

Evaluation of Micronized Ilmenite as Weighting Material in Water-Based Drilling Fluids for HPHT Applications

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Abstract

Barite is the most common weighting material for drilling fluids, which contain several heavy components including lead, cadmium, mercury, and arsenic. Some of these heavy materials can discharge into the sea, which is not allowed especially in the case of oil-based drilling fluid. The supply of barite is geographically limited, with high transportation costs. To overcome the high cost, shortage, and common problems of barite, an alternative weighting material, ilmenite (5 μ m), is introduced which is heavier than barite and more stable at high temperature. Also, the micronized ilmenite was introduced to overcome the ECD challenges in some drilling operations at reasonable cost.

Extensive lab work was done in order to: 1) optimize the rheological properties of the drilling fluid, 2) determine the optimum pH that gives stable dispersion, 3) assess the thermal stability, 4) optimize the filtration parameters (filtrate volume and filter cake thickness), and 5) characterize the ilmenite-based filter cake.

Zeta potential results showed that ilmenite was stable when mixed with water at a pH above 7 and it was dispersed and stable when mixed with the drilling fluid components. Drilling fluids have a density range from 100 to 120 pcf and a plastic viscosity of 28-32 cp. No phase separation was observed after hot rolling for 16 hrs at 300°F. The optimized water-based drilling fluid formula had a small filtrate volume (12 cm³) and thin filter cake (0.2 in.) under dynamic conditions. SEM analysis showed that ilmenite filter cake was heterogeneous and contained ilmenite particles in the layer closer to the rock surface. The layer closer to the drilling surface had a mixture of xanthan gum and modified starch, which were used to optimize the rheological properties at 250°F. This study will provide a complete evaluation of the drilling fluids with ilmenite as a weighting material and will help drilling engineers to better design drilling fluids for HPHT wells.

Introduction

Barite was introduced as a weighting agent in the 1930's. Today, drilling operations are the main applications of barite. Barite is favored for its relative abundance and market availability. About 6 million tons are produced and traded globally every year. Almost half of this is sourced from China, 12% from India and 7-8% from the USA, as well as smaller producers including: Turkey, Morocco and Iran (Newcaster et al. 2007). Prices have increased markedly in the last few years due to lack of API barite reserves suitable for drilling applications (Tran 2007). The supply of barite is geographically limited, with high transportation costs, (Blomberg 1984).

Rae et al. (2001) reported that barite was one of the biggest potential sources of pollution. It contains several heavy metal components including: lead, cadmium, mercury, and arsenic, which can be

dissolved into the sea during the discharge of mud or cuttings. They stated that the heavy toxic metal content of ilmenite was significantly lower than that of barite, and the bioavailability of the heavy metals of ilmenite was also lower.

Haaland et al. (1976) and Tuntland et al. (1981) have introduced iron-based materials such as ilmenite, hematite and others to be used as a weighting material in drilling. They mentioned that ilmenite, as a weighting material, includes attributes such as: high specific gravity, solubility in acid, similar or superior fluid properties compared with barite, and an availability of large ore deposits. Blomberg et al. (1984) evaluated ilmenite as a weighting material in drilling fluids. They mentioned some common problems when using ilmenite were: abrasion, a difficulty to water-wet and disperse, foaming occurring and excessive dust in mud room, which causes cleaning problems. By treating ilmenite with sodium silicate and caustic, the amount of flotation chemicals can be reduced, which helps to overcome problems associated with hydrophobic character, dust, air foaming, and wetting. To reduce the abrasion problem, they recommended using ilmenite with small particles, with a maximum of 3% particles larger than 45 microns.

Idris et al. (1994) studied the use of Malaysian ilmenite as a weighting material in drilling mud. They concluded that the local ilmenite has the potential to be used as a weighting material in drilling. Higher yield point and gel strength were observed, however these problems can be reduced by increasing the lignosulfonate concentration. There was no indication of incompatibility of lignosulfonate in the mud system.

Fjogstad et al. (2000) stated that no disadvantages have been observed in using ilmenite as a weighting material with a maximum of 2.5% particles larger than 45 microns and a maximum of 10% particles less than 1 micron. Ilmenite was more suited to be reused because it has a lower tendency to be ground down to finer materials and there was less of a need for fluid dilution. The laboratory work and field trials demonstrated that the performance of ilmenite was fully equal to, and in some cases represents an improvement compared to barite, as a weighting material in water-based drilling fluid.

Ismail et al. (1999) concluded that a small quantity of ilmenite could produce the same mud weight as barite, which yields lower solids content. The use of ilmenite might reduce formation damage problems, as it experienced lower fluid loss than barite. Saasen et al. (2001) concluded that ilmenite was found to be more suitable than barite for use in drilling fluids on an overall perspective. The drilling fluids had a longer lifetime, a reduced need for dilution, and a reduction in abrasion was observed when using ilmenite as a weighting material.

Lee et al. (2012) studied the effect of HPHT on the rheological properties of the drilling fluid. Amighi and Shahbazi (2010) studied effective ways to avoid barite sag in HPHT deviated wells. They mentioned that from environmental considerations, it has been desirable to exchange barite with ilmenite to eliminate the discharge of heavy metals while drilling with water-based drilling fluids. By adjusting the particle size distribution of ilmenite with the particle mean size to around 10 microns, the erosion was brought down to a level generally underneath the erosion level experienced when drilling with barite. They stated that no dynamic sag was observed while drilling or running casing and liner throughout the drilling period on the wells drilled with ilmenite.

Recently, Al-Bagoury and Steele (2011) reported the use of micronized ilmenite with an average size (D_{50}) of 5 µm as a weighting agent for drilling fluids. It was demonstrated that under similar conditions, micronized ilmenite showed low sag tendency and low plastic viscosity compared to barite. These features are of great importance and can be employed in challenging drilling operations such as horizontal drilling, low margin pressure drop (ΔP), deep water, and slimehole.

The objective of this paper is to evaluate the behavior of micronized ilmenite in water-based drilling fluids. The effect of ilmenite concentration, the type and concentration of the fluid loss agent, and viscosifier on the drilling fluid properties were investigated.

Experimental Studies Water-Based Drilling Fluid

Water-based drilling fluid contained ilmenite (1048.8 g) as a weighting material, a starch derivative (4.8 g) was used as a fluid loss agent, potassium hydroxide (0.9 g) was used for pH control, polyanionic cellulose (Pac LV, 7.2 g) was used as a secondary fluid loss agent, potassium chloride (72 g) was used for controlling the shale inhibition, and acrylic copolymer dispersant (16.8 g) was used for dispersion of ilmenite particles. The drilling fluid was mixed to form 489 cm³.

Results and Discussion

Evaluation of using Acrylic Copolymer as a Dispersant

The formed drilling fluid, which contained acrylic copolymer dispersant, has a density of 147 pcf (2.35 S.G.) and a pH of 8.5. Another formula was prepared without acrylic copolymer dispersant and it has the same properties as mentioned above.

A filtration test was performed using an HPHT filter press under static conditions. The drilling fluid was placed in the cell, the cell was placed in the heating jacket, and the temperature was adjusted to 300°F and 300 psi was the differential pressure.

Fig. 1 shows that the cumulative filtrate volume was high (86.6 cm³) and a high spurt volume was recorded (15 cm³). No filter cake was formed when the cell was opened. There was a viscous fluid which caused this filtrate volume in 30 min.

A filtration test was performed at the same conditions without using acrylic copolymer dispersant. **Fig. 1** shows that the cumulative filtrate volume was small (7.8 cm³) as compared with the previous. As the amount of ilmenite was high (2.35 S.G. drilling fluid), the thickness of the formed filter cake was 1.55 to 2.0 in.

To avoid a high filter cake thickness, it was decided to decrease the amount of ilmenite in the drilling fluid to 400 g (120 pcf mud) and do the same test with and without acrylic copolymer dispersant.

Using acrylic copolymer dispersant, the same results were obtained. The filtrate volume was more than 105 cm³ and no filter cake was formed after 30 min of filtration. While, without acrylic copolymer dispersant, the filtrate volume was 11 cm³, **Fig. 1**, and the filter cake had a thickness of 0.2 in. when using a 10 micron ceramic disk.

The water-based drilling fluid, which contained 400 g ilmenite, had a density of 120 pcf, and plastic viscosity of 2 cp. It was noticed that this formula of drilling fluid had a common problem when using ilmenite; high value of yield point 446 lb/100 ft² and high gel strength value of 18 and 26 for 10 s and 10 min, respectively.

Zeta Potential Analysis

Distribution tests for the drilling fluid were performed using zeta potential analysis. Stable drilling fluid should preferably have a zeta potential out of the range of +/- 30 mV for a water-based solution. It was noted that the drilling fluid with acrylic copolymer dispersant was not stable and precipitated ilmenite particles; while without acrylic copolymer dispersant it was stable for both high and low density drilling fluids, **Table 1**.

A mixture of water and ilmenite was prepared that contains 45 g DI water and 0.05 g ilmenite. The pH was adjusted of a range 3 to 11. **Table 2** summarizes the measurement of zeta potential at different pH values. **Fig. 2** shows that ilmenite was dispersed in DI water at pH > 7. The range of optimum dispersion should preferably be greater than 30 or lower than -30 mV (NBTC 2003).

Optimization of Drilling Fluid Properties

Table 3 summarizes the components that were used to prepare the drilling fluids. Xanthan gum and modified starch were used to control the rheological properties. The modified starch can be used up to 300°F. The amount of ilmenite was varied to control the drilling fluid density.

Drilling Fluid Properties

Table 5 illustrates the drilling fluid properties after hot rolling for 16 hrs at 250°F. As the amount of ilmenite was increased, the density and plastic viscosity were increased. The density of the drilling fluid was increased with increasing the amount of ilmenite and it changed from 100 to 120 pcf as the amount of ilmenite was from 200 to 400 g. The drilling fluid had a near constant pH value with different weights of ilmenite. **Figs. 3** and **4** show the change in the plastic and the apparent viscosity, respectively. It was noticed that the plastic viscosity was out of the safe range (25 to 30 cp) when using 300 g and more of ilmenite. **Fig. 5** shows the change in yield point and gel strength. The drilling fluid with 200 to 300 g of ilmenite has a normal value of the yield point and gel strength, while at 400 g ilmenite these values became high. Zeta potential reading showed that the drilling fluid had a good dispersion of ilmenite, the value was less than -30, which indicated good dispersion, **Fig. 6**.

HPHT Filtration Tests

Fig. 7 shows that the filter cake thickness was 0.22 in. when using 400 g ilmenite as a weighting material to prepare the drilling fluid. The filter cake was then dried at 250°F and it was noted that the top layer of the filter cake contained channels, which gave an indication of the filter cake heterogeneity. This layer contained a high percent of xanthan gum and starch with a low percent of ilmenite particles.

The cumulative filtrate volume was small when using 300 g of ilmenite, as shown in **Fig. 8**. Also the thickness of the filter cake (0.19 in.) was smaller than for 400 g (0.2 in.).

It was noted that by increasing the amount of ilmenite, the rheological properties were high, especially when using 400 g of ilmenite. To solve this issue two methods were applied. The first one was to use lignosulfonate, which acts as a thinner, to reduce the viscosity and the yield point. The second method was decreasing the amount of PAC-R, which was used as a viscofier and for filtration control. In both methods, the amount of xanthan gum was reduced to 0.25 g.

Effect of Lignosulfonate

Table 5 shows that, by adding 4 g lignosulfonate the plastic viscosity was decreased to 13 cp and the yield point was reduced to 6 lb/100 ft². The density of the drilling fluid was 110 pcf and the mixing time of 300 g of ilmenite was only 20 min, and it was easy to mix with the drilling fluid components.

The amount of KOH was increased to 1 g to adjust the pH to a range of 9-11; also the amount of ilmenite was increased to 400 g to increase the density to 120 pcf. The rheological properties of the drilling fluid were in the same range even when the amount of ilmenite was increased to 400 g, **Table 5**. The apparent viscosity of the drilling fluid was decreased from 80 to 20 cp by adding 4 g of lignosulfonate at 1000 s⁻¹, **Fig. 9**. By decreasing the amount of lignosulfonate to 2 g, the apparent viscosity was decreased to 15 cp, at 1000 s⁻¹. Zeta potential readings were done for the drilling fluid and it was noted that the dispersion of ilmenite was not good when adding 4 g of lignosulfonate, **Fig. 10**. Therefore, it was decide not to use lignosulfonate and study the effect of PAC-R.

Effect of Using 0.75 g PAC-R

Table 6 shows that by decreasing the amount of xanthan gum to 0.25 g and the amount of PAC-R to 0.75 g, the drilling fluid properties were in a good range and the mixing time for ilmenite was reduced to 20 min. The drilling fluid has a stable dispersion and the average zeta potential reading was about -48.4, which are in the stable range, **Fig. 10**.

Filtration test at 250°F and 300 psi differential pressure was performed using a 10 micron ceramic disk. The filtrate volume was high (> 60 cm³) and poor quality filter cake was formed (1.2 in. thickness).

A modified HPHT filter cell was used to do the filtration process. An Indiana limestone core of 1 in. thickness was used to simulate the reservoir section. The average permeability of the limestone core was around 100 md and the average porosity around 23 vol%. A static filtration test was conducted at 250°F and 300 psi differential pressure.

Fig. 11 shows that the cumulative filtrate volume was high even when adding 5 g of calcium carbonate with different sizes (fine and medium 25 and 50 μ m, respectively). Therefore, it was decided to increase the amount of PAC-R to 1 g to reduce the volume of filtration and increase the amount of xanthan gum to 0.5 g. The density of the drilling fluid was 110 pcf and the plastic viscosity was 35 cp. The drilling fluid had a yield point of 45 lb/100 ft² and gel strength of 9 and 14 for 10 s and 10 min, respectively.

Fig. 12 shows that the cumulative filtrate was 13 cm³ and the spurt loss was 6 cm³. The low slope of the filter cake indicated that the formed filter cake was optimum for drilling operations. The formed filter cake had a 0.2 in. thickness. A dynamic filtration test was performed at 150 rpm for the drilling fluid. The amount of XC-polymer decreased to 0.25 g to reduce the plastic viscosity. The test was performed at 250°F and 300 psi differential pressure.

Fig. 13 shows that under dynamic conditions, the filtrate volume was high (24 cm³) as compared with static conditions (14 cm³). This was due to the effect of the forces acting on the filter cake under dynamic conditions (Al-Abduwani et al. 2005). The thickness of the filter cake under dynamic conditions was large (0.25 in.) as compared with static conditions (0.2 in.).

To reduce the filtrate volume and minimize the filter cake thickness under dynamic conditions, 10.5 g CaCO₃ was added to the drilling fluid with a weight ratio of fine (25 μ m) to medium (50 μ m) 2 to 1. **Fig. 13** shows that the filtrate volume was decreased to 12 cm³. The formed filter cake had a small thickness (0.2 in.) under dynamic conditions when adding CaCO₃.

Optimized Drilling Fluid Formula

Table 7 shows the final and optimized drilling fluid formula equivalent to prepare 1 bbl. After hot rolling for 16 hrs, the drilling fluid had a density of 110 pcf and a plastic viscosity of 32 cp. The yield point was 24 lb/100 ft² and the gel strength was 4 and 6 for 10 s and 10 min, respectively. The pH was in the range of 9-10. No phase separation was observed after 16 hrs of hot rolling. This form of the drilling fluid had the required properties to solve all the common problems that were found in the literature.

Filter Cake Heterogeneity

Fig. 14 shows that the filter cake contains two layers under wet conditions. SEM analysis was performed to characterize the filter cake. Two samples were taken, one from the layer closer to the drilling fluid and one from the layer closer to the filter medium (the core surface). The two samples were dried in the oven for 3 hrs at 250°F.

Fig. 14 shows that the layer closer to the rock surface contained mainly solid particles. Chemical analysis of this layer showed that it contained mainly of Fe, Ti, O, and Ca, **Table 9. Fig. 14** shows that the layer closer to the drilling fluid contains mainly xanthan gum and starch. **Table 9** shows that this layer contained a small percent of solid particles. Due to the presence of the xanthan gum and starch in this layer, the overall permeability of the filter cake tended to be zero after 30 min of filtration.

Conclusions

Based on the results obtained, the following conclusions can be made:

- 1. Ilmenite-based filter cake was ideal for HPHT applications, 0.2 in. thickness and 12 cm³ filtrate under dynamic conditions. The filtrate volume was reduced by adding a minor amount of CaCO₃ solids that improved the particles packing.
- 2. No sag problem was observed when using the micronized ilmenite in water-based drilling fluids.
- 3. Ilmenite has a negative zeta potential in alkaline media and had a stable dispersion in water at pH > 7.

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TABLE 1: ZETA POTENTIAL MEASUREMENTS.

	1000 g	400 g Ilmenite	
Sample No.	With Acrylic Without Acrylic copolymer copolymer		Without Acrylic copolymer
	dispersant, mV	dispersant, mV	dispersant, mV
1	-23.84	-46.45	-49.03
2	-35.41	-38.89	-45.65
3	-29.65	-38.53	-45.25
Average	-29.63	-41.29	-46.64

TABLE 2: ZETA POTENTIAL AT DIFFERENT pH VALUES.

Solution	рН	Zeta Potential, mV
1	4.23	2.51
2	5.07	-3.38
3	6.17	-9.72
4	7.30	-29.44
5	8.44	-28.51
6	9.12	-31.36
7	10.08	-34.32
8	10.87	-29.01

TABLE 3: ILMENITE DRILLING FLUID FORMULA TO PREPARE 400 cm³.

Additive	Function	Lab. Unit (g)	Mixing Time, min
Water	Base	290	
Deformer	Anti-foam	0.08	1
Xanthan gum	Viscosifier	1.0	20
Modified Starch	Fluid loss	4.8	20
PAC-R	Fluid loss/ Viscosifier	1	20
KCl	Density and shale inhibition	72	20
КОН	pH control	0.5	1
Ilmenite	Weighting material	400	30

TABLE 4: DRILLING FLUID PROPERTIES AFTER HOT ROLLING AT 250°F FOR 16 HRS.

Proportios	400 g	300 g	200 g
Properties	Ilmenite	Ilmenite	Ilmenite
Density, lb/ft ³	120	110.5	99
Plastic Viscosity, cp	46	36	28
Yield Point, 100 lb/ft ²	55.5	44	27
10 s gel strength, 100 lb/ft ²	12	8	3
10 min gel strength, 100 lb/ft ²	15	11	4
рН	8.2	8.3	8.4

TABLE 5: PROPERTIES of DRILLING FLUIDS.

Properties	300 g Ilmenite	400 g Ilmenite	2 g
	and 4 g	and 4 g	lignosulfonate
	lignosulfonate	lignosulfonate	
Density, lb/ft ³	110	120	120
Plastic Viscosity, cp	13	14	11
Yield Point, 100 lb/ft ²	6	8	4
10 s gel strength, 100 lb/ft ²	2	4	2
10 min gel strength, 100 lb/ft ²	7	12	6
pН	8	9	10.3

TABLE 6: DRILLING FLUID PROPERTIES OF 400 G ILMENITE AND 0.75 G PAC-R.

Properties	Value
Density, lb/ft ³	120
Plastic Viscosity, cp	28
Yield Point, 100 lb/ft ²	16
10 s gel strength, 100 lb/ft ²	2
10 min gel strength, 100 lb/ft ²	5
рН	11

TABLE 7: ILMENITE DRILL-IN FLUID FORMULA TO PREPARE 358.5 cm³.

Additive	Function	Lab. Unit, g	Mixing Time, min
Water	Base	290	
Deformer	Anti-foam	0.08	1
Xanthan gum	Viscosifier	0.25	20
Modified starch	Fluid loss	5	20
PAC-R	Fluid loss/ Filtration Control	1	20
KCl	Density and shale inhibition	72	20
КОН	pH control	1	1
CaCO ₃ Fine (25 μm) CaCO ₃ Medium(50 μm)	Bridging Material	7 3.5	20
Ilmenite	Weighting material	300	20

TABLE 8: VSST RESULTS FOR DIFFERENT FORMULAS OF ILMENITE-BASED DRILLING FLUID.

Formula	*W ₂ , g	*W ₁ , g	VSST
400 g Ilmenite, 0.25 g Xanthan gum, 0.75 g PAC-R	18.26	18.23	0.025
300 g Ilmenite, 0.25 g Xanthan gum, 1 g PAC-R	17	16.95	0.042
300 g Ilmenite, 0.25 g Xanthan gum, 1 g PAC-R, 5 g CaCO ₃	17.08	16.96	0.100

 $[*]W_1$ = weight of the mud filled syringe in sample 1, g

TABLE 9: SEM ANALYSIS OF THE LAYER CLOSER TO THE ROCK SURFACE AT DIFFERENT LOCATIONS.

	Layer closer to the rock surface			Layer closer to the drilling fluid	
E1	Concentration, wt%			Concentration, wt%	
Element	General	1	2	3	General
0	25.37	24.36	27.44	32.5	8.36
Ti	27.80	24.02	23.95	22.22	7.37
Fe	45.89	42.17	42.44	43.03	9.14
Na	0.00	6.26	0.00	0.00	41.2
Mg	0.00	0.00	0.70	2.14	0.00
Al	0.00	1.70	1.95	0.00	0.00
Si	0.59	0.98	0.00	0.00	1.2
Cl	0.00	0.00	0.00	0.00	32.72
С	0.00	0.0	3.03	0.00	0.00
Ca	0.35	0.51	0.49	0.12	0.00
Total	100	100	100	100	100

^{*} W_2 = weight of the mud filled syringe in sample 2, g

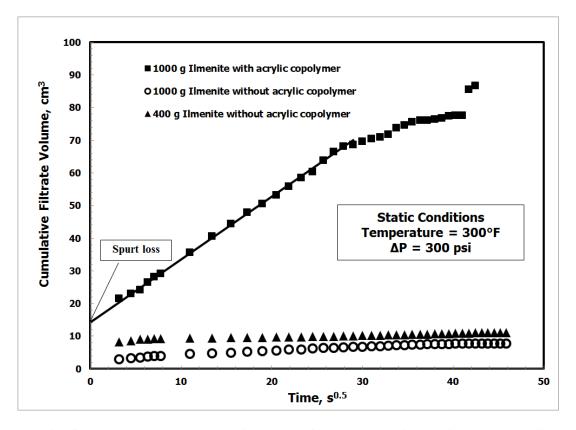


Fig. 1: Cumulative filtrate volume vs. square root of time. High filtrate volume using acrylic copolymer indicated poorly formed filter cake.

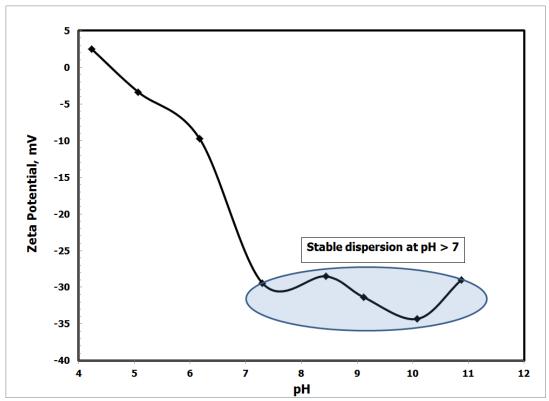


Fig. 2: Stable dispersion of ilmenite in DI water at pH > 7.

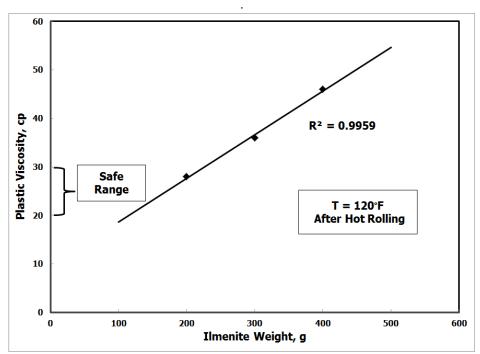


Fig. 3: High value of plastic viscosity out of the required range.

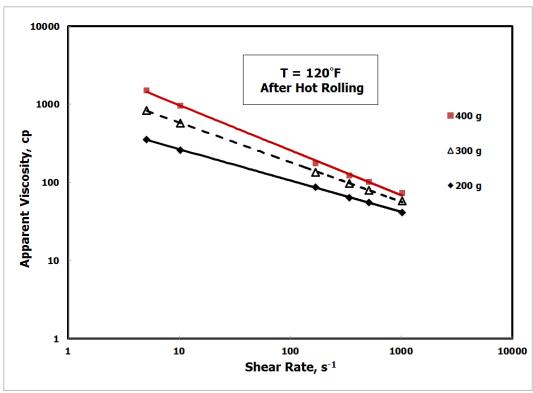


Fig. 4: Apparent viscosity of the drilling fuid with different amount of ilmenite.

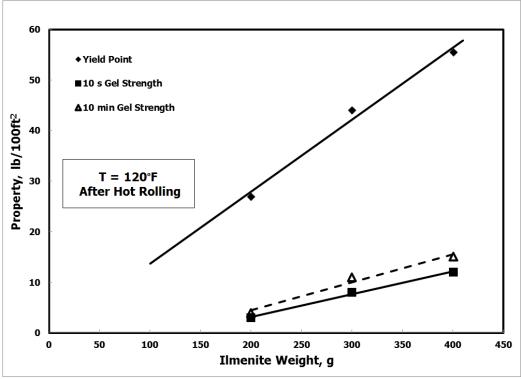


Fig. 5: High value for yield point and safe range of gel strength.

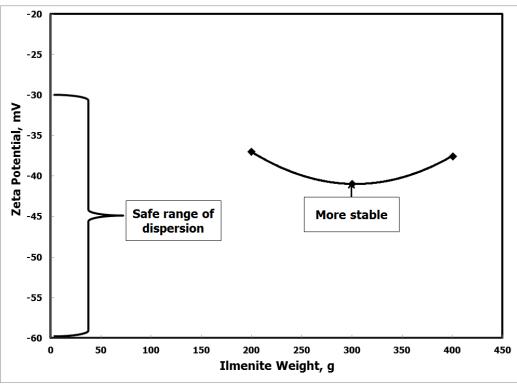


Fig. 6: Stable dispersion of ilmenite in the drilling fluid even at 400 g.



Fig. 7: 0.22 in. filter cake when using 400 g ilmenite.

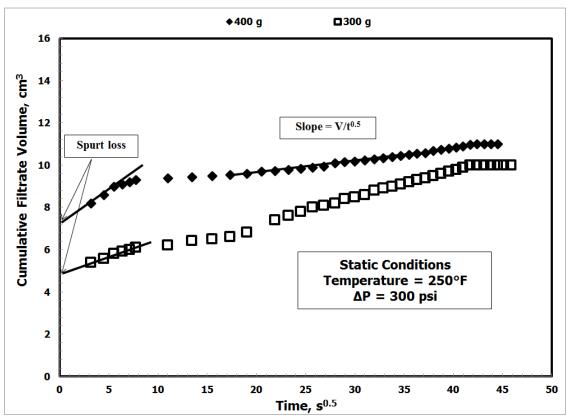


Fig. 8: Reduction of filtrate rate when using 300 g ilmenite.

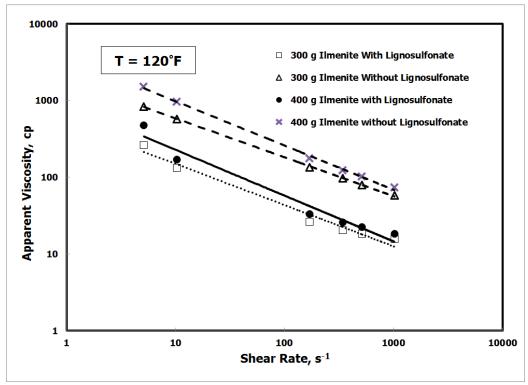


Fig. 9: Decrease in the apparent viscosity when using 4 g lignosulfonate.

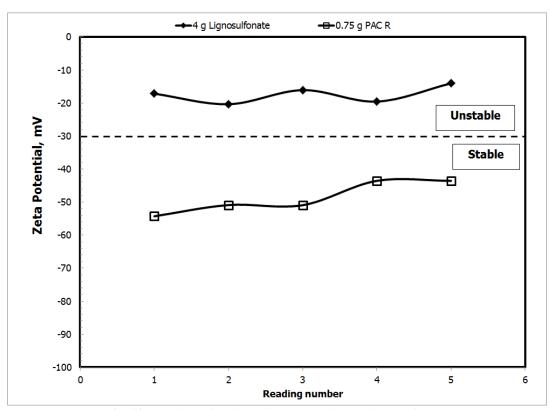


Fig. 10: Poor ilmenite dispersion when using 4 g lignosulfonate.

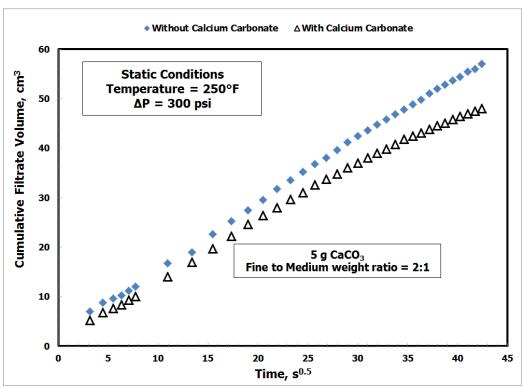


Fig. 11: High filtrate volume even when using 5 g of calcium carbonate (25 μ m and 50 μ m).

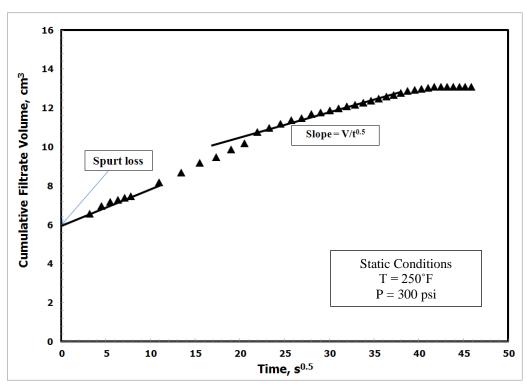


Fig. 12: Low slope of the relation between cumulative volume and square root of time indicated the lower permeability of the filter cake and optimized drilling fluid formula.

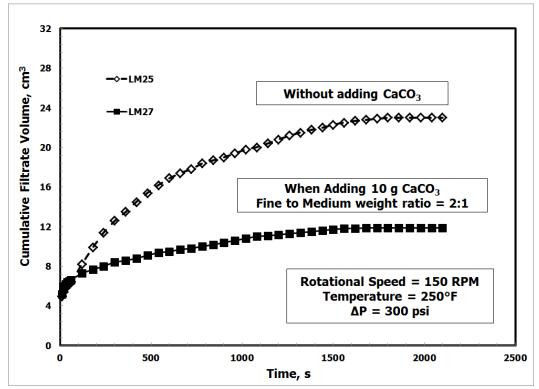


Fig. 13: Filtrate volume as a function of time.



Fig. 14: SEM analysis shows the heterogeneity of the filter cake.