



## Improved Water-Based Mud Rheological Properties and Shale-Inhibition Behavior by Using Aluminum Oxide and Iron Oxide Nanoparticles

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### HIGHLIGHTS

- Nanoparticles reduced filter loss up 41 and 32% by Fe<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub>, respectively.
- Reduction (COF) between pipe and wellbore up 48 and 34% by Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>, respectively.
- Wellbore swelling reduction up 50 and 30% by Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>, respectively.

### ARTICLE INFO

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### ABSTRACT

Nanotechnology can be used to develop drilling fluid additives that can improve the drilling fluid's properties. Using two types of nanoparticle (NP) additives in water-based drilling fluids have been studied in this paper. Three major drilling mud systems, namely potassium chloride (KCl) as a basic mud, KCl/aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) NPs, and KCl/iron (Fe<sub>2</sub>O<sub>3</sub>) NPs, were prepared and studied for enhancement of rheological properties and shale inhibition. It was found that the drilling mud contained NPs in concentrations of 0.25, 0.5, 0.75, and 1 g. Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> NPs added to KCl/polymer mud systems resulted in a 50% and 30% change in shale volume, respectively. The results demonstrated that incorporating NPs into the KCL mud system enhanced shale inhibition. Adding NPs to the KCL-WBM increased yield point, plastic viscosity, and gel strength. The COF of KCL-polymer was reduced by 48% and 34% when added Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> NPs at 0.5 and 0.75g, respectively. When Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> NPs were used, particularly at 1g, the amount of mud filtration decreased from 13.1ml to 8.8 ml and 8.4 ml, respectively. Overall, it was found that adding Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> NPs to the KCL-WBM can improve rheological, swelling, and filtration properties as well as lubrication.

## 1. Introduction

As the world's energy consumption and demand have grown, so has the production of natural gas and oil (the world's primary energy resources). Oil production is undergoing a rapid change due to the development of new technologies, such as a horizontal hydraulic fracture. In drilling oil and gas reservoirs, drilling fluids play a vital role. They are challenging topics because of technical, economic, and environmental concerns [1]. Water-based drilling fluids are the most commonly used in the drilling industry, as opposed to the more expensive oil- or synthetic-based drilling fluids that cause disposal, safety, health, and environmental problems [2]. Water, active and inactive solids, and chemical additives comprise most water-based drilling fluids [3]. In addition to transporting rock shavings removed by the drill bit to the surface [4], Hydrostatic pressure must be maintained when drilling mud pressure exceeds formation pressure [5], and Cool and lubricate drilling equipment, as well as aid in rock breaking [6].

Nanoparticles of various shapes and sizes have been tested in the production of Nano-drilling mud (being in some cases described as Nano-reinforced Drilling fluid). Drilling fluids incorporating nanoparticles are stable, filterable, and rheologically stable [7,8]. It's been shown in recent studies that Nano-fluids have interesting properties for applications such as heat transfer, gel formation, drag reduction, sand consolidation binding capability, wettability alteration, and corrosion control [9]. The thermal and electrical properties of nanoparticles are superior to those of macro and micro-sized. These nanoparticles are distinct from the bulk material in their physical and chemical properties. Drilling fluids can overcome the drilling process's

difficulties because of these properties. By their small size, nanoparticles bridge gaps in the formation's porous structure and effectively seal and plug them. They prevent fluid leakage, particularly in shale formations [10].

Some researchers, however, have shown that certain nanoparticles can improve the performance of polymer-based drilling fluids by improving their rheological, filtration, and lubrication characteristics [11]. The ability to displace oil and the flow behavior in porous media are both affected by the surfactant-polymer interactions in solution [12]. The chemical degradation effect of saline formation water has a significant influence on the performance of polymer flooding. Systematically, employing steady flow, the rheological properties of a hydrolyzed polyacrylamide mixed system were examined in relation to the effects of the inorganic salts NaCl, KCl, and CaCl<sub>2</sub>. The rheological properties of polymeric solutions are crucial for the success of enhanced oil recovery (EOR) techniques [13]. When temperatures exceed 200 °C, polymers used as viscosities and filtrate reducers to control drilling fluids' rheological properties and fluid loss is a significant challenge. In addition, under conditions of extremely high temperature, gelation of drilling fluids with low solids content and high fluid loss can be difficult to control [14]. As the base fluid for nanoparticles, the polymeric component of the drilling fluid can be used to ensure better distribution throughout the fluid composition. As a constraint on its operation, this eliminates the base effects of the nanofluid in the drilling mud [15]. Using KCl-polymer, drilling fluid loss is reduced, and shale permeation and hydration are inhibited. Since K<sup>+</sup> is a strong salt and calcium resister, it can significantly reduce solid phase particle sizes, aid in fluid entry into cavities, and increase plugging strength. A fluid loss agent for high-temperature and salt-resistant fluid loss in drilling fluid at high temperature and pressure has been developed [16]. Six different concentrations of iron (III) oxide or Hematite (Fe<sub>2</sub>O<sub>3</sub>) NPs are added to a basic KCl-PPHA polymer-based mud. Basic mud loses 5.9 ml of fluid after 30 minutes, whereas Nano-based mud loses less fluid across the board. Nanoparticles in the API LTLF filter press test result in fluid loss of 5.1 ml at a concentration of 0.5%. Plastic viscosity, yield point, and 10 s gel strength all increase by 15, 3, and 12.5 %, respectively, when nanoparticles are at 3.0 wt. % are added [17]. The oil and gas drilling industry is currently faced with the technical challenge of improving the rheological characteristics and shale inhibition of drilling muds under high temperature conditions. Shale stabilizers or conventional inhibitors found in conventional WBM are heat insulators, macro in size, and unable to block shale nanopores. Water then enters the wellbore, causing a high volume of mud filtrate and clay swelling. To seal the shale's nanopores, nanoparticles can be a great solution for the improvement of rheological properties [13], plugging characteristics [15], and shale inhibition [16], various researchers reported the use of nanoparticle-based drilling muds.

In this paper, experimental work has been done to use Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> NPs in WBM to reduce shale swelling and enhance WBM's rheological performance. Drilling muds based on nanoparticles perform more efficiently in rheology and shale inhibition than conventional KCl based mud systems.

## 2. Experimental Work

### 2.1 Characterization of the Materials

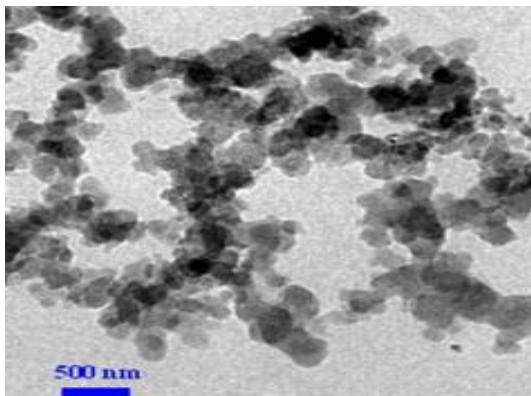
Due to their heat transfer characteristics and prices, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> are two of the most popular NPs. This led to the selection of Fe<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> NPs for this study. The suppliers of Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> were Nanjing Nano Technology and Sky Spring NPs, respectively. Tables 1 and 2 list the characteristics of nanoparticles. TEM and SEM images of Figures 1 and 2, respectively show the morphology of Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> NPs.

**Table 1:** Physical properties of Al<sub>2</sub>O<sub>3</sub> NPs.

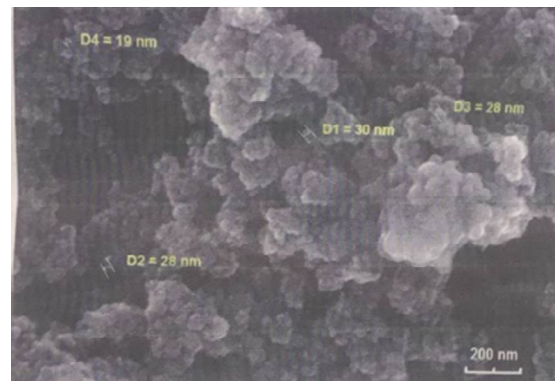
Properties	Typical value
Purity	99.9%
Appearance	White powder
Size	20 nm
Ash	<0.2 wt.%

**Table 2:** Physical properties of Fe<sub>2</sub>O<sub>3</sub> NPs.

Properties	Typical value
Purity	99.9%
Appearance	black powder
Size	20-30 nm
Ash	<0.2 wt.%



**Figure 1:** TEM images of Al<sub>2</sub>O<sub>3</sub>NPs.



**Figure 2:** SEM images of Fe<sub>2</sub>O<sub>3</sub>NPs.

## 2.2 Methodology

The drilling fluid used in this study is KCl- WBM, commonly used in southern Iraq oil fields. This mud can be prepared quickly and easily. First, bentonite fluid should be prehydrated by mixing 20 g sodium bentonite with 350 ml of fresh water for at least 20 minutes. It is done using a Hamilton Beach Mixer. Then, the mix is left to sit for 16 hours, according to API standards. After that, add 0.75 g of caustic potash to Alkalinity Control, Source of K<sup>+</sup>ion by addition 5g of KCl-polymer has a strong ability to inhibit shale permeation and hydration and reduce the loss of drilling fluid and all additions in Tables 3.A Hamilton Beach Mixer is then used to mix the mixture for 20 minutes under laboratory conditions, where the concentrations of Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> NPs range from 0.25 to 1g. After that, the fluid is placed in an ultrasonic bath for 15 minutes to ensure that the Nanoparticles disperse evenly throughout the fluid.

## 2.3 Rheological Testing

Plastic viscosity, gel strength, filter cake thickness, and filtrate loss were some of the rheological properties studied for the prepared muds. First, mud viscosity and gel strength (10sec and 10min) were measured using a Van-G meter. The API standard was used for the parameters of plastic viscosity (PV), yield point (YP), and Gel strength (GS). The Van-G meter was used to determine the PV and yield point values at both 300RPM and 600RPM motor speeds. Next, the filter cake was evaluated, and the filtration loss was calculated in a filter press. Filter press pressurized cells contain a pressurized filter medium. A nitrogen gas cylinder was connected to the filter press equipment to raise the cell pressure to 100 psi. It took about 30 minutes for each test and two of them. Afterward, the cell was disassembled, and the mud was thrown away. To avoid damaging the mud cake, take your time and be cautious when removing the components from the cake. The cake was gently scrubbed to remove any remaining mud before serving. The filter cake thickness was measured and recorded in 1/32-inch increments as a final step. To be clear, all tests were performed at 27 °C. Equations are used to calculate (PV) and (YP) [18]:

$$PV = \theta_{600} - \theta_{300} \tag{1}$$

$$YP = \theta_{300} - PV \tag{2}$$

**Table 3:** Principal Additives of KCl-Polymer muds [23].

Additive	Concentration, lb/bbl	Function
Prehydrated Bentonite	10 - 25	Viscosity Filtration Control.
Potassium Chloride	5 - 60	Inhibition Source of K <sup>+</sup> ion.
Caustic Potash	0.25 – 0.75	Alkalinity Control.
Starch	3 -6	Filtration Control.
Lignite	1 - 4	Filtration Control.
Lignosulfonate	3 - 6	Deflocculant.
Low Viscosity PAC	0.5 - 1.0	Filtration Control.

## 2.4 Lubricity

Extreme pressure/lubricity testers were used to measure COF for the lubricity test. Drill string and wellbore are analogous in that they are made of metal to metal. It was possible to calculate lubricity with this formula (3,4,5 and 6) [19]:

$$COF = \frac{\text{Torque reading}}{100} \tag{3}$$

$$100 = \frac{150 \text{ inch-lbs torque wrench reading}}{1.5 \text{ inch torque shaft lever arm}} \tag{4}$$

$$CF = \frac{\text{Meter reading for water (standerd)}}{\text{meter reading obtained in water calbration}} \tag{5}$$

$$CoF = \frac{(\text{Meter reading for water})(CF)}{100} \tag{6}$$

## 2.5 Shale Swelling Testing

Drilling fluids must be designed to provide maximum inhibition and well stability to control shale formation swelling. If the drilling fluid is unsuitable for the formation, a clay mineral-rich formation can quickly swell during drilling. Shale instability, washouts, stuck pipes, and tight holes can all be caused by clay swelling. The well's stability can be improved by using the proper drilling fluid before or during the drilling procedure. An expansion quantity meter was used to measure the length of an intact shale core over time at normal temperature and pressure to determine shale hydration or dehydration. The swelling was calculated as a percentage of the original length and measured in millimeters. The fluid cup's pressed shale powder expands and pushes against the pipe. The percentage gauge displays the value of expansion [11].

### 3. Results and Discussion

#### 3.1 Properties of Rheology

Due to the difficulty of pumping drilling fluid with a high PV, drillers avoid using it for drilling operations [18]. Due to a faster penetration rate, drilling fluids' PV should be lower, but this parameter is also affected by other rheological parameters. The PV of KCl-WBM was found to be only 7 cP. As shown in Figure 3, Adding NPs to KCl - WBM generally increased the PV. In addition, 0.25 to 1g of Al<sub>2</sub>O<sub>3</sub> NPs was added to the base mud. The PV remained constant at 7 cP. Al<sub>2</sub>O<sub>3</sub> NPs produced entirely different results than Fe<sub>2</sub>O<sub>3</sub>NP. We achieved 8 cP of PV by mixing in 0.5 g Fe<sub>2</sub>O<sub>3</sub> NPs with the base mud. When the concentration of Fe<sub>2</sub>O<sub>3</sub> NPs up 0.5 g, the PV constant is at 8 cP before increasing to 10 cP at 1 g.

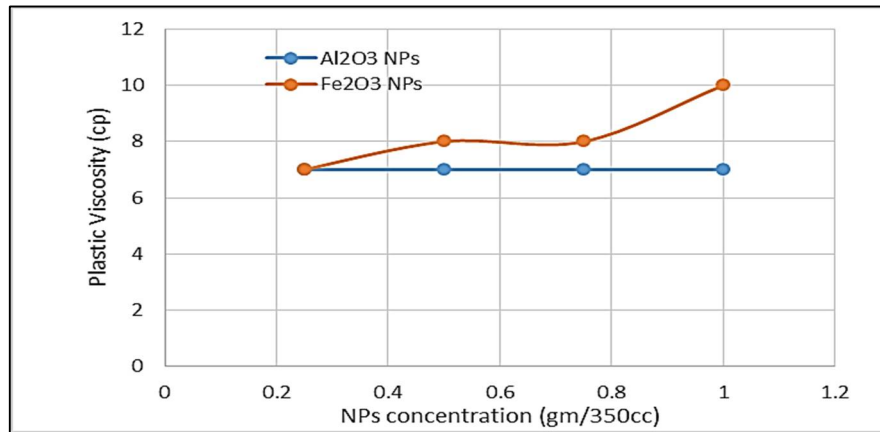


Figure 3: concentration affects by Plastic Viscosity (cp).

#### 3.1.1 Yield Point

Electrochemical forces within a fluid, including the yield point, generate fluid flow resistance. These electrochemical forces are generated by the electrical charges on the surfaces of reactive particles [6]. During testing, the (YP) of Bentonite-WBM was discovered to be 6 lb/100ft<sup>2</sup>. The (YP) of KCl-WBMs can be influenced by NPs, as shown in Figure 4. To a certain extent, NP concentration affects WBM yields. With increasing Al<sub>2</sub>O<sub>3</sub> NP concentrations, the (YP) of KCl-WBM increases. KCl-WBM by Al<sub>2</sub>O<sub>3</sub> NPs Produced 47 lb/100 ft<sup>2</sup> at 1 g. In order, the YP values of Fe<sub>2</sub>O<sub>3</sub> NPs at 1g were increased slightly to 30 lb/100ft<sup>2</sup>. When comparing Al<sub>2</sub>O<sub>3</sub> NPs to Fe<sub>2</sub>O<sub>3</sub>NPs the (YP) results at 1 g concentrations are better.

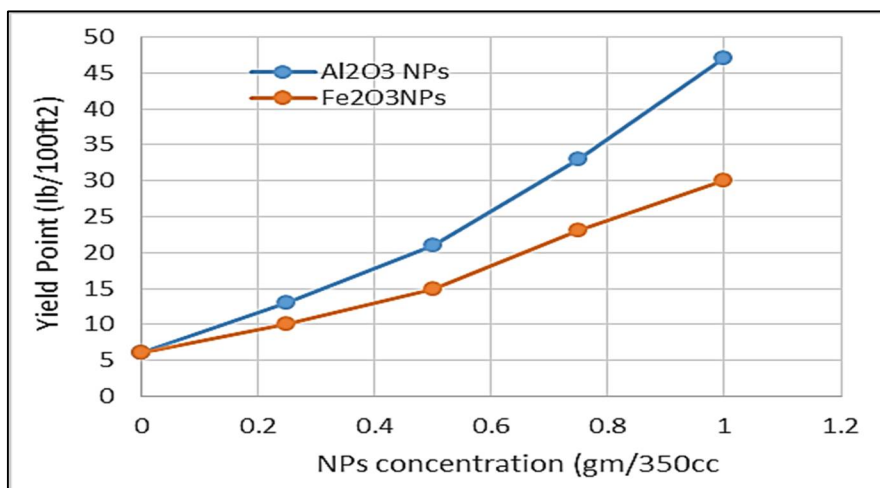


Figure 4: NPs concentration affects the yield point.

#### 3.1.2 Gel strength

Gel strength is the ability of drilling mud to enhance and maintain a gel structure when the drilling operation is paused. Appropriate gel strength is usually required to maintain the excessive circulation pressure needed to restart drilling activities. [20]. The effects of NPs on (SG) are showed in Figures 5 and 6. at 10 sec and 10 min, respectively. Base mud gel strength was tested for 10 sec and 10 min at 5 and 11 lb/100 ft<sup>2</sup>, respectively. In both short-term (10 sec) and long-term (10 min) tests, the gel strength of KCl-WBM increased when Al<sub>2</sub>O<sub>3</sub> NPs were added from 0.25 to 1 g. KCl-WBM 10 sec GS and 10 min GS were 42 and 45 lb/100 ft<sup>2</sup> at a concentration of 1 g Al<sub>2</sub>O<sub>3</sub> NPs, respectively. After 10 sec and 10 min in the presence of Fe<sub>2</sub>O<sub>3</sub> NPs, the GSs were 27 and 30 lb/100 ft<sup>2</sup>. A higher starting torque may be required to account for the fluid's high gelling characteristics. If you want to avoid a significant amount of difficult drilling situations, a strong gel will be necessary [21]. Finally, at 1 g concentration, Al<sub>2</sub>O<sub>3</sub> NPs gel is better than Fe<sub>2</sub>O<sub>3</sub> NPs.

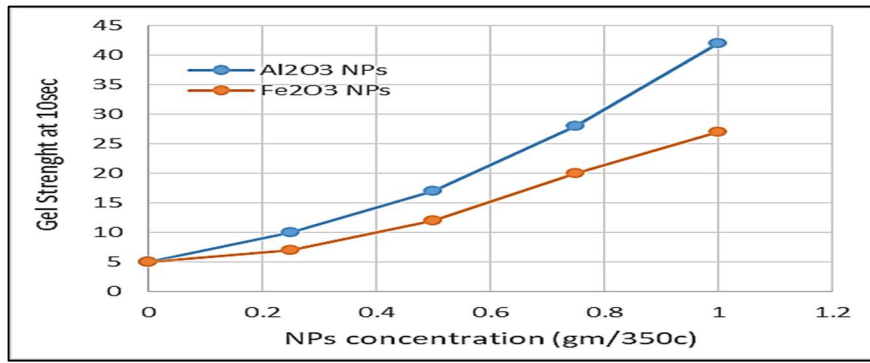


Figure 5: NPs concentration effects gel strength over a 10-sec.

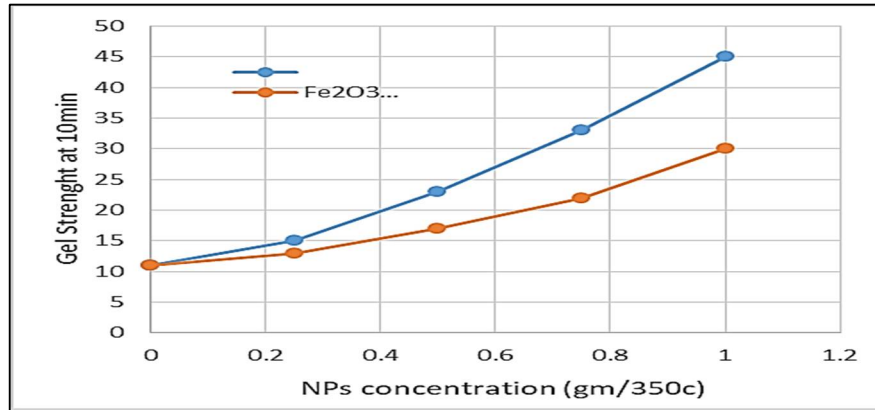


Figure 6: NPs concentration effects gel strength over a 10-min.

3.1.3 Fluid filtration and nanoparticle effects

In this test, the volume of fluid lost and the thickness of the mud cake can be measured. High filter loss is undesirable because it can result in formation instability and damage [22]. Figure 7, shows the fluid loss behavior of KCl-WBM at various NP concentrations. After 30 min, the KCl-WBM had lost 13.2 mL of fluid. The fluid loss volume after adding 1g of Al<sub>2</sub>O<sub>3</sub> NPs to the KCl-WBM was 9.6 mL. It appears that incorporating Al<sub>2</sub>O<sub>3</sub> NPs into KCl-WBM filters can reduce loss. The fluid loss was reduced to 8.8 mL when 1g of Fe<sub>2</sub>O<sub>3</sub> NPs were added to the KCl-WBM. Finally, Fe<sub>2</sub>O<sub>3</sub> NPs are more effective than Al<sub>2</sub>O<sub>3</sub> NPs at reducing fluid loss from KCl-WBM.

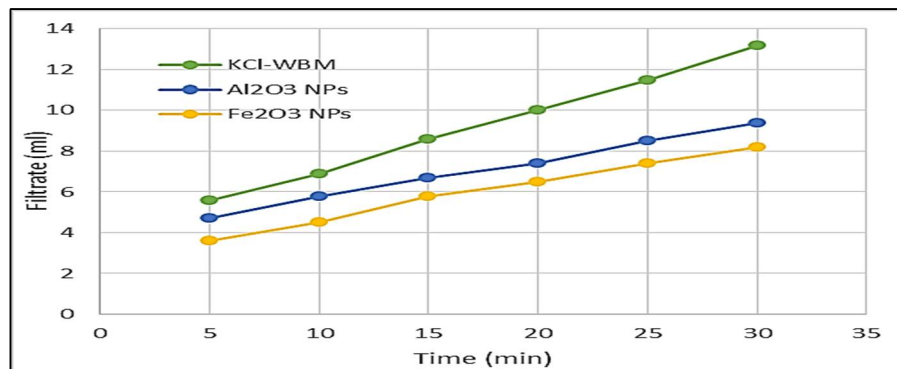


Figure 7: NPs concentration affects the filtrate (ml).

3.1.4 Lubricity and nanoparticle concentration

Drilling fluid acts as a lubricant for the drill string while drilling the wellbore. An object's resistance to movement in response to an external force is known as its coefficient of friction (COF) [19]. Figure 8, A small amount of NPs in the drilling fluid reduced COF slightly, according to this study. Consequently, the lubricity of the system was decreased by Using KCl-WBM with two different primary additives, such as Al<sub>2</sub>O<sub>3</sub> NPs and Fe<sub>2</sub>O<sub>3</sub> NPs, under ambient conditions. In general, Absolute and relative CoF reduction at different concentrations of Al<sub>2</sub>O<sub>3</sub> NPs and Fe<sub>2</sub>O<sub>3</sub> NPs are provided in Table 4. Al<sub>2</sub>O<sub>3</sub> NPs have a larger surface area than Fe<sub>2</sub>O<sub>3</sub> NPs. At 0.5g each, Al<sub>2</sub>O<sub>3</sub> NPs and Fe<sub>2</sub>O<sub>3</sub> NPs reduced torque by 28% and 30%, respectively, and the torque was increased sharply at 0.75 to 1g of NPs in KCl-WBM. KCl-WBM has higher COF values than KCl-WBM with Fe<sub>2</sub>O<sub>3</sub> NPs and Al<sub>2</sub>O<sub>3</sub> NPs because it crushes more easily while rotating.

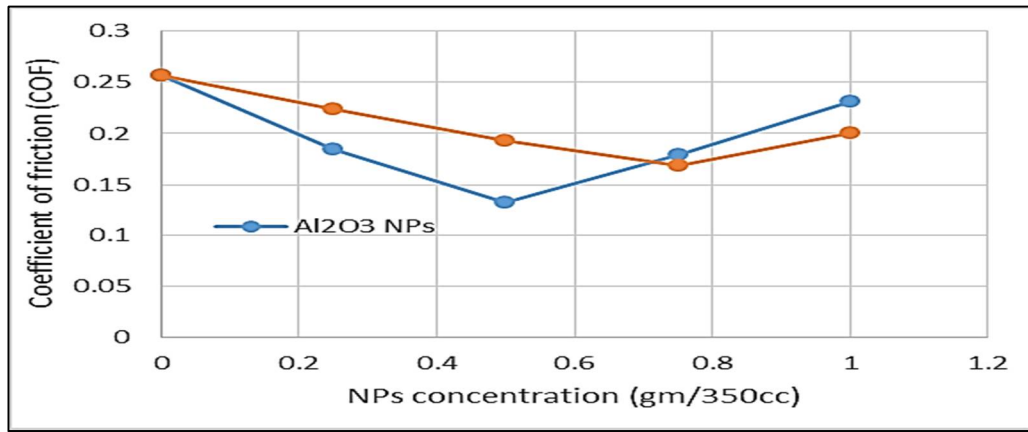


Figure 8: Friction coefficient(COF) depends on the concentration of NPs in the fluid.

Table 4: Absolute and relative CoF reductions for NPs based drilling

Concentration of NPs, (g)	<u>Al<sub>2</sub>O<sub>3</sub> NPs</u> Absolute COF	Absolute COF	<u>Fe<sub>2</sub>O<sub>3</sub> NPs</u> Absolute COF	Absolute COF
0	0.257	0	0.257	0
0.25	0.185	28	0.224	12
0.5	0.132	48	0.193	25
0.75	0.179	30	0.169	34
1	0.231	10	0.200	22

### 3.2 Swelling Behavior and Nanoparticle Effects

Figure 9, shows the expansion quantity meter results for the sodium bentonite shale using four different drilling fluids, including fresh water, Al<sub>2</sub>O<sub>3</sub> NPs, Fe<sub>2</sub>O<sub>3</sub> NPs, and KCl-WBM. The bentonite was expanded up to 12% using KCl – WBM, while Al<sub>2</sub>O<sub>3</sub> NPs expanded by less than 5% after 18 hours of exposure to these systems. Nevertheless, after 18 hours of exposure to fresh water, the bentonite had expanded by 13%. It was found that the water-based drilling fluid shale swelling was reduced to less than 5% in the presence of Al<sub>2</sub>O<sub>3</sub>NPs. However, adding Fe<sub>2</sub>O<sub>3</sub> NPs reduces swelling to less than 7% due to NPs ability to plug nanopores in clay, preventing shale swelling. Due to a stability issue, NPs do not reduce swelling at a high enough percentage, but this can be mitigated by adding KCl-WBM with the NPs. Because of the synergetic properties of NPs, to expand, it means that the bentonite in the NPs system absorbed less water, resulting in less clay swelling and increased shale strength [11].

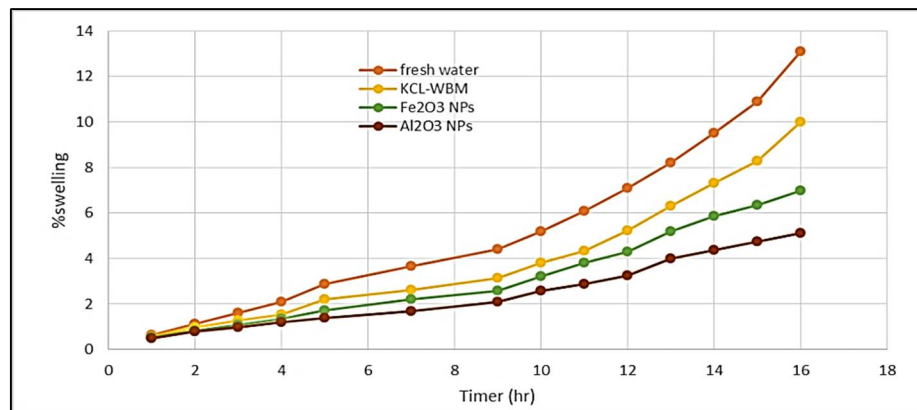


Figure 9: Exposed to Fresh Water and Drilling Fluids, Sodium Bentonite's Swelling Percentage

### 4. Conclusion

The rheology of a KCl-WBM was studied in relation to Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>NPs concentrations. There is a reasonable expectation that the low viscosity of the NPs used here will not compromise the density requirements of drilling mud, making them an excellent choice for this application. Adding Fe<sub>2</sub>O<sub>3</sub> NPs to the KCl-WBM increased yield point, plastic viscosity, and gel strength. Al<sub>2</sub>O<sub>3</sub> NPs improved the KCl-WBM mixture's rheology and the final mud product's filter loss and filter cake characteristics. Filtration losses were reduced by using both nanofluids, especially at 1g. Al<sub>2</sub>O<sub>3</sub> NPs and Fe<sub>2</sub>O<sub>3</sub> NPs were used to reduce the coefficient of friction (COF). Fluids prepared with nanoparticles were found to have better swelling inhibition properties than those without them. Overall, the results show that adding Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>NPs to KCl-WBM can improve the drilling fluid's performance.

### Author contribution

All authors contributed equally to this work.

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### Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

### Conflicts of interest

The authors declare that there is no conflict of interest.

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