



Applicability of Fe₃O₄ nanoparticles for improving rheological and filtration properties of bentonite-water drilling fluids in the presence of sodium, calcium, and magnesium chlorides

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Abstract

There are impressive efforts in conjunction with improving rheological and filtration properties of Water-Based Drilling Fluids (WBDFs) employing Nano-Particles (NPs). However, NPs' performance in the presence of different salts has not been well assessed. This study intends to investigate the effect of Fe₃O₄ NPs on rheological and filtration properties of bentonite-water drilling fluids exposed to NaCl, CaCl₂, and MgCl₂ salts. To reach the goal specified for this study, three 0.5, 1 and 2 wt% NP concentrations, separately, were added into a salt-free and three salt-contaminated bentonite-water drilling fluids (four base fluids). So, 16 different drilling fluids were prepared for this research. The rheological models obtained by six shear rates, apparent viscosity, plastic viscosity, yield point, gel strength, and cutting carrying ability of all the drilling fluids are described in the paper. Moreover, API fluid losses (under 100 psi differential pressure), the cakes' thickness, and the cakes' permeability compared to the same as the salt-free base fluid, are interpreted to evaluate the NPs' performance on filtration control ability of all the drilling fluids. The results showed that the salts weaken the rheological and filtration properties of the salt-free base fluid, while Fe₃O₄ NPs sustain and improve the rheological properties of salt-free and salty drilling fluids, significantly. Nano-sized Fe₃O₄ weakens the filtration properties of the salt-free WBDF, but it is a suitable filtration control agent for salt-contaminated drilling fluids. In a sentence, nano-Fe₃O₄ is a suitable additive to enhance salty-WBDFs' performance.

Keywords Drilling fluid · Nanoparticle · Fe₃O₄ · Sodium chloride · Calcium chloride · Magnesium chloride · Rheology · Fluid loss

Introduction

Drilling fluid, as the drilling industry blood (Meng et al. 2012) is necessary for cooling and lubricating drill bit, formation damage prevention, cutting removal, plugging permeable formations (Zakaria et al. 2012), controlling formation pressure, stabilization and supporting wellbore (Al-Yasiri et al. 2015), and providing buoyancy (Nasser et al. 2013). Two major types of drilling fluids are Water-Based Drilling Fluids (WBDFs) and Oil-Based Drilling Fluids (OBDFs). The first fluid is the more environmentally

friendly and economical drilling fluid, and the second is the preferred drilling fluid for water-sensitive, complex, and high-temperature formations (Huang et al. 2016b).

Nowadays, due to the need for producing from unconventional reservoirs such as shale oil, shale gas, and deep-water reservoirs, the drilling industry requires advanced drilling technologies (Rafati et al. 2018) and designing advanced drilling fluids is a challenge. Nano-Particles (NPs) because of their higher ratio of surface area to volume in comparison to grater particles, have more strength, better electrical and thermal properties, and are more chemically reactive (Godson et al. 2010). Suitable properties of NPs motivate researchers to overcome drilling challenges utilizing nano-fluids. Nanofluid in the oil and gas industry is defined as any fluid containing at least one additive with 1–100 nm particle size (El-Diasty and Ragab 2013).

WBDFs are composed of different additives. Polymers and clay minerals usually provide rheology and reduce

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the fluid loss of these fluids. Finding new multifunction materials that can improve WBDFs properties and minimize materials consumption is still attractive for drilling fluid engineers. Different studies have been done on the application of NPs for improving the rheological and filtration control properties of WBDFs. Some of the oxide NPs effective on WBDFs properties include SiO₂ (Ghanbari et al. 2016; Mahmoud et al. 2016; Salih et al. 2016; Srivatsa and Ziaja 2011; Taraghikhah et al. 2015; Nasser et al. 2013; Baghbanzadeh et al. 2014; Hassani et al. 2016; Bayat et al. 2018; Medhi et al. 2020), ZnO (Ponmani et al. 2016; Hassani et al. 2016; Dehtaradon et al. 2019; William et al. 2014), CuO (Dehtaradon et al. 2019; William et al. 2014; Bayat et al. 2018; Medhi et al. 2020), TiO₂ (Parizad et al. 2019; Bayat et al. 2018), SnO₂ (Parizad and Shahbazi 2016), Al₂O₃ (Smith et al. 2018; Bayat et al. 2018), Fe₄O₃ (Amarfio et al. 2015), Fe₂O₃ (Jung et al. 2011; Mahmoud et al. 2016, 2017; Vipulanandan et al. 2017) and Fe₃O₄ (Vryzas et al. 2016). Besides, carbon-based NPs, including graphene (Kosynkin et al. 2011; Aftab et al. 2017) and Multi-Walled Carbon Nano Tube (MWCNT) (Aftab et al. 2017; Sedaghatzadeh et al. 2012; Fazelabdolabadi et al. 2015) can enhance WBDFs' formulation. Moreover, nano-clay minerals such as Attapulgite (Abdo 2014), sepiolite (Abdo et al. 2016; Needaa et al. 2016; Abdo and Haneef 2013), and palygorskite (Abdo and Haneef 2013) due to their structure can improve the performance of WBDFs. Also, nanocomposites such as Fe₃O₄-PSSS (Wang et al. 2018), TiO₂-polyacrylamide (Sadeghalvaad and Sabbaghi 2015), polymer-silica (Mao et al. 2015), polyacrylamide-clay (Jain et al. 2015), and ZnO-clay (Abdo et al. 2014) can be effective additives for WBDFs.

WBDFs usually contain salt as an inhibitor in their formulation to minimize shale formations swelling capacity. Also, the drilling fluid during drilling oil and gas wells maybe become contaminated by salts and ions. So, it is necessary to evaluate WBDFs' properties while salt presents in the system. Despite the significant advances in the application of NPs in WBDFs' formulation, NPs' performance in the presence of salts has not been reviewed comprehensively. This study aims to examine nano-Fe₃O₄ performance in salt-contaminated bentonite-water drilling fluids. Different concentrations of the NP are added into a base fluid formulation, and its effects on rheological and filtration properties are evaluated. Then, the NPs' impact is assessed in the systems containing three salts, including NaCl, CaCl₂, and MgCl₂.

Materials and methodology

Materials

The commercial components used in the drilling fluid formulation and the salts were provided by drilling fluid laboratory in Petroleum University of Technology (Ahwaz). Fe₃O₄ with 15–20 nm average particle size, brown color, and 4.9 gr/cm³ density was supplied by a chemical store in Ahwaz.

Drilling fluid preparation

Mixer Zhengyuan Tongda model JB-12KD with 8000 Round Per Minute (RPM) rotary speed was used to prepare uniform drilling fluids. Two separate scenarios were considered to investigate the NPs' effect on WBDFs' properties. The first procedure which belongs to investigate the impact of the NP in salt-free WBDFs is as following:

- (1) adding 28 gr sodium bentonite slightly into 350 cm³ distilled water, and mixing for 20 min,
- (2) addition of 0.1 gr caustic soda (NaOH) to provide an alkaline medium for other components, and mixing for 10 min (steps 1 and 2: salt-free base fluid),
- (3) adding different Fe₃O₄ concentrations (0.5, 1, and 2 wt%), and mixing for 10 min.

Also, to evaluate the NPs' performance in salty-drilling fluids, 2 wt% of each of the three salts NaCl, CaCl₂, and MgCl₂ were added into the salt-free fluid after the first step (bentonite mixing). Then, the other steps implemented are the same as the salt-free fluids. Finally, the density of all the drilling fluids was measured using a mud balance (the commonest apparatus for measuring drilling fluids' density) with an accuracy of 0.1 lb per gallon (ppg). Briefly, four drilling fluid categories were assessed during the experiments, namely salt-free, NaCl-contaminated, CaCl₂-contaminated, and MgCl₂ WBDFs.

Measuring rheological properties

The rheological properties of all the fluids were measured using Fann VG meter at ambient temperature (30 °C) based on API RP for Field Testing Water-based Drilling Fluids. All the drilling fluids were exposed to six rotational speeds, including 3, 6, 100, 200, 300, and 600 RPMs to measure their corresponding dial readings (equivalent to shear stresses). Subsequently, the measured dial readings were matched on both Bingham plastic and power-law models (Eqs. 1–2), as the most well-known and most straightforward rheological models applicable for drilling fluids. The

regression coefficient values (R^2) describe the models' accuracy to predict the rheological behavior of all the drilling fluids. Experimentally values of apparent viscosity, plastic viscosity, and yield point, as the Bingham plastic parameters were calculated utilizing Eqs. 3, 4, and 5, respectively. Also, the maximum dial reading at RPM=3 after 10 s and 10 min, respectively, is considered as Gel-10 s (initial gel strength) and Gel-10 min. (final gel strength).

$$\tau = \tau_0 + \mu_p \times \dot{\gamma} \quad (1)$$

$$\tau = k \times \dot{\gamma}^n \quad (2)$$

$$AV = \frac{\theta_{600}}{2} \quad (3)$$

$$PV = \theta_{600} - \theta_{300} \quad (4)$$

$$YP = \theta_{300} - PV \quad (5)$$

τ is the shear stress in lb/100ft², $\dot{\gamma}$ is the shear rate in (s⁻¹) (1 s⁻¹ = 0.587 RPM), τ_0 is the threshold stress in lb/100ft², μ_p is plastic viscosity in cp (mPa.s). Apparent viscosity, plastic viscosity and yield point, which are respectively named AV, PV, and YP (the threshold stress), are consequently in cp, cp, and lb/100 ft². Besides, both initial and final gel strengths have the same dimension of YP.

Cuttings carrying efficiency

Hole cleaning is one of the most essential functions of drilling fluids. A clean hole is the sign of the efficient mud circulation system. Cuttings Carrying Index (CCI), shown in Eq. 6, is a dimensionless parameter to describe the drilling fluid ability to transport drilling cuttings from bottomhole to the surface. A CCI greater than or equal to unity indicates the appropriate hole cleaning, but a CCI value equal to or below 0.5 reveals the poor hole cleaning (Okon et al. 2015).

$$CCI = \frac{K \times V_{ann} \times MW}{400000} \quad (6)$$

In this equation, K is the fluid consistency index (power-law constant), determined by using Eq. 7. V_{ann} is the annular velocity in ft/min. MW is the drilling fluid density in ppg.

$$k = 511^{1-n} \times (PV + YP) \quad (7)$$

where as PV and YP are the same parameters of the Bingham plastic model, and respectively are, in cp and lb 100ft². n is the flow behavior index (dimensionless). Equation 8

calculates the flow behavior index as a function of PV (in cp) and YP (in lb/100ft²).

$$n = 3.322 \times \log \left(\frac{2 \times PV + YP}{PV + YP} \right) \quad (8)$$

It is easy to compare the studied drilling fluids' potential from the hole cleaning aspect by assuming a constant/equal annular velocity during the drilling operation for all the drilling fluids. So, CCI of the drilling fluids are divided by the CCI corresponds to the salt-free base fluid. The normalized CCI values are obtained by Eq. 9.

$$\text{Normalized CCI} = \frac{K \times MW}{K_{base} \times MW_{base}} \quad (9)$$

K and MW : consistency index and the drilling fluid density,

K_{base} and MW_{base} : consistency index and density of the salt-free base fluid.

Normalized CCI values higher and smaller than unity reveal, respectively, the more and less lifting capacity of the drilling fluid compared to the salt-free base fluid.

Measuring filtration properties

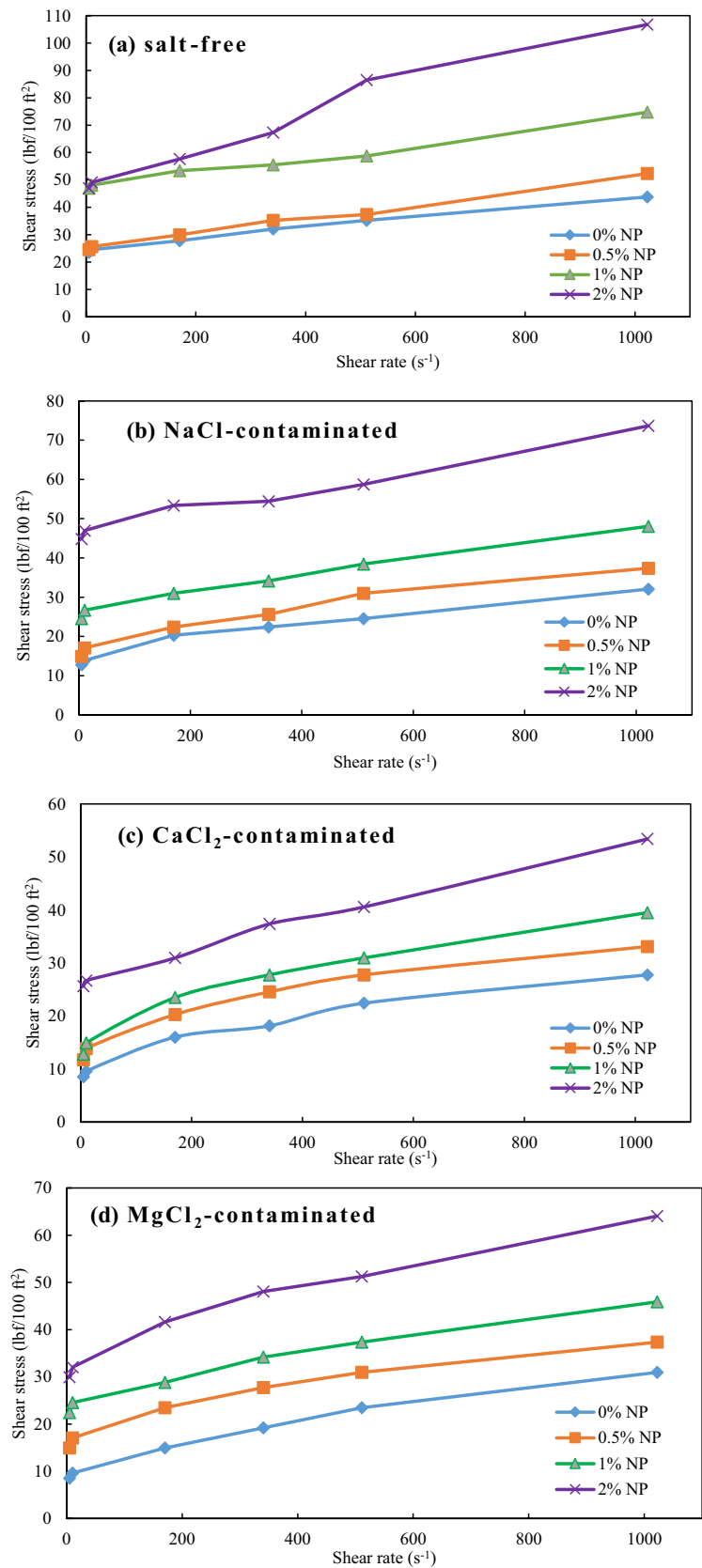
Filtration test under 100 psi differential pressure (API condition) is carried out to simulate top hole condition for the drilling fluid circulated in the mud system. Often, water-based drilling fluids are used during the drilling of the top holes. Moreover, API filtration test provides an easy, effective way to evaluate the filtration properties of a drilling fluid. So, API filtration test was implemented as a static test to predict fluid loss control ability of all the drilling fluids according to API RP for Field Testing Water-based Drilling Fluids. Also, cake thickness of all the samples was measured utilizing a vernier caliper immediately after the filtration measurements.

Permeability ratio of two mud cakes is compared by using Eq. 10 (Kök and Bal 2019).

$$\frac{K_2}{K_1} = \frac{Q_2 \times L_2}{Q_1 \times L_1} \quad (10)$$

K_i , Q_i , and L_i , respectively, correspond to permeability, filtrate volume after 30 min, and cake thickness due to the fluid i . In order to compare the cakes' permeability in this study, $i = 1$ refers to the salt-free base fluid and $i = 2$ is used for all the other drilling fluids.

Fig. 1 Rheological behavior of: **a** salt-free drilling fluids, **b** NaCl-contaminated drilling fluids, **c** CaCl₂-contaminated drilling fluids, **d** MgCl₂-contaminated drilling fluids



Results and discussion

Rheological behavior

Figure 1(a) illustrates the effect of Fe_3O_4 NPs on the rheological behavior of the salt-free base fluid. As the figure shows, the NPs increase significantly shear stresses of the base fluid at all the shear rates. To precise the significant enhancement in shear stress of the salt-free fluid provided by the NPs: iron oxide NPs are stable in bentonite-water suspensions with positive charges (Vryzas et al. 2016). So, in the cases of adding Fe_3O_4 NPs into the salt-free fluid, the positive charges (electrostatic repulsion) in the host fluid increase so much that they overcome such attractive forces and form stable colloidal suspensions (Vryzas and Kelesidis 2017). Consequently, the resistance against the fluid deformation increases, which results in more significant shear force (or shear stress) at all the shear rates. According to the regression coefficients reported in Table 1, all the salt-free drilling fluids accurately reveal the Bingham plastic behavior.

Figure 1(b–d) show the NPs' effect on the rheological behavior of the base fluid contaminated respectively by NaCl , CaCl_2 , and MgCl_2 salts. According to the figures, all the salts used in this study reduce the shear stress of the salt-free base fluid and move the salt-free fluid's rheogram down. The negative charge of bentonite-water suspensions has been proven using zeta potential test (Mekhamer 2011; Huang et al. 2016a). The reduction in shear stress of the base fluid due to the salts is a result of neutralization of a fraction of the negative charges present in the base fluid. The presence of Ca^{2+} , Mg^{2+} , and Na^+ results in disruption of bentonite charges' balance as well as bentonite particles flocculation. So, the resistance against the fluid deformation as well as the shear stresses decreases. The minimum reduction in the shear stresses among the three cations is due to Na^+ , which is a monovalent cation weaker than Ca^{2+} and Mg^{2+} (divalent cations) from the aspect of neutralizing bentonite-water suspension negative charges. Like the salt-free cases, there is a proportional relation between Fe_3O_4 concentration and shear stress in all the salty-drilling fluids. Briefly, the cation-NP combinations lead to changing the net charges in the bentonite-water suspension from negative to

Table 1 Regression coefficients (R^2) corresponding to Bingham plastic and power-law models for all the fluids

Fluid type	Rheology model	Fe_3O_4 concentration				Average R^2
		0%	0.5%	1%	2%	
Salt-free	Bingham plastic	0.9896	0.9934	0.9847	0.9744	0.9855
	Power-law	0.8385	0.8103	0.7449	0.8135	0.8018
NaCl -contaminated	Bingham plastic	0.9400	0.9439	0.9843	0.9786	0.9617
	Power-law	0.9602	0.9353	0.8632	0.7976	0.8890
CaCl_2 -contaminated	Bingham plastic	0.9223	0.8931	0.9170	0.9919	0.9310
	Power-law	0.9756	0.9800	0.9819	0.8370	0.9436
MgCl_2 -contaminated	Bingham plastic	0.9614	0.9142	0.9650	0.9457	0.9465
	Power-law	0.9501	0.9696	0.8989	0.9453	0.9409

Fig. 2 Apparent viscosity of salt-free and salty drilling fluids

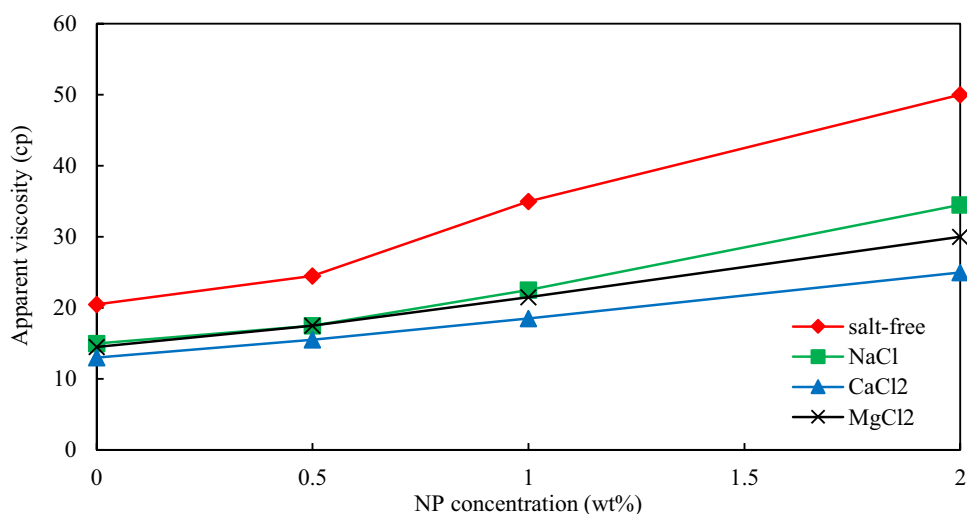
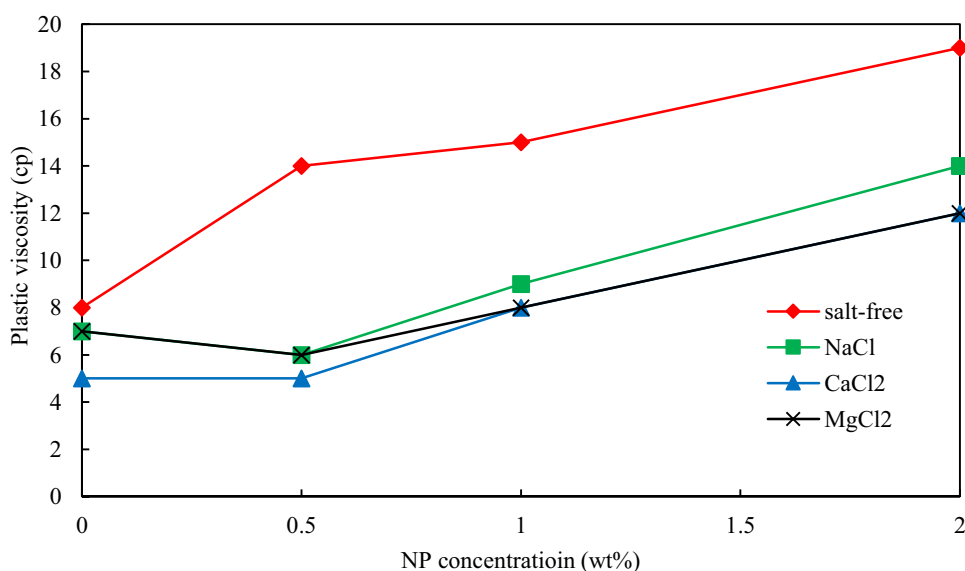


Fig. 3 Plastic viscosity of salt-free and salty drilling fluids

positive. So, the contaminated bentonite-water suspensions containing the NPs are stable like the equivalent salt-free fluids. Considering the regression coefficients listed in Table 1, minus the NP-free case, all the NaCl-contaminated drilling fluids follow the Bingham plastic model. Besides, among the CaCl₂-contaminated drilling fluids, only the fluid containing 2% Fe₃O₄ follows the Bingham plastic model more accurately than the power-law model. Moreover, the average R-squared values equal to 0.9465 and 0.9409, respectively corresponding to Bingham plastic and power-law models for Mg-contaminated cases, illustrate that Bingham plastic model is slightly more accurate than the power-law to predict rheological behavior of the MgCl₂-contaminated drilling fluids (excepting the case of 0.5% NP concentration). Eventually, in a sentence, since 11 samples of all the 16 studied drilling fluids follow the Bingham plastic model more accurately than the power-law model, this study will investigate Fe₃O₄ impact on the classical parameters of the Bingham plastic model.

Apparent viscosity

Apparent Viscosity (AV) is defined only for non-Newtonian fluids; it is the viscosity at each shear rate assuming Newtonian behavior. This study investigates AV at 600 rotational speed (corresponding to $\sim 1022 \text{ s}^{-1}$; the API recommended shear rate for AV). AV values of all the drilling fluids, as a parameter effective on the rate of penetration (ROP) during drilling (Meng et al. 2012), have been shown in Fig. 2. As can be seen in the figure, as Fe₃O₄ concentration increases, AV of the salt-free and the three salt-contaminated fluids is improved to a higher magnitude. This impressive finding is the sign of the iron oxide NPs' ability to support the bentonite used, even in harsh environments. A salt-free fluid in

comparison to a salty fluid at the same NP concentration has a higher AV. The fewer AVs of the salty base fluids in comparison to the same as the salt-free base fluid is an outcome of the neutralization of bentonite surface negative charges through Ca²⁺, Mg²⁺, and Na⁺ cations. Among the salty fluids, NaCl contamination has the minimum negative effect on AV of the salt-free base fluid because of the less positive charge of monovalent Na⁺ compared to divalent Mg²⁺ and Ca²⁺ cations. Besides, Ca²⁺ is more reactive than Mg²⁺, because it is coarser than Mg²⁺. So, CaCl₂ contamination harms the base AV more than MgCl₂. Finally, the sequence of the salts from reducing AV point of view is as following; CaCl₂, MgCl₂, and NaCl. According to the AV trends over the NPs concentration, Fe₃O₄ overall is more effective in enhancing AV of, respectively, NaCl-contaminated,

Table 2 Yield point and gel strength of all drilling fluids

Fluid type	Properties (lb/100 ft ²)	Fe ₃ O ₄ concentration (wt%)			
		0	0.5	1	2
Salt-free	YP	25	21	40	62
	Gel-10 s	26	28	52	55
	Gel-10 min	35	46	80	89
NaCl-contaminated	YP	16	23	27	41
	Gel-10 s	13	14	27	50
	Gel-10 min	21	23	36	63
CaCl ₂ -contaminated	YP	16	21	21	46
	Gel-10 s	9	11	12	24
	Gel-10 min	10	14	17	37
MgCl ₂ -contaminated	YP	15	23	27	16
	Gel-10 s	11	12	22	29
	Gel-10 min	16	19	31	47

MgCl₂-contaminated, and CaCl₂-contaminated WBDFs. For instance, 2% NP provides 137.9%, 106.9%, and 92.3% improvement in AV of the base fluid respectively contaminated by NaCl, MgCl₂, and CaCl₂.

Plastic viscosity

Plastic viscosity is one of the most effective parameters for cuttings transportation. It is affected by the mechanical friction sources in the drilling fluid, including the size, shape, and concentration of the solids and the fluid phase viscosity (Amoco-production-company 2010). As shown

Table 3 Flow behavior index, consistency index, and density (effective on cuttings carrying)

Fluid type	Parameter	0% Fe ₃ O ₄	0.5% Fe ₃ O ₄	1% Fe ₃ O ₄	2% Fe ₃ O ₄
Salt-free	n	0.313	0.485	0.348	0.304
	K	2391.956	866.404	3209.523	6216.005
	MW(ppg)	8.7	8.8	8.8	8.9
NaCl-contaminated	n	0.383	0.271	0.322	0.327
	K	1076.245	2728.996	2470.516	3653.125
	MW(ppg)	8.8	8.9	8.9	9.0
CaCl ₂ -contaminated	n	0.308	0.254	0.351	0.396
	K	1570.715	2729.604	1655.248	1643.765
	MW(ppg)	8.8	8.9	8.9	9.0
MgCl ₂ -contaminated	n	0.120	0.082	0.089	0.155
	K	5319.857	8904.756	10,241.300	5445.518
	MW(ppg)	8.8	8.9	8.9	9.0

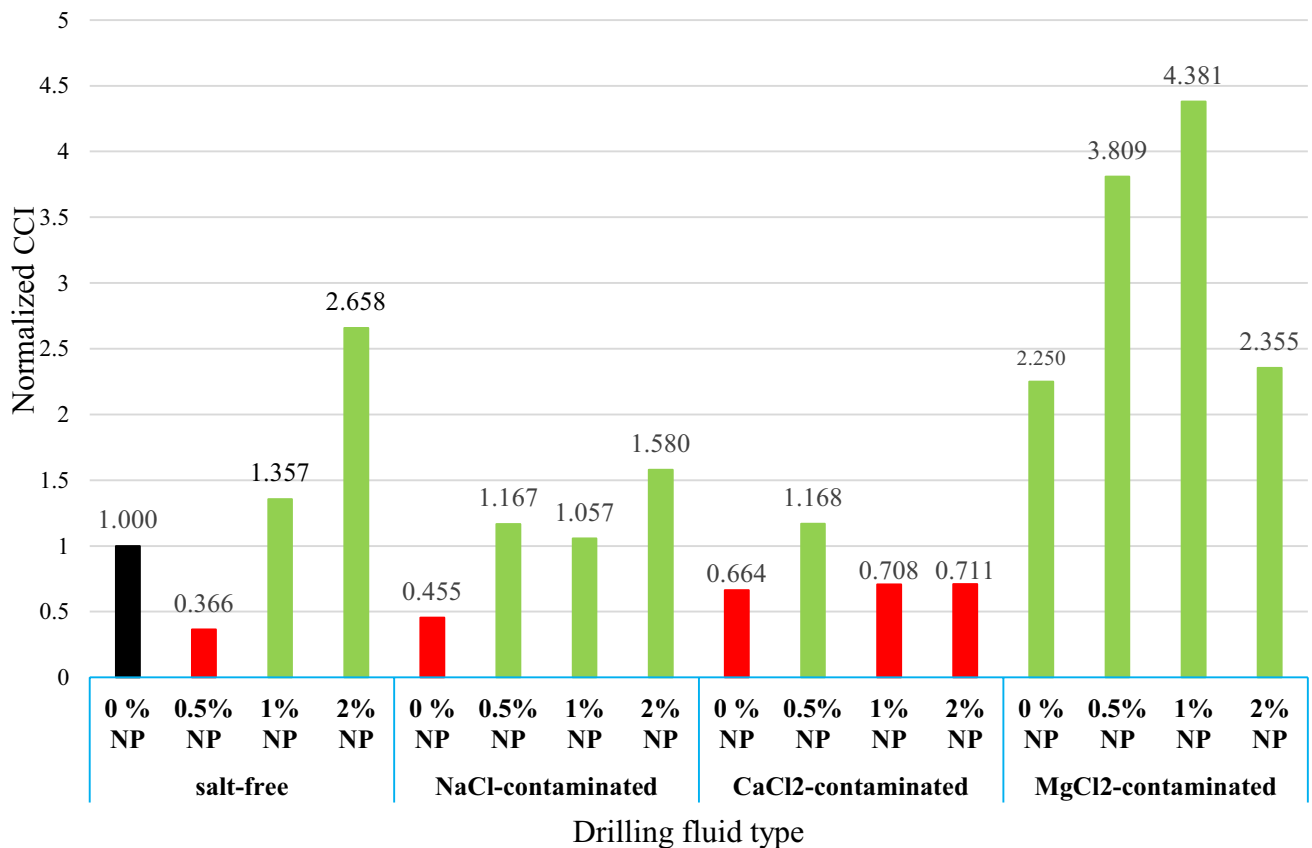


Fig. 4 Normalized CCI values

in Fig. 3, increasing the NP concentration leads to increasing PV of the base fluid. For example, the addition of 2% Fe_3O_4 into the salt-free base fluid improves the base PV by 137.5%. This improvement is due to the increase in solid content, which influences the solids' collision and mechanical fictions present in the drilling fluid. Overall, the trend of the salty fluids' PV vs. NP concentration is similar to the same as AV values. However, there is not any improvement in the PV of the salty base fluids at 0.5 wt% Fe_3O_4 concentration. The justification of the negative impacts of the salts and the positive effect of the NPs on PV of the base fluids is the same described in conjunction with AV values. PV of the NaCl-contaminated drilling fluid containing 2% NP equals to 14 cp, which is 16.7% higher than the same as both other equivalent contaminated fluids. Overall, the salts perform as inhibitors for the bentonite used, but Fe_3O_4 NPs act in the vice versa. To clarify the significant enhancement in PV provided by the NPs; bentonite layers are separated and stabilized by Fe_3O_4 particles and finally offer a higher viscosity level for WBDFs.

Yield point and gel strength

Yield point is related to electrostatic attraction forces associated with the drilling fluid particles at dynamic conditions,

but gel strength indicates the forces at static conditions. Yield point, Gel-10 s, and Gel-10 min. of all the drilling fluids have been listed in Table 2. The three salts used, reduce YP and gel strength of the salt-free base fluid. The reduction in YP and gel strength is due to the removal of attraction forces present in the fluid by use of the sodium, calcium, and magnesium cations, as precipitants (Amoco-production-company 2010). Overall, Fe_3O_4 NPs enhance the salt-free and salty base fluids' ability for cutting suspension through increasing their YP and gel strength. The ability of the NPs to improve yielding tendency and gel strength is higher for salt-free fluids compared to the salty samples. Increasing the NPs concentration leads to providing more Gel-10 s and Gel-10 min., but the relation between NP concentration and YP does not follow a specific trend. Using the NPs causes an increase in the positive charges (due to the iron oxide particles) present in the fluids and consequently improving the attraction forces effective on YP and gelation property. So, YP and gel strength are enhanced due to the bentonite sheets separation by Fe_3O_4 NPs.

Cuttings carrying efficiency

Flow behavior index, consistency index, and density of all the drilling fluids (the effective parameters on CCI) have been tabulated in Table 3. The normalized CCI values

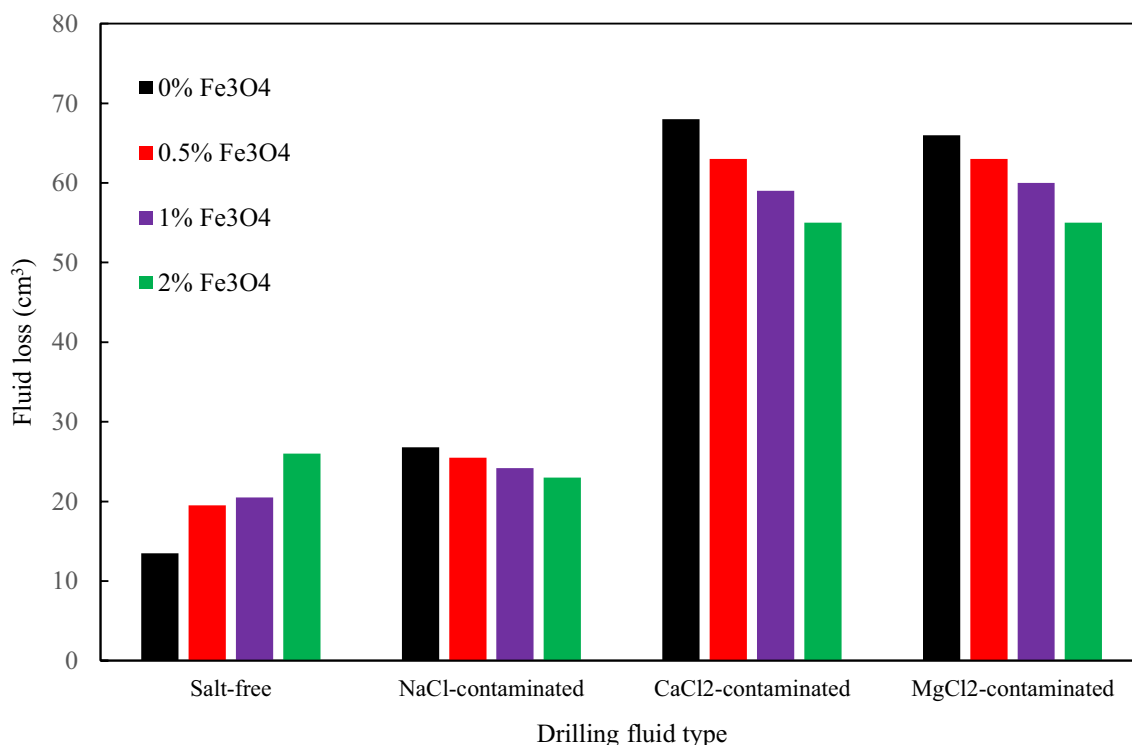


Fig. 5 Fluid loss for salt-free and salty drilling fluids in the presence of different Fe_3O_4 concentrations

obtained for all the studied fluids are used to compare the fluids' performance to clean the hole. The normalized CCI of 1.000 indicates that the drilling fluid ability to transport cuttings is the same as the salt-free base fluid. Normalized CCIs higher than 1.000 demonstrate that the drilling fluid is more potent than the salt-free base fluid to clean the hole. Besides, a normalized CCI less than 1.000 is the indicator of the unsatisfactory performance of the drilling fluid in comparison to the salt-free base fluid from the hole cleaning aspect. Figure 4 illustrates the normalized CCIs correspond to all the WBDFs. Among the salt-free WBDFs, both fluids containing 1 and 2% NP have higher CCIs over the base fluid, so Fe_3O_4 at medium and high concentrations is a potential agent to improve cutting transport ability of non-contaminated WBDFs. NaCl harms cuttings removal ability of the base fluid, but the addition of any Fe_3O_4 leads to complete retrieving the undesirable effect of NaCl. Moreover, Fe_3O_4 improves CCI of the CaCl_2 -contaminated base fluid, but only the salty WBDF of 0.5% NP can clean the hole more efficiently than the salt-free base fluid. The surprising effect of Fe_3O_4 on CCI enhancement belongs to

the MgCl_2 -contaminated WBDFs, in which all of them have normalized CCI values more significant than the salt-free base fluid. In a sentence, Fe_3O_4 is a potential additive to enhance cuttings lifting capacity of salt-free and contaminated WBDFs.

Filtration control

A suitable drilling fluid controls fluid loss into the porous and permeable formations by sealing them through forming a thin and impermeable cake on the wellbore wall. Figure 5 and Table 4, respectively, indicate filtrate volume and cake thickness for all the drilling fluids investigated in this study. Among the salt-free drilling fluids, both the cakes' thickness and the filtrate volumes are proportional to the NPs concentration. Scanning Electron Microscopy (SEM) analysis of the mud cake in the absence and presence of 2% Fe_3O_4 , shown in Fig. 6, indicates that the NPs lead to local flocculation of the bentonite sheets. Accordingly, as can be found in Table 5, the salt-free cakes made by the NPs have permeability ratios more than unity, so they are more permeable than the cake due to the salt-free base fluid. All these reasons mentioned, justify the increases in the filtrate of the salt-free base fluid by different NP concentrations. It must be noted that the NPs have been dispersed in the salt-free base fluid by using a rotational mixer (the commonest laboratory apparatus applicable for drilling fluid preparation) nor other reliable methods such as ultrasonication technique. So, the increase in the cakes' thickness is due to the instability of a portion of the NPs suspended in the fluid during the filtration tests.

As can be demonstrated from Tables 4–5 and Fig. 5, like the Fe_3O_4 NPs, the three salts increase the cake size,

Table 4 Cakes' thickness (mm)

NP concentration (wt%)	Drilling fluid type			
	Salt-free	NaCl-contaminated	CaCl_2 -contaminated	MgCl_2 -contaminated
0	0.23	0.33	0.80	0.80
0.5	0.25	0.31	0.75	0.75
1	0.29	0.30	0.72	0.73
2	0.30	0.27	0.70	0.68

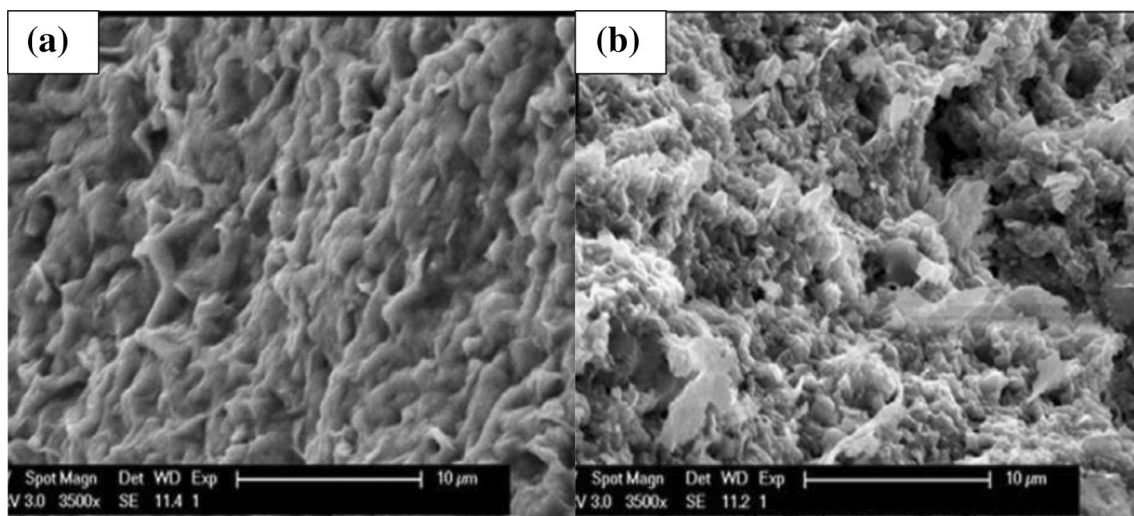


Fig. 6 SEM image of mud cake; **a** in the absence of Fe_3O_4 , **b** in the presence of Fe_3O_4

Table 5 Permeability ratio of the studied fluids

Fluid type	NP concentration (wt%)			
	0	0.5	1	2
Salt-free	1.000	1.570	1.915	2.512
NaCl-contaminated	2.848	2.546	2.338	2.000
CaCl ₂ -contaminated	17.520	15.217	13.681	12.399
MgCl ₂ -contaminated	17.005	15.217	14.106	12.399

permeability, and filtrate volume of the non-contaminated base fluid. NaCl, CaCl₂ and MgCl₂, respectively, increase the base fluid loss from 13.5 cm³ up to 26.8, 68.0, and 66.0 cm³. So, NaCl has the minimum negative effect on salt-free fluid loss.

In contrary to the salt-free cases, the Fe₃O₄ NPs decrease the cake thickness, permeability ratio, and filtrate volume of the salty-WBDFs. Moreover, as NP concentration increases, the fluid loss in all the salty fluids decreases. However, the NPs' ability overall is not enough to overcome the negative effects of the three salts, completely. Among the salty WBDFs, the NaCl-contaminated fluids have the smallest permeabilities, cakes' thickness, and filtrate volumes compared to the equivalent fluids made of other both salts. The best performance of Fe₃O₄, as filtrate reducer in salt-contaminated WBDFs, belongs to the CaCl₂-contaminated fluid contained 2 wt% NP, which has a filtrate volume 19.1% fewer than the NP-free fluid contaminated by the salt. Briefly, despite the salt-free WBDFs, Fe₃O₄ is a suitable filtration control agent for salt-contaminated WBDFs.

Conclusions

A feasibility study was done on the performance of Fe₃O₄ NPs on the rheological and filtration properties of bentonite-water drilling fluids in the absence and presence of three salts, including NaCl, CaCl₂, and MgCl₂. According to the experiments conducted, the following results have been concluded:

Fe₃O₄ NPs increase the shear stress of all the salt-free and salty drilling fluids.

Na⁺, Ca²⁺, and Mg²⁺ reduce the negative charges present in the bentonite-water suspension, so they flocculate the bentonite layers and decrease the shear stress of the salt-free fluid at all the shear rates.

Fe₃O₄ NPs provide an overall positive charge for the bentonite-water suspension, so they enhance the shear stresses correspond to all the shear rates.

Among all the 16 drilling fluid types investigated, 11 samples follow the Bingham plastic behavior model more accurately than the Power-law model.

Overall, the three salts decrease the apparent viscosity, plastic viscosity, yield point, initial gel strength, and final gel strength of the salt-free base fluid as a result of their cations' role in flocculating the bentonite sheets. Fe₃O₄ NPs enhance the rheological properties of WBDFs in either salt-free and salty systems, but their performance is better in salt-free systems.

According to the normalized CCI values, the Fe₃O₄ NPs improve the cutting carrying capacity of all the base fluids investigated, including the salt-free base fluid and the three salty base fluids. The best and weakest performance of the NPs from the hole cleaning aspect belongs to the fluids contaminated by MgCl₂ and CaCl₂, respectively.

Fe₃O₄ NPs and the three salts separately provide high-permeable and thicker mud cakes as well as increase fluid loss compared to the salt-free WBDF.

Combining the NPs with each of the salts leads to reducing the cakes' permeability and thickness, and finally, filtrate volume.

Generally, NaCl salt has the minimum negative impacts on the rheological and filtration properties of WBDFs among the salts used, while CaCl₂ provides the hardest environment for WBDFs. Nano-sized Fe₃O₄ particles could be applied to improve the rheological and filtration properties of the salt-contaminated systems.

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