A COMPARATIVE STUDY ON THE EFFECTS OF DIFFERENT WATER-BASED WEIGHTING AGENTS ON FORMATION DAMAGE

A Dissertation

by

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ABSTRACT

Utilizing fluids in oil and gas drilling operations is unavoidable. Chemical properties of the fluid are critical, as they directly affect drilling performance and most importantly, formation damage in production zones. Most drilling fluids are largely composed of solids, particularly weighting materials, which increase density and control formation pressures. However, these weighting agents can also plug pores and cause formation damage leading to significantly reduced productivity and the need for remediation. The objective of this paper is twofold: to evaluate and compare solids invasion and damage characteristics of different weighting materials in water-based drilling fluids, and to study the solubility behavior of micronized ilmenite in different acid systems. Several fluid systems were prepared using weighting agents differing in size, morphology, and chemical nature: primarily API barite, micronized barite, and micronized ilmenite. Rheological properties were measured and high pressure/high-temperature static filtration experiments were conducted to investigate filtration behavior and filter cake thickness. A modified coreflood setup was used to simulate dynamic drilling conditions downhole and accurately measure the effect of solids on formation damage. Computed-tomography scan analysis provided the extent of the damage associated with each weighting agent. Experimental results showed that the formation damage created by micronized ilmenite is much lower, compared to API and micronized barite in both Bandera, Berea and Boise sandstones. HP/HT solubility reactions with different blends of acids were monitored. The best acid system was tested using the mudloop to ensure that it results in an effective breaker system to remediate damage from drilling operations.

DEDICATION

To my family, thank you for always being supportive and for believing in me.

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NOMENCLATURE

A	Cross Sectional Area
API	American Petroleum Institute
cP	Centipoise
CT	Computed Tomography
DF	Drilling Fluid
DI	Deionized
DOCTA	Dioxaoctamethylene Dinitrilo Teraccetic acid
DTPA	Diethylenetriaminepentaacetic Acid
ECD	Equivalent Circulating Density
EDTA	Ethylene Diamine Tetra-acetic Acid
ERD	Extended Reach Drilling
HCl	Hydrochloric Acid
HEDTA	Hydroxyethyl Ethylene Diamine Triacetate
HPHT	High Pressure High Temperature
k	Core Permeability
K_d	Post Drilling Fluid Permeability
K_o	Original Core Permeability
K_R	Return Permeability
KCl	Potassium Chloride
L	Core Length
ppg	Pounds per gallon
PSD	Particle Size Distribution

psi	Pound-Force per Square Inch
PV	Plastic Viscosity
Q	Flow Rate
RPM	Revolution per Minute
ROP	Rate of Penetration
SG	Specific Gravity
WBM	Water Based Mud
WBMWAFS	Water-Based Micronized Weighting Agent Fluid System
YP	Yield Point
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence
ΔP	Differential Pressure
μ	Fluid Viscosity

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CHAPTER I

INTRODUCTION

Drilling Fluids

A drilling fluid or mud is a specially designed fluid that is used in a drilling operation in which that fluid is circulated or pumped from the surface, down the drill string, through the bit, and back to the surface via the annulus (Growcock and Harvey 2005). The successful completion of an oil well depends to a considerable extent on the properties of the drilling fluid used.

Drilling Fluid Functions

The choice of the right fluid and maintenance of its properties while drilling is of great importance. Some of the key functions of drilling fluids include:

1. Prevent the inflow of fluids- oil, gas, or water- from permeable rocks penetrated by controlling the formation pressure while minimizing any fractures in the wellbore. These functions are controlled by monitoring the fluid's density and the equivalent circulation density (ECD) (Caenn et al. 2017). Typically, the chosen fluid density value is between formation pore pressure and formation rock fracture pressure. The most common drilling method, known as overbalanced drilling, is to maintain a fluid density both static and dynamic that lies within a safe margin (about 300 psi) above the formation pore pressure (Al-Mujalhem 2021).
2. The fluid must have the required physical properties; the fluid must have a viscosity that is readily pumpable and easily circulated by pumping at pressures ordinarily employed in drilling operations, without undue pressure differentials.

3. Carry cuttings from beneath the bit, transport them up the annuls, and permit their separation at the surface.
4. The fluid must also be sufficiently thixotropic to suspend the cuttings in the borehole when fluid circulation stops. The physical properties of the fluid also need to remain unchanged if cuttings were to be suspended or dissolved.
5. Form a thin-low permeability filter cake that seals pores and prevents the loss of fluid from the drilling fluid by filtration into the formation. An effective filter cake will seal the borehole wall to inhibit any tendencies of sloughing, heaving, or cave-in of rock into the borehole (Fink 2015).
6. Maintain the stability of uncased sections of the borehole. The drilling fluid should maintain the integrity of the wellbore until the next casing setting point is reached.
7. Reduce the friction between the drilling string and the sides of the hole.
8. Cool and clean the bit.
9. Assist in the collection and interpretation of information available from drill cuttings, cores and electrical logs (Caenn et al. 2017).
10. Not corrode or cause excessive wear to the drilling equipment.
11. Minimize impact on the environment (Growcock and Harvey 2005).
12. Not cause formation damage or interfere with the normal productivity of the oil-bearing formation

Drilling Fluid Composition

The selection of the proper drilling fluid is important to the success of a drilling operation. No fluid is suitable for all situations. The drilling formulation to be chosen will depend on many

factors. The most important are safety, bottom hole conditions like temperature and pressure, environmental regulations, formation type, loss zones, shale problems, well depth and trajectory, offset well history, and economics (Bleier 1990). Traditionally, drilling fluids have been classified into three categories according to their base fluid. These are air, water and oil. Most of the worlds drilling operations use water-based drilling fluid and this will be my focus throughout my research (Caenn and Chillingar 1996). The drilling fluid system is composed of the base fluid, and a combination of different fluid additives. In water-based drilling fluids, fresh water, seawater and brines are the typical base fluids used. Oil may also be emulsified in the water; however, the water remains to be the continuous phase. Solids or weighting agents are inert high-density minerals added to increase the density of the drilling fluid to control the formation pressure (Caenn et al. 2011). A fluid-loss-control additive such as clays, dispersants and polymers are added to minimize the amount of fluid lost into the formation. Lost-Circulation materials are also another group of additives that can be used in extreme cases where large volumes of drilling fluids are lost into the formation. Viscosifiers are used to increase the viscosity of the fluid and enhance its rheological properties, xanthan gum is a polymer and it is the most commonly used type. Salts are also added to inhibit shale formations and prevent clays from swelling. Thinners or dispersants are added to reduce flow resistance and gel development. Some of the various other additives added include surfactants, defoamers, lubricants, pH control agents, corrosion inhibitors, biocides, and hydrogen sulfide scavengers (Grey and Darley 1980; Alcheikh and Ghosh 2017).

Drilling Fluid Weighting Agents

Drilling fluid weighting agents or weighting materials are high specific gravity additives that are used to adjust the density of the drilling fluids. A weighting material must provide the

necessary rheology, have low settling tendency, should be hard enough not to create any fines, should be easily cleaned and not hazardous to the environment, should not cause formation damage, should not cause any abrasion or damage to the drilling equipment, should be cost effective and readily available. There are several types of weighting agents available, each has its advantages and disadvantages, some of the most common weighting agents include: barite, hematite, ilmenite, manganese tetroxide, calcium carbonate and siderite (Al-Bagoury and Steele 2012).

Barite

Barite ($BaSO_4$) or API standard barite is by far the most used weighting agent in drilling fluids. Roughly 6 million tons are produced and traded globally every year, almost half of it is sources from China and India. Barite has been used as a weighting agent since the 1920s, it is preferred over other materials because of its high density, low abrasiveness and ease of handling. Barite specific gravity is usually around 4.1-4.2, meaning that mud densities can reach 20lb/gal or more (Civan 2011; Elkatatny et al. 2012). Barite however is a relatively soft material that over time will grind down to very small sizes and can give fluctuating viscosities and gel strength and can possibly damage the producing zone (AlAbdullatif et al. 2014). Barite may also contain several heavy metal components such as lead, cadmium, mercury and arsenic (Fjogstad et al. 2000; Elkatatny el al. 2013). Barite sag refers to the settling of drilling fluid weight material in the wellbore and this can lead to density variations as high as 4 lb/gal. Occurrence of sag is a major concern and can lead to many drilling complications such as well control problems, lost circulation, induced wellbore instability, and stuck pipe. Tehrani et al. used laboratory tests to demonstrate the sag effects of barite under static and dynamic conditions (Tehrani et al. 2009).

Another major disadvantage of barite is that it is insoluble in water and acids such as hydrochloric acid, formic, citric and acetic acid making it hard to clean and expensive to remove the filter cake. Barite has also low solubility in chelating agents like EDTA (Ethylene diamine tetra acetic acid) and HEDTA (Hydroxyethyl ethylene diamine triacetate) (Ba Geri et al. 2017; Mahmoud and Elkatatny 2019). Lakatos et al. (2002) studied the dissolution rate of barium sulphate in seven different chelating agents. The results showed that the dissolution rate of barium sulphate can be increased with the usage of some chelating agents like DOCTA (Dioxaoctamethylene Dinitrilo Teraccetic acid) and DTPA (Diethylenetriamine Pentaacetate), however the cost of these products is unreal in industrial scale applications.

Barite supplies have been diminishing worldwide which led to an increase in barite prices that exceeded 100% as well as a decrease in the quality of the commercial barite. The API has recently introduced a new standard for 4.1 SG barite in recognition of the limited availability of high-quality barite (Al-Bagoury and Steele 2012; Tehrani et al. 2014).

Ilmenite

Ilmenite ($FeTiO_3$) as a weighting material offers many benefits over barite, ilmenite has a specific gravity of 5.1. This high density helps reduce the solid content of the drilling fluid and lowers the impact of the weight material on the fluid rheology (Tehrani et al. 2014). The heavy metal content of ilmenite is lower on most components than that of barite. In-addition it is claimed that the bioavailability of the heavy metals is lower in ilmenite than in barite. Amighi and Shahbazi (2010) stated in their paper that from an environmental perspective, it is desirable to use ilmenite instead of barite to eliminate the discharge of heavy metals while drilling with water-based fluids. Unlike barite, ilmenite is also readily available.

Ilmenite was first used in Norway in 1975 to drill two wells in the North Sea. Operators reported improved rheological properties when ilmenite was used, less fine particles, very low attrition rate, and no settling. All these properties led to a stable drilling fluid that did not need chemical conditioning (Blomberg et al. 1984). In 2001 Saasen et al. reported a higher drilling penetration rate when ilmenite was used, this is mainly because a lesser colloidal solids fraction was produced during drilling. Saasen et al. (2001) also reported that it was easier to control the drilling fluid properties because ilmenite has a lower tendency of being ground down to finer particles, consequently the need of fluid dilution is reduced. It was also reported that the fluid had a longer lifetime and that the fluid can be reused. Idris et al. (1994) studied the performance of local Malaysian ilmenite and compared its performance to barite. The results showed that the local ilmenite has potential to be used as a weighting agent. Ismail et al. (1999) concluded that the use of ilmenite meant that solids content was reduced in the drilling fluid which led to higher rate of penetration due to the presence of less solids in the vicinity of the rotating drill bit. The paper also showed that ilmenite could potentially reduce formation damage as it had a lower volumes of fluid loss as compared to barite. Another advantage of ilmenite is that occupational hygiene is improved due to the fact that ilmenite is a black powder which makes it easier to locate in ventilation filters, leaking valves and mixing rooms (Fjogstad et al. 2000). Ilmenite is also acid soluble which makes it easy to clean and remove (Elkatatny et al. 2013).

A potential issue that rises with the use of ilmenite is its abrasiveness due to the high concentration of coarse material. Blomberg et al. (1984) observed higher abrasion with ilmenite than with barite particularly in the high flow velocity parts of the circulation system. Size reduction was suggested as a means to reduce abrasion, with no more than 3% of the iron-oxide particles larger than 45 μm (Tehrani et al. 2014). Another disadvantage of ilmenite is its interference with

the logging and directional drilling tools due to its para-magnetic nature. This issue can be solved by reducing the magnetic content to be less than 0.3 wt% so that the ilmenite will have very little or no effect on the tools (Al-Bagoury and Steele 2012).

Micronized Weighting Material

Recently, exploration for oil and gas has shifted towards prospects that are deeper and located in harsher conditions that require more advanced methods and trajectories for exploration (Al-Mujalhem 2021). Both high-pressure, high-temperature (HPHT) wells and long extended reach drilling (ERD) wells present extreme performance demands on the drilling fluids. In both cases there is generally a narrow operating window between the pore pressure and the fracture gradient that calls for effective management of the equivalent circulating density (ECD). Maintaining efficient hole cleaning with good suspension properties to avoid weighting agents settling is also critical (Taugbol et al. 2005; Carbajal et al. 2009). The drilling fluid must not only provide the necessary properties mentioned, it needs to also not interfere with the downhole drilling tools. All these requirements have led research to focus on identifying and enhancing alternatives to the current available weighting materials. Micronized barite and micronized ilmenite are two new alternatives that have been introduced within the past couple of years and they will be the focus of my study.

The difference between API barite and micronized barite lies in the particle size. The reduction in particle size can yield many benefits including: lower plastic viscosity (PV) and hence reduced ECD, lower friction factor which gives lower drag and torque while drilling, improved filtration properties and hence reduce the risk of differential sticking, the smaller particles can reduce the settling velocity and lower the sag properties of barite (Conn et al. 2007; Sharma at al.

2015). Mohamed et al. (2017) showed that by reducing the particle size of barite to a few microns, the drilling fluid became more stable. The results presented also show that the solubility of micronized barite was improved. There was a 5% enhancement in the removal efficiency of smaller sized barite particles.

Micronized ilmenite is dry ilmenite that has been micronized to a PSD with a D_{50} of approximately 5 µm and a D_{90} of approximately 12 µm as shown in **Figure I-1**. Micronized ilmenite is produced from the world's largest ilmenite open cast mine located in the southern part of Norway. The area has huge reserves of high-quality ilmenite ore with an estimate of >600 million tons. The production process of micronized ilmenite includes crushing, milling, separation, and refining to produce the required particle size (Al-Bagoury and Revil 2018). The morphology of the micronized ilmenite shows that the particles are almost spherical with a total circularity of 0.9. This circularity is of great interest from a rheological point of view as it provides fluids with lower plastic viscosities which means that fewer pumping requirements are needed (Al-Bagoury 2014; Ivan et al. 2018). **Table I-1** shows a comparison between the different options for weighting materials used in drilling fluids (Al-Mujalhem 2021).

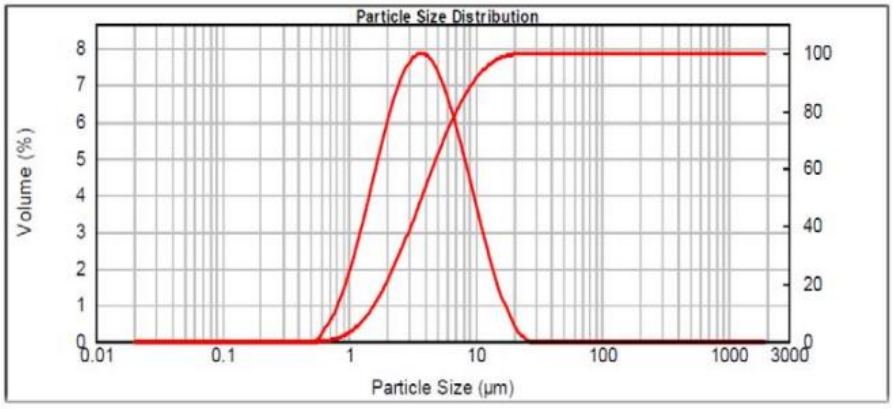

Figure I-1 Particle size distribution of micronized ilmenite. Reprinted from **(**Ivan et al. 2018)

Weighting Material	Density (SG)	D50 (µm)	Moh's Hardness	Acid Soluble?
API Barite	4.1-4.2	15-20	2.5-3.5	No
Micronized Barite	4.1-4.2	1-10	2.5-3.5	No
Ilmenite	5.1	30-45	5-6	Yes
Micronized Ilmenite	5.1	5	5-6	Yes

Table I- 1 Comparison between the different weighting agents used in drilling fluids

Recently several field trials have been utilizing micronized weighting agents as an alternative to the standard API barite. Micronized ilmenite was used to drill a HPHT appraisal well located in offshore East Malaysia. The operator stated that the implantation of the micronized ilmenite-based HPHT system was successful. The fluid demonstrated stable fluid properties while drilling up to 17 ppg. The fluid also showed no sag tendency throughout the 4-day static period of wireline logging, and this led to a 30% cost reduction per barrel of fluid (Razak and Ezani 2020). In Cairn India's first ultra-HPHT well, micronized barite was used to drill the last three HPHT sections of the well and it proved to be highly successful in reducing the ECDs as well as providing good hole cleaning with minimum maintenance. This enabled the drilling to continue through the narrow pressure margin sections of the well. The drilling fluid was also found to be very stable even after a period of 10 days under static conditions (Sharma et al. 2015). Another field trial used a Water-Based Micronized Weighting Agent Fluid System (WBMWAFS) to drill an offshore well in Columbia. The results from this trial indicated that the WBMWAFS can deliver all the technical requirements required for a smooth operation, with no significant indication of weighting agent sag or variation in the fluid properties. Furthermore, there was no significant damage following the cleanup period (Motta and Loureiro 2021).

Formation Damage

Formation damage is a generic term that refers to the impairment of the permeability of any oil and gas producing formation imparted by various adverse processes that can occur at any time during the life of a well (Bennion 1999; Vryzas et al. 2017). Near wellbore permeability impairment from drilling and completion fluids can have a substantial, yet potentially avoidable, impact on well productivity (Krueger 1988). Well productivity is critically important if oil and gas reserves are to be developed economically. With the change in economic climate and the maturation of many existing fields has come an emphasis on reduced production cost and optimized productivity. The trend towards open hole completions places additional emphasis on damage avoidance. The proper design and engineering of fluid systems to minimize productivity impairment is therefore of utmost importance (Francis 1997; Bailey et al. 2000).

Formation Damage Mechanisms

Bennion (2002) stated that there are four primary mechanisms of formation damage: mechanical, chemical, biological and thermal. Zhao et al. (2019) further categorized the mechanisms of reservoir formation damage by drilling fluids into seven mechanisms: fluid-fluid incompatibilities, rock-fluid incompatibilities, solids invasion, phase trapping or blocking, chemical adsorption or wettability alteration, fines migration and biologic.

According to Longeron et al. (1995) the two main potential sources of damage due to water-based muds are mud particles plugging the near wellbore and mud filtrate invasion, specially if the filtrate is incompatible with the rock-fluid system activity (Liu and Civan 1994). Jiao and Sharma (1993) studied the effect of water-based muds under different conditions on formation damage using sandstone cores. In their paper they observed that the first damaging process was due to mud

particles invasion which mainly occurs during the initial spurt loss. They showed that the depth of invasion was strongly depended on the mud composition. The second mechanism of damage was caused by fines release and migration, and this can be significant if the salt concentration of the mud filtrate is less than the critical salt concentration required to prevent fines migration.

Filter Cake

As mentioned earlier one of the main functions of a drilling fluid is to form a filter cake over the face of the porous medium. A well-designed filter cake will stabilize the wellbore and will assist in reducing the invasion of solids and filtrate to an acceptable level (Elkatatny 2013; Gamal et al. 2021). It is well known that mud spurt loss occurs before the filter cake is established. During the filtration process, particles of certain sizes bridge the formation and establish a base on which the filer cake can form. Particles considerably smaller than the pore opening invades the formation. However, particles of certain sizes stick at the neck of the pore channel and form an internal bridge. Once the primary bridge is established, fine particles successively fill up the interstitial spaces to form a lightly paced layer of filter cake. In order to form an effective base for the filter cake, drilling fluid must therefore contain particles ranging in size from slightly less than the largest pore size of the formation to be drilled down to about one third of that size (Azizi et al. 1997; Rahman and Marx l991).

Filtration occurs under one of two mechanisms. The first mechanism is static filtration, which occurs when the fluid pumping is interrupted and from that point on, filtration occurs due to the difference between the hydrostatic pressure in the well and the reservoir pressure. The filtration rates are controlled by the continuously increasing thickness of the filter cake. In other words, static filtration occurs when the slurry is applied to a filter cake without crossflow, which

results in particles continuously deposited to form thicker cakes until the space available is full of the filter cake. The second mechanism is dynamic filtration, and it occurs when the fluid is pumped through the well. Dynamic filtration involves crossflow through the filter cake. In this process the cake thickness is resultant from the dynamic equilibrium between solid particles deposition rate and the erosion rate due to the shear stresses generated by the fluid flow in the wellbore. Thus, the filtration rate tends to stabilize around a certain value while the cake thickness turns constant (Scheid et al. 2010). Jiao and Sharma (1993) found that at the early stages of filtration both large and small particles deposit on the cake surface, because the drag force driving the particles to the cake surface is high. Subsequently, only smaller and smaller particles are deposited. The cake growth rate gradually decreases until an equilibrium filtration rate is attained at which no particles small enough to be deposited are available in suspension. This mechanism of cake growth gives rise to a heterogenous cake with both large and small particles at the bottom and only small particles in the external portion of the cake. **Figure I-2** below is a schematic of the formation of a filter cake.

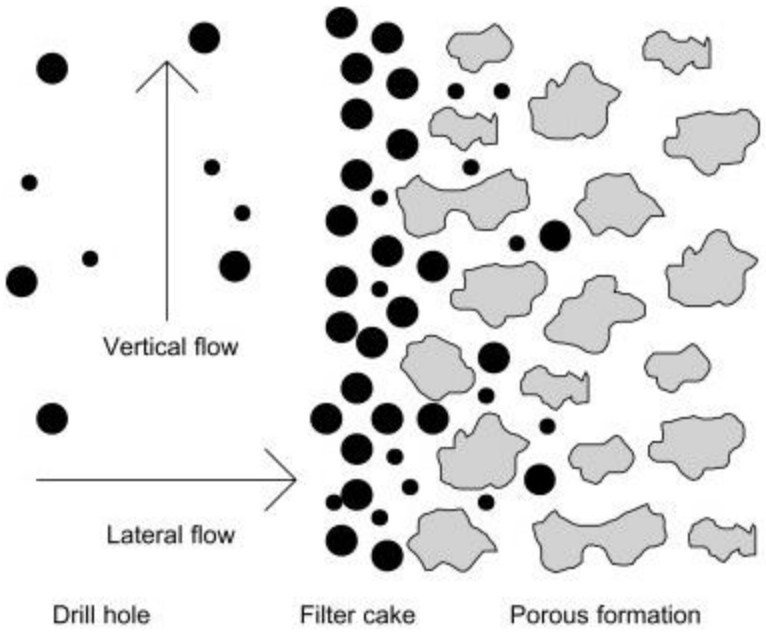

Figure I-2 Schematic of filter cake formation. Reprinted from (Reprinted from Fink 2015)

The quality of the filter cake depends on its thickness, heterogeneity and the speed by which it forms (Al-Mujalhem 2021). Thick filter cakes have high permeabilities and they cause various operational problems such as excessive torque, drag, high swab and surge pressures and differential pipe sticking (Elkatatny et al. 2012). Ershaghi (1980) concluded that the growth rate of the filter cake thickness can be greatly inhibited if the cake permeability is designed to be in the order of 10^{-7} Darcy or less. Bageri et al. (2021) stated that the filter cake permeability needs to be very low (10^{-5} to 10^{-7} Darcy) in order to be effective in protecting the hydrocarbon formation and preventing formation damage through solid invasion. Another important aspect to keep in mind is that a delay in the formation of the filter cake or its lack of bridging capabilities can cause an increase in the filtrate invasion into the rock formation, which in turn, increases the intensity of formation damage caused by the drilling fluids. In summary an ideal filter cake should be relatively

impermeable; it should also be uniformly thin and should have the ability to form rapidly (Al-Mujalhem 2021; Gamal et al. 2020).

Filter Cake Removal

The effective removal of drilling fluids filter cake during well completion is essential to reduce formation damage caused by drilling activities in production and injection wells. Wellbore clean-up treatment needs to be performed to reestablish the original permeability of the critical near wellbore area. Both mechanical and chemical means are used in the field for clean-up of the drilling fluids filter cake. Chemical means include acids, oxidizers, chelating agents and enzymes or a combination of these chemicals (Al-Ibrahim et al. 2015). The composition of the filter cake, specifically the weighting material used, is the main determinant when selecting the treatment method for the clean-up. Understanding the solubility of the weighting material used to make up the filter cake is crucial in order to achieve a successful cleanup (Al-Mujalhem 2021).

The kinetics of ilmenite dissolution in hydrochloric acid (HCl) was extensively studied by Van Dyk et al. (2002). The chemical reaction between ilmenite and HCl can be described as follows:

$$FeTiO_2 + 2HCl \rightarrow Fe^{2+} + TiO^{2+} + 2Cl^- + 2\ OH^-$$

The main parameters that can influence the rate of this reaction are particle size, acid concentration, temperature, stirring, acid-to-ilmenite mole ratio and additives. Al-Bagoury and Steele (2012) showed that the particle size of ilmenite has a great influence on the rate of dissolution, they used three different grades of ilmenite with an average particle size of 5, 18 and 70 µm. The solubility of ilmenite was tested at 100 °C using 10 and 20 wt% HCl. Their results showed that ilmenite with an average particle size of 5 µm had the highest dissolution rate. Van Dyk et al. (2002) stated in

their paper that the rate of ilmenite dissolution is strongly dependent on the acid concentration. Sinha (1984) showed that the rate of ilmenite dissolution increases rapidly as the leach temperature rises, the experiments were conducted at temperatures ranging from 80 to 108 °C. After 4 hours almost 95% of the iron was extracted at 108 °C, while less than 50% was extracted at 80 °C. Tsuchida et al. (1982) investigated the effect of the stirring speed (100-500 min^{-1}) and found that it did not have a significant effect on the amount of iron and titanium extracted. The initial acid-to-ilmenite mole ratio seems to influence whether both iron and titanium goes into solution or whether iron only goes into solution. The addition of certain additives to the leach solution can either increase or decrease the rate of ilmenite dissolution, adding bi-sulphate and fluoride to the leach solution can increase the dissolution of both iron and titanium. Furthermore, the addition of methanol, ethanol and ethylene glycol was also found to increase the dissolution rate of iron and titanium substantially (Girgin 1990). On the other hand, adding phosphoric acid and Ti(IV) to the leach solution decreased the dissolution rate of iron and titanium dramatically (Duncan and Metson 1982).

The kinetics of the dissolution of titanium and iron from ilmenite in sulfuric acid solutions was studied by Zhang and Nicol (2010), the authors tested both the presence and absence of reducing agents such as titanium (III) ions and sulfur dioxide in the solutions. The effect of particle size, temperature, solid/liquid ratio, sulfuric acid concentration and titanium (III) concentration on the kinetics of dissolution were all presented in their work. The dissolution of ilmenite in a high sulfuric acid concentration can be summarized by the reaction shown below (Fouda et al. 2010):

$$FeTiO_3 + 2H_2SO_4 = TiOSO_4 + FeSO_4 + 2H_2O$$

The dissolution rate of ilmenite was found to be enhanced under reducing conditions through the addition of metals such as zinc, tin and iron. The authors also found that both the particle size (25

to 90 µm) and acid concentration (450 to 600 g L^{-1}) have relatively minor effects on the rate of dissolution of titanium, while temperature (85 to 100 °C) has a significant effect.

The removal of water-based ilmenite filter cake was tested by Elkatatny et al. (2013), the authors concluded that 10wt % HCl dissolved 93wt% of iron after a soaking period of 10 hours and that HCl was sufficient to completely remove the filter cake after a soaking period of 16 hours at 250 °F (Siddig et al. 2020). Another study by J et al. (2021) compared the solubility of ilmenite water-based filter cake in two solutions; 7.5 wt% HCl and 7.5 wt% HCl mixed with 7.5 wt% HEDTA. The soaking time which the authors tested was six hours at a temperature of 250 °F. The authors recommended the usage of the second solution (7.5 wt% HCl + 7.5 wt% HEDTA) to prevent iron precipitation in the solution to avoid corrosion issues.

Research Objectives

The objective of this research is to analyze how different weighting materials used in water-based drilling fluids interact with the formation. Understanding the formation damage associated with the different weighting agents is imperative to help design the proper drilling fluid for each well.

1. This work will focus primarily on comparing the performance of the standard API barite to micronized barite and micronized ilmenite to evaluate the effect of the different materials as well as the effect of the particle size on the performance of the drilling fluid and its impact on formation damage.
2. The second phase of this work will evaluate the removal of the micronized ilmenite water-based filter cake. This research is unique in evaluating the filter cake clean-up methods under conditions that are very similar to the downhole conditions.

CHAPTER II

EXPERIMENTAL METHODS AND MATERIALS

This chapter will give a detailed description of the materials and equipment used. The experimental procedures followed to evaluate and compare the different weighting agents will be explained. Furthermore, a new approach will be presented to evaluate the filter cake cleanup.

Drilling Fluids Composition

The composition of the drilling fluids and the equipment used to formulate the fluids will be presented in this section. The tests conducted to condition the fluids as well as ensure that they have the necessary properties will also be described below.

Drilling Fluids Formulation

Four water-based drilling fluids were prepared using the materials listed in **Table II-1**. The four fluids have a density of 1.5 SG. The four fluids have an identical composition apart from the weighting material used. The weighting material used in fluid 1 is the standard API barite with an average particle size of 15 µm. The weighting material used in fluids 2 and 3 is micronized barite with an average particle size of 2 µm and 5 µm respectively. The weighting material used in fluid 4 is micronized ilmenite with an average particle size of 5 µm. **Table II-2** shows the particle size distribution of the four weighting materials. A fluid multimixer (shown in **Figure II-1**) was used at a speed of 11,500 rev/min to mix the different components of the drilling fluid formulation. The water that was used to prepare the drilling fluids was deionized water obtained from a water

purification system with a measured resistivity if 18.2 MΩ.cm. The chemicals used to formulate the drilling fluids were all provided by Elkem Silicon Products.

	Fluid 1	**Fluid 2**	**Fluid 3**	**Fluid 4**
Density	1.5 SG	1.5 SG	1.5 SG	1.5 SG
Material	g	g	g	g
Water	260.7	262.0	262.0	264.2
Xantham gum	0.3	0.3	0.3	0.3
ExStar HT	8.0	8.0	8.0	8.1
NaOH pellets	0.4	0.4	0.4	0.4
NaCl	91.3	91.7	91.7	92.5
CaCO3 medium (20µm)	6.1	6.1	6.1	6.2
CaCO3 large (30µm)	6.1	6.1	6.1	6.2
ESM D2, dispersant	2.0	2.0	2.0	2.1
Sodium hydrogen sulfite 39%	0.4	0.4	0.4	0.4
MgO	1.0	1.0	1.0	1.0
Microdense	0.0	0.0	0.0	140.4
API barite	146.7	0.0	0.0	0.0
Micorbarite (5µm)	0.0	0.0	143.3	0.0
Micorbarite (2µm)	0.0	143.3	0.0	0.0
Silcolapse 140 defoamer	0.2	0.2	0.2	0.2
Glutaraldehyde, 25% biocide	0.2	0.2	0.2	0.2
Total	523.4	521.8	521.8	522.1

Table II-1 Composition of the four different water-based drilling fluids

Figure II-1 Drilling fluid mixer

The micronized ilmenite that was provided by Elkem Silicon Products a trademark that is also known as Microdense®. This material comes in a black powder form. The X-ray diffraction (XRD) analysis of micronized ilmenite indicates that the mineralogy is composed of 88.12% Ilmenite, 9.42 hematite and trace amount of anorthite. **Figure II-2** shows the XRD mineral composition of the micronized ilmenite. **Table II-3** presents the elemental composition of the micronized ilmenite from the X-ray florescence (XRF) analysis. The composition of the micronized ilmenite includes iron, titanium, aluminum, magnesium and silicon oxides. Finally, **Table II-2** presents the particle size distribution of the micronized ilmenite, the D_{50} particle size is roughly 5 µm.

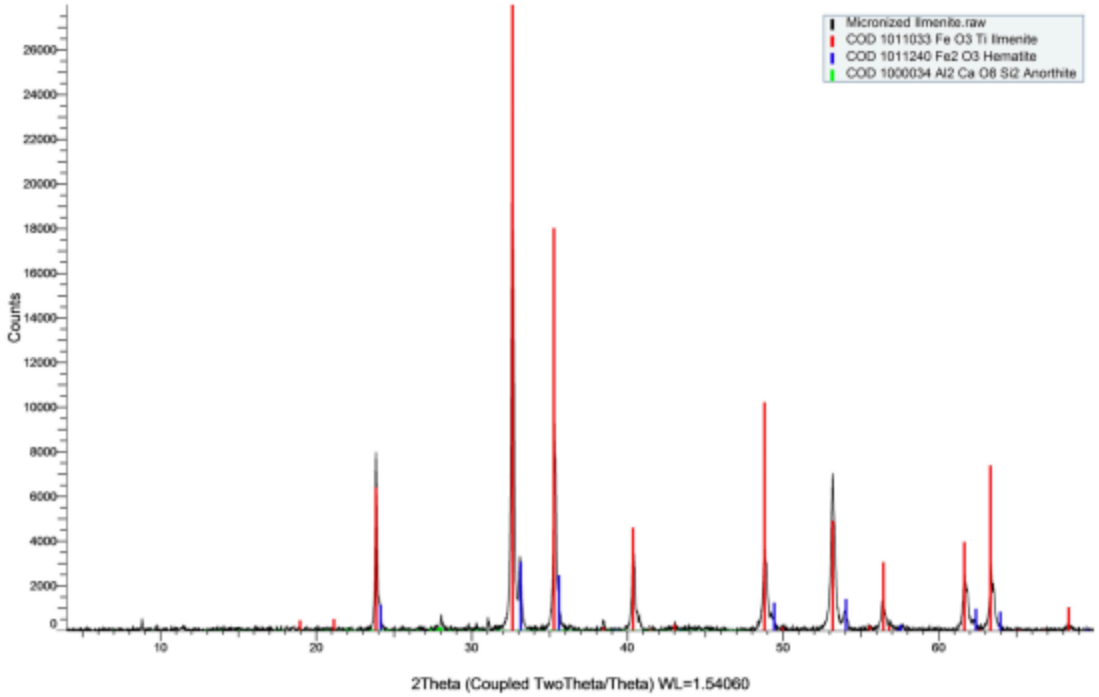

Figure II-2 XRD mineral composition of micronized ilmenite

Particle Size Distribution (µm)	D_{10}	D_{50}	D_{90}
Barite (2 µm)	1.3	2.7	5.6
Barite (5 µm)	2	5.4	11.3
Ilmenite (5 µm)	2.0	5.5	12.2

Table II-2 Particle size distribution of the weighting materials

XRF Analysis	Fe_2O_3	TiO_2	Al_2O_3	MgO	SiO_2
Composition (wt%)	51	43.1	2.4	0.54	2.97

Table II-3 XRF elemental composition of the micronized ilmenite

Drilling Fluids Conditioning

After the drilling fluids are formulated, they are exposed to high temperatures and pressures to simulate the down hole drilling conditions. This is done by using a Teflon liner which is inserted

into an aging cell as shown in **Figure II-3**. The aging cell is then tightly sealed by applying a pressure of 50 psi. The cell is then placed in a roller oven (**Figure II-4**) rotating at a speed of 25 rev/min to expose the fluid to the dynamic drilling conditions. The fluid is also exposed to high heat as the oven temperature is set at 250 °F for a period of 16 hours.

Figure II-3 Teflon liner and aging cell

Figure II-4 Roller oven

Drilling Fluids Rheological Properties

After the drilling fluids are formulated, the viscosity and gel strength are measured before and after heat exposure using the Fann 35 viscometer shown in **Figure II-5**. This is crucial to make sure that the properties of the drilling fluids are not affected by the exposure to the high temperature values. The rheology of the four drilling fluids was measured at six different shear rates: 600, 300, 200, 100, 6 and 3 rpm. The gel strength was also measured after 10 seconds and after 10 minutes.

Figure II-5 Fann 35 viscometer

Drilling Fluids Filtration Properties

High pressure high temperature (HPHT) filter press experiments were conducted to measure the filtration and filter cake properties of the four drilling fluids after they were hot rolled for a period of 16 hours. The HPHT tests were conducted at 250 °F and 500 psi differential pressure. The filtrate volume is collected after a period of 30 minutes and the filter cake thickness is measured. **Figure II-6** shows the set-up that was used.

Figure II-6 HPHT filter press set-up

Drilling Fluids pH

The pH of the four drilling fluids was measured using the pH meter shown in **Figure II-7**. This step is needed to make sure that the drilling fluid is not acidic and corrosive.

Figure II-7 pH meter

Formation Damage Evaluation

The second phase of experiments involved studying the effect of the different drilling fluids on formation damage. A modified coreflood set-up was used to assess the extent of formation damage from the four different weighting materials.

Core Preparation

Sandstone cores with different permeabilities ranges were used to represent the formation in our coreflood tests. The three main sandstone cores used were Bandera, Grey Berea and Boise sandstone cores to represent low, medium and high permeability respectively, **Figure II-8, 9, and 10** show the three core types used. The sandstone cores were cut to have a 1.5 in diameter and 6 in length. The cores were dried in the oven over-night at a temperature of 150 °F. The dry weight was then recorded, and the cores were scanned using computed tomography (CT) imaging. The cores were then saturated with the brine under a vacuum for a period of 8 hours. The composition and properties of the brine used to saturate the cores are shown in **Table II-4**. The saturated weight

of the cores was then recorded, and the core pore volume was calculated. The saturated cores were then CT-scanned.

Figure II-11 shows the CT scan machine used (Toshiba Aquilion RXL). The CT scanner measures the radiodensity value of every slice of the core to give an average CT number per slice. An image processing program, ImageJ, is then used to plot the values of the CT to give a qualitative image of the pore space distribution across the core. A change in the CT number after the core has been exposed to drilling fluids is an indication that the pore spaces are filled with a denser material (drilling fluid solids have displaced the brine) which is an indication of formation damage.

Figure II-8 Bandera sandstone core

Figure II-9 Grey Berea sandstone core

Figure II-10 Boise sandstone core

Composition		
Material	Weight (g)	
KCl	100	
DI Water	900	
Total	1000	
Properties		
Viscosity (CP)	70 °F	250 °F
	1	0.3

Table II-4 Composition and properties of the brine used

Figure II-11 Computed Tomography (CT) imaging

Mud-Loop Setup

A modified coreflood setup, also known as mudloop, was used to simulate the downhole conditions to accurately assess the effect of the four different weighting materials on formation damage. The modified coreflood set-up used was initially introduced by Ibrahim et al. (2018) to perform dynamic filtration testing that resembles the actual circulation process more precisely. **Figure II-12** shows a schematic of the set-up used. In the coreflood setup a one-liter accumulator was used to store the brine and was connected to the inlet of the core holder for initial and final permeability measurements. Two other accumulators were connected to the outlet to create a drilling fluid circulation loop. Pressure was controlled by pressure regulators, a syringe pump was used to inject the fluids, and a hydraulic hand pump was used to control the overburden pressure. A pressure transducer was used to measure the pressure differential between the core inlet and outlet during the permeability measurements, and the data was recorded using LabVIEW software. The entire set-up is inside an oven to ensure the control of the desired temperature. **Figure II-13** shows the use of a spacer at the core outlet interface, which allowed for filter cake formation and drilling fluid circulation (Al-Mujalhem 2021).

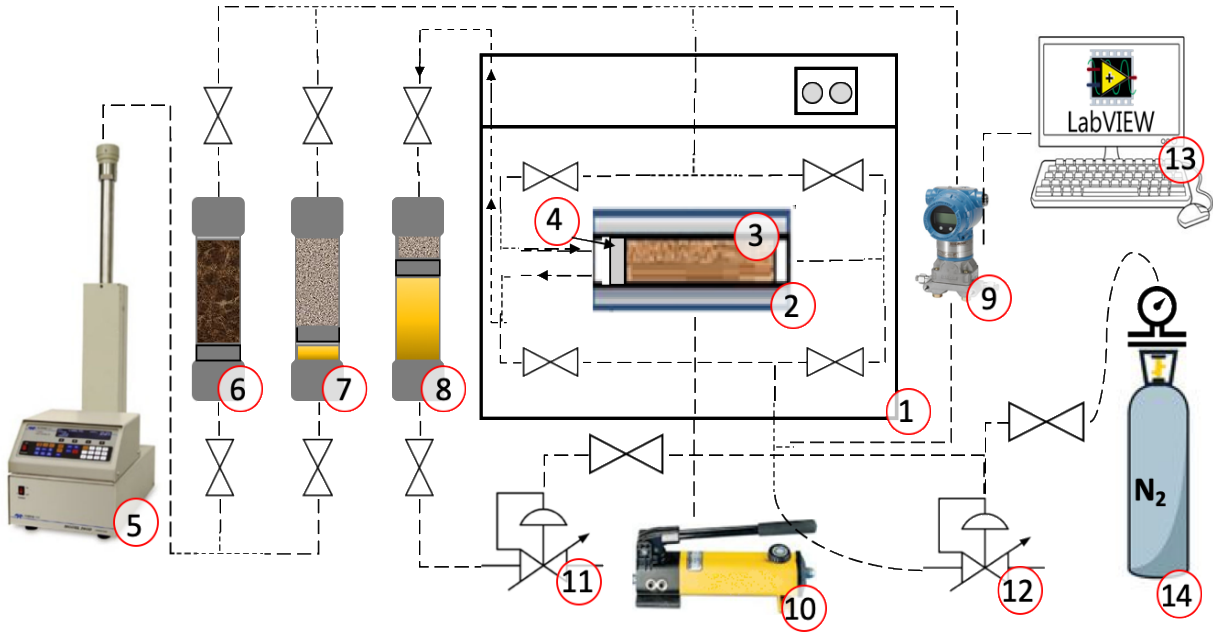

Figure II-12 Schematic of coreflood setup used, including: (1) oven, (2) core holder, (3) core, (4) spacer, (5) syringe pump, (6-8) fluid accumulators, (9) pressure transducer, (10) hydraulic hand pump, (11, 12) backpressure regulators, (13) data acquisition and recording system, and (14) compressed gas supply (Reprint from Al-Mujalhem 2021).

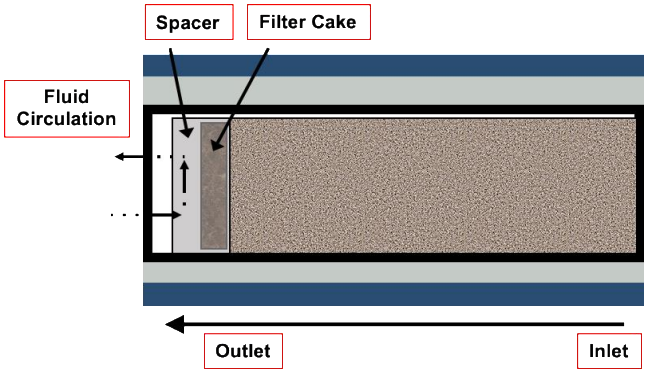

Figure II-13 Drilling fluid circulation inside the core holder (Reprinted from Al-Mujalhem 2021).

The saturated core is inserted into the core holder and the spacer is inserted from the outlet direction. The backpressure regulator was set to 100 psi, this ensures that the brine flows in the desired direction. Brine was then injected at a constant flow rate from the inlet direction for

permeability measurements. The pressure drop across the core was recorded continuously until stabilization. This process was repeated using several different flow rates. Afterward, the initial permeability was calculated using Darcy's equation (Alkhalaf et al. 2019) (eq.1).

$$k = \frac{245q\mu L}{\Delta PA} \qquad \textbf{Eq.1}$$

Where,

k : Core Permeability, md

q: Flow rate, cm^3/min

μ : Fluid viscosity, cp

L : Core length, cm

ΔP : Differential pressure, psig

A : Cross-sectional area, cm^2

Once the initial permeability at room temperature was obtained the set-up was heated to 250 °F and left to heat for 3 hours to ensure that all the lines are at the desired temperature. The inlet lines were then closed, and the drilling fluid was circulated in the drilling fluid loop at a flow rate of 1 cm^3/min. The pressure in the loop increased up to a set point of 500 psi on the second backpressure regulator [labeled (11) in **Figure II-12**]. The inlet lines were then opened and connected to backpressure regulator (11). The 500-psi set point was selected to allow for a gap between injection pressure and overburden, creating an overbalance pressure of 400 psi. Drilling fluid circulation continued for 30 minutes, and the filtrate volume was collected. The system was then left to cool down to reach room temperature. The system was then switched to direct flow again for final permeability measurements using the same brine (Al-Mujalhem 2021). The return permeability was then calculated using eq. 2 (Alkhalaf et al. 2019). Once the experiment is completed, the final

core is then CT scanned to obtain an image showing the effect of the drilling fluids invasion on the formation.

$$K_R = \frac{K_d}{K_o} \times 100 \qquad \textbf{Eq. 2}$$

Where,

K_R: Return permeability, %

K_d: Post drilling fluid permeability, md

K_o: Original core permeability, md

Filter Cake Removal

The final phase of experiments involved studying the different clean-up options for the micronized ilmenite-based filter cake. The solubility of the dry ilmenite powder was first tested under HPHT conditions using different acid systems. Following that, the best acid system was tested to remove the filter cake using the modified coreflood set-up mentioned earlier.

Ilmenite Powder Solubility

The solubility of the micronized ilmenite powder (5 µm) was tested using different acid systems. A Parr Bench Top Reactor (**Figure II-14**) was used to test the solubility under HPHT conditions. Five different acid systems were tested under different conditions as shown in **Table II-5**. The acid to solids ratio was maintained at 10:1; 20 g of micronized ilmenite were added to the acid solution to have a total mass of 200 g. **Table II-6** summarizes the exact amount of acid and water used to formulate the solutions. The dry ilmenite and acid solution and are added to the vessel where the entire system is pressurized to 800 psi, this is slightly lower than the desired test

pressure of 1000 psi to allow for some pressure increase as a result of the heating. The temperature is set, and the set-up is heated to the desired temperature through the heating jacket which is connected to the vessel. A magnetic stirrer drive is used to continuously mix the solution at 500 rev/min. Once the test is finished, the reactor is disassembled and left to cool down. The solution is then filtered using 5 µm filter paper, which is then dried over night and weighed to obtain the final weight of solids.

Figure II-14 Par Bench Top Reactor

Acid System	Composition	Temperature (°F)
1	15 wt% Hydrochloric Acid	250
2	15 wt% Acetic Acid + 1 wt% Hydrochloric Acid	200
3	10 wt% Glycolic Acid + 11.6 wt% Citric Acid	250
4	10 wt% Glycolic Acid + 15 wt% Acetic Acid	200
5	10 wt% Glycolic Acid + 13 wt% Lactic Acid	200

Table II-5 Acid systems tested

Acid System	Acid 1 (grams)	Acid 2 (grams)	Water (grams)
1	82.19	-	117.81
2	30.09	5.48	164.43
3	28.57	23.32	148.11
4	28.57	30.09	141.34
5	28.57	30.59	140.84

Table II-6 Composition of the acid systems

Ilmenite-Based Filter Cake Cleanup

After the solubility of the ilmenite powder was tested using the Parr Bench Top Reactor, the best performing acid system was tested using the modified coreflood setup. Testing a filter cake acid breaker system through the usage of the mudloop is a process that hasn't been used previously. Using a mudloop allows us to simulate the downhole drilling conditions and the filter cake cleanup process as closely as possible. The set-up shown in **Figure II-12** was used using the following steps:

1. The initial permeability of the core was measured using brine
2. The set-up was heated to 250 °F and left for 3 hours to ensure that the entire system is at the desired temperature
3. Drilling fluid is circulated for a period of 30 minutes across the face of the core using the spacer (shown in **Figure II-13**).

4. The acid system is then injected so that it circulated across the face of the core for a period of one hour and then the system is shut-off for a period of 24 hours to give time for the acid to react with the filter cake
5. Brine is injected into the core to measure the final permeability after the filter cake is removed

CHAPTER III

EXPERIMENTAL RESULTS EVALUATING THE PERFORMACE OF DIFFERENT WEIGHTING MATERIALS USED IN WATER BASED DRILLING FLUIDS

Drilling Fluids Properties

The rheological properties and thermal stability of the drilling fluid are essential parameters to be considered when selecting the proper drilling fluid. Rheological properties significantly impact drilling parameters such as wellbore hydraulics, hole cleaning, fluid stability, filter cake formation, rate of penetration, and lost circulation (Cayeux 2020). The thermal stability of the drilling fluid is vital in our current day and age where most of the wells drilled are under harsh conditions with high temperatures and pressures (Mohamed et al. 2021).

Drilling Fluid Rheology Results

The rheology and gel strength of the drilling fluids was measured using a Fann 35 viscometer. The rheology values were used to calculate the yield point and plastic viscosity of the four drilling fluids. The viscosity and gel strength were measured for each of the fluids with a shear rate ranging from 600 RPM to 3 RPM. The rheology of the four drilling fluids was measure before exposure to heat and after exposure to heat. The fluids were exposed to a temperature of 250 °F for a period of 16 hours while rolling to ensure that the drilling fluid maintains its properties as shown in **Table III 1-3**. Fluid 1, which uses API Barite has slightly higher readings across all the shear rates which is an indication of a lesser performance and a decrease in the ROP while drilling. From the data we can also notice that the plastic viscosity increased as the size of the particles increased. API barite has the largest particle size and the largest PV, followed by

micronized ilmenite and micronized barite (5 µm) and finally micronized barite (2 µm), which has the smallest particle size distribution, has the lowest PV. Another key distinction that can be seen from the data is the ratio of the yield point to the plastic viscosity (YP/PV). This ratio provides an indication of the efficiency of the drilling fluids carrying capacity and hole cleaning. The higher ratio the better the performance. Micronized ilmenite has a value of 1.25, while micronized barite (5 µm) has a value of 0.95 and micronized barite (2 µm) has a value of 1.06.

Rheology Readings	Before Hot Roll	After Hot Roll
600 RPM	64	84
300 RPM	42	60
200 RPM	32	50
100 RPM	22	38
6 RPM	5	14
3 RPM	3	12
Gel Strength- 10 sec	4	13
Gel Strength- 10 min	5	16
Plastic Viscosity	22	24
Yield Point	20	36

Table III-1 Rheology values for fluid 1 (API Barite as the weighting material)

Rheology Readings	Before Hot Roll	After Hot Roll
600 RPM	49	52
300 RPM	32	35
200 RPM	26	29
100 RPM	18	20
6 RPM	5	7
3 RPM	4	6
Gel Strength- 10 sec	4	5
Gel Strength- 10 min	6	8
Plastic Viscosity	17	17
Yield Point	15	18

Table III-2 Rheology values for fluid 2 (Micronized Barite 2µm as the weighting material)

Rheology Readings	Before Hot Roll	After Hot Roll
600 RPM	49	59
300 RPM	32	39
200 RPM	25	31
100 RPM	17	22
6 RPM	4	6
3 RPM	3	5
Gel Strength- 10 sec	4	5
Gel Strength- 10 min	4	6
Plastic Viscosity	17	20
Yield Point	15	19

Table III-3 Rheology values for fluid 3 (Micronized Barite 5µm as the weighting material)

Rheology Readings	Before Hot Roll	After Hot Roll
600 RPM	65	65
300 RPM	48	45
200 RPM	40	37
100 RPM	30	27
6 RPM	11	8
3 RPM	9	6
Gel Strength- 10 sec	11	8
Gel Strength- 10 min	15	8
Plastic Viscosity	17	20
Yield Point	31	25

Table III-4 Rheology values for fluid 4 (Micronized ilmenite as the weighting material)

Drilling Fluid Filter Press Results

Fluid loss prevention is a key performance attribute of drilling fluids. For water-based drilling fluids, significant loss of water or fluid into the formation can cause irreversible changes in the drilling fluid properties, such as density and rheology, which can create instability in the borehole (Fink 2012). High pressure high temperature (HPHT) filter press experiments were conducted to measure the static filtration and filter cake properties of the four drilling fluids after they were hot rolled for a period of 16 hours. The HPHT tests were conducted at 250 °F and 500 psi differential pressure. **Table III-5** shows the volume collected after a period of 30 minutes as well as the filter cake thickness. API Barite has the highest fluid loss and forms the thickest filter

cake, while the behavior of the three other fluids is fairly similar. An ideal drilling fluid will have a thin filter cake as well as a low volume of filtrate.

Fluid	1	2	3	4
HPHT Fluid Loss (mL)	14	10	12	10
Filter cake Thickness (mm)	2-3	1	1	1-2

Table III-5 HPHT fluid loss and filter cake thickness for the four fluids: fluid 1 uses API Barite as the weighting material, fluid 2 uses micronized barite (2 µm) as the weighting material, fluid 3 uses micronized barite (5 µm) and fluid 4 uses the micronized ilmenite as the weighting material

Drilling Fluid pH Results

The pH of the four fluids was measured using a pH meter to ensure that they are not acidic and corrosive. Results of the pH values are shown in **Table III-6**.

Fluid	1	2	3	4
pH	10.98	10.87	10.9	11.03

Table III-6 pH values for the four fluids: fluid 1 uses API Barite as the weighting material, fluid 2 uses micronized barite (2 µm) as the weighting material, fluid 3 uses micronized barite (5 µm) and fluid 4 uses the micronized ilmenite as the weighting material

Drilling Fluid SAG Factor Results

SAG is a termed used to define the separation of the solids from the drilling fluid, the weighting materials tend to settle at the bottom, and this can cause a fluctuation in the drilling fluid density. Hence it is crucial to measure the SAG factor and ensure that the drilling fluid density remains consistent. **Table III-7** shows the value of the SAG factor calculated for each of the drilling fluids, the values for the four fluids are within the range of 0.5-0.53 meaning that they are appropriate, and no SAG problems are present. The equation used to calculate SAG factor is presented below:

$$SAG\ Factor = \frac{Bottom\ desnisty}{Top\ density + Bottom\ density} \qquad \textbf{Eq. 3}$$

Fluid	1	2	3	4
Top Density	1.55	1.53	1.59	1.55
Bottom Density	1.63	1.68	1.67	1.61
SAG Factor	0.513	0.523	0.512	0.509

Table III-7 SAG factor values for the four fluids: fluid 1 uses API Barite as the weighting material, fluid 2 uses micronized barite (2 µm) as the weighting material, fluid 3 uses micronized barite (5 µm) and fluid 4 uses the micronized ilmenite as the weighting material

Formation Damage Evaluation

A modified coreflood set-up was used to examine the effect of the four drilling fluids on formation damage. A total of 12 coreflood experiments will be presented in this section. The first set of coreflood experiments used Bandera sandstone cores with permeability values less than 10 mD, which is considered as a low permeability sandstone core. The second set of coreflood experiments used the Grey Berea sandstone cores with a permeability value ranging from 60-80 mD, this is considered as a medium permeability range. The third set of coreflood experiments used the Boise sandstone cores with a permeability ranging from 800-900 mD, this is considered as a high permeability range. Results from the coreflood experiments will allow us to calculate the % return permeability as well as the dynamic filtrate volume.

Formation Damage in Low Permeability Sandstone Cores

The results from the first set of coreflood experiments is shown in **Table III- 8**. The initial permeability of each core was measured using 10% potassium chloride solution. The core was exposed to the drilling fluid and filter cake was left to be built to resemble the scenario in the field.

The dynamic filtrate was measured and recorded as well over a period of 30 minutes (**Figure III-1-4**). Finally, the final permeability was measured using the same potassium chloride solution and the return permeability (%) was calculated using **Eq. 2**.

Fluid	1	2	3	4
Initial Permeability (mD)	7.9	8.8	9.0	8.6
Final Permeability (mD)	3.5	6.7	6.5	7.9
Return Permeability (%)	44.3	76.1	72.2	91.8
Filtrate Volume (mL)	11.3	4.4	7.0	2.6

Table III-8 Results from the first set of modified coreflood experiments for the four fluids: fluid 1 uses API Barite as the weighting material, fluid 2 uses micronized barite (2 µm) as the weighting material, fluid 3 uses micronized barite (5 µm) and fluid 4 uses the micronize

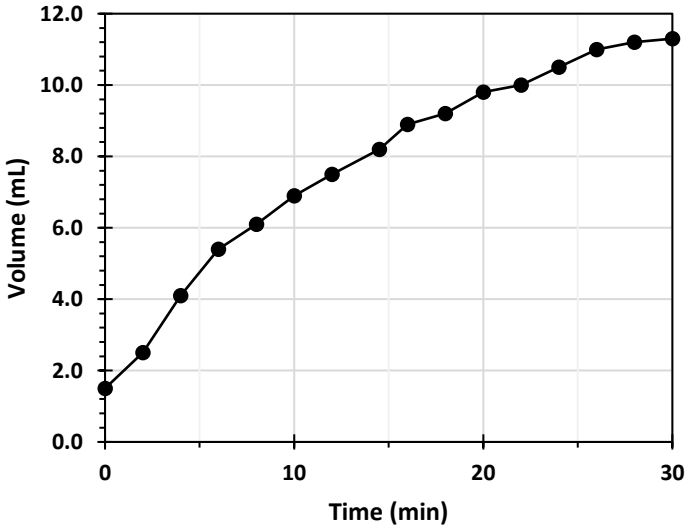

Figure III-1 Results from low permeability coreflood test showing the dynamic filtrate volume using API barite as weighting material

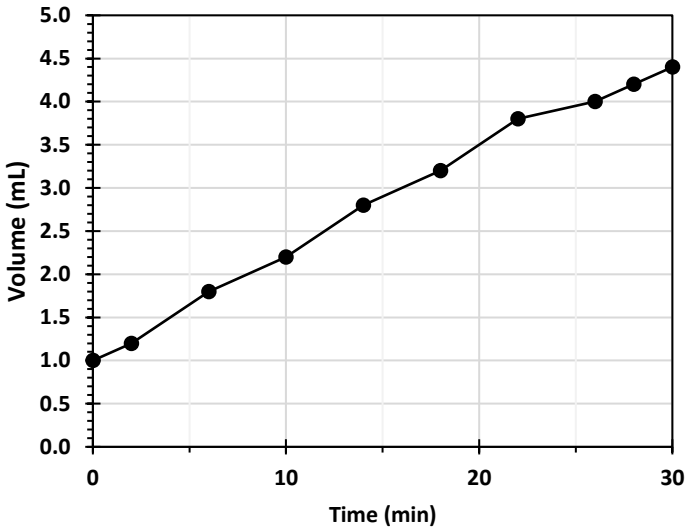

Figure III-2 Results from low permeability coreflood test showing the dynamic filtrate volume using micronized barite (2 µm) as weighting material

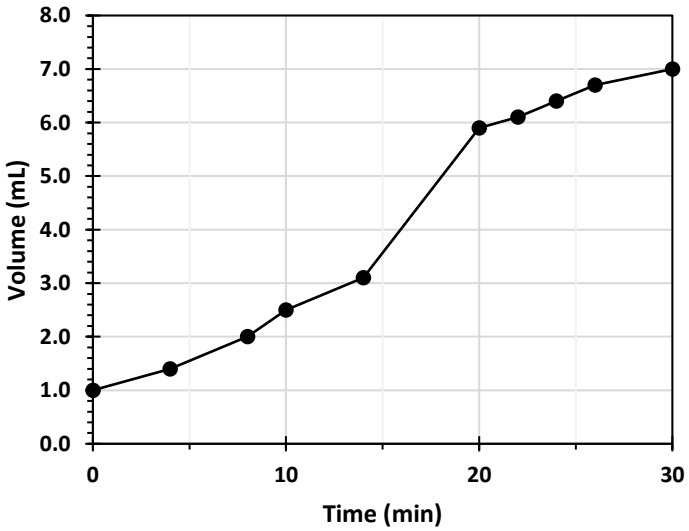

Figure III-3 Results from low permeability coreflood test showing the dynamic filtrate volume using micronized barite (5 µm) as weighting material

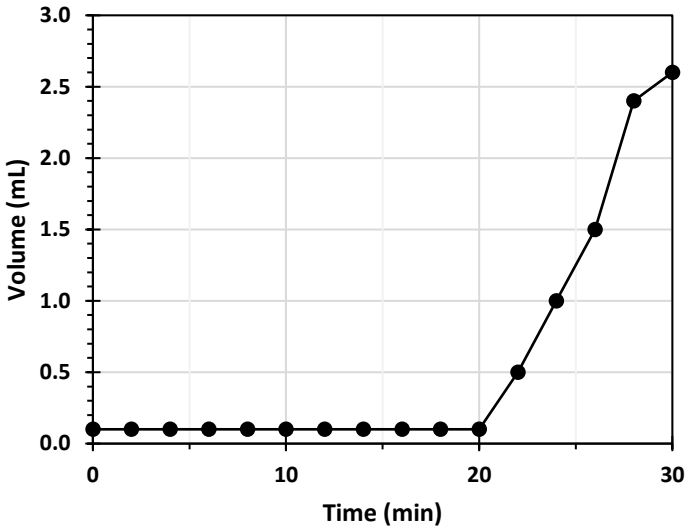

Figure III-4 Results from low permeability coreflood test showing the dynamic filtrate volume using micronized ilmenite as weighting material

Formation Damage in Medium Permeability Sandstone Cores

The results from the second set of coreflood experiments is shown in **Table III- 9**. The initial permeability of each core was measured using 10% potassium chloride solution. The core was exposed to the drilling fluid and filter cake was left to be built to resemble the scenario in the field. The dynamic filtrate was measured and recorded as well over a period of 30 minutes (**Figure III-5-8**). Finally, the final permeability was measured using the same potassium chloride solution and the return permeability (%) was calculated.

Fluid	1	2	3	4
Initial Permeability (mD)	80	81	76	83
Final Permeability (mD)	34	60	52	74
Return Permeability (%)	42.5	74.1	68.4	89.1
Filtrate Volume (mL)	12.7	5.3	9.6	3

Table III-9 Results from the second set of modified coreflood experiments for the four fluids: fluid 1 uses API Barite as the weighting material, fluid 2 uses micronized barite (2 µm) as the weighting material, fluid 3 uses micronized barite (5 µm) and fluid 4 uses the micronized ilmenite as the weighting material

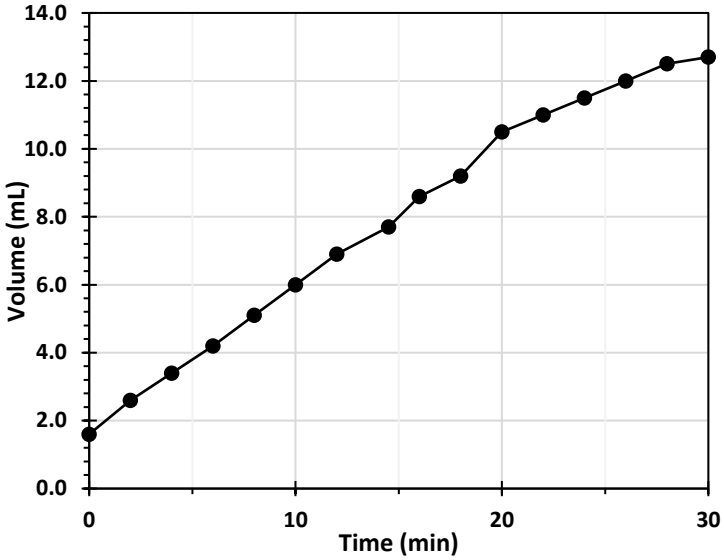

Figure III-5 Results from medium permeability coreflood test showing the dynamic filtrate volume using API barite as weighting material

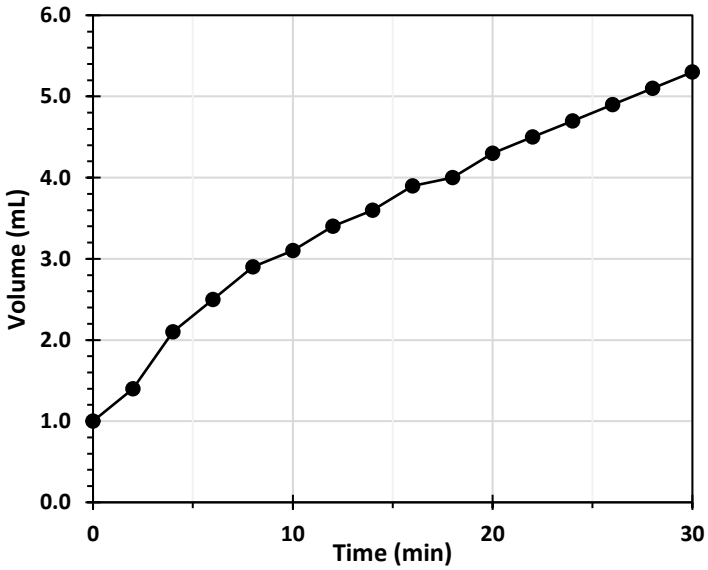

Figure III-6 Results from medium permeability coreflood test showing the dynamic filtrate volume using micronized barite (2 µm) as weighting material

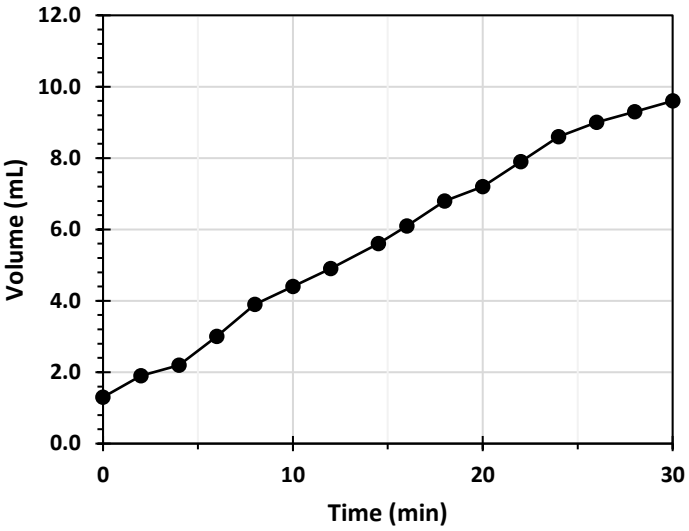

Figure III-7 Results from medium permeability coreflood test showing the dynamic filtrate volume using micronized barite (5 µm) as weighting material

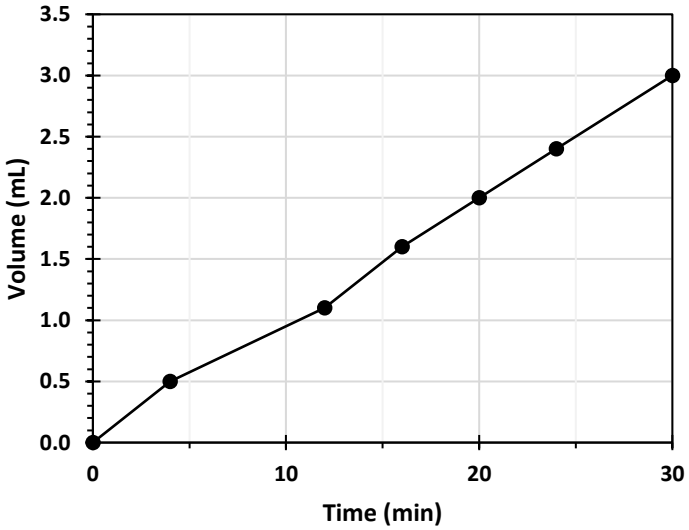

Figure III-8 Results from medium permeability coreflood test showing the dynamic filtrate volume using micronized ilmenite (5 µm) as weighting material

Formation Damage in High Permeability Sandstone Cores

Table III-10 below shows the summary of the results obtained from the third set of coreflood experiments. The initial permeability of each core was measured using 10% potassium

chloride solution. The core was exposed to the drilling fluid and filter cake was left to be built to resemble the scenario in the field. The dynamic filtrate was measured and recorded as well over a period of 30 minutes (**Figure III-9 to 12**). Finally, the final permeability was measured using the same potassium chloride solution and the return permeability (%) was calculated.

Fluid	1	2	3	4
Initial Permeability (mD)	931.2	908.3	961.6	922.8
Final Permeability (mD)	323.7	574.3	534.2	673.1
Return Permeability (%)	34.8	63.2	55.6	72.9
Filtrate Volume (mL)	15.7	8.7	10.5	6.0

Table III-10 Results from the third set of modified coreflood experiments for the four fluids: fluid 1 uses API Barite as the weighting material, fluid 2 uses micronized barite (2 µm) as the weighting material, fluid 3 uses micronized barite (5 µm) and fluid 4 uses the micronized ilmenite as the weighting material

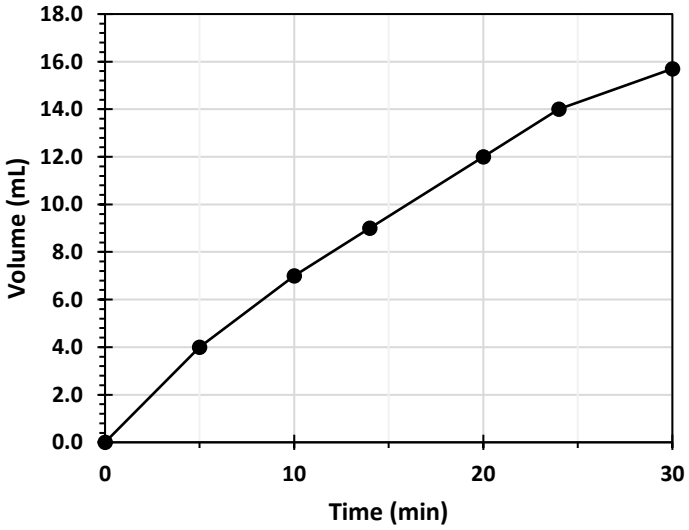

Figure III-9 Results from high permeability coreflood test showing the dynamic filtrate volume for API barite as the weighting material

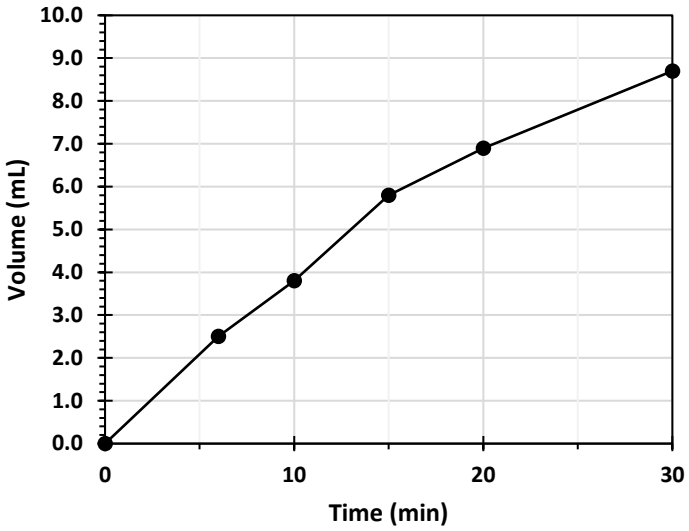

Figure III-10 Results from high permeability coreflood test showing the dynamic filtrate volume for micronized barite (2 μm) as the weighting material

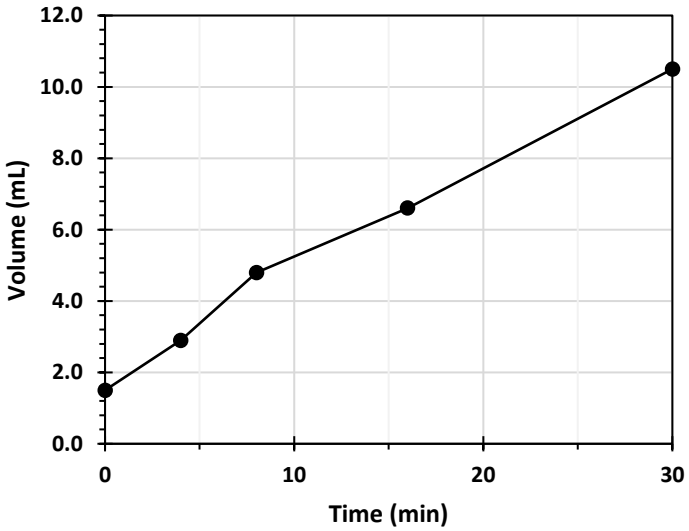

Figure III-11 Results from high permeability coreflood test showing the dynamic filtrate volume for micronized barite (5 μm) as the weighting material

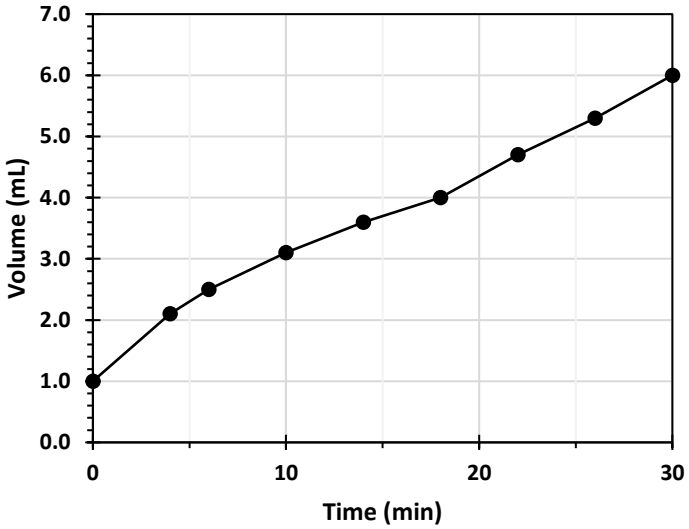

Figure III-12 Results from high permeability coreflood test showing the dynamic filtrate volume for micronized ilmenite as the weighting material

Formation Damage Comparison

The results from the three sets of coreflood presented show that the four drilling fluids cause formation damage to a certain degree, however the degree of damage varies significantly based on the weighting material used and based on the original permeability of the core tested. In all the cases micronized ilmenite had the highest return permeability, followed by micronized barite (2 µm), micronized barite (5 µm) and finally API barite had the lowest return permeability in all the cases. The first set of cores which had the tightest permeability showed the least damage and that probably is because the filter cake was formed quickly and the drilling fluid was not able to invade the pores and hence less damage was seen. As the permeability of the core increased as shown in the Boise cores, the filter cake was formed over a longer period of time which meant that more filtrate invaded the pore spaces and blocked the pore throats and as a result the return permeability for all the cases was relatively lower than that presented in Bandera and Grey Berea sandstone cores. Another indication that is used to analyze the extend of the damage caused by the

drilling fluids is the volume of the filtrate that was collected during the mudloop. The larger the volume of the filtrate the more than damage that is caused. The filtrate collected from the mudloop is considered as a dynamic filtrate while that collected from the HPHT test is considered as a static filtrate. The results from both the static and dynamic filtrate give us the same trend showing that the volume of API barite was the highest and micronized ilmenite was the lowest. The volumes of the filtrate collected from the Boise sandstone cores was all higher than the volumes collected from the Bandera and Grey Berea cores which again ties back to the extent of the damage seen in these cores. **Figure III-13** to **16** shows how the cores look like after the completion of the mudloop experiment, a sample core was picked from each fluid to be presented below.

Figure III- 13a Sandstone core after exposure to API barite water-based drilling fluid

Figure III- 13b API barite-based filter-cake formed across the face of the sandstone core

Figure III- 14a Sandstone core after exposure to micronized barite (2 µm) water-based drilling fluid

Figure III- 14b Micronized barite (2 µm) filter cake formed across the face of the sandstone core

Figure III- 15a Sandstone core after exposure to micronized barite (5 µm) water-based drilling fluid

Figure III- 15b Micronized barite (5 µm) filter cake formed across the face of the sandstone core

Figure III- 16a Sandstone core after exposure to micronized ilmenite (5 µm) water-based drilling fluid

Figure III-16b Micronized ilmenite (5 µm) filter cake formed across the face of the sandstone core

Formation Damage Evaluation Using CT-Scan

The sandstone cores used were scanned initially before exposure to the mud and after they were exposed to the mud to identify the effect of the different weighting materials on the pore space of the cores. **Figure III-17** and **18** show the results obtained from the CT scans for the Boise sandstone cores from using API barite and micronized ilmenite as weighting materials. The figures below show the extent of the damage all the way across the core and they also show that more damage is evident in the case where API barite is used as the weighting material. The results obtained from the CT scan are qualitative and they confirm the analysis shown by the mudloop experiments.

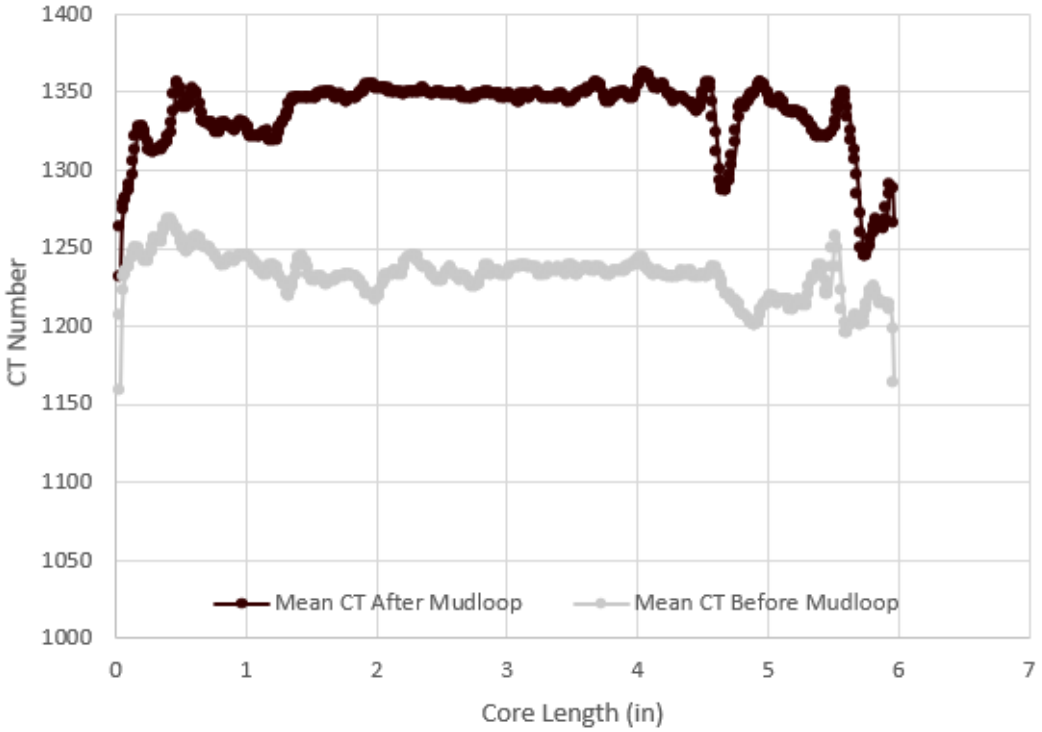

Figure III-17 CT scan results showing qualitative effect of API barite on formation damage

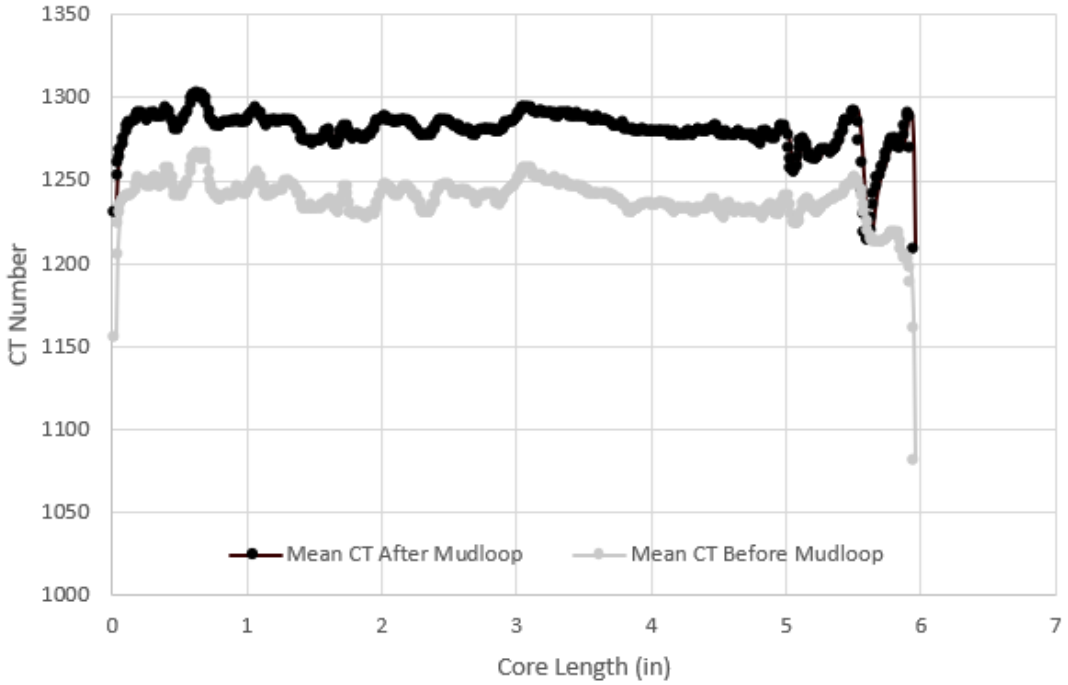

Figure III-18 CT scan results showing qualitative effect of micronized ilmenite on formation damage

CHAPTER IV

EXPERIMENTAL RESULTS EVALUATING THE REMOVAL OF MICRONIZED ILMENITE BASED FILTER CAKE IN WATER BASED DRILLING FLUIDS

Micronized Ilmenite Powder Solubility

The solubility of micronized ilmenite is a topic that is still being studied within the oil and gas industry. Several authors investigated the solubility of ilmenite-based filter cake with hydrochloric acid and with HEDTA (Al-Bagoury and Steele 2012; Elkatatny et al. 2013; J et al. 2021). This section will present the results of micronized ilmenite powder solubility with several acids systems to come up with the best possible filter cake removal system. The experiments were conducted using the HPHT Par Bench Top Reactor at 1000 psi and using an acid to ilmenite ratio of 10:1. The solubility tests were conducted over a period of 24 hours.

Results of the Acid Systems Solubility

The acid systems that were tested as well as the conditions used are shown below in **Table IV-1.** The solubility efficiency for the different systems is calculated by dividing the final weight of ilmenite solids left after the experiment is conducted by the original weight used. **Table IV-2** below shows the solubility efficiency of the different acid systems tested. The work presented in **Table IV- 1** and **2** is a continuation of the work presented by Al-Mujalhem 2021, the author had tested several acid systems (shown in **Table IV-3** and **4**) to which my work followed.

Acid System	Composition	Temperature (°F)
1	15 wt% Hydrochloric Acid	250
2	15 wt% Acetic Acid + 1 wt% Hydrochloric Acid	200
3	10 wt% Glycolic Acid + 11.6 wt% Citric Acid	250
4	10 wt% Glycolic Acid + 15 wt% Acetic Acid	200
5	10 wt% Glycolic Acid + 13 wt% Lactic Acid	200

Table IV-1 Composition of acid systems

Acid System	Initial Sample (g)	Residual Solids (g)	Dissolved Ilmenite (g)	Solubility Efficiency (%)
1	20	9.2	10.8	54
2	20	18.2	1.8	9
3	20	14.5	5.5	27.5
4	20	17.1	3.9	14.3
5	20	16.8	3.2	16

Table IV-2 Solubility efficiency of different acid systems

Acid System	Composition	Temperature (°F)
A	15 wt% Hydrochloric Acid	200
B	7.5 wt% Oxalic Acid + 1 wt% Hydrochloric Acid	200
C	7 wt% Glycolic Acid + 1 wt% Hydrochloric Acid	200
D	13 wt% Lactic Acid + 1 wt% Hydrochloric Acid	200
E	11.6 wt% Citric Acid + 1 wt% Hydrochloric Acid	200
F	20 wt% Oxalic Acid + 10 wt% Hydrochloric Acid	200
G	10 wt% Glycolic Acid + 11.6 wt% Citric Acid	200

Table IV-3 Composition of acid systems (Reprinted from Al-Mujalhem 2021)

Acid System	Initial Sample (g)	Residual Solids (g)	Solubility Efficiency (%)
A	20	11.9	40.1
B	20	24.8	-24.2
C	20	18.9	5.2
D	20	18.0	9.8
E	20	17.7	11.4
F	20	37.9	-89.4
G	20	15.4	23

Table IV-4 Solubility efficiency of acid systems (Reprinted from Al-Mujalhem 2021)

The solubility of micronized ilmenite powder using 15 wt% HCl was tested at 250 °F (acid system 1) to give a solubility efficiency of more than 50%. The solubility of ilmenite in HCl depends on many factors such as the concentration of the acid, the ratio between the ilmenite and acid, the temperature and pressure conditions and the reaction time. The incomplete solubility of the ilmenite is related to the reverse reaction of titanium Oxychloride (TiOCl). The application of hydrochloric acid is a viable solution to clean up the ilmenite-based filter cake, however the use of HCl in the field presents risks in terms of transportation and handling, corrosion of the tubulars, and the potential precipitation of solids like iron in the reservoir that may introduce more damage.

As a result, several other acid systems were considered that are safer and more cost effective to be applied in the field. The second acid system that was tested was 15 wt% acetic acid + 1 wt% hydrochloric acid at 200 °F. A very small concentration of HCl is added just to get the reaction started. The solubility efficiency was found to be 9%. Al-Mujalhem (2021) investigated the solubility of micronized ilmenite with several organic acids, the author found that the solubility of 11.6 wt% citric acid was the highest followed by 13 wt% lactic acid, 7 wt% glycolic acid and finally oxalic acid had the worst solubility with solids precipitating into the solution. The author also combined 10 wt% glycolic acid + 11.6 wt% citric acid to get the highest solubility efficiency value of 23%.

The values presented in **Table IV-4** are the basis for which acid systems 3, 4 and 5 were selected. Acid system 3 tested the solubility of micronized ilmenite in 10 wt% glycolic acid + 11.6 wt% citric acid at 250 °F. The solubility efficiency of acid system 3 was about 30%. Acid system 4 tested the solubility of micronized ilmenite in 10 wt% glycolic acid + 15 wt% acetic acid, the system had a solubility efficiency value of about 15%. Acid system 5 tested the solubility of micronized ilmenite in 10 wt% glycolic acid + 13 wt% lactic acid, the system had a solubility

efficiency of 16% which is slightly higher than acid system 4. In summary, Acid system 3 had the best performance following the 15 wt% HCl.

Results of Ilmenite-Based Filter Cake Cleanup

Once the solubility of powdered ilmenite was tested using the different acid systems, the best acid system was selected to ensure that it would be effective in cleaning up the filter cake formed. Acid system 3 was selected to clean up the filter cake formed during the mudloop experiment, this is the first time that a filter cake is formed and cleaned up under conditions very similar to the field conditions. The steps listed below were followed to utilize the mudloop set up in testing the removal efficiency of the filter cake:

1. The initial permeability of the Boise sandstone core was measured using brine, the Boise sandstone core was selected because it had the lowest return permeability compared to the grey Berea and the Bandera cores
2. The set-up was heated to 250 °F and left for 3 hours to ensure that the entire system is at the desired temperature
3. Drilling fluid is circulated for a period of 30 minutes across the face of the core using the spacer (shown in **Figure II-13**).
4. Acid system 3 is then injected so that it circulated across the face of the core for a period of one hour and then the system is shut-off for a period of 24 hours to give time for the acid to react with the filter cake
5. Brine is injected into the core to measure the final permeability after the filter cake is removed

Table IV-5 below shows the initial permeability of the Boise sandstone core to be 917 mD and the final permeability of the core after the filter cake was cleaned up to be 810 mD giving a return permeability of 88%. The results of the mudloop test show that the acid system was able to clean up the filter cake for the most part and that very little damage can be seen from using a drilling fluid system with micronized ilmenite as the weighting material. **Figure IV-1** shows a bar chart comparing the permeability values of using ilmenite-based drilling fluids with a boise sandstone core. The graphs represent the case where no breaker system was used versus the case where acid system 3 was used to clean up the filter cake. **Figure IV-2** shows the CT scan images of the original core before being exposed to drilling fluids and after the filter cake was removed.

Fluid	Boise Sandstone Core
Initial Permeability (mD)	917.1
Final Permeability (mD)	810.7
Return Permeability (%)	88.3
Filtrate Volume (mL)	7.1

Table IV-5 Mudloop results showing filter cake clean up by acid system 3

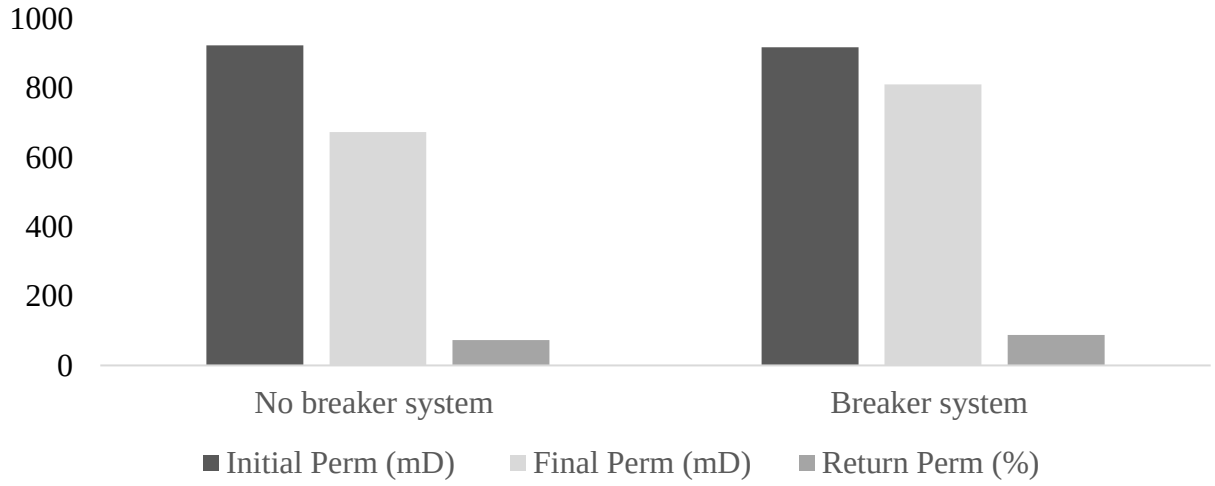

Figure IV-1 Permeability values for Boise sandstone with and without breaker system

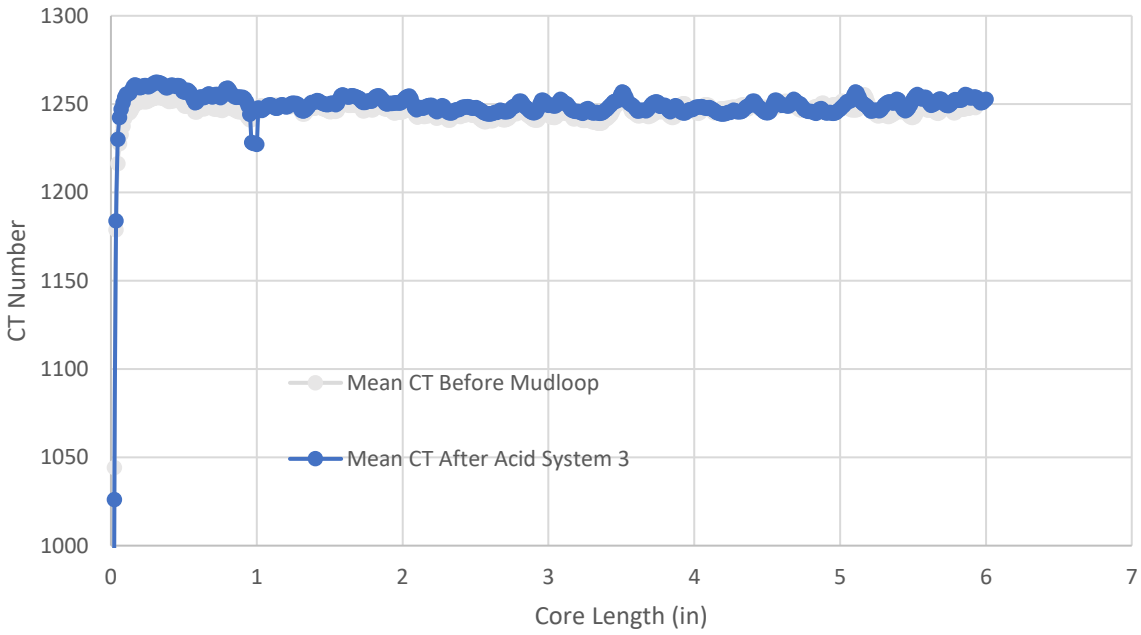

Figure IV- 2 CT Scan before and after acid system 3

CHAPTER V

CONCLUSIONS AND RECOMENDATIONS

This research project had 2 main objectives. The first objective of this research is to analyze how different weighting materials used in water-based drilling fluids interact with the formation. Understanding the formation damage associated with the different weighting agents is imperative to help design the proper drilling fluid for each formation. The goal of this research is to design a drilling fluid that will provide all the necessary rheological properties while maintaining the integrity of the formation. The second objective of this work was to evaluate different acid systems that can be used to clean the ilmenite-based filter cake without causing any field associated risks.

Conclusions

Drilling Fluid Properties and Behavior

- The properties of the drilling fluids were studied by measuring the rheology before and after exposure to heat. API Barite has slightly higher readings across all the shear rates which is an indication of a lesser performance and a decrease in the ROP while drilling.
- From the data we can also notice that the plastic viscosity increased as the size of the particles increased. API barite has the largest particle size and the largest PV, followed by micronized ilmenite and micronized barite (5 µm) and finally micronized barite (2 µm), which has the smallest particle size distribution, has the lowest PV.
- API Barite has the highest fluid loss volume and forms the thickest filter cake, while the behavior of the micronized ilmenite and micronized barite is fairly similar.

- It can be concluded that the drilling fluid properties of micronized ilmenite exceed those of API Barite and are similar to the micronized barite

Formation Damage Evaluation

- A modified coreflood set-up was used to assess the extent of formation damage from the four different weighting materials. Temperature and pressure conditions were set to simulate the field conditions. Drilling fluid was circulated across the face of the sandstone cores
- Sandstone cores with different permeabilities ranges were used to represent the formation in the modified coreflood tests.
- Micronized ilmenite was the least damaging weighting material in all the experiments conducted
- The return permeability was about 90% when micronized ilmenite was the weighting agent while it was about 40% when API Barite was the weighting agent in low and medium permeability sandstone cores
- In the cases of the high permeability sandstone cores, damage was apparent with all the four drilling fluids. Ilmenite however had the least damage with a return permeability of more than 70% followed by micronized barite (2 μm) with a return permeability over 60% and micronized barite (5 μm) with a return permeability of 55% and finally API barite was the most damaging to the formation with a return permeability of about 30%.

Ilmenite Solubility and Filter Cake Cleanup

- The solubility of micronized ilmenite powder was tested under high pressure high temperature conditions using a Par Bench Top Reactor
- The solubility of HCl with ilmenite powder was greater than 50%. The application of hydrochloric acid is a viable solution to clean up the ilmenite-based filter cake however the use of HCl in the field presents risks in terms of transportation and handling, corrosion of the tubulars, and the potential precipitation of solids like iron in the reservoir that may introduce more damage.
- Acid system 3 (10 wt% Glycolic Acid + 11.6 wt% Citric Acid) had the highest solubility efficiency following HCl, with a solubility efficiency of more than 23%
- Acid system 3 was then used to remove the filter cake using the mudloop setup. Initial permeability of the Boise sandstone core was 917 mD and the final permeability of the core after the filter cake was cleaned up was 810 mD to giving a return permeability of 88%

Recommendations

From the work presented in this research, micronized ilmenite is recommended as a weighting material for the following reasons:

1. Micronized ilmenite provides the necessary drilling fluid rheological properties
2. The industry is currently shifting towards using products that are environmentally friendly. Ilmenite is a great substituent to barite since its heavy metal content is much lower than that of barite.
3. Micronized ilmenite also causes minimal formation and is acid soluble which makes it easy to clean and remove the filter cake.

4. Unlike barite, Ilmenite is also readily available and can be easily produced in large quantities to stratify the market needs

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