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Micro-composite as a Fluid Loss Additive for Oil Well Cementing

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Abstract

To ensure long term well integrity, it is crucial to maintain the properties of an oil well cementing slurry until it sets. High fluid loss can change the properties of cement slurry greatly causing improper cement displacement and setting. It is essential to reduce the fluid loss particularly for cementing of long horizontal wells, high-pressure, high-temperature (HPHT) wells, and wells with potential for gas migration. Synthetic polymers such as vinyl sulphonates, polyethylenimine (PEI), or styrene-butadiene (SBR) latexes are the only fluid loss additives suitable for HPHT oil well cementing. The commercially available HPHT fluid loss additives are expensive and have one or more of the following drawbacks; increases the viscosity of the cement slurry at low temperature, retardation of the cement hardening process or poor functionality in salt-containing cement, etc. Moreover, in strict HSE regulated areas like the Norwegian Continental Shelf, chemicals such as SBR latex and PEI are banned.

While a wide range of HPHT fluid loss chemistries are available, there is a need for cost efficient additives for HPHT cementing. Multifunctional additives providing filtration control, mitigating the risk of gas migration and improving the cement bonding are highly desirable.

This paper presents a newly developed micro-composite material and its application as fluid loss additive for oil well cementing. Successful tests were conducted in cement slurries in a wide range of densities from 1.30 - 2.40 SG. Saturated salt-containing cement was also used. The filtration control was tested at elevated temperature up to 200°C with positive results. As such this composite material can be considered as a universal fluid loss additive for oil well cementing covering a wide range of temperatures and cement systems. Supplementary, micro-composite material provides gas migration control.

Introduction

The main function of an oil well cement is to maintain its integrity throughout its life time. Oil well cement is introduced into the well in form of a slurry. It is crucial to maintain the properties of cement slurry until it sets. Any significant change in the slurry properties such as the rheological properties, or the volume of fluid loss during the displacement until it hardens, can cause cement failure, which should be avoided at all costs. Generally, oil well cement is formulated with water content of 40-45wt% by the weight of cement. The loss of a large amount of water will lead to significant change in water/cement ratio causing improper cement displacement and unpredictable cement placement and setting. Hence, reducing the fluid loss particularly for cementing of long horizontal wells, HPHT wells with narrow pressure windows, and wells with potential for gas migration is essential.

Fluid loss control mechanisms rely on impairing the permeability of the formation through creating filter cakes both inside the formation (internal filter cake) and on the surface of the formation (external filter cake). The permeability of the filter cake is reduced through plugging pore throats by swollen particles such as hydrocolloidal particles or by inorganic particles such as Microsilica. The permeability of the formation is further reduced by increasing the viscosity of the aqueous continuous phase using highly water-soluble polymers. An increase in fluid viscosity reduces the flow rates through filter cake. Below are the commonly used fluid loss additives for oil well cementing.

Chemicals

The main selection criteria for fluid loss additives are the technical suitability and cost. The temperature of the zone to be cemented dictates the selection of the fluid loss additive. Fluid loss additives are classified according to their thermal stability into two main groups:

A) Low -medium temperature FLAC (<150°C)

There are wide varieties in chemistry, which can be used in this temperature range. Examples of additives are modified cellulose like hydroxyethyl cellulose, polyvinyl alcohol, styrene-butadiene latexes, modified humic acid and lignosulphonates. These additives function according to their water holding capacity or by plugging the pore throat of the formation.

B) High temperature FLAC (>150°C)

The fluid loss additives for HPHT oil well cementing are few and mainly synthetic polymers such as vinyl sulphonates copolymers, polyethyleneimine, or styrene-butadiene latexes. These chemicals exhibit high resistance to thermal degradations. Typical dosage for HPHT polymers is in the range 0.5-1.5%BWOC.

Styrene-butadiene latexes (SBR) used as FLA are normally 50wt.-% polymer dispersions. Since SBR is water insoluble polymeric particles, the amount needed to control fluid loss and gas migration is approximately ten times higher compared to water soluble polymers. SBR latexes require additional high-temperature stabilizer, which are surfactants, to prevent thermal flocculation for cementing application at elevated temperature >150°C.

The currently used fluid loss additives for oil well cementing, particularly the class used below HPHT conditions may have one or more of the following drawbacks:

1) High cost due to the complex production processes.
2) Generate high viscosity at low temperature.
3) Retard the cement hydration and setting process.
4) Do not function well in salt containing cement.
5) Suffer from thermal degradation at extreme high temperatures.
6) May not work at low temperatures.
7) Do not meet the HSE regulation in some regions

In this paper, we present an innovative micro-composite as a universal additive for oil well cementing. The new micro-composite eliminates the challenges mentioned above. The new composite material has a dual function; fluid loss and gas migration control, providing clear operational and cost benefits to the Operators.

Results and Discussion

Chemicals

G-cement is supplied by Dyckerhoff (fineness is 326 m²/kg). Hollow-sphere was used a light weight material. Manganese tetraoxide (Mn_3O_4) was used as a weight material. Various commercially available HPHT polymeric fluid loss additives, dispersant and retarder were used. Silica flour, Milisil M10 (23µm and 2.65sg) supplied by Sibelco were used to formulate cement slurries.

Equipment

Standard oil well cementing equipment was used: a constant speed mixer, HPHT fluid loss system at various temperatures with a differential pressure of 1000 psi, Fann 35 viscometer, HPHT consistometer, UCA, SGSA, SEM and gas migration analyzer. Cement tests were conducted according to API Recommended Practice 10B-2.

Microsilica as an additive for oil well cementing

Microsilica or silica fume consists of amorphous and spherical microfine silica particles with an average particle size (D50) of < 200nm measured by light scattering method. Microsilica is widely used in oil well cementing as a gas migration additive or as an extender to lower the density of cement slurry. The size of Microsilica particles is much smaller than the size of cement grains. As a result, cement slurry containing Microsilica builds a much tighter filter cake during filtration. Furthermore, Microsilica reacts chemically with hydrated cement and forms calcium silicate hydrates, which are cement phases with tight structure. Generally, hard or set oil-well cement with sufficient amount of Microsilica (>10%BWOC) is impermeable.

While Microsilica contributes in lowering the fluid loss, it does not control / prevent it completely and an additional fluid loss additive is required. Thus, Microsilica is combined with fluid loss additive to provide gas tight cement. The addition of Microsilica and fluid loss additive to an oil-well cement have generally synergistic effect. However, Microsilica might have a competing effect with fluid loss additive. In case of a synergistic effect, Microsilica leads to reducing the amount of fluid loss additive used to achieve a certain fluid loss volume.

Synthesis and Properties of Micro-composite

This innovative micro-composite was prepared in the laboratory using Microsilica, and water-soluble compounds.
The composite shows the following physicochemical properties.

Table 1: Properties of micro-composite

Parameter	Value
Solid content (wt.%)	20-25
pH	6-8
Viscosity at shear rate 20s^{-1} [mPa.s]	Ca. 2000
PSD measured by light scattering	2-50µm

SEM micrograph as in Figure 1 shows the microstructure of the composite as a large polymeric microstructure filled with Microsilica particles. The PSD in the range 2-50µm as shown in Figure 2.

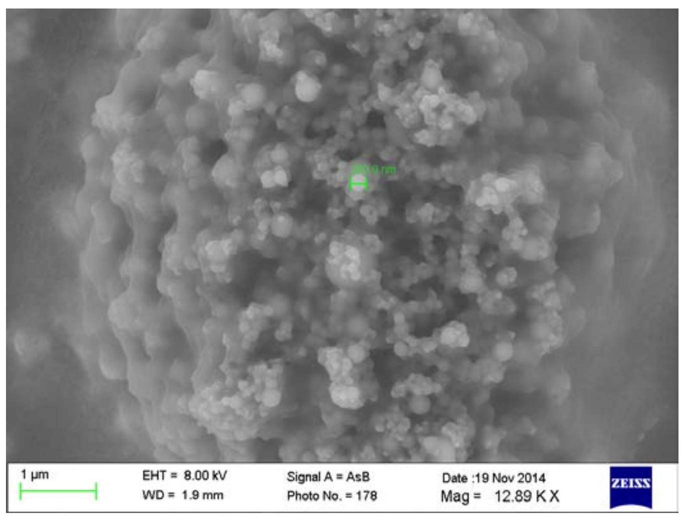

Figure 1: SEM micrograph of micro-composite material.

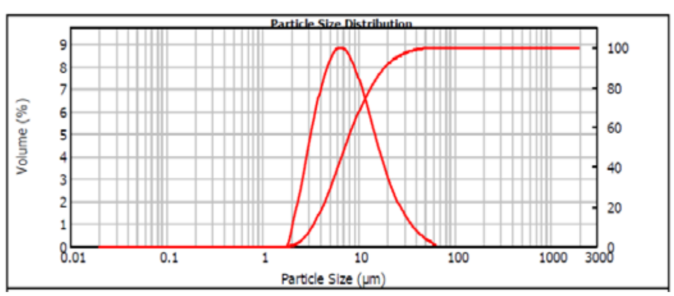

Figure 2: Light scattering particle size distribution of the micro-composite.

Testing Micro-composite as FLA in oil well cementing

This innovative material was tested successfully in cement slurries with a wide range of densities from 1.3- 2.4sg under various temperatures in the range 50-200°C. It was also tested in saturated salt cement formulation (37%BWOW). The examples below illustrate the use of micro-composite for filtration and gas migration control.

1) 1.9sg (15.8ppg) standard oil well cement

Table 1 shows the formula and properties of standard cement slurry containing g-cement, 35% silica flour, HPHT retarder 0.5%BWOC and dispersant 0.5%BWOC. The micro-composite (slurry 2) was tested against HPHT vinyl sulphonate copolymers at 150°C (slurry 1). The active-matter concentration of the fluid loss additives in both slurries 1 and 2 was 1%BWOC.

Table 2: Formulation and properties of 1.9sg standard cement slurry.

HPHT 1.9sg cement slurries	slurry 1		slurry 2	
Water	324,5		302,0	
HT dispersant	4,4		4,4	
FLAC	5,9		29,6	
HT retarder	1,8		1,8	
Silicone defoamer	2,1		2,1	
Silica flour	206,7		206,7	
G-cement	590,6		590,6	
Viscosity readings at rpm	20 ˚C	85 ˚C	20 ˚C	85 ˚C
300	209	132	193	122
200	146	89	135	84
100	79	46	71	45
60	49	28	44	28
30	26	13	23	15
6	7	2	6	4
3	4	1	4	2
PV (cP)	195,0	129,0	183,0	115,5
YP (lbs/100ft^2)	14,0	3,0	10,0	6,5
Density (sg) at 20°C	1,9		1,9	
Fluid loss (ml) at 150°C	47		36	
Filter cake, mm	40		15	

As illustrated in Table1, the new composite provides cement slurry with lower filtration and lower plastic viscosity compared to the reference. The filter cake was thinner for the micro-composite slurry compared to the reference slurry. These are desired properties from a practical point of view.

2) High density cement 2.4sg

As the density of the cement increases, the concentration of fluid loss additives decreases. As shown in Table 2, the equivalent dry concentration of micro-composite was 0.84%BWOC for 2.4sg cement slurry. Additionally, the slurry contains 35% silica flour, Micromax FF 90%BWOC as weight material, HPHT retarder 0.25%BWOC, dispersant 0.75%BWOC and defoamer 0.04gps. The W/C ratio was 45%. The slurry shows low viscosity, a fluid loss of 46mls at 150°C and filter cake of 28mm.

Table 3: Formulation and properties of 2.4sg cement slurry weighted with Micromax® FF.

HPHT 2.4sg cement slurries	slurry 3	
Viscosity readings at rpm	20 ˚C	85 ˚C
300	223	168
200	160	124
100	103	75
60	75	53
30	50	23
6	25	12
3	21	9
PV (cP)	180	139,5
YP (lbs/100ft^2)	43	28,5
Density (sg) at 20°C	2,4	
Fluid loss (ml) at 150°C	46	
Filter cake, mm	28	

4) High temperature cementing

The suitability of micro-composite for HPHT was assessed by testing the filtration at 200°C in 1.68 and 1.90 SGsg cement slurries. The retarder was a mixture of tartaric acid and synthetic polymer. The equivalent dry concentration of micro-composite in slurry 4 was 1.25%BWOC. The increase in FLA concentration is related to the high temperature as well as to the interference from high retarder concentration. Low fluid loss and thin filter cake were achieved.

Table 4: Properties of 1.9sg cement slurry tested at 200°C.

HPHT 1.9sg cement slurries	slurry 4	
Viscosity readings at rpm	20 ˚C	85 ˚C
300	295	153
200	233	108
100	129	58
60	84	37
30	48	20
6	17	5
3	13	3
PV (cP)	249	142,5
YP (lbs/100ft^2)	46	10,5
density (sg) at 20°C	1,9	
Fluid loss (ml) at 200°C	42	
Filter cake, mm	5	

Fluid loss as function of density of cement slurries

Figure 3 shows the performance of micro-composite in wide range of densities of oil well cementing (1.35-2.40 SGsg). The fluid loss was measured at 150°C (302°F). The slurries were designed to obtain a fluid loss <50ml. The concentration of micro-composite in oil well cementing is quite equal to conventional HPHT FLA. Test results showed that the concentration was slightly reduced as the density of cement slurry increased. To the contrary, at low density, higher concentration was used compared to standard cement density (1.90 SG).

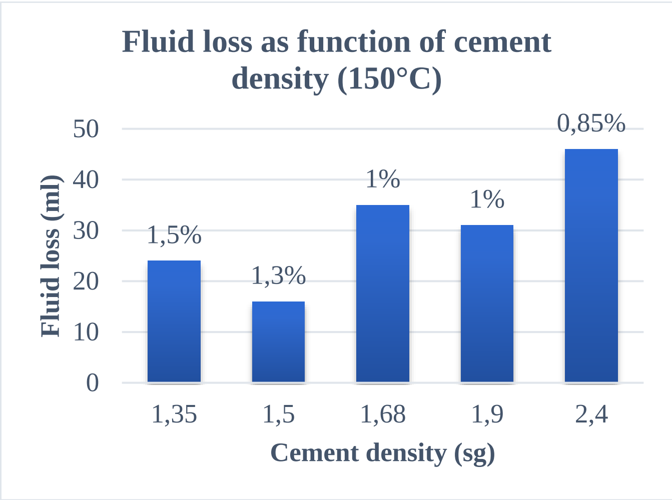

Figure 3: Fluid loss as function of the density of cement slurry. The percentage on the top of the column represents the concentration of micro-composite dry-equivalent by the weight of cement.

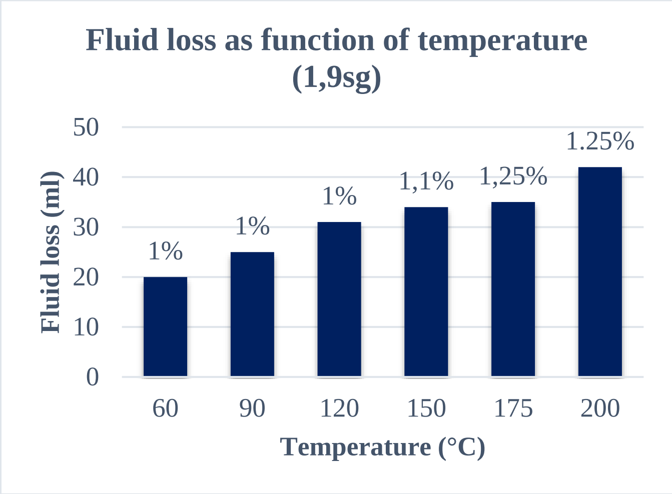

Figure 4: Fluid loss as function of temperature. The percentage on the top of the column represents the concentration of micro-composite dry-equivalent by the weight of cement.

Fluid loss as function of temperature

Results in Figure 4 illustrate the fluid loss as function of temperature for 1.90 SG cement slurries. The slurries were designed with suitable amount of retarder matching the target temperature. Silica flour was added to cement slurries for temperature >110°C to prevent strength retrogression, while it was removed from slurries tested at 60 and 90°C. As the temperature increase, the fluid loss increases. Therefore, a small additional amount of micro-compote was added to keep the fluid loss less than 50ml.

Micro-composite in salt-containing cement slurries

Saturated salt slurries are used in cement slurries through salt domes. Even if most of the drilled and cemented formations are shales (>75%), salt cement is used to protect shale zones from sloughing. Micro-composite was tested in high salinity cement slurries, namely 1.9o?SG slurries prepared with simulated sea water, NaCl 18%BWOW and 37%BWOW. The slurries were tested at a temperature of 150°C. The results listed in Table 5 indicate micro-composite is suitable for highly saturated salt cement. No gelation was observed in the three salt slurries. Salt slurries with micro-composite exhibit low viscosity particularly after conditioning at 85°C, indicating a dispersing effect.

Table 5: Performance of micro-composite in 1.9sg salt cement at 150C.

1.9sg cement slurry	Sea water	18%BWOW	37%BWOW
Fluid loss at 150C	35	20	12
Filter cake (mm)	25	6	6

Gas migration control

During the cement curing process, the cement slurry is initially converted into a gel state and finally into a solid state. To mitigate the risk of gas migration through the cement column during the hardening / curing process, the transition time should be as short as possible and the development of the compressive strength should be as fast as possible. The transition time is the time when static gel strength increases from 50 to 500 lb/100ft^2. It should be less than 40 min and preferably less than 30min to assure gas migration control.

The Static Gel Strength Analyzers (SGSA) use acoustic /ultrasonic attenuation to monitor the hardening process of an oil well cement at downhole conditions with respect to temperature and pressure. The equipment measures the static gel strength development and the compressive strength development of a cement slurry as a function of time.

We observed an additional benefit by using this the newly devolved composite in cement slurry; shortening of the transition time and prevention of gas migration. Figures 5 shows the curing process for the oil well cement slurry 2 as shown in Table 2. This test was done at a temperature of 150°C and pressure of 3000psi. The transit times were just 9min for micro-composite slurry 1 versus 36min for the reference slurry 2. In addition, the compressive strength developed much faster for the slurry 2 formulated with the new micro-composite compared to regular polymeric fluid loss additive (slurry 1). Within 12 hours' curing time, slurry 1 reach compressive strength of 1802psi while slurry 2 reach compressive strength of 2427psi.

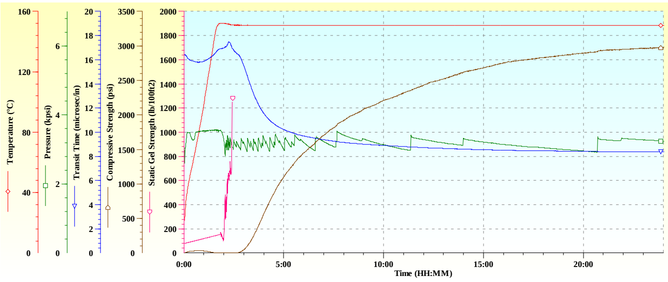

Figure 5: Static Gel Strength analyzer curve for 1.9sg cement slurry prepared with micro-composite.

The cement prepared with micro-composite provides additional benefit to cement slurry preventing the gas migration through shortening the transition time and increasing the early compressive strength. These results were confirmed by using Gas Migration Analyzer as shown in Figure 6. The gas injection pressure remained constant during the entire test until setting of cement slurry.

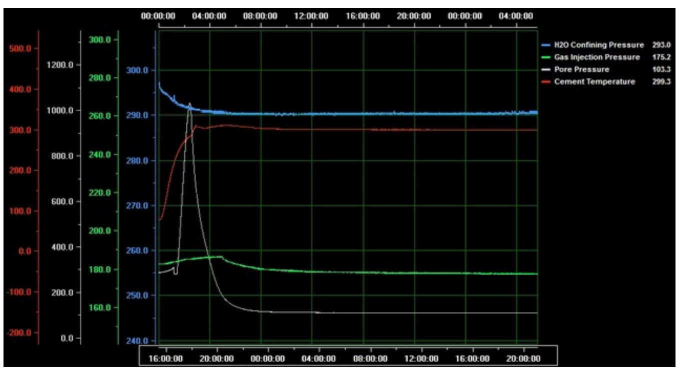

Figure 6: Gas Migration Analyzer for micro-composite 1.9sg cement slurry measured at 150°C.

Compatibility with cementing additives

Micro-composite is compatible with several of the retarders such as synthetic carboxylate polymers, lignosulphonates, or hydroxycarboxylic acids such as tartaric acid or citric acid. Furthermore it is compatible with polymerized naphthalene sulfonate condensates dispersant.

Micro-composite stability

The micro-composite exhibits high viscosity, it is stable against settling over long storage time. The freezing stability was tested in the lab by storing the material for one week at -10°C. The product was thawed at room temperature. The composite recovered its original consistency within a few hours. This indicates the usability of the product when exposed to low temperature.

Conclusions

An innovative micro-composite material has been developed for fluid loss and gas migration control of oil well cementing. This dual function has several benefits. It will minimize the number of additives handled at rig site, thereby reducing cost, and provide a gas-tight cement slurry, which assure robust well integrity. Preventing the annular gas flow through micro-composite will avoid the risk of cement failure, which can have severe consequences.

The enhanced early compressive strength using micro-composite over conventional FLA, shortens the wait on cement time, which again reduces operational time.

Micro-composite was successfully tested in various cement systems at various conditions. It can be considered as a universal fluid loss additive for oil well cementing. Micro-composite can replace various fluid loss additives currently available. This will greatly reduce the need for these various fluid loss additives.

Micro-composite provides better viscosity profile compared to conventional FLA, making it a good candidate for cementing of HPHT wells, horizontal wells and depleted zones.

Acknowledgments

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Nomenclature

API RP = American Petroleum Institute Recommended Practice
gps = gallon per sack. One sack is 94LB (42.2kg)
BWOC = By Weight of Cement
BWOW = By Volume of Water
CSH = Calcium Silicate Hydrate
SEM = Scanning Electron Microscopy
BHCT = bottom hole circulating temperature
BHST = bottom hole static temperature
HEC = hydroxyethyl cellulose
FLAC =
HPHT = high pressure and high temperature
sg = specific gravity (g/cm^3)
NaCl = sodium chloride
ppg = pound per gallon
psi = pounds per square inches
RPM = revolutions per minute
°F = temperature in Fahrenheit

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