EXPERIMENTAL STUDIES OF ILMENITE AS A WEIGHTING MATERIAL IN OIL-BASED DRILLING FLUIDS FOR HPHT OPERATIONS

A Thesis

by

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ABSTRACT

Ilmenite ($FeTiO_3$), with a mean particle size of 30 - 45 µm, was first introduced into the oil industry as weighting material in 1976. However, its use was limited mainly because of its abrasiveness to drilling equipment. Recently, a superfine ilmenite (5 µm) was introduced to address shortcomings of the traditional weighting materials. The objective of this study is to discuss the performance of oil-based drilling fluids using ilmenite as a weighting material for HPHT applications.

Oil-based drilling fluids with a density of 1.92 SG containing API standard barite and ilmenite were compared. HPHT filtration tests under static and dynamic conditions were conducted at 300°F and 300 psi. Indiana limestone cores with an average porosity of 23 vol% and an average permeability of 100 md were used in the filtration tests. The rheological properties, sag tendency, the volume of filtrate, and the filter cake thickness of the oil-based drilling fluids were determined before and after heating at 400°F for 16 hours.

Results showed that ilmenite-based drilling fluids had a sag factor of < 0.3 and a plastic viscosity of 25-30 cp, which was much lower than those of barite. HTHP filtration tests showed that under static conditions, the filter cake had a thickness of 0.18 in. and 2.9 cm^3 of filtrate volume; while under dynamic conditions, the filter cake thickness was 0.15 in. and the filtrate volume was 2.2 cm^3. The rheological properties of oil-based drilling fluids were constant after heating at 400°F for 16 hours. The filter cake thickness and sag factor were small even after 16 hours of heating. This study provides

an evaluation of oil-based drilling fluids with ilmenite as a weighting material for HPHT wells, and gives recommendations on how to use it in the field.

DEDICATION

I would like to dedicate this thesis to my parents and husband who have always given me strength to achieve my goals.

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I would like to thank my committee chair, Dr. Hisham Nasr-El-Din for his support, supervision, and assistance in this thesis. I also would like to thank my two committee members, Dr. Mahmoud El-Halwagi and Dr. Schubert, for their cooperation and support throughout the course of this research.

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Finally, I give thanks to my family for their encouragement and to my husband – Jia He for his patience and love.

NOMENCLATURE

HPHT	High Pressure High Temperature
PV	Plastic Viscosity
YP	Yield Point
API	American Petroleum Institute
VSST	Viscometer Sag Shoe Test
SG	Specific Gravity
M-I LLC	MICWACO Limited Liability Company

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1. INTRODUCTION

Weighting materials are additives used to adjust the density of drilling fluids. The choice of the weighting agent to be used in drilling fluids is determined by many factors. One of the most important factors is to provide low rheology in high density fluids and low sag (Zamora and Bell 2004; Zamora 2007).

API standard barite is, by far, the most commonly used weighting agent in drilling fluids. However, it is not suitable for all drilling fluid applications. One of the major disadvantages of barite is sag (the tendency to settle upon aging) and high rheology (plastic viscosity) particularly in high density drilling fluids (Aldea et al. 2001; Meeten 2001). Another disadvantage is the low hardness (2.5-3.5), which can, on prolonged shearing, lead to the creation of fine colloidal particles that contribute to increasing the gel strength of the drilling fluids and may cause formation damage (Guo et al. 2012). Barite cannot be used in formate brine as it dissolves. Barite is very difficult to remove from the reservoir except by using expensive chelating agents to dissolve it and remove any formation damage (Bern et al. 2010).

However, there are specialized weighting agents such as manganese tetroxide (Mn_3O_4) or treated micronized $BaSO_4$ with an average particle size of 1 μm. These can provide high density fluids with low sag, low rheology and are less damaging to the formation.

Mn_3O_4 is used in both drilling and completion fluids when low sag, low rheology and ECD (equivalent circulating density) management are required (Svendsen et al.

1995). Ilmenite ($FeTiO_3$) with a mean particle size of 30 - 45 μm was first introduced into the oil industry as a weighting material in 1976 (Haaland et al. 1976; Fjogstad et al. 2000; Saasen et al. 2001; Blomberg and Melberg 1984). However, its use was limited mainly because of its abrasiveness to drilling equipment. Abrasiveness was solved by removing the coarse particles from the ilmenite. There were magnetic issues, which were solved by reducing the magnetite content. A number of different particle size distributions have been tested since 1993. Recently, a superfine ilmenite (5 μm) was introduced to address shortcomings of the traditional weighting materials (Al-Bagoury and Steele 2012).

However, it is very challenging to drill high pressure and high temperature (HPHT) wells. Along with a number of other factors, the drilling fluid used will be of utmost importance to the progress and success of the drilling and completion operations. Literature studies show that the use of ultra-fine particle weighting agents can provide a number of technical benefits over traditional weighting agents, especially under HPHT conditions (Taugbol et al. 2005). With the application of ultra-fine particles, oil-based drilling fluids can be designed with reduced viscosity in combination with minimal settling potential of the weighting agent. The sag problem and its associated challenges, such as loss circulation or well control problems, can cause a severe economic impact on the well drilling operation (Saasen 2001).

The objective of this study is to formulate a new HPHT oil-based drilling fluid using super-fine ilmenite as the weighting material. The fluid was qualified through an extensive laboratory study on the rheological properties under high temperatures, as well as the filtration characteristics. Surface treated clay (hectorite) was used as gallant/suspension agent in the HTHP oil-based fluid system (Guichard et al. 2007). It is thermally stable up to 425°F (218°C). This study also shows the rheological properties, sag tendency, the volume of filtrate, and the filter cake thickness of the optimized oil-based drilling fluids.

2. FUNCTION AND COMPOSITION OF OIL-BASED DRILLING MUD

In this section, a basic knowledge of oil-based drilling mud is introduced, especially those properties that distinguish greatly from that of water-based drilling muds. Also, the specific chemicals used to formula the oil-based drilling muds in this study will be discussed.

2.1 Introduction

Drilling fluids, or drilling muds, are used in the drilling operations of oil and gas wells in order to lubricate and to cool the drill bit and its fittings, during rock penetration and, at the same time, to keep rock cuttings in suspension while carrying some out of the borehole.

The origin of non-aqueous drilling fluids can be traced back to the 1920s when crude oil was used as a drilling fluid. The advantage of oil as a drilling and completion fluid were obvious even then:

- Clays do not hydrate and swell.
- Wellbore stability is improved.
- Production is improved from sandstones containing clays.
- Problems are reduced when drilling evaporates (salts, anhydrite, etc.).
- Wellbore enlargement is reduced.
- Mud properties are more stable.
- Contamination resistance is increased.

In the 1940s, diesel-based muds were developed that not only tolerated water, but used emulsified water to control and maintain properties. Emulsified water droplets lowered fluid loss and raised viscosity. The continuous oil phase of these muds made them act as oil muds by wetting with an oil film and preventing emulsified water from interacting with water-sensitive shale and cuttings to provide good wellbore stability.

Environmental concerns during the 1980s led to the use of mineral oils, or highly refined oils that were less toxic and more environmentally acceptable than diesel. Diesel oil, mineral oils, and synthetic fluids are all nonpolar, non-aqueous fluids. They all have shown special economic advantages when used for:

- Troublesome shales.
- Salt, anhydrite, carnallite and potash zones.
- Deep, hot wells.
- Drilling and coring sensitive productive zones.
- Extended-reach drilling projects.
- Difficult directional wells.
- Slimhole drilling.
- Corrosion control.
- Hydrogen sulfide and carbon dioxide bearing formations.
- Perforating and completion fluids.
- Casing pack or packer fluids.
- Workover fluids.
- Spotting fluids to free stuck pipe.

2.2 Composition of Oil-Based Drilling Muds

The choice of which oil or synthetic will be used for a specific application is a matter of selecting a formulation that will provide a reasonable balance between environmental acceptability, waste disposal cost, mud cost, performance, and availability.

A typical fundamental composition of oil-based drilling mud is:

- Mineral oil and water.
- Emulsifier.
- Lime.
- Viscosifier.
- $CaCl_2$.
- Filtration control agent.
- Weighting material.

2.2.1 Mineral Oil

Mineral oils have lower aromatic (<1.0%) content than diesel and are also considered less toxic. They have higher flash points than diesel and are safer to use especially in high temperature applications. Also, they have a low viscosity when compared to diesel and crude oils which will affect the overall viscosity of the oil-based mud. Unlike diesel, mineral oils do not contain surfactants that could change the wettability of the formation.

The properties of mineral oil (Escaid 110) are shown in **Table 1**.

TABLE 1—PROPERTIES OF ESCAID 110.

	Density at 60 °F, g/cm^3	Initial Boiling Point, °F	Final Boiling Point, °F	Flash Point, °F	Aniline Point, °F	Aromatic Concentration, wt%	Viscosity at 104 °F, cSt
Escaid 110	0.806	394	459	167	162	0.05	1.63

2.2.2 Emulsifier

Emulsifier is a chemical used in the preparation and maintenance of an oil- or synthetic-base drilling fluid that forms a water-in-oil emulsion (invert emulsion). An oil-mud emulsifier lowers the interfacial tension between oil and water, which allows stable emulsions with small drops to be formed. Historically, oil-mud emulsifiers have been classified as primary and secondary. Secondary emulsifiers are generally not used alone to make a stable oil mud. There are two types of emulsifiers: primary emulsifier and secondary emulsifier.

Calcium soaps are the primary emulsifier in oil muds. These are made in the mud by the reaction of lime and long chain (C-16 to C-22) fatty acids. Soap emulsions are very strong emulsifying agents, but they take some reaction time before an emulsion is actual formed.

Secondary emulsifiers are very powerful oil wetting chemicals. Generally, these products do not form emulsions as well as the primary emulsifiers, but this oil wet solids before the emulsion is formed were used to readily emulsify any water intrusions quickly.

Fatty acids are the basic components for nearly all emulsifiers and wetting agents used in the preparation of invert emulsions. Emulsifiers can be calcium fatty-acid soaps made from various fatty acids and lime, or derivatives such as amides, amines, amidoamines, and imidazolines made by reactions of fatty acids and various ethanolamine compounds.

The emulsifiers used in this study are: CARBO-MUL HT, CARBO-TEC S, and MAGMA-VERT.

CARBO-MUL HT is a high-temperature polyamide, which is used as the primary emulsifier and wetting agent. CARBO-TEC S is a supplemental emulsifier used in the oil mud system. CARBO-TEC S requires lime to function effectively, and is used in conjunction with CARBO-MUL HT and filtration control additives. MAGMA-VERT is a specialized, high temperature, modified amide amine used as a supplementary emulsifier in HPHT oil-based mud systems.

2.2.3 Lime

Hydrated lime is added to maintain the alkalinity of oil based drilling fluids where it enhances the performance of emulsifiers and treats carbon dioxide (CO_2) or hydrogen sulfide (H_2S) contamination.

If CO_2 is present, lime additions will be needed only if an anionic emulsifier is being used. Calcium hydroxide is needed to form the calcium soap. Once the soap is formed, any introduction of CO_2 in the fluid will not affect the fatty acid and lime reaction. If H_2S is introduced to the system, the CaS compound will remain stable as long as there is excess lime present.

Lime analysis determines the amount of excess lime in the oil mud. Lime is essential for the formation of the emulsion when using fatty acid type emulsifiers. Lime content should always be checked since emulsifier additions may not be required due to deficiency in the lime content. A decrease in lime content while drilling may indicate acid gases such as H_2S or CO_2 or high temperature deterioration of products.

2.2.4 Viscosifier

The primary viscosifier in invert emulsion muds is organophilic clay. Although this clay does not hydrate, it will reduce the filtration rate by providing a colloidal solid for forming a basic filter cake. Special clays and alternative viscosifiers may be required for all-oil or high-ratio fluids (>90:10).

The typical viscosifiers used in the oil field are shown in **Table 2.**

The viscosifiers used in this study are: MAGMA-GEL and MAGMA-GEL SE. MAGMA-GEL is a specialty product, wet processed organophillic clay (modified hectorite) used as gallant/suspension agent in the HPHT oil-based mud system. MAGMA-GEL is temperature stable up to 425°F (218℃). Its performance is complemented by MAGMA-GEL SE. MAGMA-GEL SE is a organophillic clay (organo-attapulgite) used as HPHT emulsion-based drilling fluid additive to improve the suspension properties of the drilling fluid.

TABLE 2—MATERIALS USED AS VISCOSIFIERS.

Material	Principal
Bentonite	Sodium /Calcium Aluminosilicate
CMC	Sodium Carboxy-methyle cellulose
PAC	Poly anionic Cellulose
Xanthan Gum	Extracellullar Microbial Polysaccharide
HEC	Hyroxy-ethyl Cellulose
Guar Gum	Hydrophilic Polysaccharide Gum
Resins	Hydrocarbon co-polymers
Silicates	Mixed Metal Silicates
Synthetic Polymers	High molecular weight Polyacrylamides/polyacrylates

2.2.5 $CaCl_2$

Calcium chloride is added to increase the emulsified water phase salinity to provide inhibition of shales and reactive solids. Control of salinity in invert oil muds is necessary to "tie-up" free water molecules, and it prevents any water migration between the mud and the open formation such as in shales. The range for calcium chloride content is usually 25 to 35% by weight.

2.2.6 Filtration Control Agent

MAGMA-SEAL used in this study is an organophilic lignite additive used in emulsion-based drilling fluid systems to improve filtration control at high temperatures. Lignite is used as a filtration control agent and as a secondary deflocculant. To solubilize lignite, it must have a highly alkaline environment. It functions as a fluid loss additive up to 400°F. Compared to lignosulfonate, lignite provides better filtration control at elevated temperatures. It is usually added with lignosulfonate. The ratio of lignite to lignosulfonate is generally 4:1; however, the ratio will decrease depending on quality of the lignite, mud weight, formations encountered, and wellbore temperature.

Organophilic lignites are used as high temperature fluid loss additives. They also will aid in the emulsification of water especially at high temperatures. A lignite is treated with an amine to make it oil dispersible. It controls fluid loss by plugging and can be used at high concentrations without causing excessive viscosities (20 lb/bbl +/-).

2.2.7 Weighting Material

Weighting materials or densifiers are solid materials which when suspended or dissolved in water will increase the mud weight. Most weighting materials are insoluble and require viscosifier in order to be suspended in a fluid. Clay is the most common viscosifier. The properties of the most used weighting materials are shown in **Table 3**.

TABLE 3—TYPICAL SOLID WEIGHTING AGENTS AND THEIR PROPERTIES.

Weighting Agent	Chemical Nature	Density, g/cm^3	D50, µm	Acid Solubility	Mohs Hardness
Calcium Carbonate	$CaCO_3$	2.8	15-20	Soluble	3-4
Barite	$BaSO_4$	>4.2	15-20	Insoluble	2.5-3.5
Micronized Barite	$BaSO_4$	>4.2	10	Insoluble	2.5-3.5
Treated Micronized Barite	$BaSO_4$	>4.2	1	Insoluble	2.5-3.5
Ilmenite	$FeTiO_3$	4.5-4.7	15	Soluble	5-6
Micornized Ilmenite	$FeTiO_3$	4.5-4.7	5	Soluble	5-6
Manganese Tetraoxide	Mn_3O_4	4.7-4.9	<1	Soluble	5.5
Hematite	Fe_2O_3	4.9-5.2	15-50	Soluble	5.5-6.5

3. RHEOLOGY PROPERTIES AND FILTRATION CONTROL OF OIL-BASED DRILLING MUD

In this section, the physical properties of a drilling fluid, including the rheological properties, are discussed in detail to assist in optimizing the oil-based drilling mud. These physical properties contribute to several important aspects for successfully drilling a well:

- Provide pressure control to prevent an influx of formation fluid.
- Provide energy at the bit to maximize Rate of Penetration (ROP).
- Provide wellbore stability through pressured or mechanically stressed zones.
- Suspend cuttings and weight material during static periods.
- Permit separation of drilled solids and gas at surface.
- Remove cuttings from the well.

3.1 Plastic Viscosity

Plastic Viscosity is a measure of the internal resistance to fluid flow attributable to the amount, type, and size of solids present in a given fluid. It is expressed as the number of dynes per square cm of tangential shearing force in excess of the Bingham yield value that will induce a unit rate of shear. This value, expressed in centipoises is proportional to the slope of the consistency curve determined in the region of laminar flow for materials obeying Bingham's Law of Plastic Flow. When using the viscometer, the plastic viscosity in centipoise (cP) or milliPascal seconds (mPa•s) is found by subtracting the 300-rpm reading from the 600-rpm reading.

$$PV(cP) = \Theta_{600} - \Theta_{300} \tag{3-1}$$

3.1.1 Factors Affecting Plastic Viscosity

Plastic viscosity is usually described as that part of resistance to flow caused by mechanical friction. Primarily, it is affected by:

- Solids concentration.
- Size and shape of solids.
- Viscosity of the fluid phase.
- The presence of some long-chain polymers (POLY-PLUS, Hydroxyethylcellulose (HEC), POLYPACT R, Carboxymethylcellulose (CMC)).
- The Oil-to-Water (O/W) or Synthetic-to-Water (S/W) ratio in invert-emulsion fluids.
- Type of emulsifiers in invert emulsion fluids.

The solids phase is the chief concern of the fluid engineer. An increase in plastic viscosity can mean an increase in the percent by volume of solids, a reduction in the size of the solid particles, a change in the shape of the particles, or a combination of these.

3.1.2 Values of Plastic Viscosity

Plastic viscosity should be maintained at minimum values to optimize bit hydraulics and penetration rates. If the plastic viscosity trends upward over a period of time without increases in the mud weight, it usually indicates that fine solids are building up in the mud. Increases in the volume percent solids even from weight material will increase the plastic viscosity. A practical upper limit for the plastic viscosity is twice the fluid weight (lb/gal).

3.2 Yield Point

Yield point, the second component of resistance to flow in a drilling fluid, is a measurement of the electro-chemical or attractive forces in a fluid. These forces are a result of negative and positive charges located on or near the particle surfaces. Yield point is often used as an indicator of the shear thinning characteristics of drilling fluid and its ability to suspend cuttings and weight material.

3.2.1 Factors Affecting Yield Point

Yield point is a measure of these forces under flow conditions and is dependent upon the following:

- The surface properties of the fluid solids.
- Volume concentration of the solids.
- The electrical environment of these solids (concentration and types of ions in the fluid phase of the fluid).

3.2.2 Values of Yield Point

Yield Point (YP) in pounds per 100 square feet (lb/100 ft2) is calculated from Fann VG meter data as follows:

$$YP(lb/100ft^2) = \Theta_{300} - PV \tag{3-2}$$

3.3 Gel Strength

Under static conditions, 10 second and 10 minute measurements on the Fann VG meter indicate strength of attractive forces (gelation) in a drilling fluid. Excessive gelation is caused by high solids concentrations leading to flocculation.

3.3.1 Gel Strength with Time

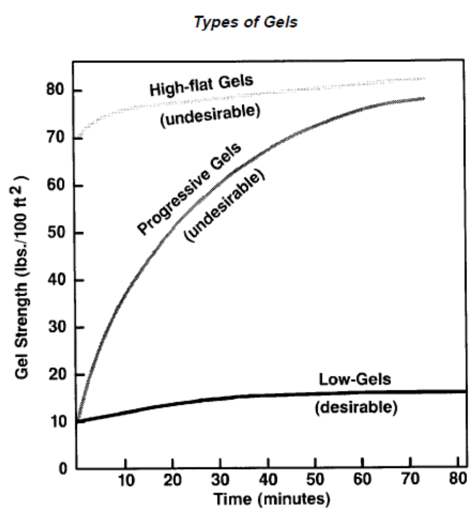

Fig. 1—Different types of gel strength.

Signs of rheological trouble in a mud system often are reflected by the gel strength development of a mud with time shown in **Fig. 1**. When there is a wide range between the initial and 10 minute gel readings they are called “progressive gels”. This is not a desirable situation. If the initial and 10 minute gels are both high, with no appreciable difference in the two, these are “high-flat gels”, also undesirable. Gelation should not be allowed to become much higher than is necessary to perform the function of suspension of cuttings and weight material. For suspension “low-flat gels” are desirable.

3.3.2 Gel Strength and Yield Point

Gel strengths and yield point, are both a measure of the attractive forces in a mud system. A decrease in one usually results in a decrease in the other. Therefore, similar chemical treatments are used to modify them both. The 10 second gel reading more closely approximates the true yield stress in most drilling fluid systems. Water dilution can be effective in lowering gel strengths, especially when solids are high in the mud.

Yield point and gel strengths are governed by two requirements. The first is the need to maintain sufficient thixotropy (gel structure) to suspend weight material and cuttings, plus provide carrying capacity. The second requirement is to minimize annular pressure losses and Equivalent Circulating Densities (ECDs).

3.3.3 Values of Gel Strength

Initial gel strength above 5 lb/100 ft^2 is usually required to suspend weight material. This is not an absolute value because of the effects of temperature on oil fluids. Additionally, initial gel strength in a weighted fluid system must be sufficient to prevent settling of weight materials. Fragile gel strength increases only slightly with time, but may be higher initially than a progressive gel.

3.4 Filtration Control

Most oil- and synthetic-based fluids are emulsions. Their fluid phase is an emulsion with oil or synthetic as the continuous phase and brine as the emulsified phase. The emulsified brine forms colloid-sized droplets, which are immiscible in the oil or synthetic. These brine droplets become trapped in the filter cake and reduce filter cake permeability and fluid loss. Invert emulsion muds may contain emulsifiers, wetting agents, organophilic clays, asphalts and/or amine-treated lignite, polymers, lime and weight material. The filtration rate of invert-emulsion muds is affected by additives other than the filtration control additives.

4. EXPERIMENTAL SET-UPS

4.1 Rheological Properties of the Oil-Based Drilling Muds

Fig. 2—M3600 Grace viscometer.

The mud properties were measured by using a mud balance and Grace M3600 viscometer shown in **Fig. 2**. The condition for density measurement is 75°F and rheological measurements are conducted at 120°F. Multiple measurements are conducted to ensure that accurate values can be obtained.

4.2 HPHT Filtration Press

HPHT filtration tests were performed using a standard HPHT filter press shown in **Fig. 3** under static and dynamic conditions (150 rpm). The cell was placed in a heating jacket, and the system was heated to a desirable temperature (300°F). The applied pressure was 300 psi differential pressure.

Fig. 3—HPHT filtration press.

4.3 Dynamic Sag Test

The viscometer sag shoe test (VSST) shown in **Fig. 4** is a well site and laboratory test to measure the sag tendency of the weight material of field and lab prepared drilling fluids under dynamic conditions, and VSST was calculated using Eq. 1

VSST = 0.833 x (W2-W1) ………………………………….. (4-1)

Where,

VSST = viscometer sag shoe test, ppg

W1 = weight of the mud filled syringe in sample 1, g

W2 = weight of the mud filled syringe in sample 2, g

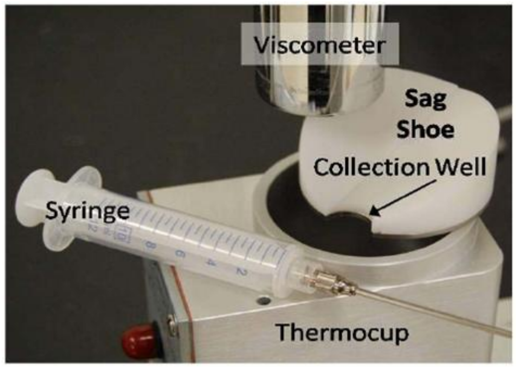

Fig. 4—Sag test setup.

4.4 Effect of Aging at High Temperatures

A hot rolling oven shown in **Fig. 5** was used to examine the effect of heat on the rheological properties of oil-based drilling fluids after heating at 400°F for 16 hours. Two hundred cm^3 of drilling fluid was pressurized inside a Teflon liner at 300 psi using nitrogen, and was put into the rolling oven at 400°F for 16 hours. Then the rheological properties of the fluids were measured at 120°F by a Grace M3600 viscometer. HPHT dynamic filtration and sag tests were conducted after the drilling fluid was heated for 16 hours. A high permeability Indiana limestone core with a thickness of 0.25 in. and a diameter of 2.5 in. was used in the dynamic filtration test.

Fig. 5—Aging effect setup.

5. RHEOLOGICAL PROPERTIES OF FORMULATED OIL-BASED DRILLING MUD

5.1 Experimental Materials

Micronized ilmenite and API standard barite were used as weighting materials to prepare oil-based drilling fluids for HPHT applications. Micronized ilmenite was supplied by Elkem A/S Company. **Table 4** summarizes the components that were used to prepare the oil-based drilling fluids. The chemicals used in this work such as viscosifiers, emulsifiers and fluid loss agents were kindly supplied by the Baker Hughes Company.

TABLE 4—RECIPE OF OIL-BASED DRILLING FLUID CONTAINING ILMENITE.

Additive	Weight, g	Mixing Time, min
Mineral Oil	242.5	
Viscosifier - Organically clay (modified hectorite)	6	10
Viscosifier - Organically clay (organo-attapulgite)	6	10
Lime	20	10
HT Primary Emulsifier - Polyamide	15	10
HT Secondary Emulsifier – Modified amide amine	15	10
Deionized Water	24.5	10
$CaCl_2$ 97%	8	10
HT Filtration Control - Organophilic lignite	12	10
HT Secondary Emulsifier - Polymerized fatty acid	1.5	10
Weighting Agent – Ilmenite	400	20

The properties of the weighing agents used in this study are shown **Table 5**. The particle size distribution of micronized ilmenite is shown in **Fig. 6**.

TABLE 5—PROPERTIES OF DIFFERENT WEIGHTING AGENTS.

Weighting Agent	Formula	Density, g/cm^3	D_{50}, µm
Barite	$BaSO_4$	4.2	15-20
Micronized ilmenite	$FeTiO_3$	4.5-4.7	5
Manganese tetraoxide	Mn_3O_4	4.8	1

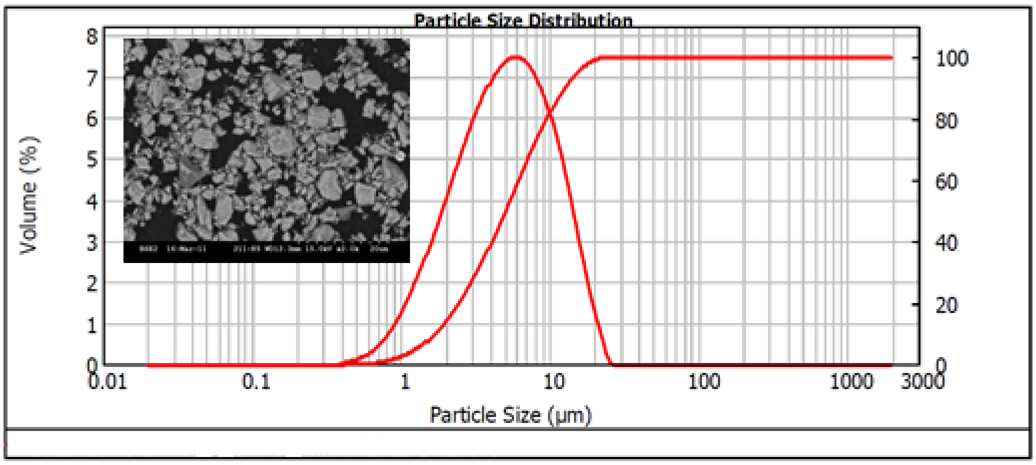

Fig. 6—Particle size distribution of micronized ilmenite measured by light scattering with a refractive index of 2.8.

To produce micronized ilmenite a specially selected ilmenite ore suited for oil well drilling was subjected to grinding and refining processes to remove the associated minerals. The material was further ground and classified to obtain the desired average size (D50) of 5 µm. A refractive index of 2.8 was used to calculate the size distribution using a laser diffraction method. The total specific surface area of 1.6 m^2/g was measured by the BET method. Also the mineralogical composition of the ilmenite sample was analyzed using the XRD. The micronized ilmenite is mainly composed of (FeO, MgO)TiO_2 (> 94 wt%). The material also contains less than 3 wt% silica phases such as orthopyroxene (Mg, Fe)SiO_3, plagioclase (($NaAlSi_3O_8$)($CaAl_2Si_2O_8$)), and biotite. The morphology of micronized ilmenite was studied using a scanning electron microscope and particle imaging analyzer. The material has a high circularity of greater than 0.85, which contributes to the lowering of the viscosity of its dispersions.

To simulate a reservoir formation, Indiana limestone cores with an average porosity of 23 vol% and an average permeability of 100 md were used in the filtration tests.

5.2 Rheological Properties

The first set of drilling fluids was formulated to assess the effects of ilmenite solids loading on the rheological properties of the non-aqueous drilling fluid. **Table 6** illustrates the drilling fluid properties with 400 to 800 g of ilmenite. With the addition of 400 g of ilmenite as the weighing material, the values of yield point, 10 s gel strength, and the 10 min gel strength are too low and that might cause a sag problem. The low viscosity is related to the low viscosifier concentration, which was 3 g of an organically modified clay (hectorite) and 2 g of an organically modified clay (organo-attapulgite). The two types of organically modified clays act as the viscosifiers and depend on the fluid density. A certain concentration of viscosifiers is used to provide enough viscosity for the drilling fluid. Therefore, it was decided to increase the total viscosifiers concentration to 12 g (40 g/l mineral oil).

When the amounts of viscosifiers were increased to 6 g, respectively, the gel strength as well as the value of yield point increased greatly. However, literature studies show that a practical upper limit for the plastic viscosity is twice the fluid weight (lb/gal) and the initial gel strength above 5 lb/100 ft^2 is usually required to suspend the weighting material (M-I LLC. 1998). The value of 10 s gel strength is lower than the value that can be sufficient to prevent settling of the weighting materials. Therefore, the amount of ilmenite was increased to obtain the desired values for 10 s gel strength.

TABLE 6—PROPERTIES OF OIL-BASED DRILLING FLUIDS WITH DIFFERENT AMOUNTS OF ILMENITE.

Additive	400 g Ilmenite	400 g Ilmenite	500 g Ilmenite	600 g Ilmenite	700 g Ilmenite	800 g Ilmenite	800 g Ilmenite
Mineral Oil, g	242.5	242.5	242.5	242.5	242.5	242.5	242.5
Viscosifier - Organically clay (modified hectorite), g	3	6	6	6	6	6	3
Viscosifier - Organically clay (organo-attapulgite), g	2	6	6	6	6	6	3
Lime, g	20	20	28	28	28	28	28
HT Primary Emulsifier - Polyamide, g	15	15	15	15	15	15	15
HT Secondary Emulsifier – Modified amide amine, g	15	15	15	15	15	15	15
Deionized Water, g	24.5	24.5	24.5	24.5	24.5	24.5	24.5
$CaCl_2$ 97%, g	8	8	8	8	8	8	8
HT Filtration Control - Organophilic lignite, g	12	12	12	12	12	12	18
HT Secondary Emulsifier - Polymerized fatty acid, g	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Density, SG	1.52	1.52	1.70	1.81	1.92	2.02	2.02
Plastic Viscosity, cp	11	15	16	20	30	55	28
Yield Point, lb/100ft^2	2	7	6	8	10	12	4
10 s Gel Strength, lb/100ft^2	0	4	4	6	9	11	3
10 min Gel Strength, lb/100ft^2	1	11	9	12	16	17	5

When the amount of ilmenite was increased to 800 g, it was found that the yield point, 10 s gel strength, and 10 min gel strength reached the desired range (above 5 lb/100 ft^2). However, the plastic viscosity is a bit higher than the desired value (**Fig. 8**). When the amount of ilmenite decreased to 700 g, the results show that all the values are in the desired range in Table 6. **Fig. 7** shows that the density of the drilling fluid increased with the increment of ilmenite and it changed from 1.52 to 2.02 SG as the amount of ilmenite was increased from 500 to 800 g. **Figs. 8 to 10** show the safe range for those values, where the upper limit for the plastic viscosity is twice the fluid weight (lb/gal) and the initial gel strength should be above 5 lb/100 ft^2 to suspend the weighting material (M-I LLC. 1998). It can be seen clearly that the one with the addition of 700 g ilmenite has all the desired values when the amount of additives are kept constant.

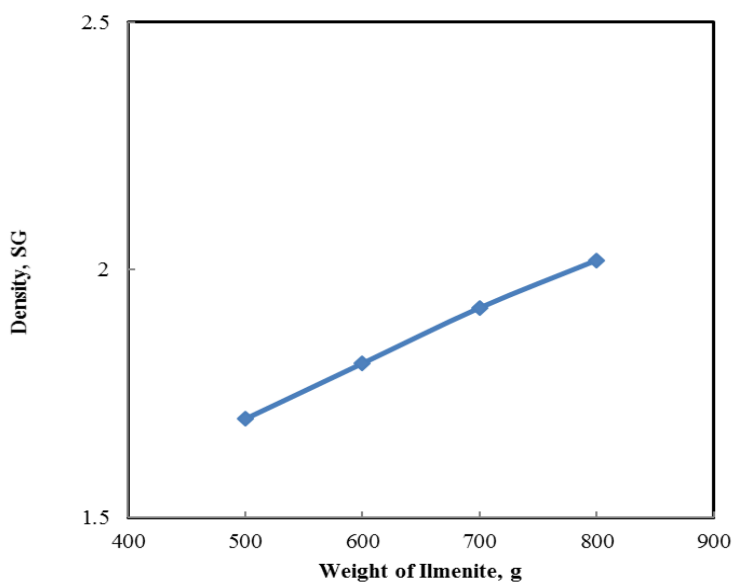

Fig. 7—Density of oil-based muds using ilmenite as the weighting agent.

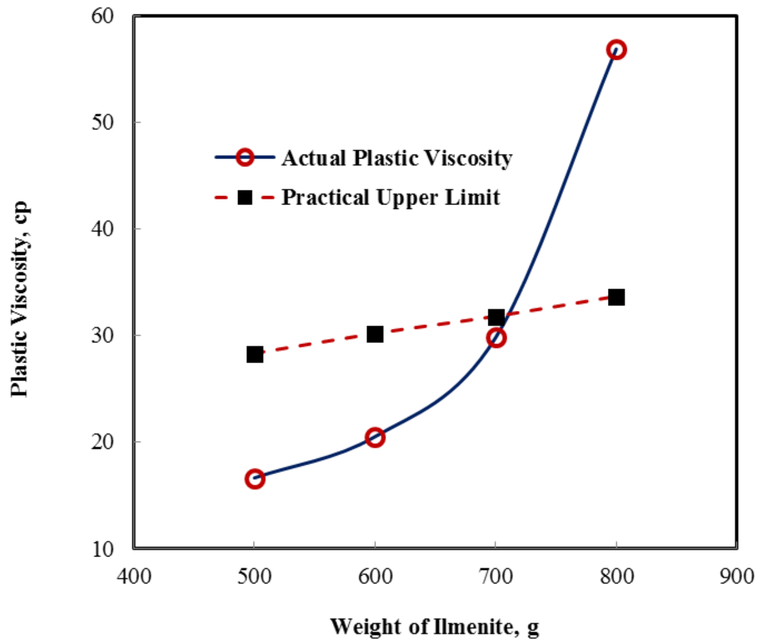

Fig. 8—Plastic viscosity as a function of weight of ilmenite.

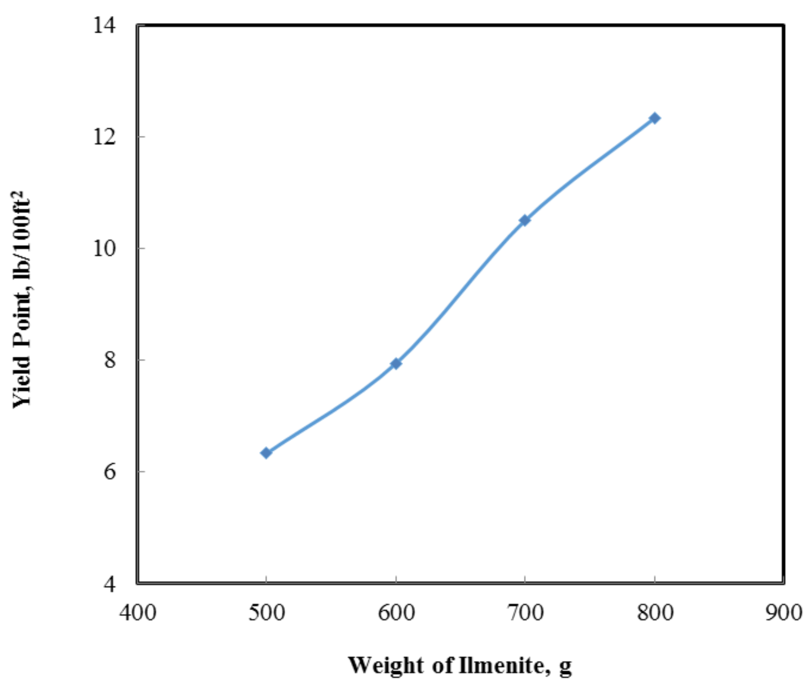

Fig. 9—Yield point as a function of weight of ilmenite.

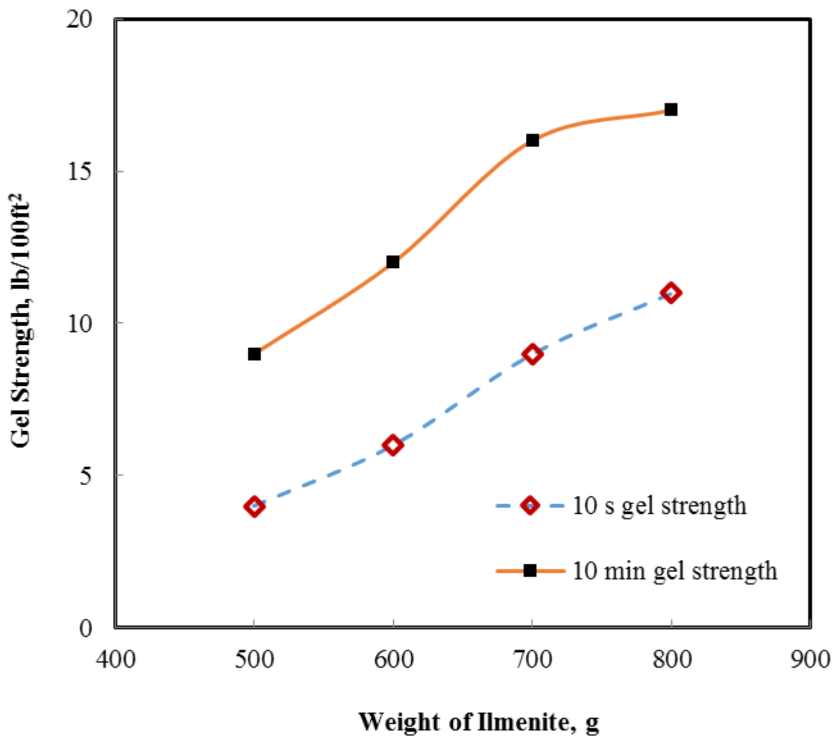

Fig. 10—The gel strengths as a function of weight of ilmenite.

Besides, this large increase in plastic viscosity with the addition of 800 g ilmenite is a sign of high particle-particle interaction due to the high solid content. One way to reduce such interaction is to lower the viscosifier concentration which is mainly used to suspend the solid and/or increase the wetting agent concentration. It seems in this particular system with a density of 1.92 SG the fluid becomes self-suspending. Therefore fluid was designed using a lower viscosifier concentration of 20 g/l mineral oil. The plastic viscosity was significantly diminished to 28 cp. This example shows how important is to take into consideration the effect of the specific surface area of the micronized material in formulating drilling fluids, particularly at high density. So with using micronized material less than 10 μm at high density the viscosifier concentration should be reduced and the oil-wetting concentration increased to satisfy the high specific surfaced area of the fine particles to produce a well dispersed fluid. A well dispersed fluid generally has low filtration properties and also shows low sag tendencies and low viscosities.

5.3 Effect of Various Drilling Fluid Additives

As we discussed above, good rheological properties of oil-based drilling muds can be achieved with the addition of 700 g ilmenite for specific amount of additives. More experiments are conducted to examine the effects of additives on the rheological properties.

Tables 7 and 8 show the rheological properties of oil-based drilling fluids with various amounts of additives. When 700 g ilmenite was used as the weighting agent, the following observations can be drawn.

- When we decreased the amount of filtration control agent from 25 to 18 g without the addition of $CaCO_3$, generally, the PV, YP, 10 s gel strength, and 10 min gel strength were constant. The filtration control agent in this study is organophilic lignites. It controls fluid loss by plugging and can be used at high concentrations without causing excessive viscosities. However, the thickness of the filter cake formed increased to 1.7 in. with the addition of 18 g filtration control agent.

TABLE 7—PROPERTIES OF OIL-BASED MUDS WITH 700 G ILMENITE.

	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7
Additive	700 g Ilmenite	700 g Ilmenite	700 g Ilmenite	700 g Ilmenite	700 g Ilmenite	700 g Ilmenite	700 g Ilmenite
Oil/Water Ratio	92.5:7.5	92.5:7.5	92.5:7.5	92.5:7.5	92.5:7.5	92.5:7.5	92.5:7.5
Mineral Oil, g	242.5	242.5	242.5	242.5	242.5	242.5	242.5
Viscosifier - Organically clay (modified hectorite), g	6	6	6	3	3	6	6
Viscosifier - Organically clay (organo-attapulgite), g	6	6	6	3	3	6	6
Lime, g	28	28	28	28	28	28	28
HT Primary Emulsifier - Polyamide, g	15	15	15	15	20	15	20
HT Secondary Emulsifier – Modified amide amine, g	15	15	15	15	20	15	20
Deionized Water, g	24.5	24.5	24.5	24.5	24.5	24.5	24.5
$CaCl_2$ 97%, g	8	8	8	8	8	8	8
HT Filtration Control - Organophilic lignite, g	25	18	18	18	18	12	18
HT Secondary Emulsifier - Polymerized fatty acid, g	1.5	1.5	1.5	1.5	1.5	1.5	1.5
$CaCO_3$ (50 µm)			17.5	17.5	17.5	17.5	
$CaCO_3$ (150 µm)			17.5	17.5	17.5	17.5	
Density, pcf	120	120	121	120	120.5	120	121
Plastic Viscosity,cp	31	33	28	22	22	28	28
Yield Point, lb/100ft^2	10	10	9	4	4	9	11
10 s Gel Strength, lb/100ft^2	7	7	6	3	2	6	7
10 min Gel Strength, lb/100ft^2	14	14	9	4	4	13	16

TABLE 8—PROPERTIES OF OIL-BASED MUDS WITH VARIOUS FORMULAS.

	Test 8	Test 9	Test 10	Test 11	Test 12	Test 13	Test 14
Additive	700 g Ilmenite	700 g Ilmenite	700 g Ilmenite	800 g Ilmenite	800 g Ilmenite	800 g Ilmenite	800 g Ilmenite
Oil/Water Ratio	80:20	80:20	80:20	92.5:7.5	92.5:7.5	92.5:7.5	92.5:7.5
Mineral Oil, g	209.6	209.6	209.6	242.5	242.5	242.5	242.5
Viscosifier - Organically clay (modified hectorite), g	3	6	3	6	3	3	6
Viscosifier - Organically clay (organo-attapulgite), g	3	6	3	6	3	3	6
Lime, g	28	28	28	28	28	28	28
HT Primary Emulsifier - Polyamide, g	15	15	15	15	15	15	20
HT Secondary Emulsifier – Modified amide amine, g	15	15	15	15	15	15	20
Deionized Water, g	65.7	65.7	65.7	24.5	24.5	24.5	24.5
$CaCl_2$ 97%, g	8	8	8	8	8	8	8
HT Filtration Control - Organophilic lignite, g	18	25	25	12	18	25	25
HT Secondary Emulsifier - Polymerized fatty acid, g	1.5	1.5	1.5	1.5	1.5	1.5	1.5
$CaCO_3$ (50 µm)	17.5				17.5		
$CaCO_3$ (150 µm)	17.5				17.5		
Density, pcf	123	121.5	121	126	127.5	125.5	124
Plastic Viscosity,cp	34	43	34	57	28	27	39
Yield Point, lb/100ft^2	10	20	11	12	4	5	12
10 s Gel Strength, lb/100ft^2	5	10	6	11	3	3	8
10 min Gel Strength, lb/100ft^2	10	20	9	17	5	5	16

- When we decreased the amount of filtration control agent from 25 to 18 g and added 17.5 g of $CaCO_3$ with average particle sizes of 50 and 150 μm, respectively, the PV, YP, 10 s gel strength and 10 min gel strength both decreased. The calcium carbonate acts as a bridging agent to rapidly establish an effective filter cake across permeable intervals during drilling operations, and it may interact with the other additives in this study to provide good rheological properties of the oil-based muds.
- When we increased the amount of emulsifiers from 15 to 20 g, the PV decreased a little while the YP, 10 s gel strength, and 10 min gel strength increased. The oil-based mud emulsifiers lowered the interfacial tension between the oil and the water, which allows more stable emulsions with smaller drops to be formed.

If we decreased the oil/water ratio from 92.5:7.5 to 80:20, the PV, YP, 10 s gel strength and 10 min gel strength increased dramatically; however, when we decreased the viscosifiers from 6 to 3 g with oil/water ratio 80:20, the PV, YP, 10 s gel strength and 10 min gel strength will change back to reasonable values, where the upper limit for the plastic viscosity is twice the fluid weight (lb/gal) and the initial gel strength should be above 5 lb/100 ft^2 to suspend the weighting material (M-I LLC. 1998).

When the amount of ilmenite was changed from 700 to 800 g, the following points were observed:

- When the viscosifier concentration decreased from 6 to 3 g and the amount of filtration control agent increased from 12 to 25 g, the PV, YP, 10 s gel strength, and 10 min gel strength all decreased greatly; especially, the YP, 10 s gel strength and 10 min gel strength became very small. The viscosifiers used in this study are organophillic clays, and they are used to viscosify oil-based drilling muds. They help increase the low-shear and static suspension capabilities of oil-based drilling muds. The decrease in the concentration of viscosifiers greatly reduced the viscosity of oil-based drilling muds.

- When we increased the amount of emulsifiers from 15 to 20 g and increased the amount of filtration control agent from 12 to 25 g, the PV decreased greatly; the YP, 10 s gel strength, and 10 min gel strength decreased a little. As we discussed before, the addition of emulsifier can help form more stable emulsions and increase the rheology properties of drilling muds. The addition of filtration control agent can function not to cause excessive viscosities, and therefore decreases the rheology properties of drilling muds.

The most important point of this set of tests is that using microfine particles as a weighting material in non-aqueous fluids requires a careful selection for the concentration of the emulsifiers and the viscosifiers.

5.4 Rheological Properties with Different Weighting Agents

Table 9 illustrates an example of the 1.92 SG oil-based drilling fluid weighted with API standard manganese tetraoxide, ilmenite, and barite. Table 6 also illustrates the drilling fluid rheology properties with different amounts of manganese tetraoxide, ilmenite, and barite.

The results in Table 9 show that the values of yield point, 10 s gel strength, and the 10 min gel strength when ilmenite was used and maintained at a reasonable value, were quite similar with that of manganese tetraoxide based drilling mud. When barite was used as the weighting agent, the values were low.

TABLE 9—PROPERTIES OF 1.92 SG OIL-BASED DRILLING FLUIDS WITH DIFFERENT WEIGHT AGENTS.

Additive	Value			Mixing Time, min
Mineral Oil, g	242.5	242.5	242.5	
Viscosifier - Organically clay (modified hectorite), g	6	6	6	10
Viscosifier - Organically clay (organo-attapulgite), g	6	6	6	10
Lime, g	28	28	28	10
HT Primary Emulsifier - Polyamide, g	15	15	15	10
HT Secondary Emulsifier – Modified amide amine, g	15	15	15	10
Deionized Water, g	24.5	24.5	24.5	10
$CaCl_2$ 97%, g	8	8	8	10
HT Filtration Control - Organophilic lignite, g	25	25	25	10
HT Secondary Emulsifier - Polymerized fatty acid, g	1.5	1.5	1.5	10
Weighting Agent- Mn_3O_4, g	670			20
Weighting Agent - $FeTiO_3$, g		700		20
Weighting Agent - $BaSO_4$, g			740	20
Density, pcf	120	120	120	
Plastic Viscosity, cp	22	31	34	
Yield Point, $lb/100ft^2$	15	10	8	
10 s Gel Strength, $lb/100ft^2$	7	7	5	
10 min Gel Strength, $lb/100ft^2$	13	14	13	

6. FILTRATION CONTROL OF FORMULATED OIL-BASED DRILLING MUD

In this section, the results of sag tests used to examine the sag tendency of the formulated oil-based drilling mud were first discussed. Then, the HPHT filtration tests were performed using a standard HPHT filter press under static and dynamic conditions (200 rpm). The cell was placed in a heating jacket, and the system was heated to a desirable temperature (300°F). The applied pressure was 300 psi differential pressure. Indiana limestone cores with an average porosity of 23 vol% and an average permeability of 100 md were used in the filtration tests.

6.1 Dynamic Sag Test

It appears that a VSST value of 1.0 ppg or less would imply the presence of a drilling fluid with minimal sag tendency (Aldea et al. 2001). A VSST value over about 1.6 ppg would indicate the beginning of a possible sag problem (Bern et al. 2010). **Table 10** shows that the drilling fluids using manganese tetraoxide, ilmenite, or barite as a weighting material have VSST values of less than 1 ppg, which indicates that the drilling fluids had no sag problems. The sag factor of ilmenite-based drilling mud is lower than that of barite-based based drilling mud. According to stokes' law, the settling velocity for the micronized ilmenite (5 µm) with a density of 4.5-4.7 g/cm^3 is less than the barite (15-20 µm) with a density of 4.2 g/cm^3.

TABLE 10—VSST RESULTS FOR OIL-BASED DRILLING FLUIDS.

Formula	W_2, g	W_1, g	VSST, ppg
700 g Ilmenite	18.01	17.88	0.108
670 g Manganese tetraoxide	18.34	18.22	0.100
740 g Barite	18.94	18.64	0.250

6.2 HPHT Static Filter Press

Table 11 shows the properties of 700 g ilmenite oil-based drilling fluid. It was noticed that this formula of oil-based drilling fluids has good gel strength and viscosity properties for Tests #1 to #3.

A filtration test was performed using an HPHT filter press under static conditions. The drilling fluid was put in the cell, the cell was placed in the heating jacket, and the temperature was adjusted to 300°F and 300 psi differential pressure. High permeability Indiana limestone cores with a thickness of 1 in. and a diameter of 2.5 in. were used in this experiment.

Fig. 11 shows that the amount of filtration was high (59.2 cm^3) and a low spurt volume (1.6 cm^3) was recorded when 12 g filtration control agent was used, which indicated a poor quality of the formed filter cake.

To reduce the amount of filtration, Test #3 was performed at the same conditions by increasing the amount of filtration control agent to 25 g. Fig. 11 shows that the amount of filtration decreases from 59.2 to 2.9 cm^3. **Fig. 12** shows that filter cake was formed with a thickness of 0.18 in., which is mainly due to the low volume of filtration.

TABLE 11—PROPERTIES OF OIL-BASED DRILLING FLUIDS WITH DIFFERENT AMOUNTS OF FILTRATION CONTROL AGENTS.

Additive	Test #1	Test #2	Test #3
Mineral Oil, g	242.5	242.5	242.5
Viscosifier - Organically clay (modified hectorite), g	6	6	6
Viscosifier - Organically clay (organo-attapulgite), g	6	6	6
Lime, g	28	28	28
HT Primary Emulsifier - Polyamide, g	15	15	15
HT Secondary Emulsifier – Modified amide amine, g	15	15	15
Deionized Water, g	24.5	24.5	24.5
$CaCl_2$ 97%, g	8	8	8
HT Filtration Control – Organophilic lignite, g	12	18	25
HT Secondary Emulsifier – Polymerized fatty acid, g	1.5	1.5	1.5
Weighting Agent - $FeTiO_3$, g	700	700	700
Density, pcf	120	120	120
Plastic Viscosity, cp	30	33	31
Yield Point, lb/100ft^2	10	10	10
10 s Gel Strength, lb/100ft^2	9	7	7
10 min Gel Strength, lb/100ft^2	16	14	14

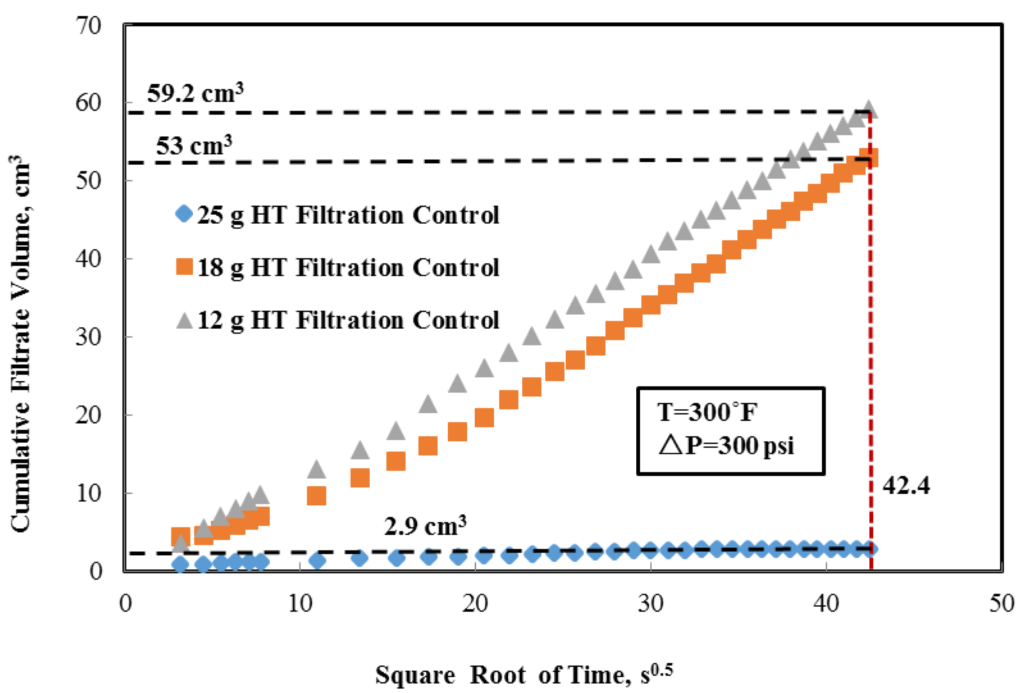

Fig. 11—Cumulative filtrate volume as a function of the square root of the time under static conditions.

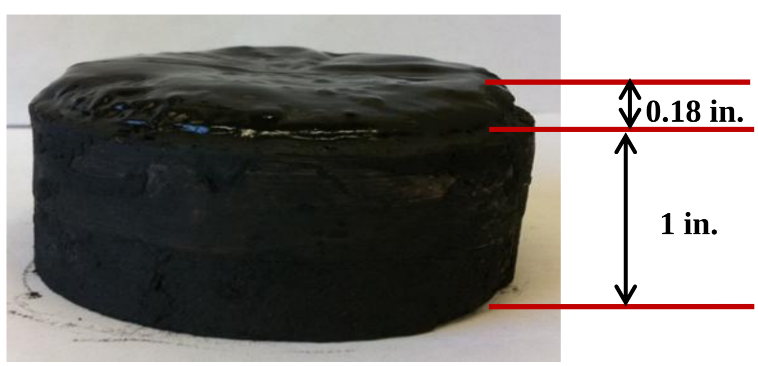

Fig. 12—Filter cake was formed when ilmenite was used as the weighting agent after 30 min. of filtration for Test #3 under static conditions.

6.3 HPHT Dynamic Filter Press

The volume of filtration was 2.9 cm^3 and the thickness of filter cake was 0.18 in. from previous static HPHT filter press test. These results show that current formula of oil-based drilling mud has a good fluid loss control behavior under static conditions. However, under dynamic conditions, the hydrodynamic effect of the mud flow, the filter cake erosion, and the variation in the erosional resistance of filter cake will make the fluid loss characteristics of drilling fluid differ significantly from those of static filtration. A dynamic filtration test (150 rpm) was conducted to evaluate the dynamic fluid loss characteristics of the oil-based drilling mud which was the same formula used in last static filtration test. A high permeability Indiana limestone core with a thickness of 1 in. and a diameter of 2.5 in. was used. The oil-based drilling fluid was put in the cell, the cell was placed in the heating jacket, and the temperature was adjusted at 300°F and 300 psi differential pressure. The filtrate rate was measured over a 30 minute period. **Fig. 13** shows that the volume of filtration was 2.2 cm^3 when 25 g HT filtration control was used. **Fig. 14** shows that filter cake was formed, with a thickness of 0.15 in., which is mainly due to the low volume of filtration.

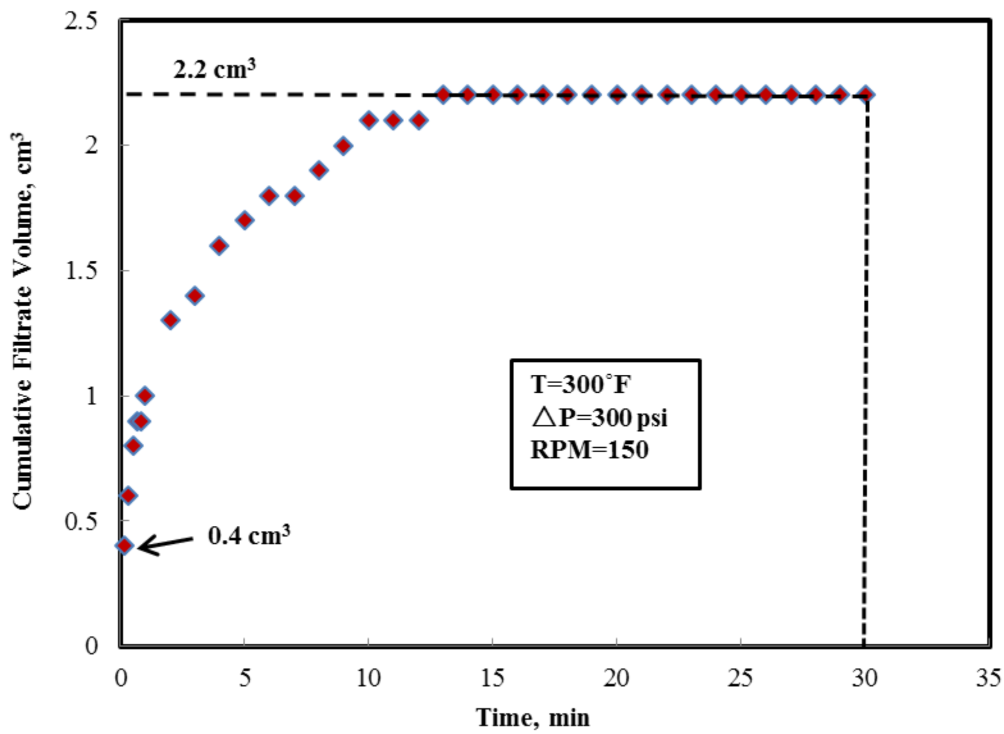

Fig. 13—Cumulative filtrate volume as a function of time under dynamic conditions (150 rpm).

Fig. 14—Filter cake was formed when ilmenite was used as the weighting agent after 30 min. of filtration under dynamic conditions (150 rpm).

6.4 Implications from the Sag Tests

The settling tendency of the weight material, known as barite sag, is a major drilling problem and it has been extensively investigated in literature. There are two main methods for testing sag by means of static and dynamic. Static sag simulates the settling situation under static conditions where dynamic sag simulates sag under conditions of shear. Prevention of sag may be managed by optimizing the fluid rheology to have good suspending properties or by lowering the size of the weighting material or a combination of both.

The former option works to a certain fluid density. The latter is called micronization of weighting material such as the one we present here. The dynamic sag (VSST) as of several ilmenite containing fluids were measured and were generally below 0.3 indicating no sag issue to be anticipated during drilling. Also the sag values were significantly less for ilmenite fluids compare to API barite. This is in line with the prediction applying Stokes' law, the settling velocity for the micronized ilmenite (5 µm) with a density of 4.6 g/cm^3 is less than the barite (15-20 µm) with a density of 4.2 g/cm^3.

7. EFFECT OF AGING AT HIGH TEMPERATURES

A hot rolling oven was used to examine the rheological properties of oil-based drilling muds shown in Table 9 after heating at 400℉ for 16 hours. A volume of 200 cm^3 drilling mud inside a Telfon liner was pressurized at 300 psi using a nitrogen source, and it was put into the hot rolling oven to be heated up to 400℉ for 16 hours. Then, the rheological properties of drilling muds were measured at 120°F by Grace M3600 viscometer. A HPHT dynamic filtration test and a sag test were also conducted after the drilling mud was heated for 16 hours.

A high permeability Indiana limestone core with a thickness of 0.25 in. and a diameter of 2.5 in. was used in the dynamic filtration test. The results in **Table 12** clearly show that the rheological properties of oil-based drilling muds using ilmenite or manganese tetraoxide as the weighting agents were almost constant after heating at 400℉ for 16 hours. However, the oil-based drilling mud using barite as the weighting agent did not maintain good rheological properties after heating at 400℉ for 16 hours. The 10 s and 10 min gel strengths are both below 5 lb/100 ft^2, which is too low to suspend the weighting material for HPHT applications. For the oil-based mud using ilmenite as the weighting agent, the sag factor was kept low (0.298 ppg). The filtration volume of the mud under dynamic conditions was low (2.3 cm^3) in **Fig. 15**, and the thickness of the filter cake was thin (0.21 in) shown in **Fig. 16**. The filter cakes shown in **Figs. 17 and 18** were also very thin when manganese tetraoxide or barite was used as the weighting agent in the oil-based muds. Those results indicate that the optimized oil-based drilling fluids using either ilmenite or manganese tetraoxide as the weighting agents are very suitable for HPHT applications.

The same observations are made for the new optimized formulas of 1.92 SG oil-based drilling muds with different weighting agents shown in **Tables 13 and 14**. Those new formulas clearly show that the rheological properties of oil-based drilling mud using barite as the weighing agents are much higher than the others. However, in this case, the filtrate volume was low, and the formed filter cake under dynamic conditions was thinner. All of the filter cakes formed before and after hot rolling are shown in **Figs. 19 to 21**. The aging at 400℉ for 16 hours can slightly increase the rheological properties as well as the filtration volume and thickness of the filter cake for all of the oil-based muds using different weighting agents.

TABLE 12—PROPERTIES OF OIL-BASED DRILLING FLUIDS AFTER HEATING AT 400 ℉ FOR 16 HOURS.

Test Results	**Manganese tetraoxide**		**Ilmenite**		**Barite**	
	BHR	AHR	BHR	AHR	BHR	AHR
Density, pcf	120	120	120	120	120	120
Plastic Viscosity, cp	22	22	31	39	34	36
Yield Point, $lb/100ft^2$	15	12	10	6	8	1
10 s Gel Strength, $lb/100ft^2$	7	7	7	6	5	2
10 min Gel Strength, $lb/100ft^2$	13	17	14	13	13	5
VSST, ppg	0.100	0.267	0.108	0.298	0.250	0.500
Filter Cake Thickness Under Dynamic Conditions, in.	0.21	0.26	0.15	0.21	0.20	0.23
Volume of Filtrate, cm^3	2.6	3	2.2	2.3	1.7	2.2

*BHR: Before Hot Rolling

*AHR: After Hot Rolling

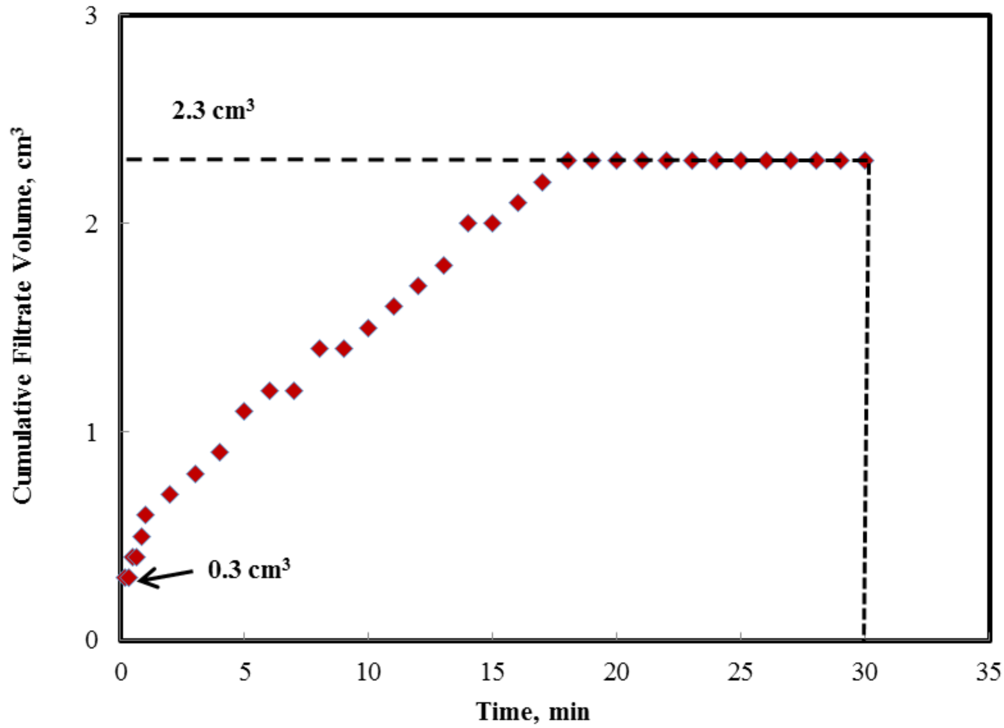

Fig. 15—Cumulative filtrate volume as a function of time under dynamic conditions (150 rpm) after heating at 400 ℉ for 16 hours.

Fig. 16—Filter was cake formed when ilmenite was used as the weighting agent under dynamic conditions (150 rpm) after heating at 400 ℉ for 16 hours.

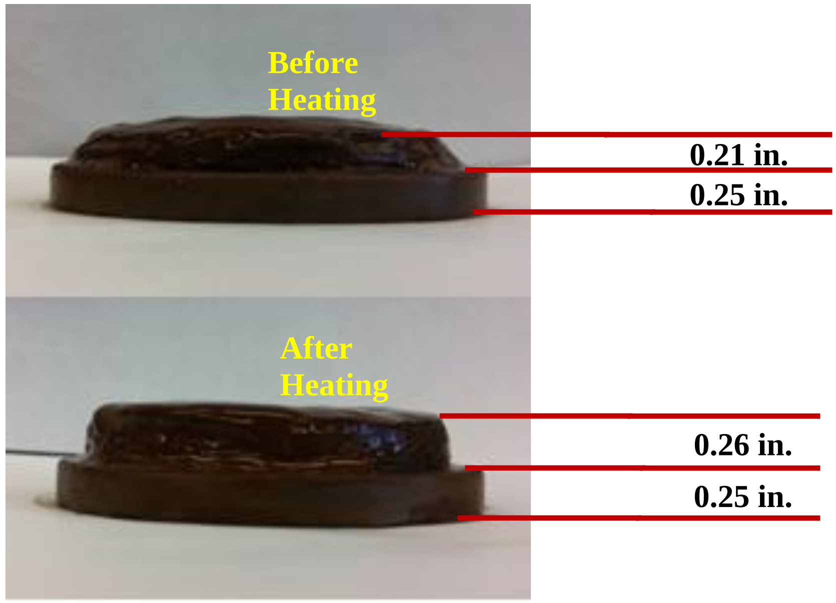

Fig. 17—Filter cake was formed when manganese tetraoxide was used as the weighting agent under dynamic conditions (150 rpm) before and after heating at 400 °F for 16 hours.

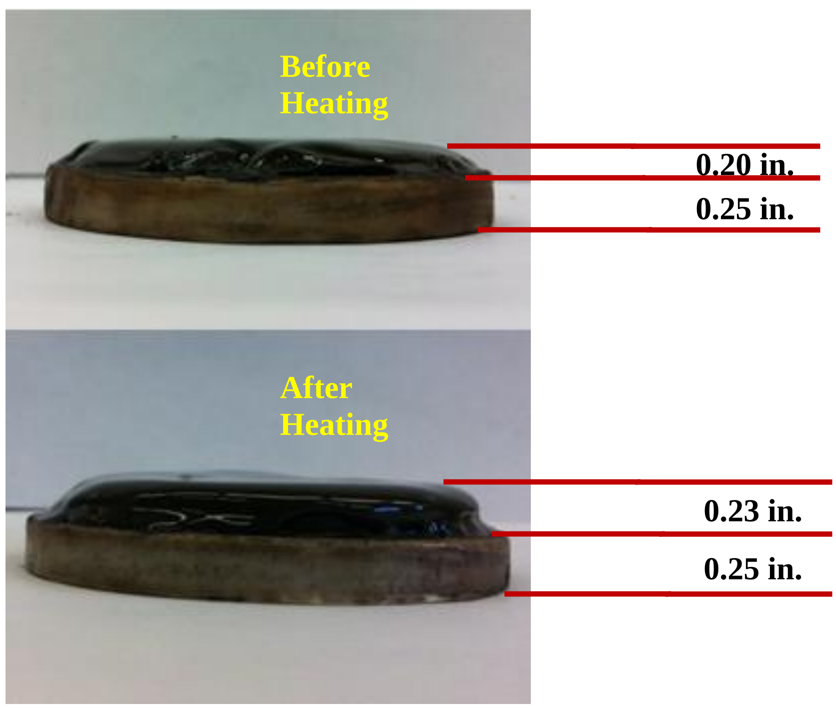

Fig. 18—Filter cake was formed when barite was used as the weighting agent under dynamic conditions (150 rpm) before and after heating at 400 °F for 16 hours.

TABLE 13—PROPERTIES OF 1.92 SG OIL-BASED DRILLING FLUIDS WITH THE NEW FORMULA.

Additive	Value			Mixing Time, min
Mineral Oil, g	242.5	242.5	242.5	
Viscosifier - Organically clay (modified hectorite), g	3	3	4	10
Viscosifier - Organically clay (organo-attapulgite), g	3	3	4	10
Lime, g	30	30	30	10
HT Primary Emulsifier - Polyamide, g	30	30	30	10
HT Secondary Emulsifier – Modified amide amine, g	25	25	25	10
Deionized Water, g	24.5	24.5	24.5	10
$CaCl_2$ 97%, g	8	8	8	10
HT Filtration Control - Organophilic lignite, g	23	23	23	10
HT Secondary Emulsifier - Polymerized fatty acid, g	2.8	1.8		10
Weighting Agent- Mn_3O_4, g	665			20
Weighting Agent - $FeTiO_3$, g		700		20
Weighting Agent - $BaSO_4$, g			760	20

TABLE 14—PROPERTIES OF 1.92 SG OIL-BASED DRILLING FLUIDS WITH NEW FORMULA AFTER HEATING AT 400 ℉ FOR 16 HOURS.

Test Results	Manganese tetraoxide		Ilmenite		Barite	
	BHR	AHR	BHR	AHR	BHR	AHR
Density, pcf	118	118	120	120	121	121
Plastic Viscosity, cp	17	18	23	25	27	28
Yield Point, $lb/100ft^2$	9	8	5	5	4	4
10 s Gel Strength, $lb/100ft^2$	4	4	3	3	3	3
10 min Gel Strength, $lb/100ft^2$	7	6	5	6	6	4
VSST, ppg	0.116	0.216	0.150	0.308	0.175	0.441
Filter Cake Thickness Under Dynamic Conditions, in.	0.3	0.38	0.23	0.27	0.1	0.12
Volume of Filtrate, cm^3	8	11	3.1	3.5	1.4	1.6

*BHR: Before Hot Rolling

*AHR: After Hot Rolling

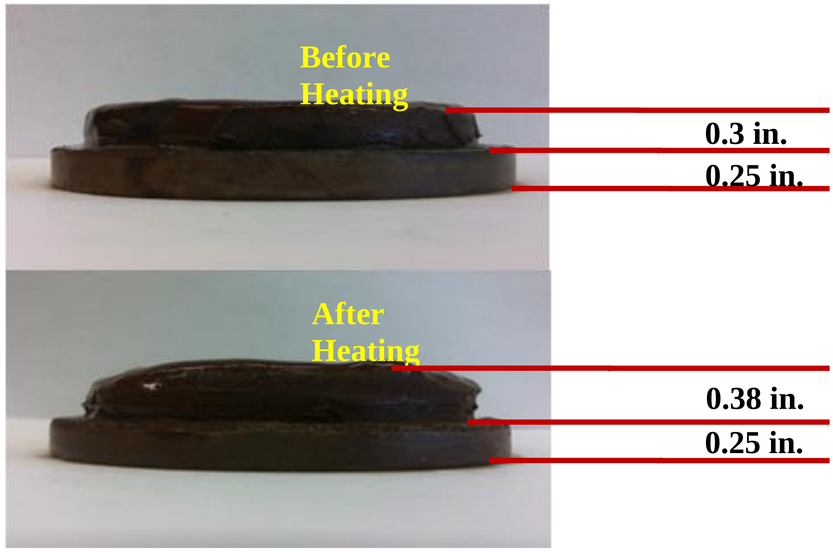

Fig. 19—Filter cake was formed when manganese tetraoxide was used as the weighting agent under dynamic conditions (150 rpm) with the new formula before and after heating at 400 °F for 16 hours.

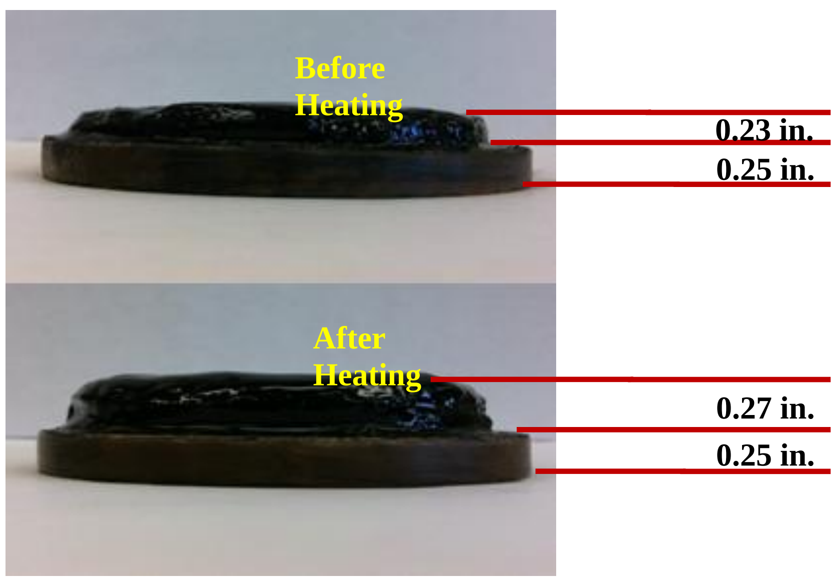

Fig. 20—Filter cake was formed when ilmenite was used as the weighting agent under dynamic conditions (150 rpm) with the new formula before and after heating at 400 °F for 16 hours.

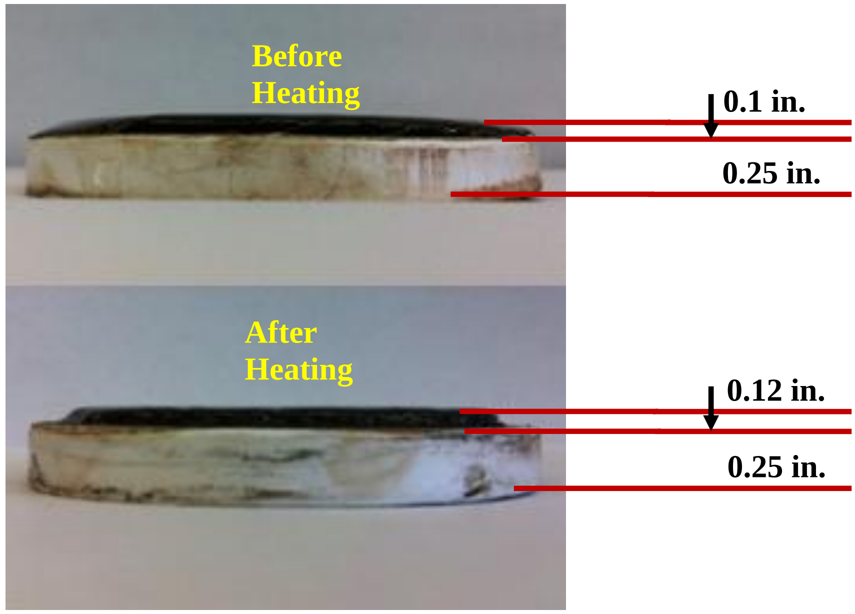

Fig. 21—Filter cake was formed when barite was used as the weighting agent under dynamic conditions (150 rpm) with the new formula before and after heating at 400 °F for 16 hours.

8. CONCLUSIONS

A series of experiments, including the rheological properties, sag test and filtration test were conducted to examine the performance of oil-based drilling fluids using ilmenite as a weighting material for HPHT applications. Based on the experimental results, the following conclusions can be made:

- Micronized ilmenite can be successfully used as a weighting material in non-aqueous drilling fluids with a wide density range.
- The specific surface area and the finesses of micronized material should be taken into consideration when formulating new fluids. Generally micronized ilmenite requires less viscosifier to be suspended, but to be well dispersed it needs slightly more dispersant and wetting agent when compared to API barite.
- Micronized ilmenite containing fluids show good filtration properties (a thin filter cake and the small filtrate volume) under static and dynamic conditions at 300°F and 300 psi.
- Micronized ilmenite shows low dynamic sag (< 0.3), which make it suitable for challenging drilling such as HPHT, narrow pressure margin drilling and extended reach wells.
- Oil-based drilling fluids using ilmenite as the weighting material are suitable for HPHT drilling operations with good rheological properties and low filtration volumes, even after heating at 400°F for 16 hours.

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