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Experimental Investigation of Using Manganese Monoxide as a Hydrogen Sulfide Scavenger for Aqueous Drilling Fluids

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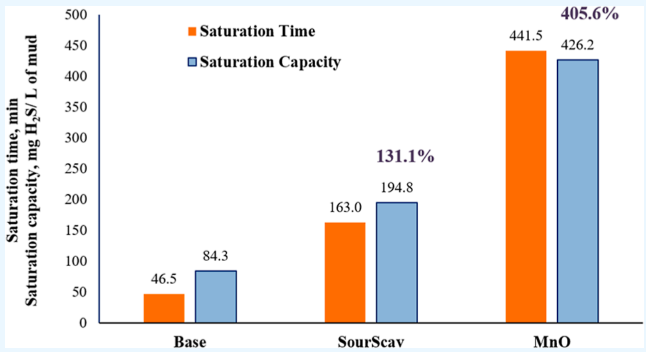
ABSTRACT: One of the most serious safety and health concerns during drilling oil and gas wells is the potential release of hydrogen sulfide (H_2S) to the surface, exposing workers to high risks. Serious corrosion-related damage to handling equipment is also inevitable in the presence of H_2S. Various H_2S scavengers have been utilized, but each has its pros and cons; hence, research is continuing to develop an optimum and feasible scavenger. Since manganese monoxide (MnO) is a reactive metal oxide with high oxidation and absorption capabilities, it may have the potential to effectively scavenge H_2S during drilling operations when included in drilling mud formulations. Consequently, the key aim of this work is to investigate the H_2S scavenging performance of the aqueous drilling fluid containing MnO. This work studied the impact of MnO addition on the drilling mud's alkalinity, rheological behavior, filtration performance, and corrosion tendency. The experiments were also conducted for mud without a scavenger and a fluid containing the SourScav commercial scavenger, which serves as a benchmarking reference. The findings demonstrated that MnO performed exceptionally well for H_2S scavenging where it boosted the aqueous mud's scavenging capacity from 84.3 to 426.2 mg of H_2S/L of mud, showing more than 400% improvement relative to the base mud. Additionally, this scavenging performance is about 2.1 times higher than that of the commercial scavenger. As opposed to SourScav, MnO maintained the mud's pH at a safe level above 10. The addition of either MnO or SourScav did not weaken the mud rheology and provided practically satisfactory rheological parameters. Both SourScav and MnO marginally increased the formed filter-cake thickness from 2.9 to 3.9 mm with a slight increment in the filtrated volume but still within the acceptable limits. The corrosion test indicated the noncorrosive characteristics (i.e., the corrosion rate was nearly zero) of MnO and the commercial scavenger. This study illustrates the promising utilization of MnO as a cost-effective H_2S scavenger, enhancing the efficiency and safety of drilling operations.



1. INTRODUCTION

Drilling fluids consist of suspended active/inactive solids and chemicals in a colloidal continuous phase (either liquid or gas). They can be split into three groups depending on whether they are water-based, oil-based, or gaseous-based. Aqueous drilling muds are the most commonly used because they are relatively inexpensive, have a number of technical advantages, and are less harmful to the environment than other types of drilling muds.[1−4] The efficient design and selection of drilling fluid additives are essential to ensure providing the desired mud functions while also preserving the required mud characteristics for successful drilling operations.[5] One of these specified mud additives is the hydrogen sulfide (H_2S) scavenger that must be included in the drilling mud composition in the sour environment (i.e., H_2S bearing formations).[6]

This toxic and corrosive gas is common to encounter in geological subterranean formations from different sources. The natural existence of H_2S in formations arises from geological origins or as a byproduct of sulfate breakdown by anaerobic bacteria.[7,8] Additionally, H_2S can be formed through the thermal decomposition of sulfur-containing compounds.[9] The exposure of personnel to this highly hazardous gas can lead to severe health and safety repercussions, encompassing irritation, respiratory failure, loss of consciousness, collapse, and even fatality.[10] In addition, because of its acidic nature, H_2S can cause various corrosion problems, such as under-deposit corrosion, sulfide-stress cracking, pitting, and hydrogen embrittlement.[11,12] Therefore, encountering H_2S during drilling operations may lead to substantial economic losses and

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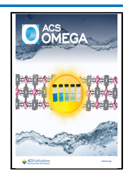

cause safety and health hazards unless effective in situ scavenging measures are implemented.[13]

Using H_2S scavengers (sorbent materials) is a feasible approach for the downhole scavenging of H_2S and avoiding its release to the surface. These scavengers are compounds that engage with H_2S to generate less hazardous substances.[11] They come in two forms: solids that adsorb H_2S and liquids that operate as H_2S absorbents.[9,14] Additionally, H_2S scavengers can be regenerative or nonregenerative. An efficient H_2S scavenger should be able to remove H_2S quickly and completely in a broad variety of pressure and temperature conditions[8] without compromising the properties of the drilling mud.[15]

Certain H_2S-scavenging chemicals possess regenerative properties, enabling their repeated utilization (such as amine-based compounds). These substances find application in extensive amine facilities tasked with processing substantial quantities of sulfurous fluids. When the H_2S concentrations within these fluids fall within the percentage range, amine plants stand as the most efficient and cost-effective means of treatment.[16] Different amine compounds are used as sour scavengers. Alkanolamines such as monoethanolamine (MEA), diethanolamine (DEA), *N*-methyl-diethanolamine (MDEA), and diglycol amine (DGA) are the most used amine compounds.[17] These compounds can be described as regenerative scavengers as they can be regenerated and reused. They are employed mainly in the gas process stream by controlling the process parameters such as pressure, temperature, and amine concentrations to minimize operational problems.[18]

Oxidizers, triazines, and metallic substances are instances of nonregenerative scavengers that are commonly utilized in drilling operations, with a preference for metallic scavengers.[8,19] Oxidizing agents such as hydrogen peroxide and potassium permanganate are utilized for the purpose of eliminating H_2S from drilling muds. Nevertheless, hydrogen peroxide displays instability at elevated pH levels and acts as a nonselective reactant, thereby exhibiting high reactivity with other mud components.[9,20,21] On the other hand, potassium permanganate demonstrates a more effective scavenging performance, accompanied by improved rheological properties. However, it proves ineffective when dealing with heavy mud.[6,19]

Triazines react quickly and efficiently with H_2S; however, they can lead to undesirable consequences by generating dithiazine as a precipitated byproduct. Challenges associated with these scavengers include the formation of intractable solids, relatively sluggish reaction rates when applied through direct injections, limited thermal stability, and a propensity for scaling. Consequently, for downhole applications, triazines are not favored unless they undergo suitable modifications to suit the specific conditions of the wellbore.[22−25]

Salts, oxides, and chelates of metals are employed as H_2S scavengers in drilling operations. The commonly used metals are copper, zinc, and iron.[9] While copper-containing substances serve as effective scavengers because they react quickly with H_2S, they should be used with caution in drilling fluids as they can potentially cause bimetallic corrosion.[8,15,20,26] Zinc compounds also effectively remove H_2S, especially when they are in the nanoparticle form.[27] These substances possess several appealing attributes, including an amphoteric nature, a predictable reaction, and thermal stability.[28−30] However, the generation of zincate ions results in mud flocculation and rheology degradation.[11,31−33] Iron-based scavengers are also used; however, they exhibit complex reactions with H_2S. Additionally, their effectiveness is contingent on pH levels. They exhibit greater efficacy within acidic pH ranges; however, drilling fluids with low pH levels are unusual.[33,34]

As discussed above, applied H_2S scavengers have their own benefits and drawbacks. Consequently, researchers are still looking for feasible scavengers with optimum performance.[8,19]

As far as the authors are aware, there has not been any prior research that has been documented in the published literature for assessing the efficacy of manganese oxide in removing H_2S from drilling muds, which is the stimulus for this study. Manganese oxides have been reported to be very reactive metal oxides that can operate as sorbents/catalysts for H_2S scavenging at high temperatures.[35] Manganese oxides exhibit two modes of reaction with H_2S: sulfidation and reduction−oxidation. In general, simple sulfidation occurs at lower temperatures and higher pH ranges, while reduction−oxidation is more likely to occur at higher temperatures and lower pH levels.[36,37] Among the different manganese oxides, manganese monoxide (MnO) is the most stable form under reducing environments since the other oxides are reduced to MnO when exposed to a reducing atmosphere.[38] Also, MnO has a faster reaction rate compared to that of other metal oxides.[39,40] Several researchers addressed the scavenging performance of combined manganese oxides with support substances, such as aluminum oxides, showing competent stability and performance for removing H_2S from gas streams.[35,41−45]

In this research, the incorporation of MnO in the aqueous drilling mud and the investigation of the H_2S scavenging performance of the developed fluid have been performed. The performance of the MnO-containing mud has been compared to that of the base mud as well as to the mud containing the commonly used SourScav commercial scavenger. When developing new mud additives, it is necessary to conduct extensive laboratory and field testing to evaluate the rheological properties, filtering behavior, corrosion affinity, and other key characteristics in order to determine the new mud formulation's applicability. Thus, these mud properties were extensively studied at the laboratory scale. The primary novelty of this study lies in the introduction and assessment of MnO as a scavenger to remove H_2S in situ from drilling fluids, aiming to enhance the drilling efficiency and safety.

2. MATERIALS

The drilling mud utilized in this research was created using a real-field mud recipe. This involved sequentially adding various mud additives to distilled water under ambient circumstances using a high-speed blender. The procedure for preparing the base mud commenced by introducing an antifoaming agent, known as a defoamer, into the water. Subsequently, xanthan viscosifier polymer, starch (to manage fluid losses and enhance viscosity development), sodium chloride (for shale stabilization), and caustic soda (to regulate alkalinity) were added. To achieve a density of 9.8 pounds per gallon (ppg), calcium carbonate was incorporated as a bridging and weighting material. Following this, the commercial or proposed H_2S scavengers were blended into the base mud. The specific sequence and quantities of each additive in the mud composition are listed in Table 1.

The proposed scavenger (i.e., MnO), which has a density of 5.37 g/cm^3, is an inorganic metal oxide compound that is dry and green, powdery, and insoluble in water. As a reference

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Table 1. Drilling Mud Formulation

component	amount
distilled water	316 cm^3
defoamer	0.08 cm^3
xanthan polymer	1 g
starch	6 g
sodium chloride	34 g
caustic soda	0.25 g
calcium carbonate (50 μm + 25 μm)	36 + 24 g
H_2S scavenger	0/1 g

point for comparison, a commercial H_2S scavenger called SourScav (provided by a drilling fluid service provider) was used. SourScav is an iron-gluconate-base powder that is water-soluble and has a specific gravity of 0.70 and a pH of 4.5.

3. EXPERIMENTAL WORK

The methodology employed in this investigation is illustrated in Figure 1 and described below.

To comprehensively investigate the capability of MnO as a H_2S scavenger, its scavenging capacity and impact on the characteristics of the water-based mud were assessed and compared to those of the base mud and SourScav-containing fluid. The H_2S scavenging experiment was carried out by exposing 10 cm^3 of the prepared fluids in a bubble column (buret) to a H_2S gas stream. The H_2S source is a cylinder containing 100 ppm of H_2S (methane balance gas). The gas inlet flow rate was maintained constant at 150 cm^3/min using a flowmeter equipped with a control valve. Using a gas detection analyzer (MultiRAE Lite-PGM-6208) connected to the bubble column outlet, the H_2S concentration in the effluent stream was continuously monitored and analyzed until the saturation point. This point is achieved when the H_2S concentrations at the outlet and inlet are equal (i.e., 100 ppm). The test was carried out at room temperature. The H_2S scavenging capacity at saturation point was quantified using the measured concentrations and the following equation[17]

$$\text{Saturation capacity(mg/L)} = 150\times10^{-7}\times\rho\times\int_0^{t_s}(100 - C_{out})dt \quad (1)$$

where ρ represents the H_2S density (1.391 mg/cm^3 at 25 °C and atmospheric pressure [6]), t_s is the time at saturation point, and C_{out} denotes the H_2S concentration at the bubble column outlet.

In a sour environment, mud alkalinity is a key parameter that profoundly influences its rheology and filtration performance.[46] As such, the alkalinity of each mud sample was analyzed by determining its pH value under ambient conditions using a calibrated digital pH meter from Mettler Toledo.

The rheological indices of the freshly prepared drilling fluids, including the plastic viscosity (PV), apparent viscosity (AV), yield point (YP), and gel strength, were obtained using a direct-indicating viscometer (Grace M3600 Rheometer) at 120 °F. This equipment was operated by shearing the fluid in the annular space between the rotor and the bob. The reading of the bob was specified by a dial deflection reading (Φ) at various shear rates ranging from 3 to 600 rpm. The Bingham plastic model was employed to determine the viscosities and YPs of the fluids using the below equations.

The initial gel strength (10 s-gel) was determined directly by measuring the highest dial reading at 3 rpm after a static period for 10 s, while the final gel strength (10 min-gel) was measured after the fluid had remained undisturbed for 10 min.

$$\text{AV} = \frac{\emptyset_{600\text{ rpm}}}{2} \quad (2)$$

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

$$\text{YP} = 2\times\emptyset_{300\text{ rpm}} - \emptyset_{600\text{ rpm}} \quad (4)$$

To evaluate the filtration conduct of the formulated muds, a static fluid loss test was performed in accordance with the recommended practices of the American Petroleum Institute (API). The experiment was performed in a high-temperature high-pressure (HTHP) OFITE filter press at 250 °F for 30 min with a differential pressure up to 300 psi, and a 40 μm ceramic disc was utilized as the filtration media. During the test, the filtrate was obtained in a cylinder, and its volume was recorded at different time intervals. Additionally, the thickness of the filter cake was also determined.

The corrosion rate of the drilling fluid containing MnO was assessed from the corrosive test and compared with those of the base- and SourScav-containing muds. The test was performed by immersing a metal coupon made of N80-grade casing steel in the mud inside an aging cell for a period of 6 h at a temperature of 250 °F and a pressure of 300 psi. The weight of the metal coupon was measured both prior to and after aging. The corrosion rate was then determined by

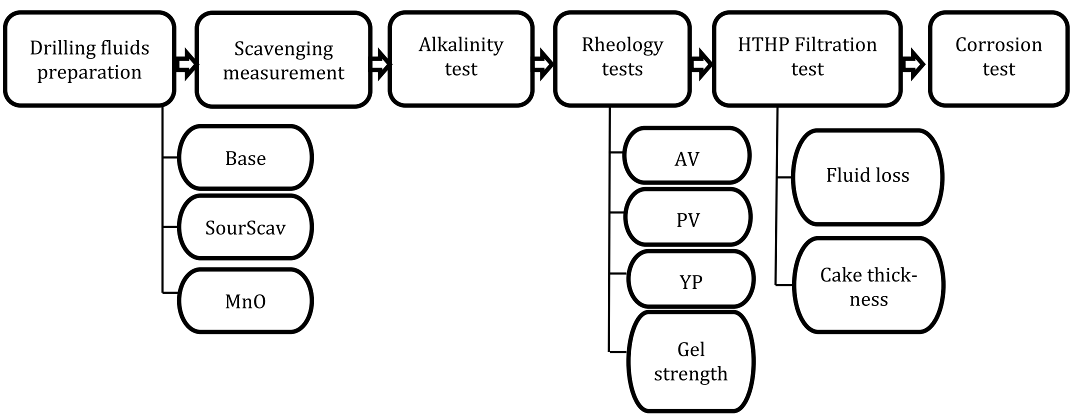

Figure 1. Experimental approach.

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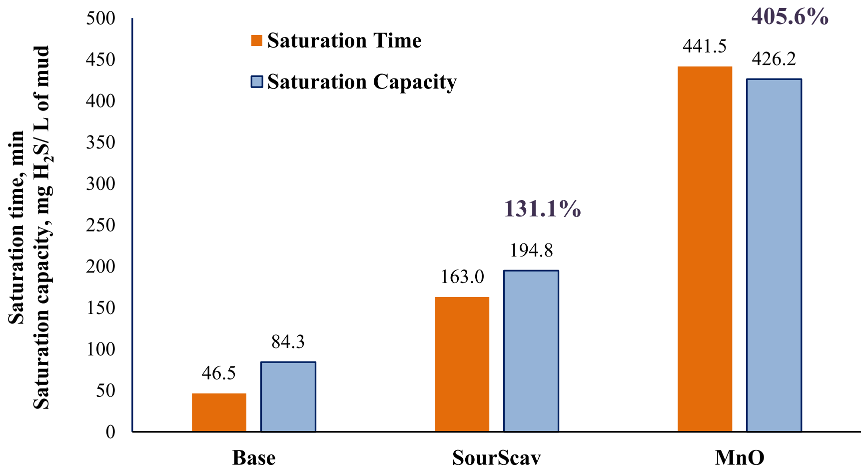

Figure 2. Saturation time and H_2S scavenging capacities for the prepared fluid samples.

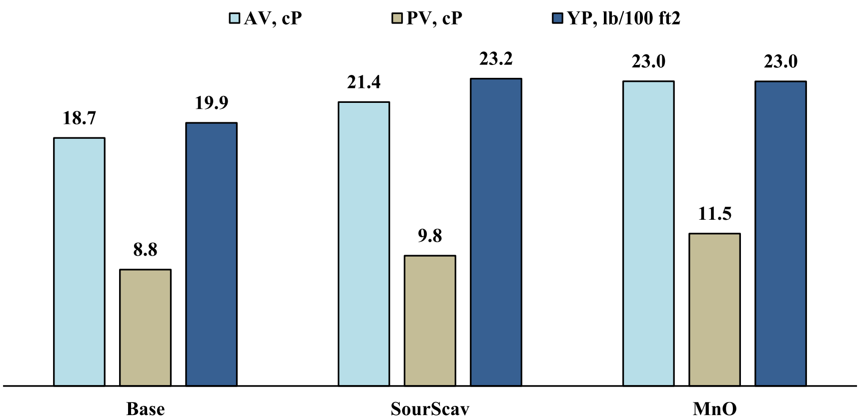

Figure 3. Rheological features of the addressed muds.

dividing the lost weight by the surface area of the metal coupon.

4. RESULTS AND DISCUSSION

4.1. H_2S Scavenging Test. The capacity of the formulated muds to remove H_2S was assessed by introducing H_2S gas to the prepared mud samples in a bubble column and using eq 1 to determine the scavenging capacity at the point of saturation.

As shown in Figure 2, H_2S saturation was achieved after 46.5 min in the case of the base drilling fluid, while the inclusion of SourScav delayed the saturation until 163.0 min. The drilling fluid containing MnO continued to scavenge H_2S for 441.5 min. The outcomes revealed that the H_2S scavenging capacity of the base mud was 84 mg of H_2S/L of mud, while the addition of SourScav improved the scavenging capacity at saturation by 131.1%, resulting in a value of 194.8 mg/L. However, the inclusion of MnO significantly enhanced the scavenging capacity to 426.2 mg/L, representing a 405.6% improvement compared to the base drilling mud and approximately 2.1 times that of the SourScav-based scavenger.

MnO reacts with H_2S according to the following stoichiometric equation

$$MnO + H_2S \rightarrow MnS + H_2O \quad (5)$$

The reaction shows that one mol of manganese oxide reacts with 1 mol of H_2S to yield 1 mol of manganese sulfide and 1 mol of water. Given that MnO, H_2S, MnS, and H_2O have molecular weights of 70.9, 34.1, 87.0, and 18.0 g/mol, respectively, each gram of MnO should consume about 480.9 mg of H_2S when it is completely utilized. Comparing this theoretical H_2S consumption value with the experimentally determined scavenging capacity at the saturation point (426.2 mg of H_2S) demonstrates the ability to scavenge H_2S with around 89% of MnO utilization in this process.

Although manganese oxides have not been used so far to scavenge H_2S during drilling operations, to the best of the author's knowledge, they have been utilized for sour gas sweetening (under dry conditions) at surface facilities. For instance, Li et al.[47] stated that manganese oxides supported on alumina can remove about 120 mg/g of H_2S when the reaction temperature is above 930 °F. Huang et al.[48] also demonstrated that manganese oxides, which were produced by calcining manganese nitrate and supported on MCM-48, can be used to scavenge H_2S at 1022 °F from a hot coal gas with 66.1% utilization. Additionally, according to Wang et al.,[35] compounds of manganese and aluminum oxides can remove H_2S with up to 96% utilization of manganese.

4.2. pH Measurement. The alkalinity measurements for the base drilling fluid and the muds containing SourScav and MnO exhibited pH values of 11.0, 7.9, and 11.3, respectively. Using SourScav caused a significant pH drop due to its low pH and the presence of ferrous sulfide byproducts.[49] The addition of MnO to the base drilling fluid resulted in a marginal increase in pH. In general, the pH range for aqueous muds is typically between 9.0 and 11.0, while in the presence of H_2S, it is recommended to maintain a higher pH level in order to lessen

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its acidifying effect.[13] Also, the regulations for drilling in sour environments mandate a minimum pH level of 10.[50] Based on these requirements, the pH achieved by the MnO-containing fluid is in compliance with these regulations, while using SourScav raises a concern and necessitates more consideration for alkalinity control.

4.3. Rheological Characteristics. For the hydraulic design of drilling fluids, the rheological properties are key features that must be carefully controlled. Pumping pressure, cuttings removal, cuttings holding in static conditions, rate of penetration, and hole cleaning are all affected by mud rheological parameters, such as the AV, PV, YP, and gel strength.

The rheology experiments revealed that the base mud had AV, PV, and YP values of 18.7 cP, 8.8 cP, and 19.9 lb/100 ft^2, respectively. Figure 3 illustrates that muds containing SourScav and MnO had greater AV values of 21.4 and 23.0 cP, respectively. The PV increased to 9.8 cP for SourScav and 11.5 cP for MnO. The YP values were boosted to 23.2 lb/100 ft^2 with SourScav and 23.0 lb/100 ft^2 with MnO.

To ensure efficient drilling operations, it is important for drilling muds to have a low PV and high YP. A low PV (recommended to be below 25 cP) allows for improved fluid flowability with reduced pressure loss, thus minimizing the pumping pressure.[51,52] A high YP value, not exceeding 50 lb/100 ft^2, indicates a greater carrying capability during circulation.[53] Also, to achieve better fluid loss behavior, it is recommended that the AV exceed 15 cP and preferably fall within the range of 20 to 35 cP.[51,54]

Another crucial metric for assessing the effectiveness of carrying capacity and hole cleaning is the YP to PV ratio (YP/PV ratio),[55] with recommended values greater than 0.75.[19] The results demonstrated that all addressed muds have YP/PV ratios greater than 2.

It is possible to infer from the observed rheological data that both MnO and SourScav-containing muds match the requirements within the given ranges and demonstrate suitable AV, PV, YP, and carrying capabilities.

The gel strength of mud is a measure of its ability to suspend cuttings and prevent them from settling when drilling operations are halted. The 10 s-/10 min-gel values for the base, SourScav, and MnO-containing fluids, respectively, were 5.8/6.9, 6.1/6.5, and 6.7/8.0 lb/100 ft^2, indicating that the addition of SourScav and MnO slightly increased both the initial and final gel strengths, as presented in Table 2, with a better gel structure for MnO-contained mud.

Table 2. Gel Strength Values for the Prepared Drilling Fluids

mud	10 s-gel, lb/100 ft^2	10 min-gel, lb/100 ft^2
base mud	5.8	6.9
SourScav	6.1	6.5
MnO	6.7	8

If the mud has a weakened gel structure or insufficient gel strength, the cuttings will settle and accumulate at the bottom, which could present several operational challenges. However, once the circulation is broken, exceedingly high values also result in fluid loss, ineffective solid control, and increased torque and pressure.[19] The recommended 10 s-/10 min-gel values are not to exceed 15/35 lb/100 ft^2.[53]

4.4. Filtration Performance. Another crucial factor that must be controlled in order to create an efficient aqueous drilling fluid is filtration performance. In general, the filtration process results in water loss from drilling fluids into the formation, and elevated levels can cause formation damage, clay swelling, and instability in formations that have high clay contents due to water−clay interactions. Also, the fluid loss may dramatically increase under HTHP conditions,[56] making it inappropriate for use in drilling operations. Therefore, it is important to carry out the HTHP filtration test in order to ensure that the filtration parameters, such as the filtrate volume and filter-cake thickness, are maintained within acceptable limits.

As presented in Figure 4, the filter-cake thickness and fluid loss after 30 min for the base mud were 2.9 mm and 4.7 cm^3, respectively. The inclusion of SourScav and MnO increased the filtrate from 21 and 6% to 5.7 and 5.0 cm^3, respectively, and the filter-cake thickness was increased to 3.9 mm, which was expected due to the increment in the solid content of the mud. Nevertheless, the filtration properties of the addressed muds stayed within their practical limits.

4.5. Corrosion Investigation. According to the corrosion investigation, the base fluid corrodes at a rate of 1.1×10^{-4} lb/ft^2, whereas the inclusion of either SourScav or MnO results in almost no corrosion. The nearly zero corrosion rate of SourScav and MnO proved how noncorrosive these substances are.

Based on the findings of this work, MnO can be appended to aqueous muds for successfully removing H_2S during the drilling of sour formations. This would result in safe and effective drilling operations. Additionally, MnO produces acceptable drilling fluid rheology, filtration performance, and corrosion affinity. MnO also results in a safe pH value, confirming its applicability for drilling sour hydrocarbon formations. However, before commencing field trials, more study is required to evaluate the scavenger efficacy under HTHP conditions as well as to account for any change in mud composition.

5. CONCLUSIONS

In this study, MnO was used as a H_2S scavenger to improve the scavenging efficiency of aqueous drilling mud. The scavenging performance of MnO and its impact on mud characteristics were investigated and compared to a commercial scavenger (i.e., SourScav), giving the following key conclusions.

- SourScav enhanced the ability of the aqueous mud to scavenge H_2S by 131.1%, whereas utilizing MnO exceeded this commercial scavenger by increasing scavenging capacity by 405.6%.
- Unlike SourScav, which caused the pH of the mud to drop to 7.9, the addition of MnO held the mud's pH at 11.3, maintaining the 10-pH threshold for drilling in sour formations.
- Both SourScav and MnO-containing drilling fluids have the AV, PV, YP, and gel strength within the practical recommended limits, satisfying the rheological performance criteria.
- Both SourScav and MnO had a noncorrosive nature, as demonstrated by the near-zero corrosion rates attained with these substances.

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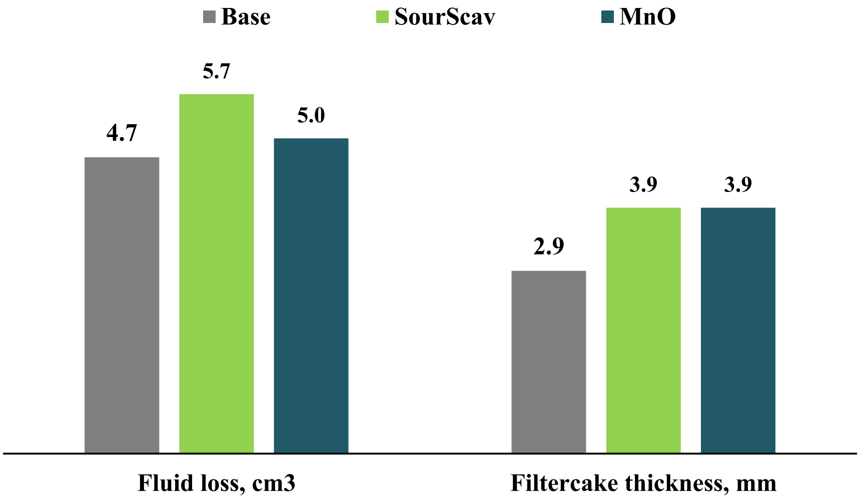

Figure 4. Results of the filtration test including the fluid loss and filter-cake thickness after 30 min.

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Author Contributions

Conceptualization, S.E. and S.O.; methodology, A.A.; formal analysis, A.A.; investigation, A.A.; resources, S.E. and S.O.; data curation, A.A.; writing—original draft preparation, A.A.; writing—review and editing, S.O.; visualization, A.A.; supervision, S.E.; all authors have read and agreed to the published version of the manuscript.

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Notes

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