer:Mud</td>
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<td>95:5 Spacer:Mud</td>
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</tr>
</tbody>
</table>

**R-Index:** -4

Table 7. Spacer/Synthetic-base mud compatibility evaluation.

Table 8 shows the compatibility-test results for the following fluid combination — 1.50 kg/L NOVAPLUS™ mud; 1.62 kg/L spacer fluid: 50.0 wt% microemulsion of Table 1, 0.9 wt% CLAYTONE™ AF, 0.5 wt% RHEFLAT™, 48.6 wt% barite. The test temperature was 48.9°C (120°F). The R-Index was -2, indicating fluid compatibility.

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<thead>
<tr>
<th>Fluid</th>
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<tr>
<td>1.50 kg/L (12.5 lbm/gal) NOVAPLUS™ Mud</td>
<td>18</td>
</tr>
<tr>
<td>1.62 kg/L (13.5 lbm/gal) Microemulsion Spacer Fluid</td>
<td>33</td>
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Table 9 shows the compatibility-test results for a scavenger slurry/microemulsion spacer fluid combination. The scavenger slurry had the following composition: LaFarge Class G + 8% fumed silica (BWOC) + 0.2% polyamine sulfonate (BWOC) + 0.8% polyvinyl alcohol (BWOC) + 1.5% sodium glucoheptonate (BWOC) + 0.2% polypropylene glycol (BWOC) + 0.1% hydroxyethyl cellulose (BWOC) + 25% ceramic microspheres (BWOC) + 0.2 diutan gum. The spacer fluid had the following composition: 53.5 wt% microemulsion of Table 1, 1.2 wt% CLAYTONE™ AF, 0.5 wt% RHEFLAT™ (available from Mi-SWACO), 44.8 wt% calcium carbonate. The test temperature was 48.3°C (119°F). The R-Index was 1, indicating that the fluids were compatible, but caution should be exercised with regard to friction pressure.

<table>
<thead>
<tr>
<th>Fluid</th>
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<tr>
<td>1.46 kg/L (12.2 lbm/gal) Scavenger Slurry</td>
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<td>1.41 kg/L (11.8 lbm/gal) Microemulsion Spacer Fluid</td>
<td>60</td>
</tr>
<tr>
<td>5:95 Spacer:Scavenger Slurry</td>
<td>183</td>
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<td>25:75 Spacer:Scavenger Slurry</td>
<td>177</td>
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<td>50:50 Spacer:Scavenger Slurry</td>
<td>140</td>
</tr>
<tr>
<td>75:25 Spacer:Scavenger Slurry</td>
<td>63</td>
</tr>
</tbody>
</table>
Table 9. Spacer/Synthetic-base mud compatibility evaluation.

Table 10 shows the compatibility-test results for a scavenger slurry/microemulsion spacer fluid combination. The scavenger slurry had the following composition: LaFarge Class G + 0.08% hydroxyethyl cellulose (BWOC) + 5% bentonite (BWOC) + 0.2% polypropylene glycol (BWOC) + 1.0% polynaphthalene sulfonate (BWOC) + 0.3% AMPS/acrylamide copolymer (BWOC) + 0.1% sodium glucoheptonate (BWOC) + 0.2% diutan gum. The spacer fluid had the following composition: 53.5 wt% microemulsion of Table 1, 1.2 wt% CLAYTON™ AF, 0.5 wt% RHEFLAT™ (available from MI-SWACO), 44.8 wt% calcium carbonate. The test temperature was 48.3°C (119°F). The R-Index was -8.5, indicating that the fluids were compatible, but caution should be exercised with regard to friction pressure.

<table>
<thead>
<tr>
<th>Fluid</th>
<th>100-RPM Reading</th>
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<tbody>
<tr>
<td>1.46 kg/L (12.2 lbm/gal) Scavenger Slurry</td>
<td>164</td>
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<tr>
<td>1.41 kg/L (11.8 lbm/gal) Microemulsion Spacer Fluid</td>
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<td>95:5 Spacer:Scavenger Slurry</td>
<td>63.5</td>
</tr>
</tbody>
</table>

Table 10. Spacer/Synthetic-base mud compatibility evaluation.

Compatibility testing was performed for a spacer fluid/synthetic-base mud combination. The spacer fluid had the following composition: 1.68 kg/L (14.0 lbm/gal)
MUDPUSH™ II spacer (of Table 2), weighted with barite + 10 vol% microemulsion of Table 1. The synthetic-base mud was 1.41 kg/L (11.8 lbm/gal) NOVAPLUS™. The test temperature was 48.3°C (119°F). The R-Index result was -4, indicating fluid compatibility.

[0071] Compatibility testing was performed for a spacer fluid/cement-slurry combination. The spacer fluid had the following composition: 1.68 kg/L (14.0 lbm/gal) MUDPUSH™ II spacer (of Table 2), weighted with barite + 10 vol% microemulsion of Table 1. The cement slurry had the following composition: 1.97 kg/L (16.4 lbm/gal) LeHigh Class H cement + 35% silica flour (BWOC) + 0.2% polypropylene glycol (BWOC) + 0.2% polynaphthalene sulfonate (BWOC) + 0.2% AMPS/Acrylamide Copolymer (BWOC) + 0.04% sodium glucoheptonate (BWOC). The test temperature was 48.9°C (120°F). The R-Index result was 6, indicating that the fluids were compatible, but caution should be exercised with regard to friction pressure.

Example 3—Casing Water Wetting Test

[0072] A casing water wetting evaluation was conducted involving a spacer fluid and an 1.52 kg/L (12.7 lbm/gal) RHELIANT™ mud. The spacer fluid had a density of 1.74 kg/L (14.5 lbm/gal), and the composition was as follows: MUDPUSH™ II spacer (of Table 2), weighted with barite + 10 vol% microemulsion of Table 1. The contact angle was 33.8°, inducing good water-wetting properties.

Example 4—Grid Cleaning

[0073] A grid test was conducted with a spacer fluid and a synthetic-base mud. The spacer-fluid density was 1.68 kg/L (14.0 lbm/gal) and its composition was as follows: MUDPUSH™ II spacer (of Table 2), weighted with barite + 10 vol% microemulsion of Table 1. The mud was 1.42 kg/L (11.8 lbm/gal) NOVAPLUS™.

[0074] The results of the grid test, shown in Fig. 1, indicate mud removal efficiency of about 100%. Most of the mud was removed within about 10 min.
Example 5—Wettability (Reverse Emulsion)

A wettability test was conducted involving a spacer fluid and a synthetic-base mud. The test temperature was 48.9°C (120° F). The spacer-fluid density was 1.68 kg/L (14.0 lbm/gal) and its composition was as follows: MUDPUSH™ II spacer (of Table 2), weighted with barite + 10 vol% microemulsion of Table 1. The mud was 1.42 kg/L (14.8 lbm/gal) NOVAPLUS™. A spacer volume of about 65% was required to reach the 3-mA set point.

Example 6—Contamination Evaluation

The effect of spacer contamination on cement-slurry performance was evaluated. The spacer-fluid density was 1.74 kg/L (14.5 lbm/gal) and its composition was as follows: MUDPUSH™ II spacer (of Table 2), weighted with barite + 10 vol% microemulsion of Table 1. The cement-slurry density was 1.97 kg/L (16.4 lbm/gal) and its composition was as follows: Lehigh Class H + 35% silica flour (BWOC) + 0.2% polypropylene glycol (BWOC) + 0.2% polynaphthalene sulfonate (BWOC) + 0.2% AMPS/acrylamide copolymer (BWOC) + 0.15% sodium glucoheptonate (BWOC).

The thickening-time and compressive-strength performance of the neat cement slurry, and a 90:10 by volume blend of cement slurry and spacer, was measured. The tests conformed to the recommended practices published in American Petroleum Institute Publication API RP 10B-2, "Recommended Practice for Testing Well Cements." The results are shown in Table 1.

<table>
<thead>
<tr>
<th>Mixture</th>
<th>Thickening Time to 70 Bc (hr:min)</th>
<th>50 psi Compressive Strength (hr:min)</th>
<th>500 psi Compressive Strength (hr:min)</th>
<th>12 hr Compressive Strength (psi)</th>
</tr>
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<td>Cement</td>
<td>3:54</td>
<td>6:22</td>
<td>7:03</td>
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Table 3. Effects of spacer-fluid contamination on cement-slurry performance.
CLAIMS

1. A wellbore-cleaning composition, comprising a microemulsion that comprises a solvent, a co-solvent, a water-wetting surfactant, a cleaning surfactant, an emulsifying surfactant and an aqueous fluid.

2. The composition of claim 1, wherein the microemulsion comprises:
   - 10%-45% by weight solvent, wherein the solvent comprises an aliphatic solvent;
   - 10%—40% by weight co-solvent, wherein the co-solvent comprises a glycol ether;
   - 1%—10% by weight water-wetting surfactant, wherein the water-wetting surfactant comprises a linear ethoxylated alcohol;
   - 5%-20% by weight cleaning surfactant, wherein the cleaning surfactant comprises one or more salts of n-alkylbenzenesulfonic acid;
   - 1%-5% by weight emulsifying surfactant, wherein the emulsifying surfactant comprises a polysorbate; and
   - 1%—20% by weight aqueous fluid.

3. The composition of claim 1, wherein the microemulsion further comprises:
   - a non-ionic surfactant comprising an alkylpolyglycoside, present at a concentration between about 1% and 6% by weight; and
   - a non-ionic co-surfactant comprising an alcohol alkoxylate, present at a concentration between about 1% and 5% by weight.

4. The composition of claim 1, further comprising a viscosifying agent that comprises one or more members of the group comprising: aluminum phosphate ester, bentonite, alkyl quaternary ammonium bentonite, alkyl quaternary ammonium montmorillonite, an inorganic cement, xanthan gum, gelatin, pectin, cellulosic
derivatives, gum arabic, guar gum, locust bean gum, tara gum, cassia gum, agar, n-octenyl succinated starch, porous starch, alginates, carrageenates, chitosan, scleroglucan, diutan, welan gum and an organophilic clay.

5. The composition of claim 1, further comprising a carrier fluid, the carrier fluid comprising an aqueous fluid and a viscosifying agent that comprises one or more members of the group comprising: aluminum phosphate ester, bentonite, alkyl quarternary ammonium bentonite, alkyl quarternary ammonium montmorillonite, an inorganic cement, xanthan gum, gelatin, pectin, cellulose derivatives, gum arabic, guar gum, locust bean gum, tara gum, cassia gum, agar, n-octenyl succinated starch, porous starch, alginates, carrageenates, chitosan, scleroglucan, diutan, welan gum and an organophilic clay.

6. The composition of claim 5, wherein the microemulsion concentration in the carrier-fluid/microemulsion mixture is between about 5 vol% and 20 vol%.

7. A method for cleaning a section of a wellbore prior to a cementing operation, the wellbore having a casing suspended therein, and containing or having been treated with an oil- or synthetic-base drilling fluid, the method comprising the pumping of a wellbore-cleaning composition into the wellbore, the composition comprising a microemulsion, the microemulsion comprising a solvent, a co-solvent, a water-wetting surfactant, a cleaning surfactant, an emulsifying surfactant and an aqueous fluid.

8. The method of claim 7, wherein the microemulsion comprises:

30%–45% by weight solvent, wherein the solvent comprises an aliphatic solvent;
30%–40% by weight co-solvent, wherein the co-solvent comprises a glycol ether;
1%–10% by weight water-wetting surfactant, wherein the water-wetting surfactant comprises a linear ethoxylated alcohol;
5%-20% by weight cleaning surfactant, wherein the cleaning surfactant comprises one or more salts of n-alkylbenzenesulfonic acid;
1%-5% by weight emulsifying surfactant, wherein the emulsifying surfactant comprises a polysorbate; and
1%-20% by weight aqueous fluid.

9. The method of claim 7, wherein the microemulsion further comprises:
a non-ionic surfactant comprising an alkylpolyglycoside, present at a concentration between about 1% and 6% by weight; and
a non-ionic co-surfactant comprising an alcohol alkoxylate, present at a concentration between about 1% and 5% by weight.

10. The method of claim 7, wherein the composition further comprises a viscosifying agent that comprises one or more members of the group comprising: aluminum phosphate ester, bentonite, alkyl quarternary ammonium bentonite, alkyl quaternary ammonium montmorillonite, an inorganic cement, xanthan gum, gelatin, pectin, cellulosic derivatives, gum arabic, guar gum, locust bean gum, tara gum, cassia gum, agar, n-octenyl succinated starch, porous starch, alginites, carrageenates, chitosan, scleroglucan, diutan, welan gum and an organophilic clay.

11. The method of claim 8, wherein the composition is pumped into the wellbore by itself or diluted in water, ahead of a conventional spacer fluid or scavenger slurry, behind a conventional spacer fluid or scavenger slurry, or is incorporated into a carrier fluid and pumped within the resulting mixture.

12. The method of claim 11, wherein the carrier fluid comprises an aqueous fluid and a viscosifying agent that comprises one or more members of the group comprising: aluminum phosphate ester, bentonite, alkyl quarternary ammonium bentonite, alkyl quaternary ammonium montmorillonite, an inorganic cement, xanthan gum, gelatin, pectin, cellulosic derivatives, gum arabic, guar gum, locust bean gum, tara gum, cassia
gum, agar, n-octenyl succinated starch, porous starch, alginates, carrageenates, chitosan, scleroglucan, diutan, welan gum and an organophiliic clay.

13. The method of claim 11, wherein the microemulsion concentration in the carrier fluid/microemulsion mixture is between about 5 vol% and 20 vol%.

14. A method for cementing a subterranean well having a wellbore, the wellbore having a casing suspended therein, and containing or having been treated with an oil-base or synthetic-base drilling fluid, comprising:
(i) providing a wellbore-cleaning composition comprising a microemulsion, the microemulsion comprising a solvent, a co-solvent, a water-wetting surfactant, a cleaning surfactant, an emulsifying surfactant and an aqueous fluid;
(ii) pumping the composition into the region between the casing and the wellbore;
(iii) providing a cement slurry; and
(iv) pumping the cement slurry into the region between the casing and the wellbore.

15. The method of claim 14, wherein the microemulsion comprises:

10%-45% by weight solvent, wherein the solvent comprises an aliphatic solvent;
10%-40% by weight co-solvent, wherein the co-solvent comprises a glycol ether;
1%-10% by weight water-wetting surfactant, wherein the water-wetting surfactant comprises a linear ethoxylated alcohol;
5%-20% by weight cleaning surfactant, wherein the cleaning surfactant comprises one or more salts of n-alkylenesulfonic acid;
1%-5% by weight emulsifying surfactant, wherein the emulsifying surfactant comprises a polysorbate; and
1%-20% by weight aqueous fluid.

16. The method of claim 14, wherein the microemulsion further comprises:
a non-ionic surfactant comprising an alkylpolyglycoside, present at a concentration between about 1% and 6% by weight; and a non-ionic co-surfactant comprising an alcohol alkoxy!ate, present at a concentration between about 1% and 5% by weight.

17. The method of claim 14, wherein the composition further comprises a viscosifying agent that comprises one or more members of the group comprising: aluminum phosphate ester, bentonite, alkyl quarternary ammonium bentonite, aikyl quarternary ammonium montmorillonite, an inorganic cement, xanthan gum, gelatin, pectin, cellulosic derivatives, gum arabic, guar gum, locust bean gum, tara gum, cassia gum, agar, n-octenyl succinated starch, porous starch, alginates, carrageenates, chitosan, scleroglucan, diutan, welan gum and an organophilic clay.

18. The method of claim 14, wherein the composition is pumped into the wellbore by itself or diluted in water, ahead of a conventional spacer fluid or scavenger slurry, behind a conventional spacer fluid or scavenger slurry, or is incorporated into a carrier fluid and pumped within the resulting mixture.

19. The method of claim 18, wherein the carrier fluid comprises an aqueous fluid and a viscosifying agent that comprises one or more members of the group comprising: aluminum phosphate ester, bentonite, alkyl quarternary ammonium bentonite, alkyl quarternary ammonium montmorillonite, an inorganic cement, xanthan gum, gelatin, pectin, cellulosic derivatives, gum arabic, guar gum, locust bean gum, tara gum, cassia gum, agar, n-octenyl succinated starch, porous starch, alginates, carrageenates, chitosan, scleroglucan, diutan, welan gum and an organophilic clay.

20. The method of claim 18, wherein the microemulsion concentration in the carrier fluid/microemulsion mixture is between about 5 vol% and 20 vol%.
Figure 1
INTERNATIONAL SEARCH REPORT

PCT/EP2011/056804

A. CLASSIFICATION OF SUBJECT MATTER
INV. C09K8/524
ADD.
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

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

Electronic database consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<td>WO 2009/136364 A1 (SCHLUMBERGER CA LTD [CA]; SCHLUMBERGER SERVICES PETROL [FR]; SCHLUMBERGER) 12 November 2009 (2009-11-12) claimed 1-8 the whole document</td>
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Date of the actual completion of the international search: 15 July 2011
Date of mailing of the international search report: 02/08/2011

Name and mailing address of the ISA:
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NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040,
Fax: (+31-70) 340-3016

Authorized officer:
Straub, Thomas

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