

## Effect of Iron (III) Oxide Nanoparticles without Sonication on Properties of High Viscous Bentonite Water Base Mud

Nayem Ahmed<sup>1</sup>, Md. Saiful Alam<sup>1,\*</sup>, M. A. Salam<sup>2</sup>

<sup>1</sup> Department of Petroleum and Mining Engineering, Shahjalal University of Science & Technology, Sylhet-3114, BANGLADESH

<sup>2</sup> Bangladesh Petroleum Exploration and Production Co. Ltd. (BAPEX), Dhaka-1215, BANGLADESH

### ABSTRACT

Fluid invasion into the near-wellbore formation for bentonite treated spud mud is a common obstacle during the drilling operation, which increases non-productive time (NPT) and cost. To address this impediment, researchers recently introduced nanoparticles (NPs) base fluid, since these particles prove their advantage in different stages of petroleum exploration and recovery. Most of the researchers claimed that nanoparticles require a sonication before the inclusion of it into the mud system. However, the sonication of NPs at the field level is not so easy to implement. In this article, in-house prepared iron (III) oxide (Fe<sub>2</sub>O<sub>3</sub>) NPs without sonication is used in high viscous bentonite (HVB) water-base mud (WBM) to assess the effect of nanoparticles on the characteristics of drilling fluid. The experimental analysis shows that NPs reduces the fluid loss compared to bentonite base mud at all concentrations. At NPs concentration of 0.5 weight (wt)% a 10.72% reduction in the API low pressure low temperature (LPLT) filter volume is obtained. While at 3.0 wt% the plastic viscosity (PV), 10s, and 10min gel strength are enhanced by 43.75%, 37.50%, and 33.33%, respectively. The yield point (YP) is also enhanced by 20% at an optimal concentration of 0.5 wt%, that reflects a better cutting transport capacity of drilling mud.

Keywords: Iron (III) oxide; Nanoparticles; Fluid Loss; Water Base Mud.

### 1. Introduction

Drilling fluid is the major concern for any drilling operation, especially in the oil and gas industry. Drilling fluid characteristics are one of the main factors that determine the success and safety of any drilling operation. Drilling fluid serves very crucial functions such as balance formation pressure, drill bit lubrication and cooling, cuttings removal, prevent formation damage, maintain wellbore stability, suspend cuttings and weighting materials, and cake off the permeable formation [1-4]. A large number of drilling accidents and expense evolves during operation are directly or indirectly the results of malfunctioning of drilling mud [5, 6]. Researchers have been investigating several materials to solve these difficulties.

Over the past decade, nanotechnology has contributed to different industries, such as medicine [7], chemistry [8], environment [9], material and surface treatment [10], heavy industry, and food processing [11]. In recent years, nanotechnology has offered promising opportunities for the development of petroleum industry-related issues, such as enhanced oil recovery processes, oil and gas exploration, well stimulation, drilling engineering, cementing operation, sensors development, and production operation [12-18]. The application of nanotechnology, more specifically nanoparticles, has gained considerable interest; to enhance the properties of drilling mud by effectual regulation of fluid loss and shale swelling, accelerating the creation of low-permeable, thin, and smooth mud cake. The primary challenge for any nanoparticle application in drilling fluid is the suspension stability. To ensure the stability of nanofluid in suspension, researchers previously utilized ultrasonication [19, 20].

Ultrasonication is the process by which agglomeration of the particles can be disrupted using sound energy frequency (frequency in inaudible range to human).

Researchers have introduced the ultrasonicated NPs solution to modify the rheology and filtration properties of the drilling fluid. Jung, et al. [21] examined the sonicated iron oxide nanofluid influence on drilling fluid and found a considerable improvement in viscosity and yield stress. They also demonstrated a 6.14% fluid loss reduction by mixing 30 nm-sized NPs at 0.5 wt% concentration. Mahmoud, et al. [22] inquired <50 nm sized sonicated NPs by mixing with bentonite WBM at several concentrations (0.3, 0.5, 1.5, and 2.5 wt%). They observed that dosing NPs in base mud provides a substantial improvement in apparent viscosity profile. Their experiment revealed an increment in plastic viscosity up to 46.5% at 2.5 wt%, while at a concentration of 0.5 wt%, plastic viscosity decreased up to 26.85% with increasing temperature. For 0.5 wt% NPs, they demonstrated an increment of yield stress up to 78.19% relative to the base drilling fluid at 140°F. For concentrations ranging from 0.2 to 1.0 wt% the increment in 10 s and 10 min, gel strength was recorded as 50 to 130% and 50 to 58%, sequentially. At an optimum concentration of 0.5 wt%, the fluid loss reduction was 42.50% compared to base mud under high temperature high pressure (HTHP) conditions. But ultrasonication of nanoparticles for the inclusion in drilling fluid is quite hazardous in field scale. The inconsistent application of sonicator can create aerosols and foaming issues, as well as cavitation erosion [23]. Moreover, any kind of insincerity of workers could lead to tremendous health hazards like hearing impairment. In this article, nanoparticles are introduced in high

\* Corresponding author. Tel.: +88-01711954946

E-mail addresses: saifulraju@yahoo.com

viscosity bentonite water-based mud in the absence of nanofluid sonication. The paper presents the iron (III) oxide NPs' impact on high viscous bentonite mud to enhance the fluid loss control and rheology, apart from the sonication process.

## 2. Experimental Procedures

Standardized guidelines API RP 13I [24] and API RP 13B-1 [25] is followed to evaluate and characterize the performance of nano-based drilling fluids. Experimental protocols are ensured to conduct this laboratory-scale investigation.

### 2.1 Collection of Materials

All the purchased reagents including Ferric chloride hexahydrate ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ , 97% extra pure), ammonia hydroxide ( $\text{NH}_4\text{OH}$ , 25%), ethyl alcohol ( $\text{C}_2\text{H}_6\text{O}$ , 99.8%) are analytical grade. High viscosity bentonite mud ingredients are collected from Bangladesh Petroleum Exploration and Production Company Ltd. (BAPEX). Laboratory water distillation apparatus driven deionized (DI) water is used without further purification to prepare the solutions.

### 2.2 Synthesis of iron (III) oxide nanoparticles

Having advantages like fast production rate with relatively low cost and low reaction temperature, high uniformity, high purity, and high crystallinity; iron (III) oxide nanoparticles are synthesized following chemical precipitation route [26]. Synthesis procedure starts with the preparation of 0.2M solution of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  precursor after that homogeneity of the solution is confirmed with a magnetic stirrer by stirring 30 min at  $80^\circ\text{C}$ . Then without the interruption of stirring, a solution of 3M  $\text{NH}_4\text{OH}$  as a precipitating agent is dropwisely added at ambient conditions. Upon continuous stirring for 3.0 hours, the obtained material is permitted to precipitate entirely. Subsequently, draining the precipitate, the unreacted precipitating agent is eliminated repeatedly with DI water and ethyl alcohol. It is then dried for 1.5 hours in a hot-air oven at  $80^\circ\text{C}$ . This dry material then ground into a powder form that will be further calcined at  $400^\circ\text{C}$  in a muffle furnace for 4 hours. The final form of iron (III) oxide NPs is collected as a reddish-brown powder. The X-ray powder diffraction (XRD) technique is employed to characterize the synthesized NPs.

### 2.3 Preparation of drilling fluid

High viscous bentonite (2% bentonite) base mud sample is formulated with a proper quantity of DI water and mud additives. All drilling fluid additives are mixed, using a beach mixer having a rotating speed of 36,000 rpm. The sequential inclusion of additives into a solution is essential to ensure proper mixture. Additives are introduced gradually, and 10 minutes of agitation is ensured to avoid the creation of fish-eye, before the addition of the following products. Once all the additives were subsequently dosed, mixing proceeded for 20 minutes. Eight different concentrations (0.1, 0.3,

0.5, 0.8, 1.0, 1.5, 2.0, 3.0 wt%) of hematite NPs are mixed with these additives for the formulation of nano based high viscosity bentonite mud. Hematite NPs are properly mixed in deionized water using a beach mixture. Nanoparticles are blended with other mud additives in the subsequent sequence to prepare nano-based mud:

Water  $\rightarrow$  Caustic soda  $\rightarrow$  Iron (III) Oxide Nanoparticles  $\rightarrow$  Bentonite  $\rightarrow$  PAC-L  $\rightarrow$  Xanthan Gum  $\rightarrow$  Barite  $\rightarrow$  Bara-Defoam.

### 2.4 Nano-based drilling mud properties analysis

#### 2.4.1 Rheological properties measurement

The rheological characteristics of the drilling fluids under this research are determined using a rotational Fann viscometer at a standard test temperature of  $120^\circ\text{F} \pm 2^\circ\text{F}$  [25]. This viscometer has 6 specific rotational speed of RPM 3, 6, 100, 200, 300, and 600 which corresponds to Newtonian shear rate of 5.11, 10.21, 170.23, 340.46, 510.69, 1021.38  $\text{s}^{-1}$  [27]. To evaluate the shear rate versus shear stress relationship of drilling fluid; a rotational viscometer is used by which the Bingham Plastic parameters PV and YP are measured directly.

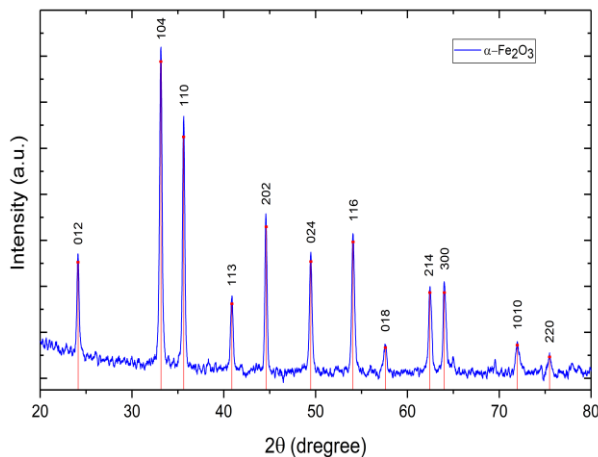
#### 2.4.2 Filtration properties measurement

In this investigation, API LTLP apparatus utilized to characterize the filtration properties of nano-based mud. A Fann (Model 300 LTLP) filter press mud cup poured with sample and a filter paper, placed on the mesh screen then a base cup is used to close the cell body. To apply the differential pressure, a  $\text{CO}_2$  charge as a pressure source used and, a graduated cylinder kept below this metallic filter press cell body. The amount of fluid that passes through the filter paper for 30 minutes is collected and measured using the graduated cylinder. The temperature selected to run this experiment is room temperature. The filter cake is shaped over the filter paper and the thickness is determined after conducting a 30-minute filter test.

## 3. Results and Discussions

### 3.1 Nanoparticles Characterization

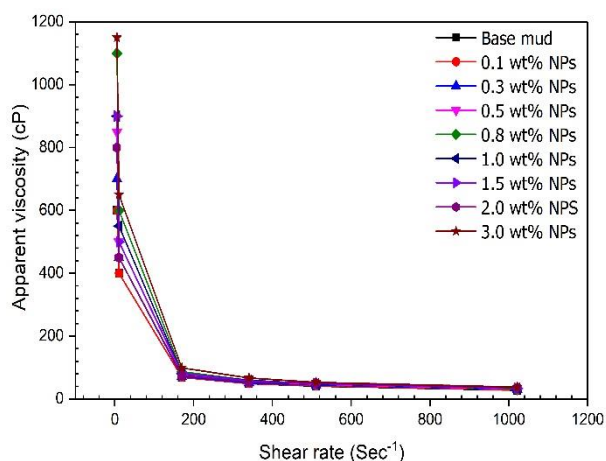
Figure 1 presents the XRD patterns for the synthesized reddish-brown powder of iron (III) oxide NPs. The XRD of synthesized NPs shows a distinct peak for 300, 214, 018, 116, 024, 202, 113, 110, 104, and 012 planes, which reflect the X-ray at an angle ( $2\theta$ ) of  $64.05^\circ$ ,  $62.48^\circ$ ,  $57.58^\circ$ ,  $54.08^\circ$ ,  $49.47^\circ$ ,  $44.59^\circ$ ,  $40.87^\circ$ ,  $35.63^\circ$ ,  $33.14^\circ$ , and  $24.12^\circ$ , respectively. In addition, the XRD analysis confers the crystallographic system of iron (III) oxide NPs as rhombohedral (hexagonal). These distinct reflection planes, peak positions, and crystal structures confirm the synthesis of pure iron (III) oxide [28, 29]. The analysis of XRD reveals that the synthesized NPs has an average crystal size of 40 nm.



**Fig. 1** Synthesized iron (III) oxide NPs XRD patterns.

### 3.2 Rheological Characteristics

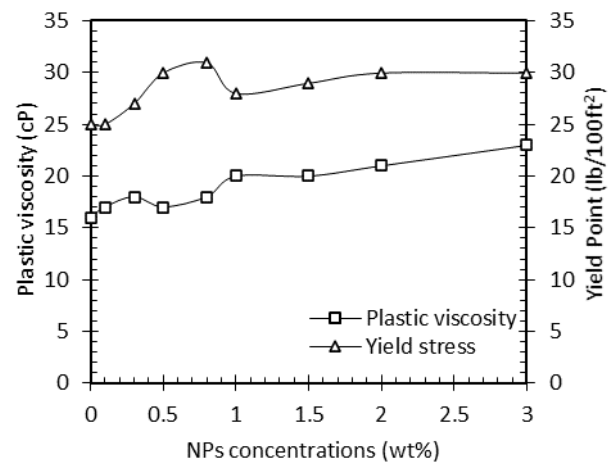
Viscosity is the dominant properties of fluid which characterize the flow behavior. Figure 2 shows the NPs influence on the apparent viscosity of HVB mud at a broad shear range. Analogous to typical drilling fluid, non-Newtonian shear thinning viscous profile is distinctive to the nano drilling fluid. All the muds have almost similar viscosities in the high shear range although NP-based muds are slightly thicker in the low shear range than base HVB muds. This high viscous profile is due to an electric charge imbalance between NPs (positively charged) and bentonite clay (negatively charged) in the aqueous state, which causes flocculation at a high concentration during low shear rate [30]. This viscosity profile of drilling fluid is benevolent since tripping and drilling operations demand high viscosity and low viscosity, respectively, to suspend and clean the cuttings from the bottom of the hole [31, 32]. Drilling fluid with lower-viscosity at a high shear rate and higher-viscosity at a lower shear rate exhibits good pumpability.



**Fig. 2** Apparent viscosity of high viscous bentonite mud at different NPs concentrations

Plastic viscosity (PV) is the measure of mechanical counteraction offered by solid and liquid inside the mud

suspension during the circulation of mud. PV of the mud system and how they vary with the concentration of nano-hematite presented in fig. 3. It is obvious from the figure that the increase in the concentration of NPs increases plastic viscosity. For NPs concentration of 0.5 wt% and 3.0 wt% PV enhanced by 6.25% and 43.75% respectively. PV increases because the solid percentage of the drilling fluids tends to rise as the number or quantity of nanoparticles rises, which forces the mechanical friction towards upward. In the context of pumping and drilling efficiency, PV must be maintained within the desired ranges otherwise, it may be deleterious to the rate of penetration [33].

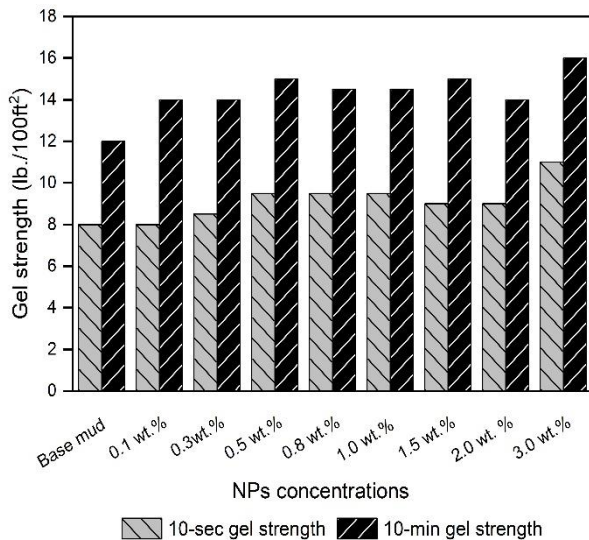


**Fig. 3** Plastic viscosity and yield stress of high viscous bentonite mud at different NPs concentrations.

Yield Points (YP) influences the cutting carrying capacity, which echoes the dynamic properties of drilling fluid. A higher YP is often demanded in large holes for effective hole cleaning. Figure 3 explains the behavior of yield points (YP) at different NPs concentrations in HVB-based mud. YP increases until concentration touches 0.8 wt%, after that with some decrement is observed, and then YP attains a stable value at high NPs concentrations. The addition of 0.5% of hematite NPs increases the YP by 20%, while 0.8% NPs shows an increase in YP by 24%. YP increases at higher NPs concentrations because positively surface charge hematite interacts with the clay platelets and with themselves, which triggers the flocculation, and flocculated slurry contributes to flow resistance [30].

Gel strength is the static (non-flow time) properties of drilling fluid, which measures the holding capacity of drill solids and mud weighting materials. The gel strength behavior of drilling mud with the inclusion of NPs is shown in Fig. 4. The gel strength behavior of nano-based mud is the representative of the progressive type of gel strength (a sharp increase in gel strength with time). A similar remark has been recorded by Amanullah, et al. [34]. From fig. 4, it indicates that 10-sec gel is increased by 18.75%, and 10-min gel strength is increased by 25% at a NPs concentration of 0.5 wt%.

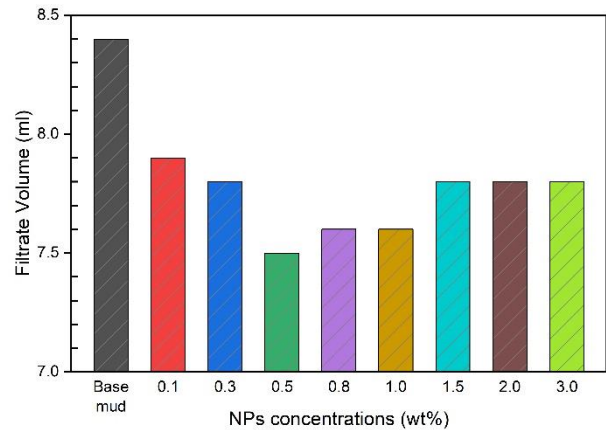
While significant improvement in gel strength is recorded at 3.0 wt%, which results in 37.5% and 33.33% increment in 10-sec and 10-min gel strength, respectively. This signifies the flocculating characteristics of nano-based mud at a concentration level of NPs. All the rheological properties evolution after introducing iron (III) oxide NPs without the sonication process reveals a significant improvement that was previously obtained using the sonication process [21, 30, 35, 36].



**Fig. 4** Effect NPs concentrations on 10s and 10min gel strength of high viscosity bentonite mud

### 3.3 Filtration Characteristics

The filtrate volumes obtained at different NPs concentration are shown in fig. 5. API LTLF loss of fluid for all NP concentrations decreases compared to base mud that reflects NPs tremendous capacity to regulate the loss of fluid. Initially, at low concentrations, there is a distinctive drop in filtrate volume relative to the base mud. API LTLF filter test confers 8.4 ml of filtrate volume in 30 min for base mud. On the other side, 0.5 wt% concentration of NPs generates 7.5 ml of filtrate, which reflects an improvement of 10.72% in regulation of fluid loss relative to base mud. From experimental measurement, at 0.5 wt% of NPs concentration, the total fluid loss is observed to be minimal (7.5 ml). While the cumulative fluid losses are found marginally higher (7.6~7.8 ml) for higher NPs concentrations, which validates the best iron (III) oxide nanoparticles efficiency at an optimal concentration of 0.5 wt%. It may be that at low concentrations, owing to a sufficient quantity of particles in dispersion, the nanoparticles may efficiently block the pores, whereas at higher concentrations they may begin agglomerating. These agglomerated nanoparticles generate a profoundly porous/permeable layer underneath the main filter cake, thus extending fluid loss. [22].



**Fig. 5** Comparison of total fluid loss at different NPs concentrations in high viscosity bentonite mud at 30 minutes.

### 4. Conclusions

In this article, the effects of non-sonicated  $Fe_2O_3$  NPs on the filtration and rheological properties of high viscosity bentonite mud; are experimentally analyzed. From the experimental results, the following conclusions are made:

- The best performing NPs concentration of Iron (III) oxide in the mud system is found to be 0.5 wt%.
- Nanoparticles blend with base mud contributes 6.25% and 20% increase in plastic viscosity and yield stress, respectively, at a NPs concentration of 0.5 wt%.
- At NPs concentration of 0.5 wt%, initial gel strength and final gel strength are found to be increased by 18.75% and 25%, respectively.
- The fluid loss is reduced by 10.72% when NPs inclusion is maintained at 0.5 wt%.
- Iron (III) oxide NPs without sonication can be used in a field to enhance the drilling fluid characteristics.

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