

Application of Anhydrous Calcium Sulfate as a Weighting Agent in Oil-Based Drilling Fluids

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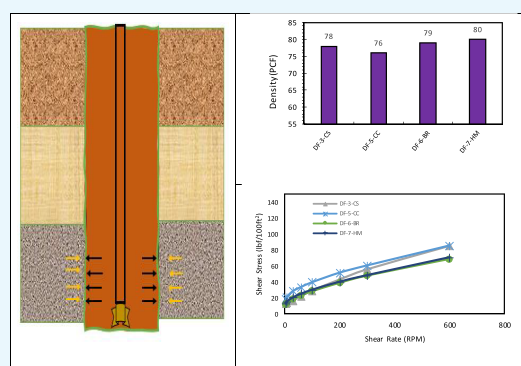
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ABSTRACT: The hydrostatic pressure exerted during the drilling operation is controlled by adding a weighting agent into drilling fluids. Various weighting materials such as barite, calcium carbonate, hematite, and ilmenite are used to increase the density of drilling fluids. Some weighting additives can cause serious drilling problems, including particle settling, formation damage, erosion, and insoluble filters. In this study, anhydrite (calcium sulfate) is used as a weighting additive in the oil-based drilling fluid (OBDF). Anhydrite is an abundantly available resource used in the preparation of desiccant, plaster of Paris, and Stucco. Anhydrite application in drilling fluids is discouraged because of its filter cake removal issue. This study investigated anhydrite (anhydrous CaSO_4) as a weighting agent and its filter cake removal procedure for OBDFs. The anhydrite performance as a weighting agent in OBDFs was evaluated by conducting several laboratory experiments such as density, rheology, fluid loss, and electrical stability and compared with that of commonly used weighting materials (barite, calcium carbonate, and hematite). The anhydrite was mixed in three different concentrations (62, 124, and 175 ppb) in a base-drilling fluid. The results showed that calcium sulfate enhanced rheological parameters such as plastic viscosity, yield point, apparent viscosity, and gel strength. CaSO_4 reduced the fluid loss and provided better control over the fluid loss than other tested weighting materials tested at the same concentration of 124 ppb. Similarly, the emulsion stability was decreased with the increase in the amount of calcium sulfate in the OBDF. The calcium sulfate filter cake can be removed easily from the wellbore with an efficiency of 83 to 91% in single-stage and multistage removal processes, respectively using the newly developed formulation consisting of 20 wt % potassium salt of glutamic acid-*N,N*-diacetic acid (K_4GLDA) as a chelating agent, 6 wt % potassium carbonate, and 10% ethylene glycol monobutyl ether. The introduction of anhydrite as a weighting agent can be more beneficial for both academia and industry.



1. INTRODUCTION

Drilling fluids play a significant role in drilling operations as the success and failure of drilling are primarily based on the selection of drilling fluids. The type and composition of drilling fluids are vital elements to serve many purposes such as carrying drilled cuttings to the surface, cleaning, cooling, and lubrication of the drilling bit, reducing friction, maintaining the borehole integrity, making a thin and low permeable filter cake, and preventing gas or oil kicks from prematurely flowing into the wellbore.^{1–5} Two common types of drilling fluids used in oil and gas drilling operations that differ in application and composition are oil-based drilling fluids (OBDFs) and water-based drilling fluids (WBDFs). WBDFs are cheaper and environmentally friendly, but they have limitations such as their application in high-pressure high-temperature (HPHT) wells and shale-bearing formations. On the other hand, OBDFs are recommended in HPHT wells and shale-bearing formations to provide wellbore stability. The drilling fluid

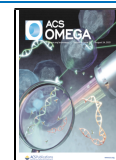
properties that control many vital functions of drilling fluids are density, viscosity, filtration, pH, and rheological properties such as plastic viscosity (PV), yield point (YP), and gel strength.^{6,7}

Weighting agents are those materials that increase the density of drilling fluids by their suspension and dissolution in drilling fluids. Their primary objective is to control formation pressure by creating enough hydrostatic pressure in the hole (overbalanced drilling), which ensures borehole stability. Also, they reduce the amount of fluid loss into the formation by making a mud cake on the borehole walls.

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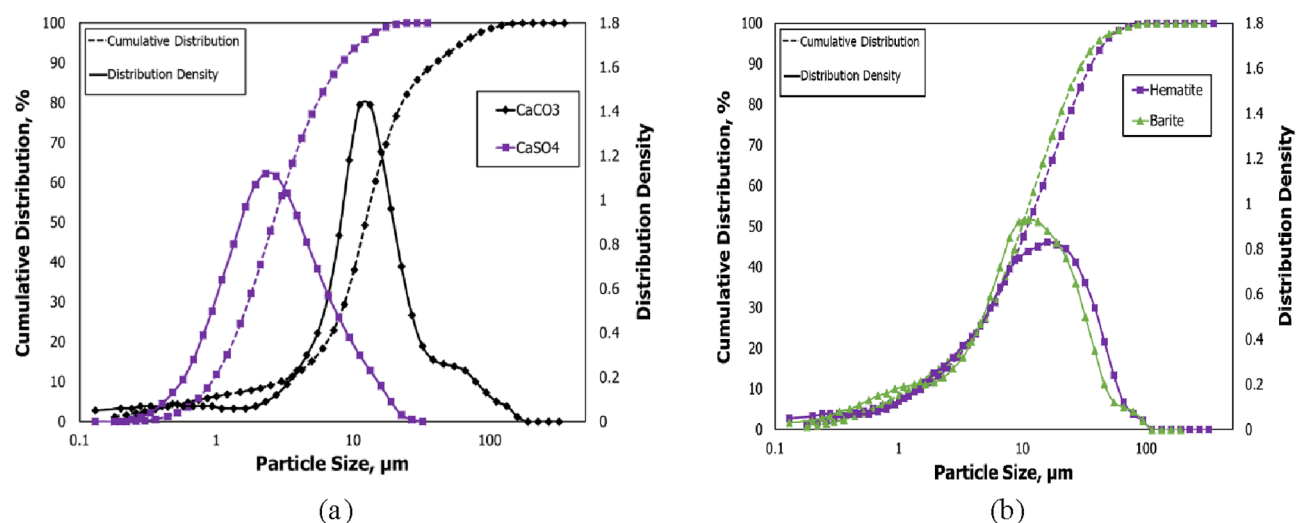


Figure 1. Particle size distribution and distribution density of: (a) CaSO_4 and CaCO_3 and (b) hematite and barite.

Drilling fluids evolved with time from a simple mixture of water and clay to complex formulations and contain organic, inorganic, and polymeric compounds to meet operational demands and challenges. In general, drilling fluids must be easy to handle, cheaper, and environmentally friendly.^{8–10} Commonly used weighting materials are calcium carbonate, barium sulfate or barite, hematite, manganese tetroxide, and ilmenite.^{11,12}

During the drilling operation, the drilling fluids impair the permeability of the formation upon their invasion into the formation. The invaded particles block the pore throat and lead to a loss of permeability of the produced formation by building a filter cake on the wall of the permeable formation. Most of the time, the build filter cake contains an insoluble weighting material, which is difficult to remove as it is composed of a higher content of weighting materials.^{13–15} The composition of the filter cake depends on several factors, such as solid additives in the drilling fluid, the differential pressure between mud and formation, and the geology of formation.¹⁴ The filter cake removal process involves the chemical dissolution of weighting materials.

Calcium carbonate exists in abundance as a limestone. Calcium carbonate is prioritized over other weighting materials in drill-in, completion, and workover fluids because of its high dissolution in hydrochloric acid (HCl). However, calcium carbonate has a weight limitation of 14 lb/gal for drilling fluids as compared to barite and other weighting materials. Several chemicals such as acids, oxidizers, acid precursors, enzymes, and chelating agents such as glutamic-di-acetic acid (GLDA) are used to dissolve the filter cake formed by calcium carbonate.^{15–17}

Barite is a mainly used weighting agent in drilling fluids because of its high specific gravity of 4.2 and inertness toward many chemical additives mixed in drilling fluids. However, barite applications have some limitations, such as dissolution in formate-based drilling fluids, high PV, low acid solubility, and significant sagging.^{18,19} Furthermore, the filter cake formed by barite is insoluble in hydrochloric acid (HCl), citric acid, formic acid, and acetic acid; in addition, it has low solubility in chelating agents such as N-hydroxy-ethylene-di-amine-tri-acetic acid (HEDTA) and ethylene-di-amine-tetra-acetic acid (EDTA). Many barite filter cake removal methods were developed; for example, Bageri et al.²⁰ applied modified EDTA

and HEDTA chelating agents, and Mahmoud and Elkatatny²¹ used potassium carbonate (K_2CO_3) at a pH of 12.0 to convert the barite to barium carbonate (BaCO_3). Recently, Mahmoud²² applied combined thermochemical/chelating agent fluids for barite filter cake removal. However, a recent increase in consumption and staggering supplies of premium barite have led to a significant rise in its price across the world²³ and pushed industry for other sources.

Later, ilmenite and hematite were investigated as weighting materials in drilling fluids.^{24–26} There are several benefits associated with both materials, such as the minimum impact on rheology, improvement in the rate of penetration, less dilution, and better solid tolerance.^{25,27,28} However, the potential issues with them are the abrasiveness and magnetic characteristics of iron oxide, as both affect the tubular and directional tools, respectively.^{24,29} The filter cake formed using ilmenite and hematite as weighting materials can be dissolved in sulfuric acid (H_2SO_4) and HCl.^{30–32} The removal efficiency can be improved by adding EDTA and HEDTA and glycolic acid into HCl.³³

In the past, the application of CaSO_4 was discouraged as an additive in drilling fluids. It was treated as a contaminant to the drilling fluids. It could not be used as a weighting material because of the poor solubility of its filter cake. For the first time, Murtaza et al.³⁴ reported CaSO_4 as a weighting agent in WBDFs. They reported numerous advantages of CaSO_4 , such as higher solubility in the drilling fluid and improved rheology over CaCO_3 . Calcium sulfate has a higher density of 2.96 g/cm^3 than calcium carbonate (2.70 g/cm^3); consequently, a drilling fluid with a less quantity of solids can be used to drill HPHT wells. However, calcium sulfate has never been tried in OBDP formulation because of the poor solubility of its filter cake in acids.

In this work, we developed a new formulation to dissolve the filter cake of CaSO_4 in OBDP, and the developed formulation consists of a 20 wt % K_4GLDA chelating agent, 6 wt % potassium carbonate, and 10% EGMBE (ethylene glycol monobutyl ether). Different tests, including density, rheology, and fluid loss, were conducted. The impact of CaSO_4 on the various properties of OBDPs, such as rheology, fluid loss, density, and emulsion stability (ES), was investigated. The performance of CaSO_4 was compared with that of other weighting materials such as CaCO_3 , barite, and hematite by

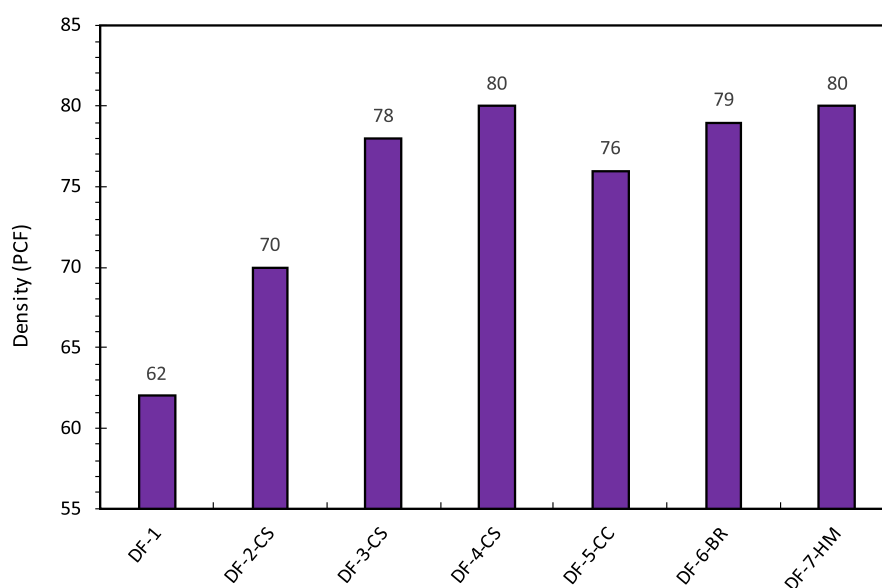


Figure 2. Density change of drilling fluids containing various concentrations of CaSO_4 (DF-1 to DF-4-CS) and other weighting materials (DF-5-CC, DF-6-BR, and DF-7-HM).

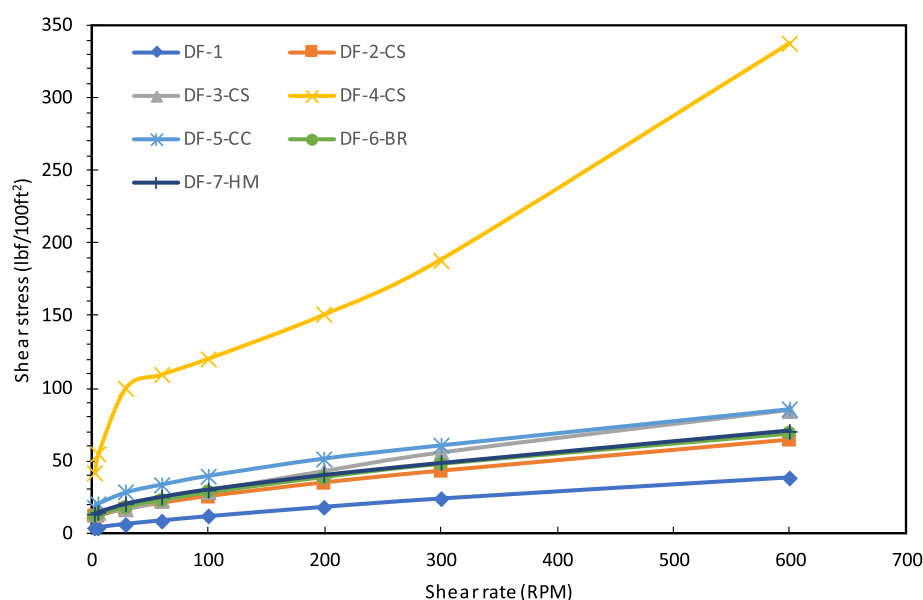


Figure 3. Shear stress at 120 °F for drilling fluids containing various concentrations of CaSO_4 (DF-1 to DF-4-CS) and other weighting materials (DF-5-CC, DF-6-BR, and DF-7-HM).

formulating different drilling fluids that contained an equal amount of weighting materials. In the final phase, the filter cake removal method based on a single stage and multiple stages for CaSO_4 has been reported and discussed in detail.

2. RESULTS AND DISCUSSION

2.1. Particle Size Analysis. The particle sizes of all weighting materials were measured using a HELOS particle size analyzer (Model#H3533). The nominal median particle sizes (D_{50}) of CaCO_3 and CaSO_4 were 12.37 and 2.62 μm , respectively, as Figure 1a provides the cumulative distribution and distribution density of both materials. The average particle size of the provided material of CaSO_4 is smaller as compared to that of CaCO_3 . Furthermore, median particle sizes (D_{50}) of barite and hematite were 9.91 and 11.03 μm , respectively, as

shown in Figure 1b. Out of all tested weighting materials, CaSO_4 consisted of smaller size particles.

2.2. Density and Rheological Properties. Different OBDs were prepared and tested for density and rheology. Three different concentrations of CaSO_4 were mixed in OBD (DF-1), and their impact on density and rheology was investigated. Furthermore, the performance of CaSO_4 was evaluated against that of CaCO_3 , barite, and hematite-based drilling fluids (DF-5-CC, DF-6-BR, and DF-7-HM) by conducting rheology and density tests.

The densities of all drilling fluids were measured using a FANN mud balance (see, Figure 2) and reported in pounds per cubic foot (PCF). The density of the base-drilling fluid (DF-1) was increased with an increase in the concentration of CaSO_4 ; for example, the addition of 62 ppb of CaSO_4 increased the density of DF-1 from 62 PCF to 70 PCF, and

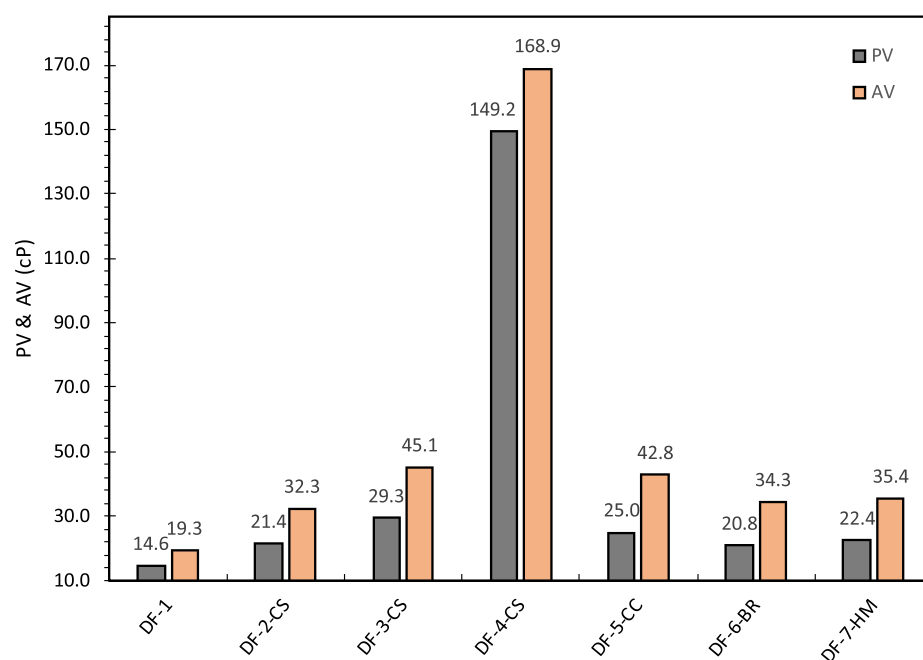


Figure 4. PV and AV change of drilling fluids containing various concentrations of CaSO_4 (DF-1 to DF-4-CS) and other weighting materials (DF-5-CC, DF-6-BR, and DF-7-HM).

124 ppb of CaSO_4 increased the density to 78 PCF. Similarly, other additives (barite and hematite) increased the density of drilling fluids based on their specific gravities. DF-6-BR and DF-7-HM had densities of 79 PCF and 80 PCF, respectively, though weighting materials were added in equal amounts in DF-1. Furthermore, it was observed that CaSO_4 resulted in a high-weight drilling fluid compared to CaCO_3 mixed at a similar amount. This indicates that CaSO_4 can be used as an alternate weighting material to prepare high-weight drilling fluids.

From experimental work, it was noticed that CaSO_4 and other weighting materials affected the rheological properties.

The change in shear stress for different shear rates of other weighting materials and different concentrations of CaSO_4 was investigated, as shown in Figure 3. The illustrated curves indicated that all OBDs responded as non-Newtonian drilling fluids. The shear stress/rate curves of OBDs loaded with different concentrations of CaSO_4 displayed that shear stresses increased with the amount of CaSO_4 added into the OBD. Furthermore, CaCO_3 , barite, and hematite materials increased shear stresses compared to the base-drilling fluid (DF-1). At high concentration (175 ppb) of CaSO_4 , shear stress values of DF-4-CS were raised by many fold compared to its lower concentrations and other weighting material-containing drilling fluids. The shear stress/rate curves of DF-3-CS and DF-5-CC were quite similar and overlapped with each other. Similarly, DF-6-BR and DF-7-HM resulted in very close shear stress/rate curves.

The drilling fluid viscosity needs to be controlled and maintained at a sufficient value to suspend the drilled cuttings among rheological parameters. The viscosity of the drilling fluid also controls the performance of solid control equipment. PV and apparent viscosity (AV) play a significant role in transporting cuttings and their suspension. Both rheological parameters were impacted by adding CaSO_4 and other weighting materials, as shown in Figure 4. The PV of OBDs loaded with different concentrations of CaSO_4

increased with an increase in the concentration of CaSO_4 in DF-1 (base-drilling fluid). For instance, the PV of DF-2-CS and DF-3-CS was higher by 46.6 and 100.7% compared to DF-1.

Further addition of CaSO_4 by 175 ppb in DF-1 enhanced the PV by many fold. DF-4-CS showed a lot of solid content and precipitation of CaSO_4 at this concentration. The CaSO_4 reached a supersaturating level and, as a result, precipitated. The precipitated solids of CaSO_4 offered high PV. Similarly, CaCO_3 at high concentration precipitates. It was reported that precipitation of CaCO_3 is more complex than CaSO_4 .³⁵ The high PV increases the drilling fluid ability to suspend and transport cuttings during drilling and provides better wellbore cleaning, as displayed by DF-2-CS and DF-3-CS.

On the other hand, DF-4-CS resulted in the highest PV among all the tested OBDs. The recommended PV values of OBDs should be in the range of 10 to 60 cP, preferably (15–40 cP), according to Gautam and Guria³⁶ and Li et al.³⁷ As a result, all drilling fluids employed in this investigation met this criterion except DF-4-CS. The friction among particles became evident with the increase in particles in drilling fluids, which enhanced PV. Very high PV provides severe friction losses and might result in formation breakdown because of the increased pumping pressure required for its circulation. Furthermore, high-PV drilling fluids have a powerful impact on the drilling process and slow down the penetration rate.

The performance of DF-3-CS was compared with that of DF-5-CC, DF-6-BR, and DF-7-HM, which were mixed with an equal amount (124 ppb) of CaCO_3 , barite, and hematite. The DF-3-CS showed a bit high viscosity based on the PV results compared to that of the DF-5-CC, DF-6-BR, and DF-7-HM.

The AV plays a significant role in cutting transportation, drilling rate, and efficiency of solid removal equipment. In the Bingham plastic model, it is one-half of shear stress measured at 600 rpm. The AV increased with CaSO_4 and other weighting materials compared to the unweighted drilling

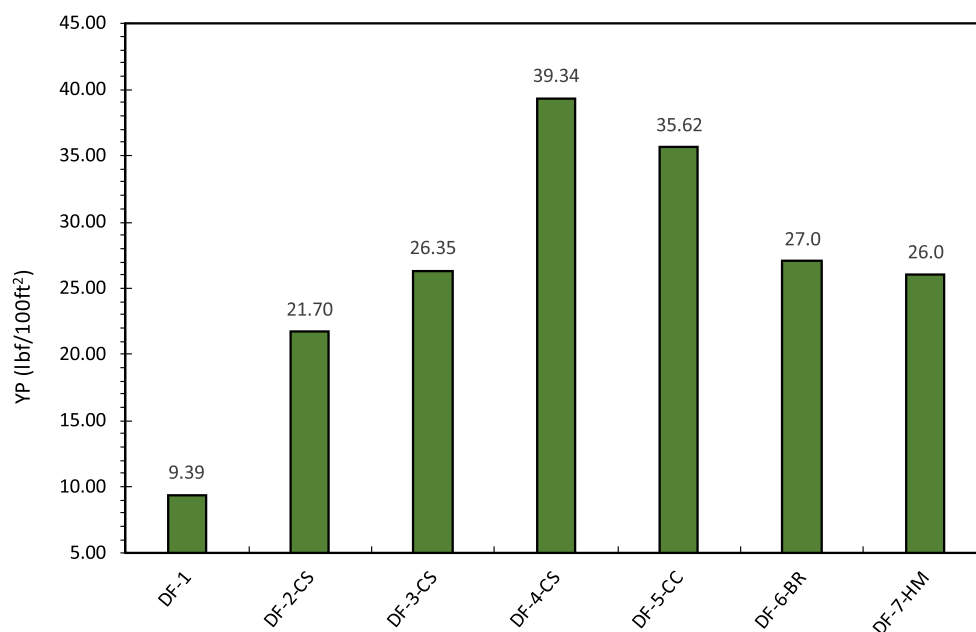


Figure 5. YP change of drilling fluids containing various concentrations of CaSO_4 (DF-1 to DF-4-CS) and other weighting materials (DF-5-CC, DF-6-BR, and DF-7-HM).

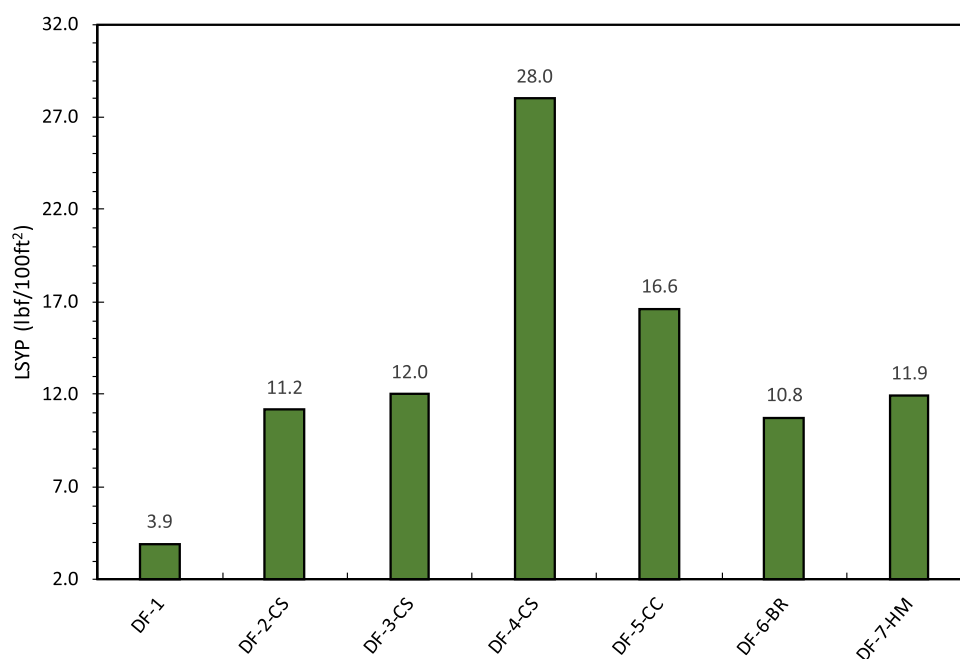


Figure 6. LSYP change of drilling fluids containing various concentrations of CaSO_4 (DF-1 to DF-4-CS) and other weighting materials (DF-5-CC, DF-6-BR, and DF-7-HM).

fluid (DF-1). The trend of AVs of all OBDFs (DF-1 to DF-7-HM) was similar to that of PV.

Figure 5 shows the YPs of OBDFs (DF-1 to DF-7-HM) laden with different concentrations of CaSO_4 and different weighting materials (CaCO_3 , barite, and hematite). YP develops because of electrochemical attraction among particles of drilling fluids, and it plays a significant role in the carrying capacity of drilling fluids during circulation. YP is the ultimate stress required to initiate the distortion or flow of drilling fluids. It impacts many other parameters, such as wellbore cleaning, barite sag, equivalent circulating density (ECD), swab, and surge pressures.³⁸ The addition of CaSO_4 increased

YP. For instance, adding 62 ppb of CaSO_4 into DF-1 increased the YP from 9.39 to 21.70 lbf/100 ft². A further increase in the concentration of CaSO_4 brought a slight change in YP, as noticed in DF-3-CS. Later, mixing 175 ppb CaSO_4 in DF-1 increased the YP by many fold, which occurred because of very high solid contents and precipitation of CaSO_4 solids. Similarly, CaCO_3 -, barite-, and hematite-containing OBDFs enhanced the YPs by many fold compared to DF-1. At similar concentrations of all materials (124 ppb), the CaSO_4 -containing OBDF had a YP of 26.35 lbf/100 ft², which was the same as that of DF-7-HM, and the CaCO_3 -containing OBDF provided the highest YP (35.62 lbf/100 ft²).

Low shear yield point (LSYP) plays a significant role in wellbore cleaning and carrying capacity. It is as vital as YP during the drilling operation. For better rheology, it is recommended to have better YP and a low value of LSYP. The desirable range of LSYP falls in the range of 7–15 lbf/100 ft².^{39,40} LSYP improved with the addition of weighting agents (Figure 6). It was noticed that DF-2-CS and DF-3-CS resulted in low values of LSYP, which fall in the optimum range.

On the other hand, DF-4-CS exceeded the range and provided the highest value as the high value resulted from high solid contents detrimental to the drilling operation. The CaCO₃-containing drilling fluid provided 16.6 lbf/100 ft² and was close to the high limit of LSYP. For barite- and hematite-containing OBDFs, the LSYP existed in the optimum range.

The carrying capacity of OBDFs containing various weighting materials was evaluated by the ratio of (YP/PV) given in Table 1. It has been reported in the literature that the

Table 1. Carrying Capacity Property (YP/PV) Drilling Fluids Containing Various Concentrations of CaSO₄ (DF-1 to DF-4-CS) and Other Weighting Materials (DF-5-CC, DF-6-BR, and DF-7-HM)

formulations	YP/PV
DF-1	0.64
DF-2-CS	1.01
DF-3-CS	0.89
DF-4-CS	0.29
DF-5-CC	1.42
DF-6-BR	1.29
DF-7-HM	1.16

most suitable values of YP/PV range from 0.75 to 1.00 (lbf/100 ft²/cp).⁴¹ All the tested drilling fluids provided good wellbore cleaning and cutting suspension, except DF-4 containing 175 ppb of CaSO₄. A low YP/PV value results in

the settling of weighting materials and drill cuttings and causes sticking of the drilling string. On the other hand, a high YP/PV ratio increases the annular frictional pressure losses, consequently increasing the drilling fluid's ECD. The YP/PV results further strengthen our observation that a higher concentration of CaSO₄ impacted the wellbore cleaning. DF-2-CS, DF-3-CS, DF-5-CC, DF-6-BR, and DF-7-HM provided better wellbore cleaning and carrying capacity.

The gel strengths at 10 s and 10 min data are shown in Figure 7. All drilling fluids provided developed gel strengths, which means that gel strength increased with time from 10 s to 10 min except for the barite-containing drilling fluid (DF-6-BR). The highest gel strength was gained in DF-4-CS composed of a high content (175 ppb) of CaSO₄. It severely impacted gel strength development. The CaCO₃-containing drilling fluid (DF-5-CC) results in higher gel values than other weighting materials mixed with the same concentration (124 ppb). The gel strengths of all drilling fluids fall in the range of (4–15 lbf/100 ft²)³⁷ except DF-4-CS, which provided high gel strength and precipitation of solids at higher concentrations.

2.3. Fluid Loss Test. The fluid loss test was conducted for CaSO₄-containing drilling fluids prepared with three different concentrations (62, 124, and 175) ppb. The impact of CaSO₄ as a weighting agent and other weighting materials including CaCO₃, barite, and hematite on fluid loss was investigated. All weighting materials were evaluated at the same concentrations irrespective of their different weight. It was observed that fluid loss decreased with an increase in the concentration of CaSO₄ except at a higher concentration (175 ppb); there, it was increased by twice as compared to DF-1 (see Figure 8). At low concentrations, the fine particle size and water solubility of CaSO₄ reduced the fluid loss. CaSO₄ precipitated out of the drilling fluid at higher concentrations and built a permeable filter cake resulting in high fluid loss.

Furthermore, the performance of DF-3-CS mixed at 124 ppb of CaSO₄ in DF-1 was compared with that of DF-5-CC, DF-6-

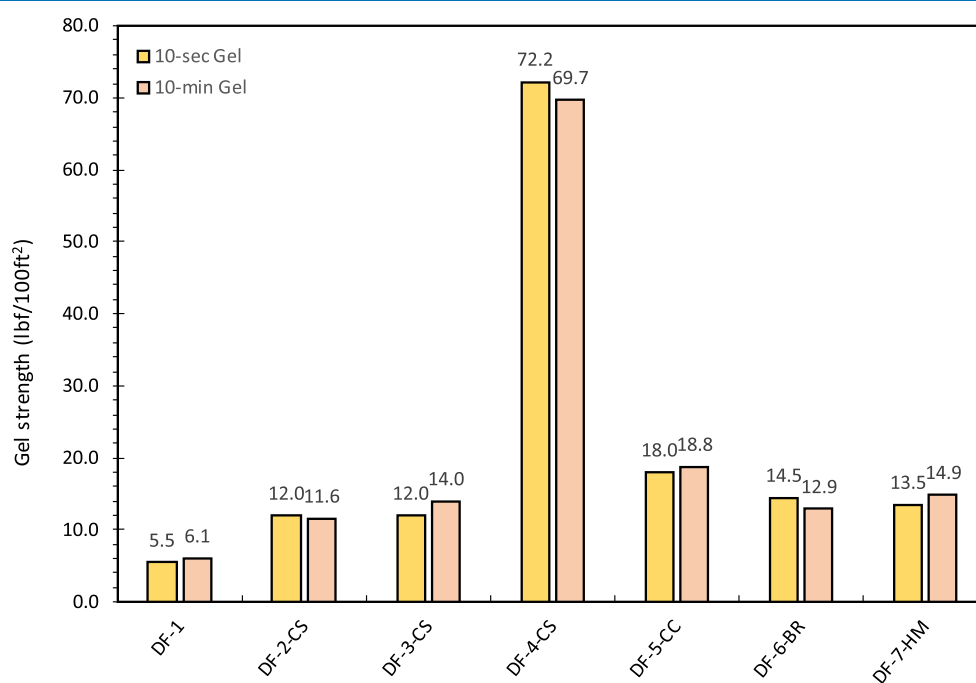


Figure 7. Gel strengths (10 s and 10 min) of drilling fluids containing various concentrations of CaSO₄ (DF-1 to DF-4-CS) and other weighting materials (DF-5-CC, DF-6-BR, and DF-7-HM).

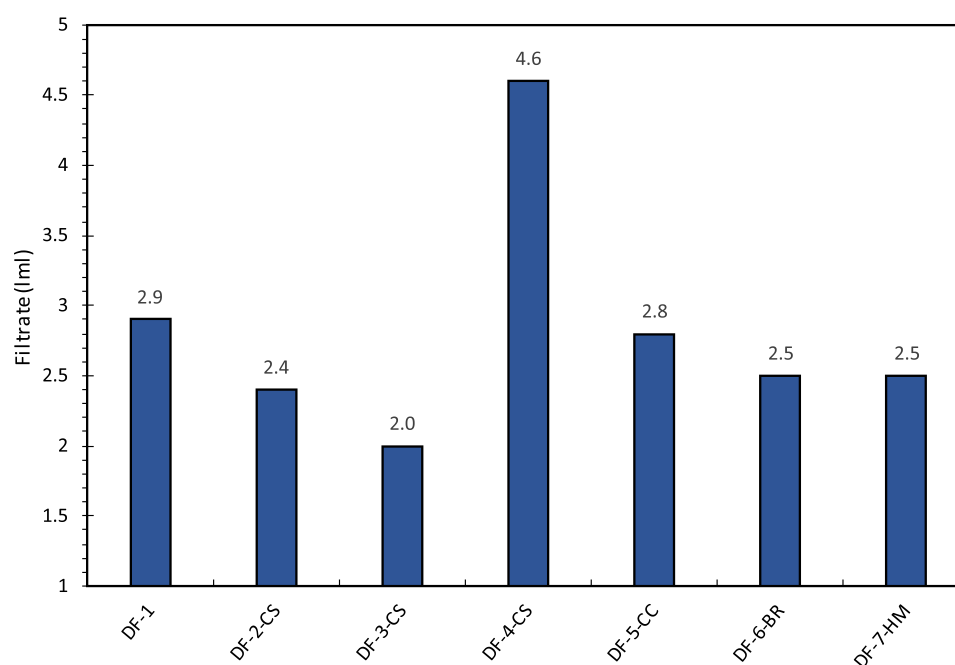


Figure 8. Fluid loss test results of drilling fluids (DF-1 to DF-7-HM).

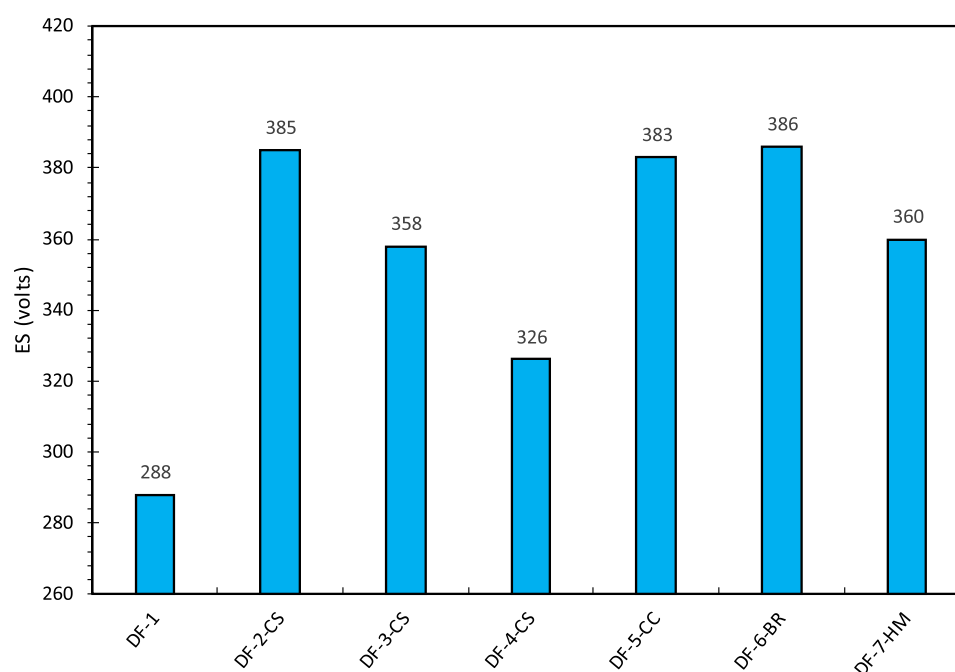


Figure 9. ESs of drilling fluids (DF-1 to DF-7-HM).

BR, and DF-7-HM. It was observed that DF-3-CS resulted in the lowest fluid loss of 2 mL, among other tested fluids. For instance, CaCO₃, barite, and hematite-containing drilling fluids provided 2.8, 2.5, and 2.5 mL as filtrates, respectively.

2.4. Electrical Stability. The ES of CaSO₄-containing drilling fluids was evaluated using an electrical stability tester. Furthermore, CaSO₄ performance was compared with that of other weighting material-containing drilling fluids (DF-5-CC, DF-6-BR, and DF-7-HM) mixed at the same amount (124 ppb) in DF-1.

Figure 9 provides the ESs of all the tested OBDs (DF-1 to DF-7-HM). It was observed that the addition of CaSO₄ increased the ES as compared to DF-1. Further increase in

concentration decreased the ES value as noticed in DF-4-CS. The high solid contents of CaSO₄ reduced ES. The ES values of DF-2-CS, DF-3-CS, DF-5-CC, and DF-6-BR were quite similar with slight variation. DF-3 and DF-7 resulted in almost the same ES values.

2.5. Filter Cake Removal. The process of filter cake removal of the calcium sulfate OBDs can be conducted in a single- and two-stage removal process.

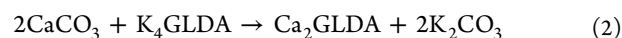
2.5.1. Single-Stage Filter Cake Removal. The single-stage remover consists of 20 wt % K₄GLDA chelating agent +6 wt % potassium carbonate +10% EGMBE (ethylene glycol mono-butyl ether). The soaking time for this formulation was

optimized and found to be 24 h. The mechanism of filter cake removal using this formulation is as follows:

- i. The 10 vol % EGMBE can remove the oil coat from the filter cake.
- ii. Potassium carbonate will convert the calcium sulfate to calcium carbonate in the presence of a high-pH GLDA chelating agent as follows:



In addition, the 20 wt % K_4GLDA will dissolve the resulting calcium carbonate from the conversion process as follows:



This single-stage removal process requires 24 h to obtain a removal efficiency of 83%, as shown in Figure 10. After 24 h,

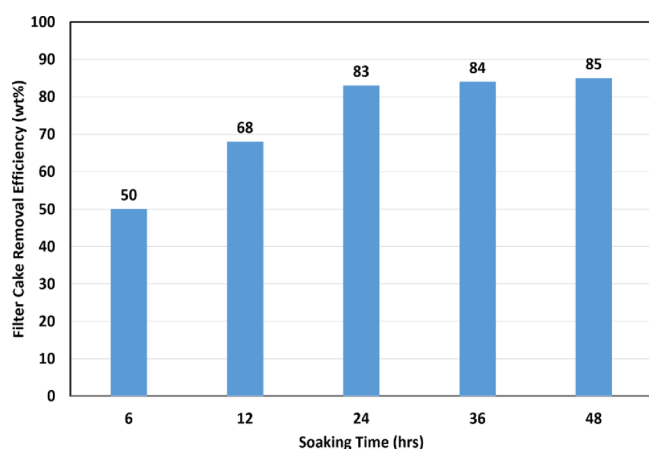


Figure 10. Removal efficiency in a single stage.

there was no significant enhancement in the filter cake removal efficiency. Therefore, the optimum soaking time is 24 h. The calcium sulfate conversion to calcium carbonate is a continuous process. Once the oil is removed from the cake in an OBDF, the calcium sulfate will be exposed for reaction with potassium carbonate. It results in calcium carbonate that

will react with GLDA to reproduce potassium carbonate again, and this process will continue until the majority of calcium sulfate is converted. The filter cake removal experiments were conducted at 100 °C and 300 psi differential pressure.

2.5.2. Multistage Filter Cake Removal. In this method, the formulation will be pumped on a stage mode; three stages of chemicals will be injected into the well as follows:

- i. Inject 10 vol % of EGMBE to remove the oil for 6 h.
- ii. Inject 1 wt % water wetting surfactant to remove the residual oil from the filter cake for 4 h.
- iii. Inject 20 wt % K_4GLDA + 6 wt % K_2CO_3 for 24 h.

This formulation resulted in a filter cake removal efficiency of 91% compared to 83% in the single-stage method. The concentration of the solvent (EGMBE) was selected based on interfacial tension reduction, 10 vol % was the optimum concentration, and higher concentrations did not result in a significant decrease in the interfacial tension, as shown in Figure 11. In addition, the same for the water wetting surfactant, 1 wt % was the optimum concentration that resulted in the maximum water wetness of the cake surface, as shown in Figure 12. The first and second stages were selected based on the oil removal from the filter cake and the degree of wettability change and interfacial tension reduction that indicated the equilibrium of the process, as shown in Figure 13. The third-stage time was selected on the maximum conversion of the calcium sulfate, and it was found to be 24 h in the case of the oil-based filter cake.

3. CONCLUSIONS

The drilling fluid formulation containing calcium sulfate showed comparable rheological properties to the drilling fluid formulation containing calcium carbonate as a weighting material. The use of calcium sulfate as a weighting material could have several advantages over calcium carbonate, such as:

1. Calcium sulfate affects the rheology and fluid loss properties of the OBDF.
2. The drilling fluid with calcium sulfate produced lower filtration losses than other weighting materials (calcium

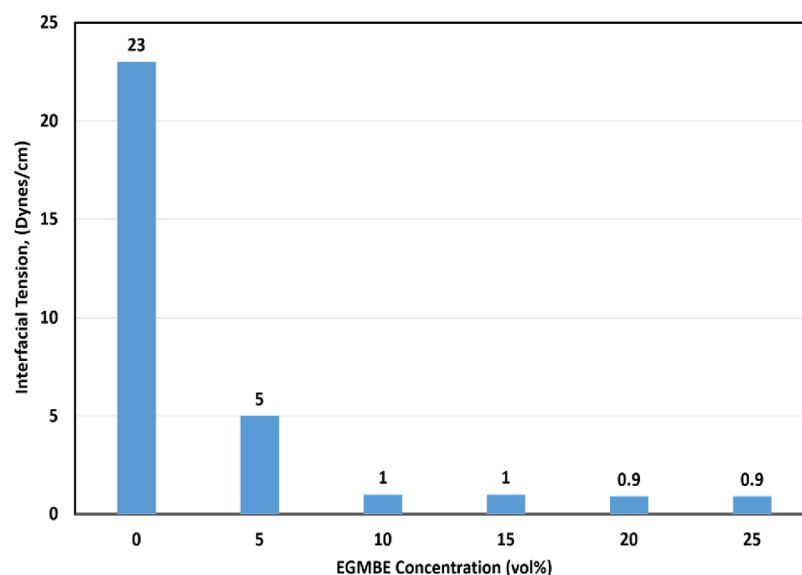


Figure 11. Effect of solvent concentration on the interfacial tension between diesel oil and water for the multistage filter cake removal process.

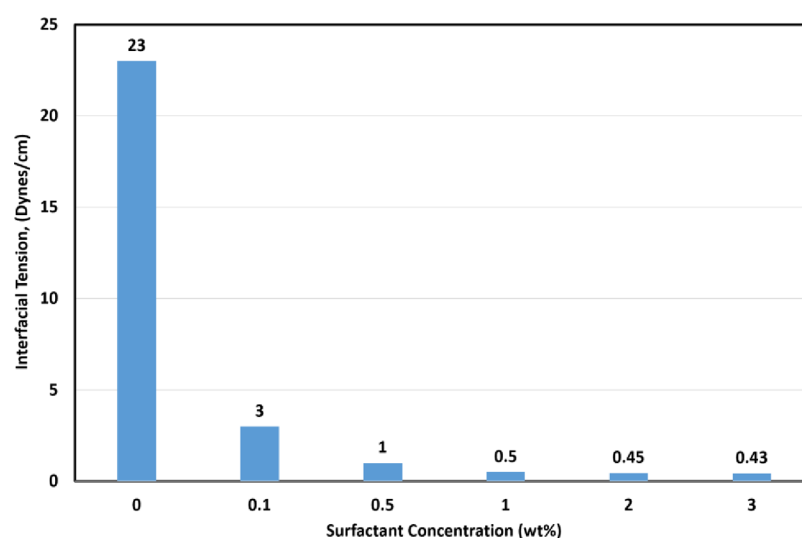


Figure 12. Effect of surfactant concentration on the interfacial tension between diesel oil and water for the multistage filter cake removal process.

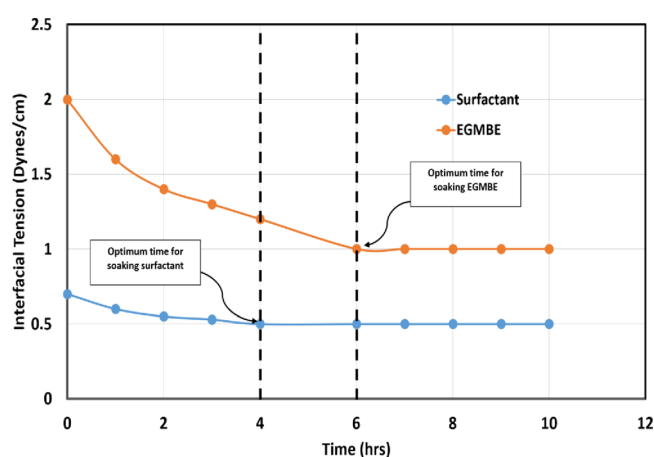


Figure 13. Optimum time for multistage filter cake removal for the surfactant and EGMBE stages.

carbonate, barite, and hematite) mixed at an equal amount.

3. The fluid loss increased with the content of calcium sulfate in the drilling fluid.

4. The ES was affected by the addition of CaSO_4 , and it reduced at high contents (175 ppb) of CaSO_4 .

5. Calcium sulfate has a higher density than calcium carbonate; therefore, it can be used in drilling HPHT wells with less quantity.

6. Calcium sulfate provides high carrying capacity because of high-generated PV at a concentration of 124 ppb concentration.

7. The calcium sulfate filter cake can be removed easily from the wellbore with an efficiency of 83 to 91% in single-stage and multistage removal processes, respectively, by the newly developed formulation in this study.

4. MATERIALS AND METHODS

In this study, calcium sulfate was introduced as a novel weighting material in OBDs. The calcium sulfate was purchased from ACROS ORGANICS with purity above 99%. It appears as an odorless, white powder or a colorless, crystalline solid with a specific gravity of 2.96. It is slightly

soluble in water with a solubility of 0.2 g/100 mL of water at 20 °C. The Mohs hardness of CaSO_4 is 3.5. The nominal median particle sizes (D_{50}) of CaSO_4 were 2.62 μm . Halliburton, Saudi Arabia supplied other weighting materials (CaCO_3 , barite, and hematite). Table 2 provides the properties of barite, hematite, and CaCO_3 .

Table 2. Properties of Weighting Materials (Barite, Hematite, and CaCO_3)

weighting material	formula	specific gravity	Mohs scale hardness	color	median size (d50)
barite	BaSO_4	4.1	2.5–3.5	grayish	9.91
hematite	Fe_2O_3	5.02	5.5–6.5	reddish brown	11.03
calcium carbonate	CaCO_3	2.7	2.5–3	off white	12.37

4.1. Drilling Fluid Preparation. The performance of CaSO_4 as a weighting agent was investigated by mixing it in different concentrations such as 62, 124, and 175 ppb in OBDs and compared with that of CaCO_3 , barite, and hematite-mixed OBDs (DF-5-CC, DF-6-BR, and DF-7-HM). The compositions and sequence of mixing of the tested drilling fluids are given in Table 3. All the additives were mixed using a high-speed Hamilton beach mud mixer. The OBD was prepared by mixing emulsifiers (primary and secondary) in diesel followed by lime (emulsion stabilizer), calcium chloride brine solution (shale stabilizer), a viscosifier, and a fluid loss controller. In the end, weighting materials were mixed in the base-drilling fluid, and their impact on density, rheology, fluid loss, and ES was investigated. Furthermore, CaSO_4 formed filter cake removal methods were discussed in detail.

4.2. Density and Rheology Test. The drilling fluid density is essential in measuring the hydrostatic pressure exerted by drilling fluids during drilling operations. The density of drilling fluids plays a critical role in the stability of the wellbore as it mainly controls kicks and lost circulations. The drilling fluid density was measured using a FANN mud balance apparatus (Model#140). The density measurement test was performed according to American Petroleum Institute (API) 13B-1.⁴²

Table 3. Compositions of OBDFs

additives	mixing time (min)	DF-1	DF-2-CS	DF-3-CS	DF-4-CS	DF-5-CC	DF-6-BR	DF-7-HM
diesel, bbl	0	0.6	0.6	0.6	0.6	0.6	0.6	0.6
primary emulsifier, ppb	6	6	6	6	6	6	6	6
secondary emulsifier, ppb	2	3	3	3	3	3	3	3
lime, ppb	10	8	8	8	8	8	8	8
CaCl ₂ brine salt/water, ppb/bbl	5	44/0.17	44/0.17	44/0.17	44/0.17	44/0.17	44/0.17	44/0.17
viscosifier, ppb	10	8	8	8	8	8	8	8
fluid loss controller, ppb	10	8	8	8	8	8	8	8
CaSO ₄ , ppb	10		62	124	175			
CaCO ₃ , ppb	10					124		
barite, ppb	10						124	
hematite, ppb	10							124

Rheology plays a pivotal role during the drilling operation as removing cuttings and suspension is controlled by rheological parameters.⁴³ In this study, the rheology of the OBDFs was evaluated using a digital atmospheric viscometer (Model#M3600). Different rheological parameters were measured by conducting standard procedures of the API.⁴⁴ The shear stress was evaluated by applying a shear rate ranged from 3 to 600 rpm for all OBDF. PV, YP, AV, and LSYP were calculated using eqs 1234 of the Bingham plastic model.

$$PV = \phi_{600 \text{ rpm}} - \phi_{300 \text{ rpm}} \quad (3)$$

$$YP = \phi_{300 \text{ rpm}} - PV \quad (4)$$

$$AV = \phi_{600 \text{ rpm}}/2 \quad (5)$$

$$LSYP = 2 \times \phi_{3 \text{ rpm}} - \phi_{6 \text{ rpm}} \quad (6)$$

where $\phi_{600 \text{ rpm}}$, $\phi_{300 \text{ rpm}}$, $\phi_{6 \text{ rpm}}$, and $\phi_{3 \text{ rpm}}$ are dial readings at 600, 300, 6, and 3 rpm, respectively. The PV and AV were reported in centipoise (cP) unit, and YP, LSYP, and gel strengths were reported in lbf/100 ft².

The gel strengths (10 s and 10 min) were measured at the end of the static period of 10 s and 10 min by shearing at 3 rpm. The maximum shear stress obtained at a low shear of 3 rpm after a static period defines gel strength.

4.3. Electrical Stability Test. The ESs of OBDFs were determined using an electrical stability tester (Model 23E, FANN) at 120 °F. In this test, the probe of the electrical stability tester was immersed in the OBDF, making sure that the fluid covered the electrode surfaces. The drilling fluid was briskly stirred for 10 s. Later, the test button was pressed on the instrument. The voltage started ramping up and stopped at breakdown voltage, the point at which current flows between the electrodes. The average voltage of the three readings was reported for each sample as electrical stability.

4.4. Filtration and Filter Cake Removal Test. The fluid loss potential of CaSO₄ and other weighting material-containing drilling fluids was investigated by conducting a filtration test using HPHT filter press from OFITE. The test was conducted based on the API standard procedure (API 13-B). In this test, the filtration cell was filled with 175 mL of drilling fluid and loaded in the filtration instrument. The test was conducted under 150 °F temperature and 300-psi differential pressure conditions for 30 min. Upon loading the cell, the temperature of the cell was raised to 150 °F temperature. Upon reaching the targeted temperature, 300-psi differential pressure was applied in the cell at the top of the drilling fluid using nitrogen gas. The bottom cap valve of the

cell was opened, and the filtrate was collected in a graduated cylinder in milliliter (mL). The filtrate was collected for 30 min. At the end of the test, the heater was switched off, and the pressure was released.

The formed filter cake in the calcium sulfate-based drilling fluid was placed in an HPHT cell. The cell was filled with the remover fluid; two different scenarios were suggested to remove the filter cake of the calcium sulfate OBDF.

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Notes

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