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Potential of co-precipitated synthesized iron oxide nanoparticles on the rheological properties and filtration loss control of water-based drilling fluids at high temperature and high pressure conditions

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ABSTRACT

The antagonistic conditions such as high temperature and pressures encountered as drilling operations cut deeper into formations require powerful drilling mud formulae that would provide the thermal stabilization of the drilling fluids while maintaining their rheological integrity. The present work investigates the synthesis of hematite (Fe_2O_3) nanoparticles through a simple chemical precipitation method to be used as failure resistance modifier for water-based drilling fluids (WBFs) at high temperature/high pressure (HTHP) conditions. The surface morphology revealed by transmission electron microscopy (TEM) strategy suggested that the employed iron oxide nanoparticles (IONPs) are mainly present as big spherical shaped particles and are well crystallize in nature with an average diameter less than 50 nm. Scanning electron microscopy (SEM) technique confirmed the shape and size of the produced nanoparticles. The chemical composition of the synthesized Fe_2O_3 nanoparticles was confirmed by energy dispersive analysis of X-rays (EDX). Three water-based drilling fluids (WBFs) samples; blank unmodified, surface modified 1 g iron oxide nanoparticles (IONPs-A) and modified 2 g (IONPs-B) were prepared and studied. The rheological properties and filtration ability were investigated for the utilized samples at both 500 psi and 350°F (HTHP). The results of this study demonstrated that the addition of 1 g of IONPs to the drilling fluid sample displayed that the plastic viscosity (PV) was recorded at 8 cP and thixotropy at 2 lb/100 ft2 under HTHP conditions without mud failure due to establishing highly stable multiple bentonite-IONPs bridging effect. Also, filtration for IONPs-1 sample showed enhanced sturdy filter mud cake and confirmed the resistance of this modified sample to the failure occurred for the blank drilling mud at these harsh thermal conditions.

Introduction

The advancement of drilling operations into HTHP formations demand the usage of drilling fluid formulae that will withstand high temperatures by stabilizing the integrity of the rheology of the drilling fluid under such conditions [1-3]. Increasing demand for energy and depletion of conventional reserves forces the drilling industry to drill deeper and more complicated wells. Drilling operations are now at the advanced level where complex drilling situations including high temperature and high pressure zones are encountered. More concern in the drilling operations now is the need to circumvent problems posed by the high temperature zones during drilling operations [4,5]. However, water-based mud (WBM) deteriorates under high-temperature/high-pressure (HT/HP) conditions, and there is a great need to improve their properties and extend their use in extreme down-hole conditions. This can be achieved by using different additives [6].

Different types of polymers are used in designing the drilling mud to meet some functional requirements such as the appropriate mud rheology, density, mud activity, fluid loss control property and temperature [7,8]. Studies of nanoparticles have shown their unique abilities in their functionalities such as thermal conductivity, electrical conductivity, optical features etc. Particularly, the thermal ability and the larger surface area of these nanoparticles are the motivation for this study to show

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the extent of stability that will be imposed on the drilling fluids against temperature [9,10].

Some investigators used Palygorskite (Pal), which was purified, synthesized, characterized, functionalized, and tested in nano-form (10 – 20 nm). The added nanoparticles were found to provide drilling fluid with stable rheological properties under HTHP environments. They concluded that Pal NP can be used as an effective rheological modifier [11]. Another study [12] investigated the enhancement in yield point at high pressure and high temperature wells by using polymer nanocomposites based on ZnO and $CaCO_3$ nanoparticles.

The Principle aim of this investigation is to illustrate the potential of co-precipitated synthesized iron oxide nanoparticles (IONPs) after their characterizations on the rheological properties and filtration loss control of water-based drilling fluids under HTHP conditions.

Subjects and Methods

Reagents

All reagents used in the investigation were analytical grade and used without further purification. Iron(III) chloride hexahydrate ($FeCl_3$, $6H_2O$) (Sigma-Aldrich) was the iron precursor, while ammonia hydroxide (NH_4OH) was the precipitating agent, and ethanol (C_2H_6O) (Sigma-Aldrich) was used for washing. All solutions were prepared with deoxygenated distilled water. Drilling-grade bentonite clay with API/ISO specifications was gained from OCMA- Schlumberger Oilfield Glossary, free zone, Alex., Egypt.

Synthesis of iron oxide nanoparticles (IONPs)

Pure iron oxide (hematite) nanoparticles were synthesized with the chemical precipitation method. In this procedure, aqueous solution was prepared by dissolving 50 g of iron(III) chloride hexahydrate ($FeCl_3$, $6H_2O$) in 100 mL of deoxygenated distilled water under magnetic stirring for 30 min at 80^{o}C to obtain a 0.05 M concentration solution (the same principle to obtain solutions of different concentrations (0.1, 0.2 and 0.4 M)). 50 mL aqueous solution of 2 M of NH_4OH was used as the precipitating agent. Base solution (NH_4OH) was added gradually drop wise to maintain a pH value of 11. The reaction vessel was heated up to the temperature of 80^{o}C under magnetic stirring for 3 h. The resulting precipitations were collected and centrifuged at 6000 rpm and then washed with distilled water and ethanol for several times and finally dried in air at 80^{o}C and calcined at 700^{o}C for 4 h [13] as displayed in **Scheme 1**.

Transmission electron microscope (TEM) characterization of iron oxide nanoparticles (IONPs)

Transmission electron microscope of the IONPs was conducted at an accelerated voltage of 200 kV electron microscopes (JEM 2100 La B6 Japan)**.** In the TEM, the solid samples were dispersed in ethanol solution using ultrasonicator and then dropped on a copper grid coated with carbon film prior to inserting the samples in the TEM column, the grid was vacuum dried for 15 min.

Scanning electron microscope (SEM) and Energy dispersive analysis of X-rays (EDX) characterizations of iron oxide nanoparticles (IONPs)

Scanning electron microscopic (SEM) micrographs of iron oxide nanoparticles (IONPs) were clarified by QUANTA FEG 250 scanning electron microscope utilized for this investigation [14]. All micrographs of these particles were carried out at magnification of 100,000 X. EDX system attached with QUANTA FEG 250 scanning electron microscope was employed for elemental analysis of the employed IONPs. It depends on the investigation of sample via interaction between electromagnetic radiation and the matter. Then, a detector was used to convert X-ray energy into voltage signals. These signals are sent to a pulse processor, which measure the signals and passed them onto an analyzer or data display and analysis.

Preparation of drilling fluid samples

API bentonite was incorporated as a base for the preparation of the drilling fluid for this investigation. In this approach, 350 ml of fresh water was measured using a measuring cylinder and was added to 26 g of bentonite using the measuring scale and stirred in the bucket until no more lumps were observable by the help of an electric mixer. The drilling fluid was left to stay overnight (16 hours) to swell to establish blank mud sample. Bentonite drilling fluids with iron oxide nanoparticles (IONPs) were prepared by adding 1 and 2 grams of the nanoparticles after the fluid is left overnight and stirred vigorously then, homogenized with an electronic mixer for about 2 - 5 minutes to establish IONPs-1 and IONPs-2 drilling mud samples. The blank bentonite fluid and the modified bentonite fluids were used for the various experiments.

Mud viscosity determination

The mud viscosity of the samples was determined using Fann V-G meter. The Fann V meter was filled to the 350-cc mark and placed on the movable work table [14]. The table was adjusted until the mud surface was at the scribed line on the rotor sleeve. The motor was started with a high-speed position (600 rpm) and the reading was taken from a steady indicator dial value. The reading was also obtained at the low speed of 300 rpm. This was repeated for both samples and at different mud weights. The following parameters are calculated as:

$$\boldsymbol{Plastic\,Viscosity}\ (\boldsymbol{PV}) = \theta_{600} - \theta_{300}\ (cp) \quad (1)$$

$$\boldsymbol{Apparant\,Viscosity}\ (\boldsymbol{AV}) = {}^{\theta_{600}}/_{2}\ (cp) \quad (2)$$

$$\boldsymbol{Bingham\,Yield\,Point}\ (\mathbf{Y}_{\boldsymbol{b}}) = \theta_{300} - PV\ ({}^{\mathrm{lb}}/_{100\ \mathrm{ft}^2}) \quad (3)$$

In which $\theta_{600} and\, \theta_{400}$ are the 600 and 300 dial readings, respectively.

Gel strength determination

The Fann V-G meter was also used to determine the gel strength of the mud samples. The mud samples were stirred thoroughly at 600 rpm. The lift gear was shifted slowly to the first position, and the motor was shut off. The motor switch was turned to low after 10 seconds. The dial was read at maximum deflection units in lb/100ft^2 that is 10 second gel. The steps were repeated for 10 minutes.

Mud filtration loss and cake morphology

API filtration loss examination (HTHP testing) is a procedure utilized to determine mud filtration at 500 psi and 350^{o}C by utilizing API liquid test HTHP outfit. Mud estimations are limited to the static filtration. Filtration attributes of a mud are controlled by methods for a filter press. The analysis comprises controlling the rate at which liquid is penetrated from a filter press under designated states of time, temperature and weight. The fluid misfortune standard test is prepared more than 30 minutes. The filtrate amount ascends with expanding the square foundation of the time. The surface morphology of mud cake established by the different blank and IONPs drilling mud sample was surveyed by QUANTA FEG 250 scanning electron microscope utilized for this investigation.

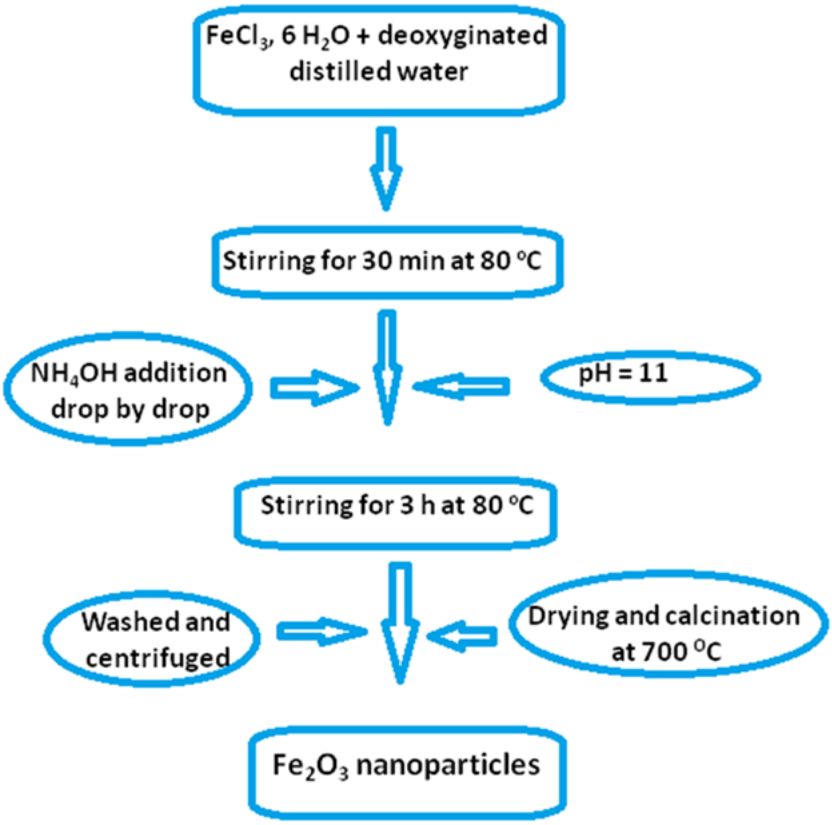
Results and discussion

TEM characterization of the synthesized iron oxide nanoparticles (IONPs)

The internal morphology of the synthesized iron oxide nanoparticles (IONPs) was characterized by transmission electron microscope (TEM). TEM image of IONPs shows the homogenized surface of material and provide a unique scope to directly visualize nano-particle morphology. As shown in **Fig. 1** it can be noted that, the white bar in the left bottom corner is a 50 nm-length reference for the TEM image. The size of the investigated particles could be visually seen in the range of 50 nm size. Also, the TEM figure shows the non-agglomeration of the IONPs in addition to the approximately spherical shape of these particles.

SEM and EDX micrographs of the synthesized iron oxide nanoparticles (IONPs)

The SEM image of the synthesized iron oxide nanoparticles (IONPs) is illustrated in **Fig. 1**. This clearly shows the realistic nature of the nano-sized particles used in this investigation. This characteristic micrograph demonstrated that, IONPs have a spherical-shaped morphology as shown in **Fig. 2** and illustrated the network arrangement of these nanoparticles similar to retinal clusters in which affords a conception about the bridging effect can be produced during the well dispersion of these particles via the API bentonite mud sample. **Fig. 3** offers the EDX chemical analysis of the synthesized IONPs that confirms the chemical composition of the synthesized nanoparticles.



Scheme 1: Flow chart for the preparation process of iron oxide nanoparticles (IONPs).

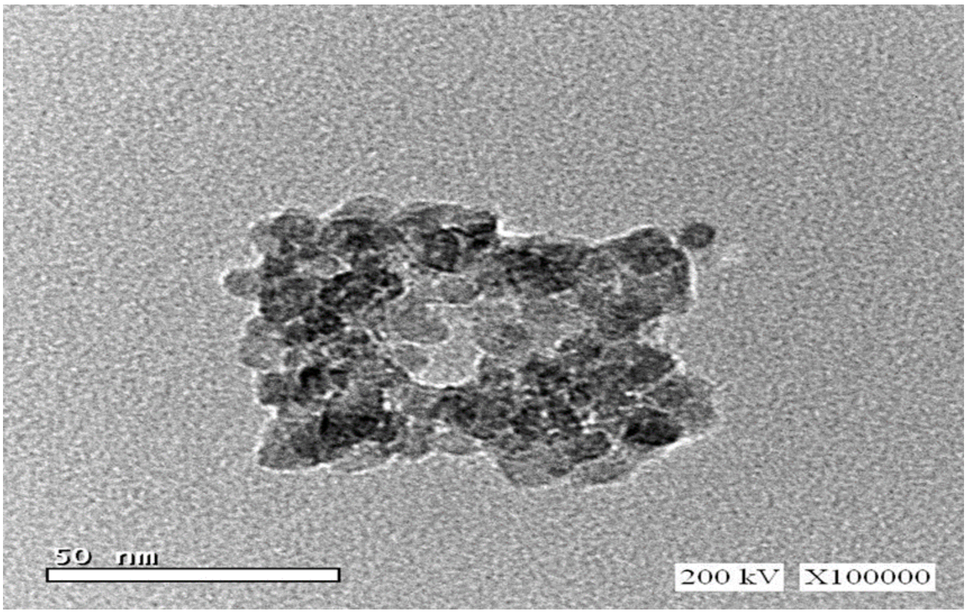


Fig. 1: TEM micrograph of the synthesized iron oxide nanoparticles.

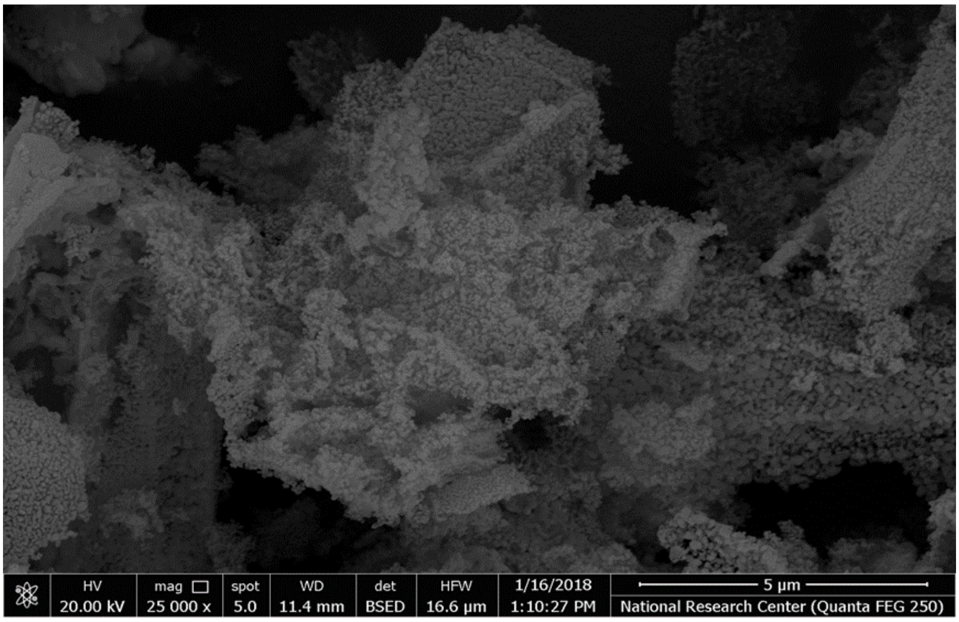


Fig. 2: SEM image of the synthesized iron oxide nanoparticles.

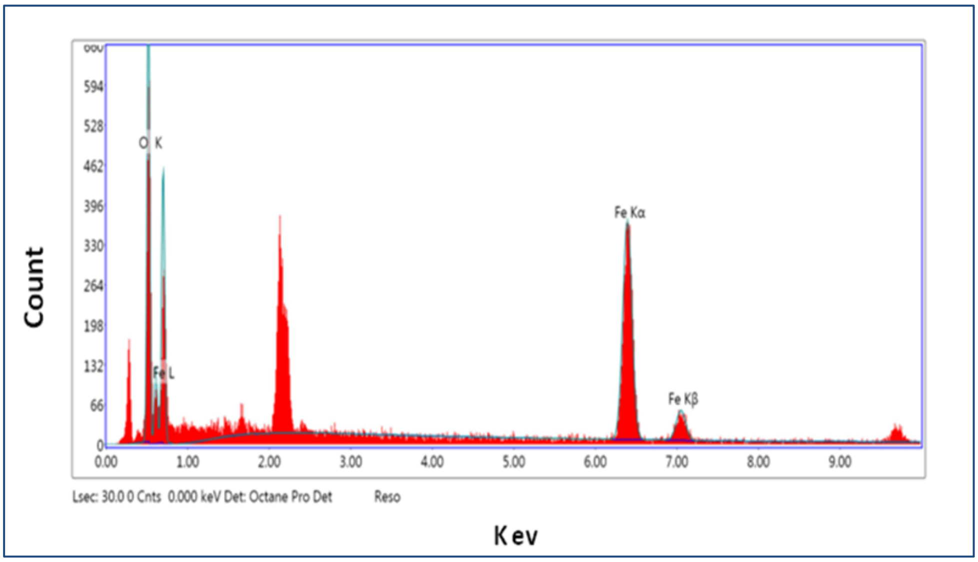


Fig. 3: EDX image of the synthesized iron oxide nanoparticles.

Influence of incorporating the synthesized iron oxide nanoparticles (IONPs) on the bentonite-based drilling fluids at HTHP conditions

The potential of alteration the shear stress with shear rate for blank drilling fluids sample at different temperatures (65 and 100, 150 and 200^{0}F) was shown in **Fig. 4.** According to the results depicted in **Table 1**, there is a linear increase in shear rate with increasing the shear stress. It was observed that viscous characteristics for the blank drilling fluid sample were decreased with increasing the temperature. A plot of shear stress/shear rate shown in **Fig. 4** assumed a Bingham Plastic model and indicated an effective rearrangement of gel-networks of API bentonite particles produced from the thixotropic effect by Na-bentonite particles in water under shear stress at ambient temperature. Then, in the case of high temperatures application as 150 and 200^{0}F, there is a disturbance produces in API bentonite gel-networks and then, a decrease in viscous properties is observed as illustrated in **Table 1**. At the elevated temperature of 350^{0}F, it could be observed that a complete failure in rheology of blank drilling sample and this behavior was done and attributed to the deterioration of gel-network established by API bentonite particles. In the case of 1 g addition of synthesized iron oxide nanoparticles (IONPs) to the water-based drilling mud sample at 350^{0}F, the IONPs modified drilling fluid sample displayed highly failure resistance in the swelling properties than that of the bentonite mud without nanoparticles as offered by **Fig. 5** and listed in **Table 2**. This could be attributed to the well-dispersion of IONPs with polar terminals via the bentonite mud suspension in which increasing the interface surface interactions between these particles and clay grains then stick to Na-bentonite layers surface through hydrogen bonding or electrostatic attractions [14,15]. This produces further connection with adjacent clay particles, which can make multiple clay- IONPs mixture particles bridged together during the excellent swelling behavior of API bentonite in drilling fluids as shown in the predicted mechanism of bridging effect produced between iron oxide nanoparticles and API bentonite layers in **Fig. 6**. This behavior improved the thermal stability of drilling mud suspension and the affinity to hold drilling cuttings at high temperature /high pressure (HTHP) conditions.

The synthesized Fe_2O_3 nanoparticles fixed in randomly formed pore structure on the surface of clay particle confer link between bentonite particles, which might promote gel-networks of the bentonite particles [15,16]. In the case of 2 g IONPs modified drilling mud sample (IONPs-2), there was an increase in the shear rate values at 200, 300 and 600 shear stress readings than that for 1 g IONPs treated drilling fluid as shown in **Fig. 7** and illustrated in **Table 3**. This might be assigned to the enhancement in the bridging effect and interface interactions produced with increasing the concentration of IONPs in drilling mud suspension **[17]**. The variation of shear stress with shear rate showed that all mud samples were non-Newtonian that follow Bingham plastic model.

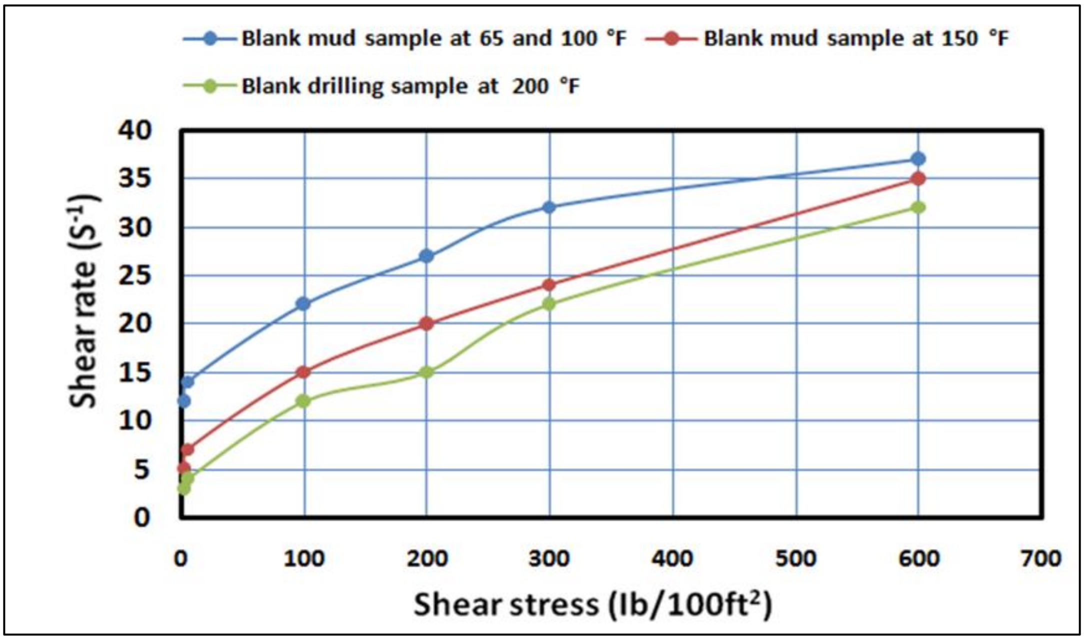


Fig. 4: Shear rate and shear stress values for blank drilling mud sample at different temperatures.

Table 1: Shear rate and shear stress at different temperatures for blank drilling fluid sample

Shear rate (S^{-1}) at different temperatures	**Shear stress ($lb/100ft^2$)**					
Mud sample	3	6	100	200	300	600
Blank mud sample at 65 and 100^0F	12	14	22	27	32	37
Blank mud sample at 150^0F	5	7	15	20	24	35
Blank drilling sample at 200^0F	3	4	12	15	22	32
Blank mud sample at 350^0F	**Complete failure notice**					

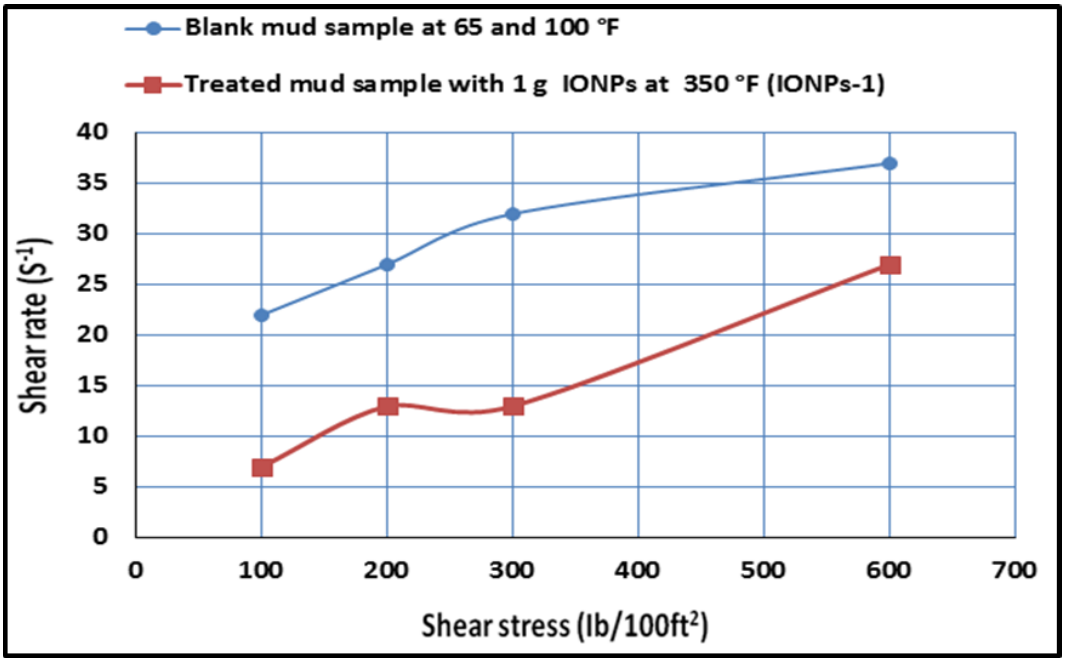


Fig. 5: Shear rate and shear stress values for blank drilling mud sample at ambient temperature and 1 g treated mud sample with IONPs at 350^0F.

Table 2: Shear rate and shear stress at HTHP in case of adding 1 g of iron oxide nanoparticles (IONPs) at 350^0F against blank drilling fluid sample at 65 and 100^0F

Shear rate (S^{-1}) at different temperatures	**Shear stress ($lb/100ft^2$)**					
Mud sample	3	6	100	200	300	600
Blank mud sample at 65 and 100^0F	12	14	22	27	32	37
Blank mud sample at 350^0F	**Complete failure notice**					
Treated mud sample with 1 g IONPs at 350^0F (IONPs-1)	3	3	7	13	13	27

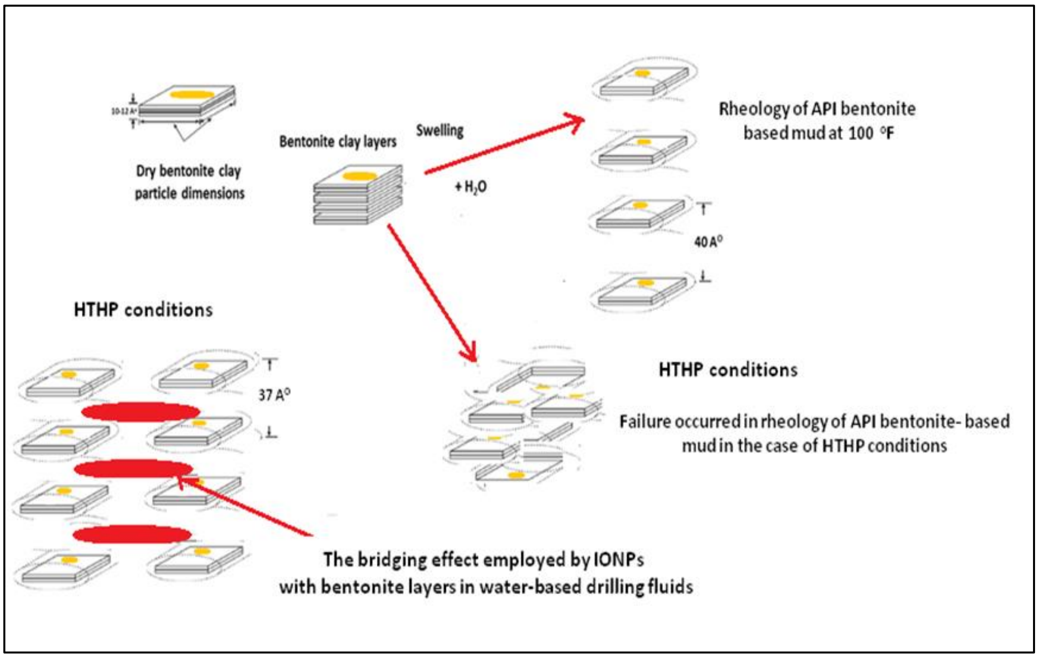


Fig. 6: Mechanism of bridging effect produced by surface modified IONPs drilling mud and blank drilling mud samples at 350^0F.

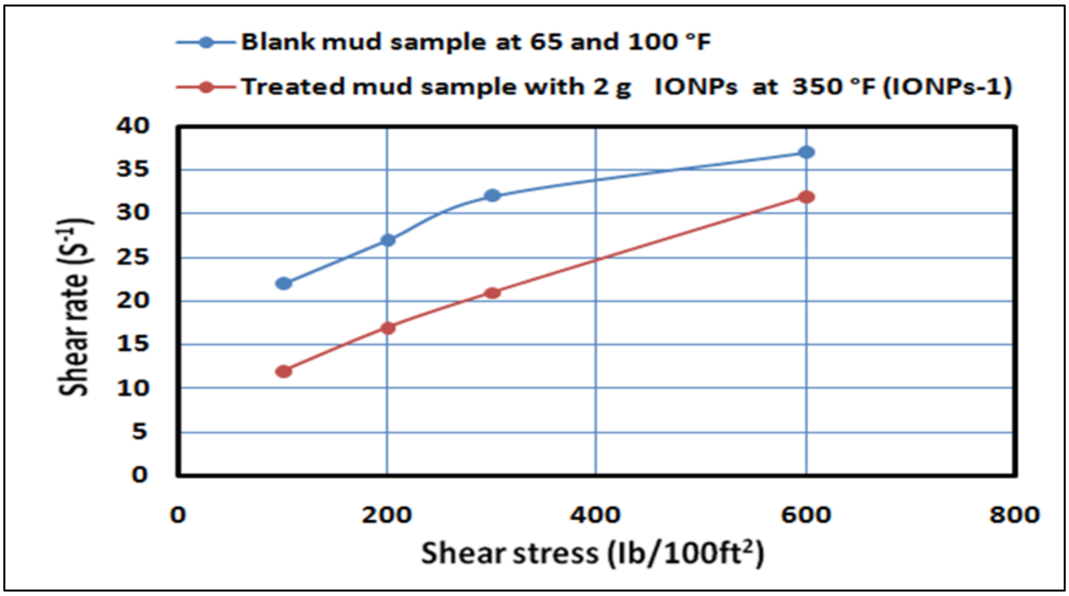


Fig. 7: Shear rate and shear stress values for blank drilling mud sample at ambient temperature and 2 g treated mud sample with IONPs at 350^0F.

Table 3: Shear rate and shear stress at HTHP in case of adding 2 g of iron oxide nanoparticles (IONPs) at 350^0F against blank drilling fluid sample at 65 and 100^0F

Shear rate (S^{-1}) at different temperatures	**Shear stress (lb/100ft^2)**					
Mud sample	3	6	100	200	300	600
Blank mud sample at 65 and 100^0F	12	14	22	27	32	37
Blank mud sample at 350^0F	**Complete failure notice**					
Treated mud sample with 2 g IONPs at 350^0F (IONPs-1)	6	7	12	17	21	32

Rheological properties of the synthesized iron oxide nanoparticles (IONPs) treated drilling fluid against blank API bentonite suspension

The considered rheological properties such as apparent viscosity (AV), plastic viscosity, yield point (YP), gel strength (GL) and thixotropy (Thixo) were measured for blank drilling mud sample at different temperatures (100, 150, 200 and 350^{0}F) as shown in **Fig. 8** and illustrated in **Table 4.** According to the tabulated results in **Table 4** it could be observed that there was a decrease in the apparent and plastic viscosities recorded 17.5 and 11 cPs, at 150^{0}F and, 16 and 10 cPs, at 200^{0}F, respectively. Also, yield point property decreased to be listed at 12 lb/100ft^2 at 200^{0}F. In addition, gel strength and thixotropy parameters were decreased with increasing the applied temperature. The rheological properties of blank drilling fluid sample offered complete failure notice at elevated temperature of 350^{0}F. This decrease in the rheology values at high temperatures could be attributed to deterioration in the gel-networks of API bentonite particles at HTHP conditions. In the case of adding 1 g iron oxide nanoparticles (IONPs-1) to the water-based drilling mud, thermal stability against deterioration effect and resistance to rheology failure for the drilling mud sample was observed. Apparent and plastic viscosities were calculated at 14 and 8 cPs, respectively as illustrated in **Table 5** and shown in **Fig. 9**. This could be attributed to the generated bridging effect due to the sticking between API bentonite layers and iron oxide nanoparticles (IONPs) in which resisted the influence of high temperatures and pressures and enabled the drilling emulsion in preserving its thixotropic behavior then hindered the failure occurred in the case of blank drilling fluid sample at elevated HTHP conditions. It was observed that, the thermal stability and resistance to rheology failure for the drilling mud sample were increased in the case of IONPs-2drilling sample. As shown in **Fig. 10** and listed in **Table 6**, apparent and plastic viscosities were increased and recorded at recorded at 16 and 12 cPs, respectively in compared to their calculated values for 1 g IONPs treated sample recorded at 14 and 8 cPs, respectively. This could be attributed to the increasing in concentration of IONPs in drilling mud suspension and supporting the bridging behavior produced between API bentonite particles and [17]. On the other hand, an increase in the yield point and gel strength rheological properties values was observed with increasing the IONPs concentration through the drilling mud suspension as illustrated in **Table 6**. These properties were recorded at 17 and 9 lb/100ft^2, respectively for 2 g IONPs modified sample in compared to recognized values at 11 and 3 lb/100ft^2, respectively for 1 g IONPs modified sample. This behavior could be attributed to the enhancement in installed bridging effect than the effect of homo-agglomeration and the interface surface interactions with Na-bentonite layers produced due to the re-arrangement of IONPs in mud suspension.

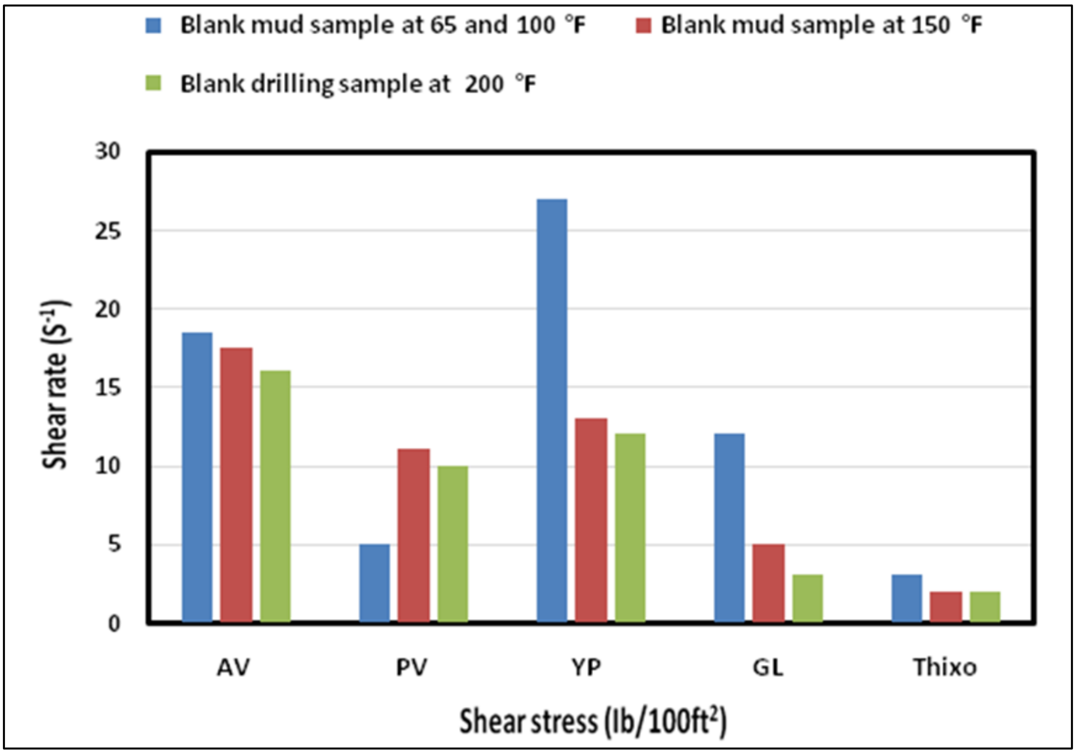


Fig. 8: Rheology parameters for blank drilling mud sample at different temperatures.

Table 4: Rheological properties for blank drilling fluid sample at different temperatures

Rheology parameter **Mud sample** **At different temperature**	**AV (cP)**	**PV (cP)**	**YP (Ib/100ft^2)**	**GL (lb/100ft^2)**	**Thixotropy (lb/100ft^2)**
Blank mud sample at 65 and 100 ^{0}F	18.5	5	27	12	3
Blank mud sample at 150^{0}F	17.5	11	13	5	2
Blank drilling sample at 200^{0}F	16	10	12	3	2
Blank mud sample at 350^{0}F	**Complete failure notice**				

Table 5: Rheological parameters for blank drilling fluid sample at 65 and 100^{0}F and treated mud sample with 1 g IONPs at 350^{0}F (HTHP)

Rheology parameter **Mud sample** **At different temperature**	**AV (cP)**	**PV (cP)**	**YP (Ib/100ft^2)**	**GL (lb/100ft^2)**	**Thixotropy (lb/100ft^2)**
Blank mud sample at 65 and 100 ^{0}F	18.5	5	27	12	2
Blank mud sample at 350^{0}F	**Complete failure notice**				
Treated mud sample with 1 g IONPs at 350^{0}F (IONPs-1)	14	8	11	3	2

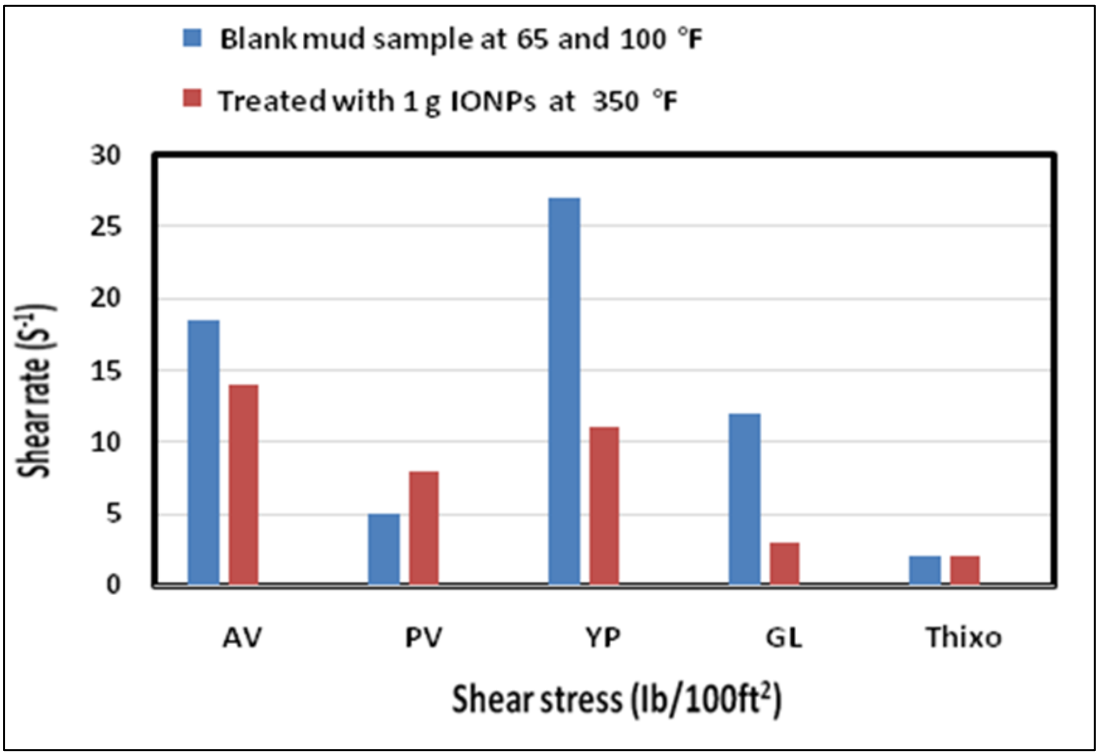


Fig. 9: Rheology parameters for blank drilling mud sample at ambient temperature and 1 g treated mud sample with IONPs at 350^{0}F.

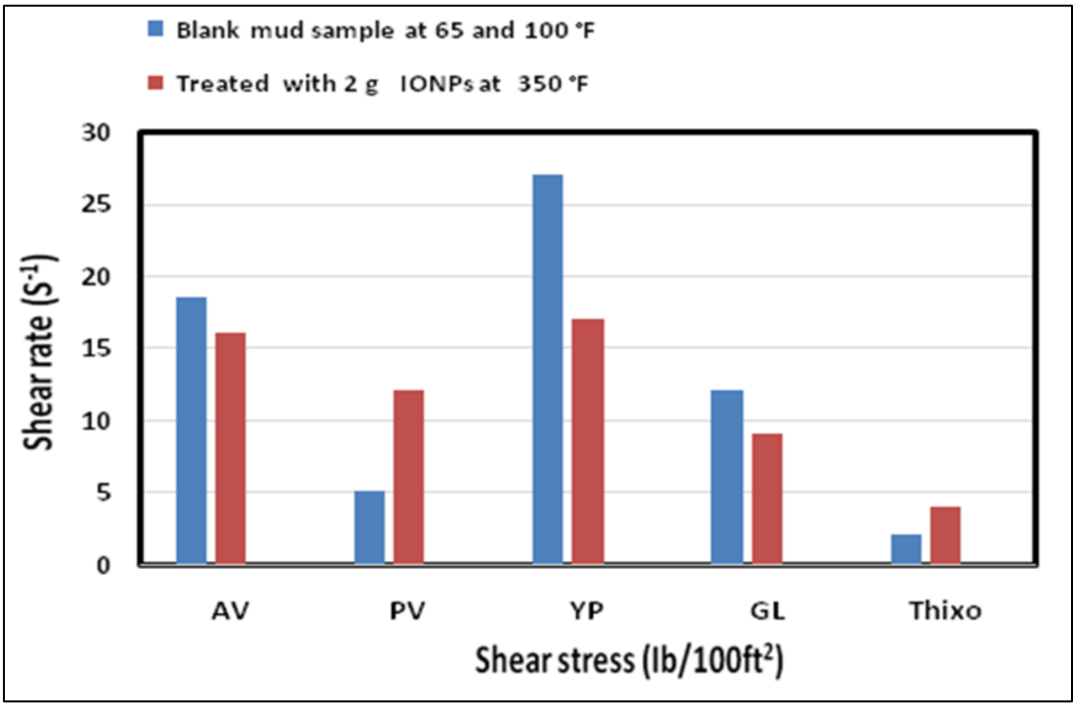


Fig. 10: Rheology parameters for blank drilling mud sample at ambient temperature and 2 g treated mud sample with IONPs at 350^{0}F.

Table 6: Rheological parameters for blank drilling fluid sample at 65 and 100^{0}F and treated mud sample with 2 g IONPs at 350^{0}F (HTHP)

Rheology parameter **Mud sample** **At different temperature**	**AV (cP)**	**PV (cP)**	**YP (Ib/100ft^2)**	**GL (lb/100ft^2)**	**Thixotropy (lb/100ft^2)**
Blank mud sample at 65 and 100^{0}F	18.5	5	27	12	2
Blank mud sample at 350^{0}F	**Complete failure notice**				
Treated mud sample with 2 g IONPs at 350^{0}F(IONPs-2)	16	12	17	9	4

Filtration loss control by IONPs

The obtained results for blank bentonite-based drilling fluid and modified IONPs mud samples manifested filtrate loss values per unit volume at 500 psi and temperature 350^{0}F as shown in **Fig. 11.** The potential of treatment of the drilling mud with nanoparticles on enhancing its thermal stability and rheological properties was recently studied [17]. From the illustrated data in **Table 7**, it was observed that the blank drilling mud sample displayed higher fluid loss of 22 ml at ambient temperature and complete failure notice at 350^{0}F compared to thermal stability observed in the case of 1 g IONPs treated samples (filtrate volume = 19 ml) at elevated HTHP conditions. With increasing the concentration of IONPs via the drilling fluid suspension, the filtrate loss volume decreased and was recorded at 12 ml. The phenomenon was due to the ability of differential pressure to compress larger quantity of particles presented in the mud sample with iron oxide nanoparticles, thus prevented the formation of thin and low permeability mud cake which excessive fluid loss that can cause formation damage and lead to reduce the well production. Failure was progressed at HTHP conditions could be attributed to the clay-aggregation and causing the mud cake to have high permeability [18,19]. The increase in mud cake thickness is attributed to the well-dispersion of spherical iron oxide nanoparticles which stick with bentonite clay layers (bridging effect) in mud emulsion and improves the hydration behavior and swelling process then, increases thickness of the formed cake and block any bores, thereafter, decreases the filtration loss per unit volume **[20, 21]**. This behavior increases the thermal stability of the drilling fluid sample modified with the IONPs to be suitable for HTHP applications and resists the failure can be progressed in the drilling suspension.

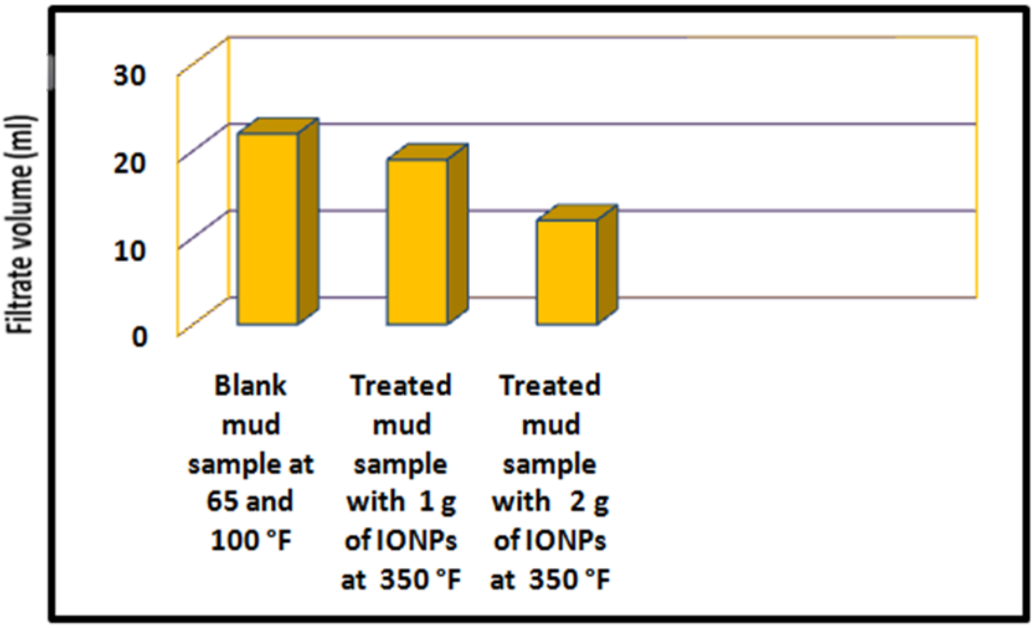


Fig. 11: Filtration loss volume for blank drilling mud sample at ambient temperature and for 1 and 2 g treated mud sample with IONPs at 350⁰F.

Table 7: Filtration loss for blank drilling fluid sample at 65 and 100⁰F and treated mud sample with 1 and 2 g of IONPs at 350⁰F (HTHP)

Mud Samples at different Temperatures	Blank mud sample at 65 and 100⁰F	Blank mud sample at 350⁰F	Treated mud sample with 1 g of IONPs at 350⁰F	Treated mud sample with 2 g of IONPs at 350⁰F
Filter loss values (ml)	22	Complete failure notice	19	12

Conclusions

Iron oxide (Fe_2O_3) nanoparticles (IONPs) were synthesized and utilized to increase the thermal stability of water-based drilling fluids at HTHP conditions. The shear stress/shear rate plot showed the shear thinning behavior of blank drilling mud samples at ambient temperature (65⁰F) and a lower viscous property in the mud sample was demonstrated with increasing temperature gradually till 200⁰F in addition to complete failure produced at elevated temperature (350⁰F). The studied drilling fluid samples assumed non-Newtonian effect that follow Bingham plastic model. The modified 1 g IONPs drilling fluid sample resists the deterioration effect of HTHP conditions in which the rheological properties were upholded at 350⁰F. Filtration loss per unit volume evaluation indicated an obvious improvement for treated mud sample with iron oxide nanoparticles (IONPs) at 350⁰F against complete failure occurred in the case of blank drilling mud.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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