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Experimental Investigation of Nano Iron Oxide on Physical Properties of Salt-Saturated Drilling Mud

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ABSTRACT

The process of drilling wells has become more complex today due to greater depths and advanced drilling techniques. Therefore, obtaining ideal specifications for drilling fluids has become of great importance to overcome drilling problems and reduce costs. Therefore, studies have begun to investigate the effect of nanomaterials on drilling fluids. This study is an investigation into the effect of nano-iron oxide on drilling fluid specifications. Saturated salt mud was used, and different physical properties such as viscosity, density, and filtrate loss were measured at different temperatures (30 ˚C, 50 ˚C, and 70 ˚C), and adding different proportions of nano iron oxide (0.75 and 1.5 gm). This study is characterized by the combination of field and laboratory work. The addition of nano iron oxide leads to an increase in gel strength while the other rheological properties remained relatively constant, and the change in filtration and mud cake is small except at high temperature. Rising temperature leads to a decrease in rheological properties, except for gel strength. But at 70 C˚, the addition of nano iron oxide reduced the effect of rising temperature, especially on the yield point, and this is an important property when drilling a salt section.

Keywords: Salt saturated drilling mud, Nano iron oxide, Filtration, Rheological properties, Lower fars formation.

1. INTRODUCTION

Drilling fluids play an important part in the drilling of oil and gas reservoirs. The drilling fluid has some significant jobs such as: clean the bottom of wells from cuttings of rocks and remove them at surface, work on cooling drill bits and strings and lubricate them, make a thin mud cake that has low permeability to close pores of rock formation, provide hydrostatic pressure that lead to banning formation fluid enters into wells and decrease the coefficient of friction that occur between the hole and drilling string **(Skalle, 2012)**. In general, drilling fluids are categorized into two types: water-based mud and oil-based mud **(Caenn et al., 2011).** 80% of all wells are drilled by water-based fluids. They may be fresh water, seawater, brine, saturated brine, or a format brine **(Ahmed and Ekrem, 2019)**. Salt

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structures appear in some big oil and gas reservoirs. They are present in Domal structures in the Gulf of Mexico (GoM – Jurassic salt emplaced during the Tertiary), Williston basin (Mid US Continent Devonian age) the North Sea (Zechstein age salt emplaced in the Cretaceous), Iran (Zagros salt plugs, which in some areas outcrop), Brazilian and West African offshore basins, and other areas **(Dusseault et al., 2004)**. Drilling fluid must have Salinity at or near saturation when salt rocks are drilled in order to avoid salt dissolution and to maintain a gauge hole **(Amer et al., 2016)**. Also, drilling fluid must have high density because the Porosity and permeability of salt are very low, and salt creep can happen, which means it flows plastically through other geological rock beds under stress. Therefore, overbalance of Boreholes in salt layers tends to occur **(Weijermars et al., 2014)**. This research was made on salt-saturated drilling mud that was used to drill the salt section (Lower Fars Formation) in Fauqi oilfield (FQCN-96) in Iraq. The diameter of this section is 2-1/4", and its length is about 712 m (from 2266.5m to 2978m).
Missan oilfields are located in the Missan province in southeastern Iraq and close to the Iraq-Iran border. It is about 175km north of Basra city. The area consists of three producing oilfields, namely Abu Ghirab, Buzurgan, and Fauqi oilfields. The Fauqi oilfield ranges about 30km X 7km with two blocks in the north and south, respectively. It is a NW-SE long-axis anticline. Well, FQCN-96 is a Vertical Producer, and is located about 500 m to the SE of FQ-2. **Figs. 1 and 2** show the Location of the well.

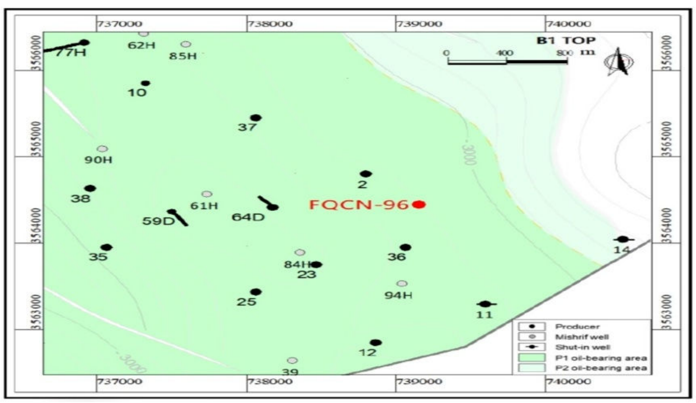

Figure 1. Well location on top B1 of the Asmari Formation in Fauqi oilfield **(COSL, 2023)**

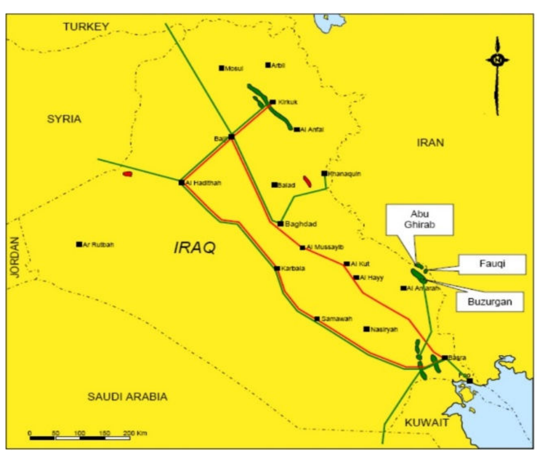

Figure 2. Location map of Missan oilfields **(COSL, 2023)**

Drilling the Lower Fars Formation faced many challenges due to its high formation pressure, high temperature, and included layers of salt **(Hassan and Mohammed, 2023),** so it became necessary to conduct studies that can prevent these difficulties or reduce them. The geothermal gradient of the Asmari reservoir in Fauqi oilfield is 2.37 °C/100m, and the pressure coefficient of Lower Fars is about 2.20-2.24(g/cc), and the ECD at the bottom is 2.295 (g/cc). The description of the lithology column of well FQCN-96 is listed in **Table 1**.

Table 1. Description of lithology column of well **(COSL, 2023)**

Strata		Expected Top depth m, TVD	Lithology description
Tertiary	Upper Fars		Upper mainly clay, lower mainly shale, interbedded sand
	Lower Fars(mb5)	2186.5	Alternation of anhydrite, shale
	Lower Fars(mb4)	2517.5	Alternation of anhydrite, shale, and salt
	Lower Fars(mb3)	2755.5	A thick bed of anhydrite, interbedded with salt and shale, with rare limestone.
	Lower Fars(mb2)	2949.5	Massive salt
	Lower Fars(mb1)	2977.5	Anhydrite with thin intercalation of shale and shaly dolomite
	Jeribe-Euphrate(A1)	3012.5	Massive dolomite, locally limestone, and anhydrite interbedded with reddish brown shale
	Jeribe-Euphrate(A2)	3025.5	Massive dolomite, locally limestone, and anhydrite interbedded with reddish brown shale
	Jeribe-Euphrate(A3)	3047.5	Massive dolomite, locally limestone, and anhydrite interbedded with reddish brown shale
	Upper Kirkuk(B1)	3064.5	Mainly dolomite interbedded with sandstone, limestone and mudstone
	Upper Kirkuk(B2)	3095.5	Mainly dolomite interbedded with sandstone, limestone and mudstone
	Upper Kirkuk(B3)	3124.5	Mainly dolomite interbedded with sandstone, limestone and mudstone
	Upper Kirkuk(B4)	3162.5	Mainly dolomite interbedded with sandstone, limestone and mudstone
	Top Middle-Lower Kirkuk(C)	3210.5	Massive shale and sandstone, locally limestone
TD		3223.5	

Nanotechnology is one of the advanced scientific solutions to problems **(Patel et al., 2024)**. The results of analyses and investigations show that drilling fluid properties can be improved by using nanoparticles as additives because of their large area-to-volume ratio **(Gokapai et al., 2024)**. The main features of using nanoparticles in drilling fluids are reduced formation damage and total cost of drilling fluid, separated hazardous materials, and made perfect change in properties such as rheological, heat transfer, lubrication, and control of fluid loss and mud cake **(Cheraghian, 2021)**. Iron Oxide nanoparticles are used in this research. γ-Fe_2O_3 (maghemite) is the chemical formula of gamma iron oxide. It is the second most stable form of iron oxide. It has many features that make researchers investigate it widely, such as abundance, low cost, non-toxicity, and high chemical and physical stability **(Biswas et al., 2024)**. Nano Iron Oxide applications can cover a wide range, such as coatings, gas sensors, ion exchangers, flocculants, pigments, magnetic recording devices, wastewater treatment adsorbents, magnetic data storage devices, catalytic materials, bio separation, and medicine **(Mohapatra and Anand, 2010)**. Nano γ-Fe_2O_3 is also used in magnetic materials **(Agarwal et al., 2011)** and in plants **(Hu et al., 2017)**. In drilling fluids of oil wells, Nano iron oxide can be used as an additive. There is research by Barry to study the effect of adding nano-iron oxide to water-based mud with 5.0 wt% bentonite under two conditions (low-temperature low-pressure and high-temperature high-pressure), and the results showed an increase in the rheological properties **(Barry et al., 2015)**. In a study conducted by Muhammed, the effect of adding iron oxide nanoparticles to two types of drilling fluids, bentonite and KCl-based drilling fluids, was studied. The results showed a decrease in the amount of filtrate loss and also reduced the coefficient of friction **(Alvi et al., 2020)**. Likewise, Jung added nano Fe_2O_3 (3 and 30 nm) to water-based mud (5% wt bentonite), and that led to an increase in viscosity **(Jung et al., 2011)**. Contreras used oil-based mud (Oil/Water Ratio (90/10)) in their research and concluded that adding nano Fe_2O_3 led to fluid loss control **(Contreras et al., 2014)**.

Several physical properties indicate the behavior of the drilling fluid, such as density, which controls the pressure of formations and their effect on wellbore stability **(Khan et al., 2021)**. Also, Mud Filter Loss is another property that shows the amount of filtrate that enters through porous permeable formation **(Peng, 1990)**. Borehole stability is excellent when fluid loss is Low. In addition to the above, the rheological properties refer to the deformation and flow behavior of fluids under a variety of different conditions. These rheological properties are apparent viscosity (AV), plastic viscosity (PV), gel strength, and yield point (YP) **(Okorie et al., 2021)**. The Marsh funnel is a crude method for measuring the consistency of a fluid **(Alsabaa et al., 2020)**. Presently, a significant number of rheological models describing the flow of drilling fluids are known. Such as: Newton model (Eq. (1))**(Newton, 1999)**, Bingham model (Eq. (2)) **(Frigaard, et al.,2017; Bingham, 1922)**, Ostwald model (Eq. (3)) **(Ostwald, 1925; Khalil et al., 2011)**, Herschel and Bulkley model (Eq. (4)) **(Herschel and Bulkley, 1926; Hemphill et al., 1993)**, Casson model (Eq. (5)) **(Casson, 1959)**, Schulman model (Eq. (6)) **(Lykiva and Smilkogo, 1968)** and Robertson and Stiff model (Eq. (7)) **(Robertson and Stiff, 1976)**

$$\dot{\gamma}(\tau) = \tau / \eta \tag{1}$$

$$\dot{\gamma}(\tau) = (\tau - \tau_\circ) / \eta \tag{2}$$

$$\dot{\gamma}(\tau) = (\frac{\tau}{k})^{1/n} \tag{3}$$

$$\dot{\gamma}(\tau) = ((\tau - \tau_\circ) / \mathrm{k})^{1/n} \tag{4}$$

$$\dot{\gamma}(\tau) = \left(\sqrt{\tau} - \sqrt{\tau_\circ}\right)^2 / \eta \tag{5}$$

$$\dot{\gamma}(\tau) = (\tau^{1/n} - \tau_\circ{}^{1/n})^n / \eta \tag{6}$$

$$\dot{\gamma}(\tau) = (\frac{\tau}{k})^{\frac{1}{n}} - \beta \tag{7}$$

Where $\dot{\gamma}$ And τ are the velocity gradient and shear stress; k, n, η, $\tau_\circ$,β are the indices of rheological properties.

2. EXPERIMENTAL AND FIELD WORKS

The type of drilling fluid used in this research is salt-saturated drilling mud. It was prepared in the field during drilling FQCN-96 by using the mud system of rig No. 55, which belongs to the Iraqi Drilling Company, in cooperation with the Chinese company (COSL). The concentration of materials used has been listed in **Table 2**.

Table 2. Concentration of materials used in preparing drilling fluid

Products	**Concentration Kg/m^3**
NaOH	3
Na_2CO_3	3
PF-VIS	3
XC D	3
NaCl	250
Barite	Weighting
PAL FLC	40
KCL	50
CONa	80
NanoFe_2O_3 0.75 gm	1.25
NanoFe_2O_3 1.5gm	2.5

Caustic soda is used to raise the pH value, and Soda ash is used to treat out calcium ions. PF-VIS and (XC D) Xanthan Gum are primarily a viscosity modifier. Sodium Chloride (NaCl) and Potassium Chloride (KCL) are used to increase the fluid density, inhibit hydrate formation, provide shale control, and prevent the dissolution of salt sections **(Hassan and Mohammed, 2023)**. PAL FLC is utilized to control filtration, and Barite is used for density. Also, a CONa was added to give better efficiency to the mud cake and thus maintain the stability of the well. After completing the preparation of the mud, its properties were measured in the laboratory, and the results were listed in **Table 3**. The properties were taken repeatedly, but at different temperatures (30 ˚C, 50 ˚C, and 70 ˚C) and with the addition of different concentrations of nano Fe_2O_3 (0 gm, 0.75gm ,1.5 gm) Mix them for at least 20 minutes using the Hamilton Beach mixer.

Table 3. Physical properties of drilling fluid

Fluid type	**HIBDRILL**
Density (g/cc)	2.28
FV (s)	50
PV (cp)	38
YP (lb/100ft^2)	4
ϕ600/ϕ300	76/40
ϕ200/ϕ100	29/16
ϕ6/ϕ3	3/2
Gel 10sec/10min (lb/100ft^2)	3/6
Cake thickness(mm)	1
API FL (mL)	4
pH	7.5
Cl^- (mg/L)	150000
Ca^{++} (mg/L)	100

The properties of Gamma nano Fe_2O_3 Have been listed in **Table 4**.

Table 4. Properties of Gamma nano Fe_2O_3

Purity	+99%
APS	20-40nm
Form	powder
Appearance	red
Manufacturer	Sky Spring Nanomaterials, Inc.

All tests of samples are done according to American Petroleum Institute **(API RP 13B-1, 2003)**. Many instruments and devices were used in laboratory work. They were manufactured by the Fann Instrument Company. Such as: Hamilton High Speed Mixer that used for mixing fluid and other additives, Electrical Weigh Balance and measuring cylinder that used to measure different samples of different chemical materials, Phydrion pH paper dispenser to test the acidity or alkalinity nature, Mud balance for measuring the weight of mud, Chemical solutions for measuring chloride ion content and total hardness as calcium, Six speed rotational viscometer to obtain the viscosity and gel strength, Filter press for measuring mud filtration and Others include marsh funnel, stop watch, mud cups. From Fann V-G Meter readings, rheological data are obtained. Linear regression analysis is run for these data to choose the model closest to the actual flow curve. By using Eq. (8), the model with the squared correlation coefficient **(Lenschow, 1992)** closest to unity will be chosen. In this research, a comparison was made between two models, which are: Bingham and the Power Law Model.

$$R^2 = \left(\frac{p*\sum X*Y-\sum X*\sum Y}{\sqrt{(P*\sum X^2-\sum X^2)((P*\sum Y^2-\sum Y^2))}}\right)^2 \quad (8)$$

Where x and y are the shear rate and shear stress. P is the number of fann readings made.

3. RESULTS AND DISCUSSION

This research is characterized by the combination of field work and laboratory work. About 340 cubic meters of salt-saturated mud were prepared to drill the salt section (Lower Fars Formation). Before drilling began, drilling fluid was contaminated with cutting of rock cuttings, and samples were taken to the laboratory and tested. Plastic viscosity, apparent viscosity, gel strength (10 sec and 10 min), and yield point are measured from the relationship between shear stress and shear rate. Results are shown in **Figs. 3 to 7** sequentially.

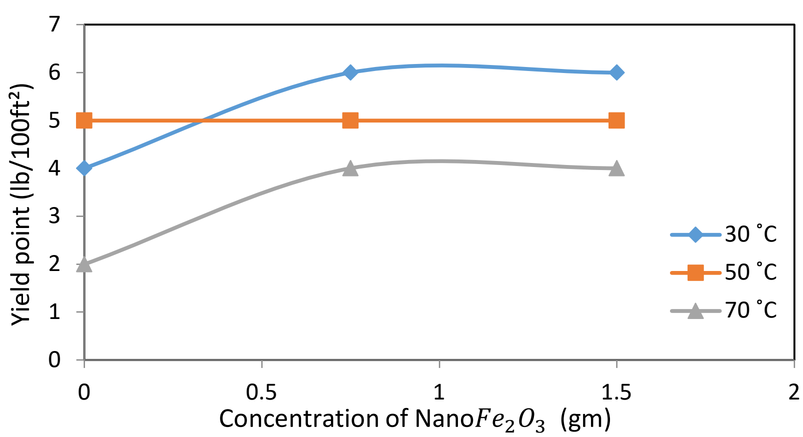

Figure 3. Yield point changes with nanomaterial concentration at different temperatures.

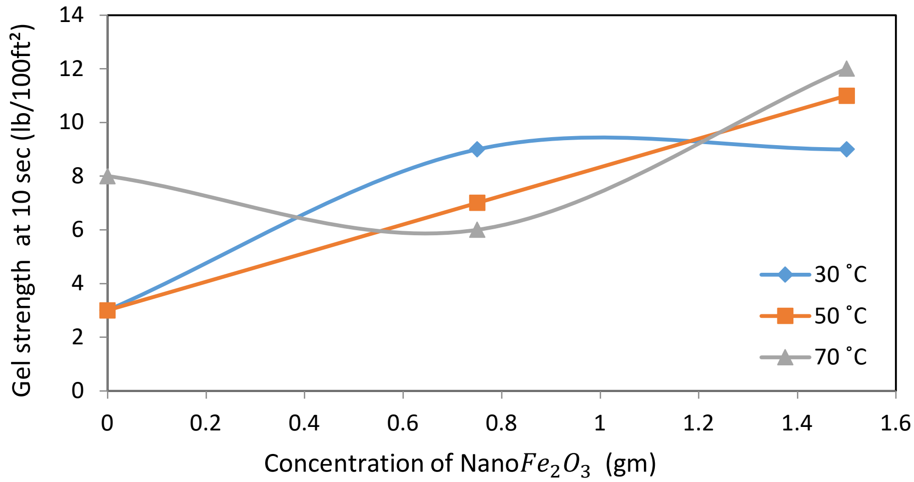

Figure 4. Gel strength at 10 sec changes with nanomaterial concentration at different temperatures.

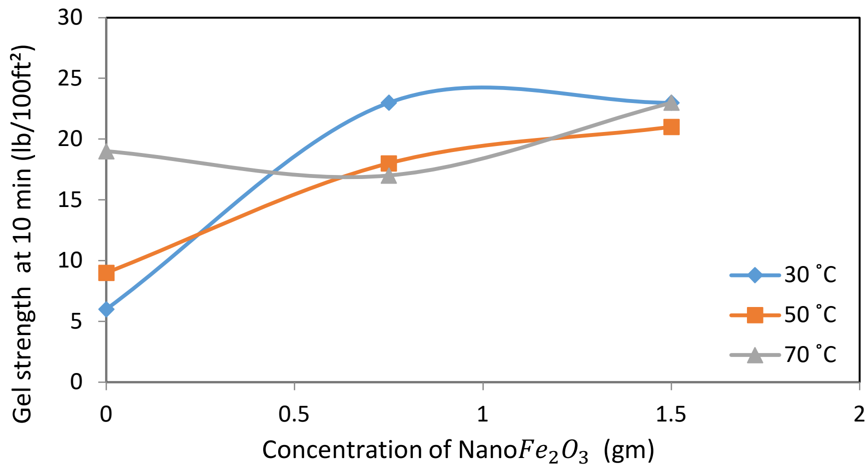

Figure 5. Gel strength at 10 min changes with nanomaterial concentration at different temperatures.

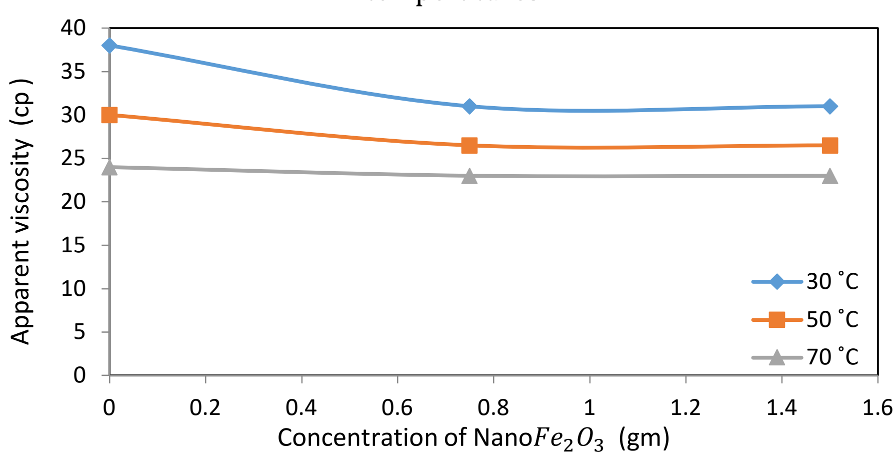

Figure 6. Apparent viscosity changes with nanomaterial concentration at different temperatures.

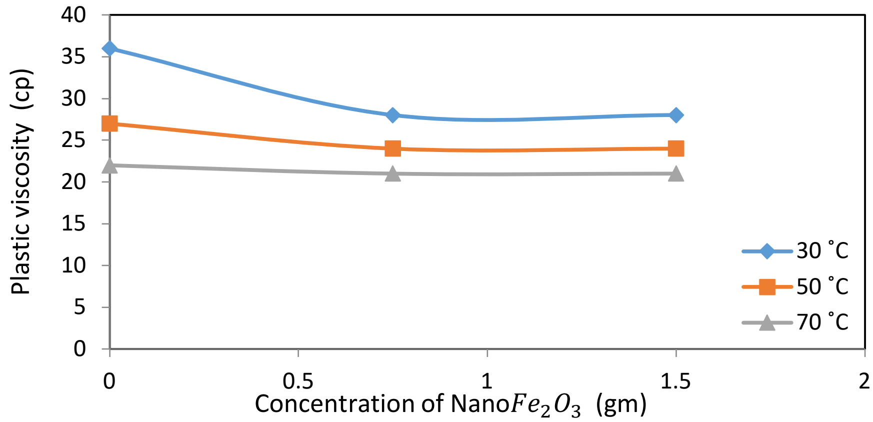

Figure 7. Plastic viscosity changes with nanomaterial concentration at different temperatures

The addition of nano Fe_2O_3 Leads to an increase in gel strength while the other rheological properties remained relatively constant. The results of R^2 for the two models are shown in **Table 5**. These models (power-law, Bingham) represent the relationship between shear stress and shear rate.

Table 5. Squared correlation coefficient of two rheological models

Sample	(R^2) Bingham Model	(R^2) Power Law Model
At 30 ˚C without Fe_2O_3	0.998	0.993
At 50 ˚C without Fe_2O_3	0.999	0.964
At 70 ˚C without Fe_2O_3	0.999	0.965
At 30 ˚C with 0.75 Fe_2O_3	0.998	0.949
At 50 ˚C with 0.75 Fe_2O_3	0.997	0.877
At 70 ˚C with 0.75 Fe_2O_3	0.997	0.958
At 30 ˚C with 1.5 Fe_2O_3	0.998	0.949
At 50 ˚C with 1.5 Fe_2O_3	0.999	0.909
At 70 ˚C with 1.5 Fe_2O_3	0.996	0.97

The effect of nano Fe_2O_3On drilling fluid, measured in terms of filtration and mud cake, is shown in **Figs. 8 and 9**.

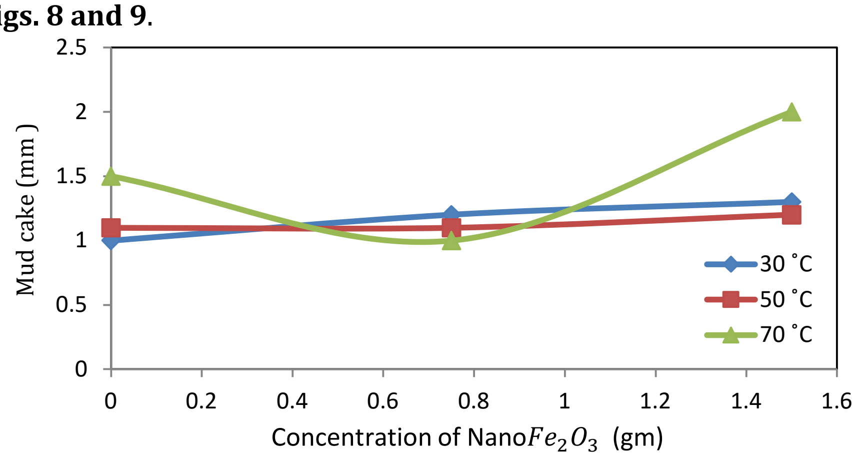

Figure 8. Mud cake changes with the nanomaterial's concentration at different temperatures.

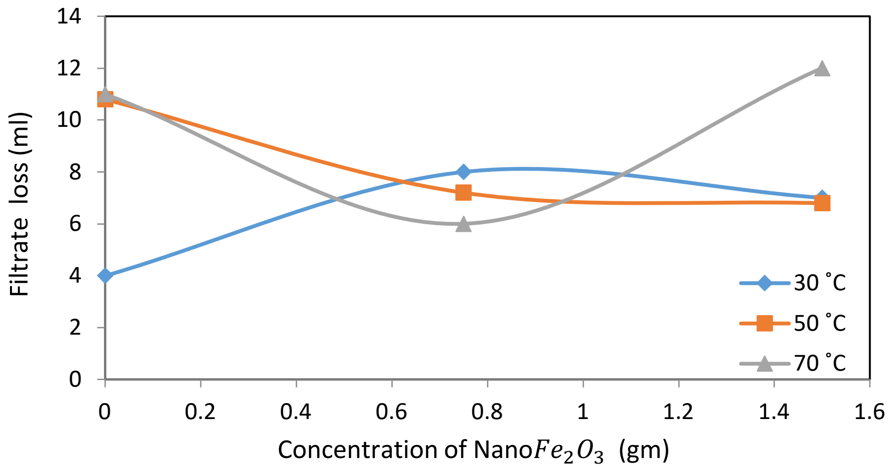

Figure 9. Filtrate loss changes with nanomaterial concentration at different temperatures.

The change in these properties is small except at high temperatures. Drilling mud filtration increases as the temperature rises due to: viscosity of filtrate decreases, change in electrochemical equilibria, and chemical degradation of components **(Peng, 1990)**. In general, the results of this research are similar to the results of previous studies in some specifications and are close to or different from them in other specifications. This is because this research was conducted on a drilling fluid saturated with salt and also of high density (2.28 gm/cc) and does not contain bentonite, while other studies **(Contreras et al., 2014; Jung et al., 2011; Barry et al., 2015; Alvi et al., 2020)** were conducted on fresh water bentonite-based drilling fluid with or without barite, KCl, and on oil-based mud. For example, when comparing the results of rheological properties with the results of other studies, despite the difference in the size of the nano Fe_2O_3, their concentrations, and the type of the drilling fluid, we find similarity, as adding nano Fe_2O_3 leads to an increase and improvement of the rheological properties. On the other hand, we can notice that the filtration increases at normal temperature but decreases when the temperature increases. This result is similar to the study made by **(Barry et al., 2015)**. But this rule does not apply when the concentration is 1.5 gm and the temperature is 70 ˚C, **Fig. 9.**

4. CONCLUSIONS

According to the results, we can conclude some points. These conclusions, although few, are very important because any change, even a small one, in the drilling fluid properties will affect all pressure loss measurements, drill fluid flow measurements, and also impact drilling speed and drilling efficiency:

- The experimental results show that an increase in temperature leads to an increase in gel strength due to the rise in electromechanical resistance and the addition of nano Fe_2O_3 Leads to another rise because of an increase in solid particles.
- Increasing the temperature decreases the yield point due to its effect on the polymers, but these effects decrease at 70 ˚C with nano Fe_2O_3. Therefore, we can take advantage of this feature because the lower Fars formation has high pressure and temperature.
- According to R2, the best model that represents the relationship between shear rate and shear stress is the Bingham model.

NOMENCLATURE

Symbol	Description	Symbol	Description
AV	Apparent viscosity, cp.	KCl	Potassium chloride
FL	Filtrate loss, ml.	PV	plastic viscosity, cp.
FV	Marsh funnel viscosity, sec.	R^2	squared correlation coefficient
Gel	Gel strength, lb/100ft^2.	YP	yield point, lb/100ft^2.

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Credit Authorship Contribution Statement

Aya Abaulkareem: Review & editing, Validation, and Supervising. Hassan Abdul Ameer: Writing –review & editing, Methodology, Laboratory work.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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التحري المختبري لأوكسيد الحديد النانوي على الخصائص الفيزيائية لطين الحفر المشبع بالملح

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الخلاصة

أصبحت عملية حفر الآبار أكثر تعقيدًا اليوم بسبب الأعماق الكبيرة وتقنيات الحفر المتقدمة. لذلك، أصبح الحصول على المواصفات المثالية لسوائل الحفر أمرًا ذا أهمية كبيرة للتغلب على مشاكل الحفر وخفض التكاليف. لذلك، بدأت الدراسات في التحري في تأثير المواد النانوية على سوائل الحفر. هذه الدراسة هي تحري في تأثير أكسيد الحديد النانوي على مواصفات سائل الحفر. تم استخدام سائل حفر المشبع بالملح وتم قياس الخصائص الفيزيائية المختلفة مثل اللزوجة والكثافة وفقدان الترشيح عند درجات حرارة مختلفة (30 درجة سيليزية، 50 درجة سيليزية، 70 درجة سيليزية) وإضافة نسب مختلفة من أكسيد الحديد النانوي (0.75 غرام، 1.5 غرام). تتميز هذه الدراسة بالجمع بين العمل الميداني والمختبري. أدى إضافة أكسيد الحديد النانوي إلى زيادة قوة الهلام بينما ظلت الخصائص الريولوجية الأخرى ثابتة نسبيًا وكان التغيير في الترشيح وكعكة الطين صغيرًا باستثناء عند درجة الحرارة العالية. يؤدي ارتفاع درجة الحرارة إلى انخفاض في الخصائص الريولوجية، باستثناء قوة الهلام. ولكن عند 70 درجة سيليزي، فإن إضافة اوكسيد الحديد النانوي يقلل من تأثير ارتفاع درجة الحرارة وخصوصا على نقطة المطاوعة وهذه خاصية مهمة عند حفر المقطع الملحي.

الكلمات المفتاحية: سائل الحفر المشبع بالملح، أوكسيد الحديد النانوي، الترشيح، الخصائص الريولوجية، تكوين فارس السفلي.