Investigating the effects of nano-Fe$_3$O$_4$ and MWCNTs on the filtration and rheological properties of water-based muds at elevated temperature and pressure

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HIGHLIGHTS

• HPHT well drilling often faces fluid loss issues, commonly addressed with expensive and environmentally harmful OBMs.
• Cheaper and more eco-friendly WBMs degrade quickly under HPHT conditions.
• This study used nanomaterials (MWCNTs, Fe$_3$O$_4$) to improve WBM filtration at high temperature and pressure.
• MWCNTs showed significant promise, reducing filtrate volume and enhancing filtration properties for high-temperature applications.

ABSTRACT

Thermal degradation of water-based muds is a common occurrence while drilling wells in elevated to high temperature and pressure formations. Mud breakdown is usually accompanied by changes in the composition of the mud and subsequent loss of properties required by the mud to perform its functions optimally. Water-based muds are cost effective and environmentally compatible but degrade at higher temperatures. Thus, the need for continuous research for water-based muds with improved performance that can meet the technical requirements of drilling at higher temperatures. This study investigated the effects of two nanomaterials, Multi-Walled Carbon Nanotubes and Iron (II, III) Oxide (Fe$_3$O$_4$) on the filtration and rheological properties of WBMs at elevated temperature and pressure (150°C and 500 psi). Conventional additives were used with the nanomaterials at 0.2, 0.5 and 0.8 wt% concentrations to prepare the nano-mud systems which were aged in a static oven at 150°C for 16 hours before conducting filtration tests at 500 psi and 150°C and rheological tests at ambient temperature and pressure. The morphology of the filter cake samples was investigated using SEM and the permeability was obtained using an existing correlation. The nanomaterials enhanced the filtration properties with carbon nanotube muds exhibiting considerable improvement at all concentrations. The highest improvement was obtained with 0.8 wt% Multi-Walled Carbon Nanotubes with a 15.79 % reduction in filtrate volume relative to the base mud.
while a slight improvement was achieved with 0.2 wt% FeO₄. The carbon nano tube mud systems also gave lower filter cake thicknesses and permeability values relative to the base mud and the FeO₄ system. The nano-mud samples exhibited lower rheological properties relative to the base mud for all the measured parameters (10-second gel strength, 10-minute gel strength, yield point and plastic viscosity) indicating improved dispersion of the mud particles. The results obtained indicate the suitability of these nanomaterials for the modification of water-based mud properties at elevated temperature and pressure conditions.

1. Introduction

Breakdown of drilling mud additives can occur at elevated temperatures and pressures leading to degradation of the mud and impairment of mud properties. Degraded muds will fail to perform the required functions optimally leading to associated challenges such as filtrate loss, formation damage, barite sag, stuck pipe, inadequate hole cleaning and low rate of penetration among other challenges [9,11,16,45,61,74]. Drilling muds are therefore designed and formulated to retain stability by preserving their rheological and filtration properties during drilling operations to ensure that the fluids can meet up with the technical challenges of drilling wells in elevated temperature and pressure terrains.

Oil-based muds (OBMs) have been the fluids of choice for elevated to HPHT applications in terms of high technical performance due to their inherent desirable properties such as preservation of mud properties, shale stabilisation and drilling of gauge/near gauge wellbores. However, OBM are expensive and detrimental to the environment so the use of OBM is either prohibited or severely restricted in many areas due to the toxic nature of the base fluids and concerns associated with disposal of spent mud. Consequently, increasing awareness and more stringent environmental regulations have led to an increase in the use of water-based muds (WBMs) for elevated temperature and pressure applications ([16,18,30,36,45]; [94]).

WBMs are the most extensively used drilling fluids; generally easy to formulate, inexpensive to maintain and can be easily modified to overcome most drilling problems. However, conventional WBMs are generally not as technically efficient as non-aqueous mud systems in elevated temperature fields due to their relatively poor shale inhibition, lubricity and thermal stability characteristics. The drawbacks associated with WBMs, as well as the need for affordable and environmentally friendly options have led to intensified efforts in research towards the development and identification of drilling fluid additives that can improve WBM properties to compete favourably with OBM and synthetic-based muds (SBMs) in terms of technical efficiency [16,18,34,36,40,57,61,93].

The application of nanomaterials for the modification of drilling mud properties has been under investigation since the early 2000s [69,75]. The addition of NMs to drilling fluids improves the properties of the fluid in terms of thermal stability, rheology and hole-cleaning capabilities, fluid loss reduction and prevention of formation damage and minimising shale hydration and wellbore instability. Using NMs in drilling fluid formulations enables the mud engineer to control the mud properties by adjusting the type, size and quantity of nanomaterials in drilling fluid to cater for special needs. Furthermore, less proportion of nanomaterials are required relative to micro-sized additives to achieve a similar effect, due to the large surface area per volume of nanomaterials. The small volumes required increase the rate of penetration, decreasing the time for drilling and drilling-related activities ([8,17]; [21]; [22,57,68,81]).

Different types of NMs including nano-ZnO, CuO, SiO₂, FeO₄, FeO₃, Al₂O₃, MgO and Multi-Walled Carbon Nanotubes (MWCNTs) have been used by different researchers for the modification of WBM properties in order to improve the technical performance of the muds with varying degrees of improvement [12,18,28,36,51,57,72,93].

FeO₄ nanoparticles and MWCNT have been used for the modification of WBMs in diverse studies. FeO₄ nanoparticles possess high chemical stability and exhibit a very high surface area to volume ratio providing more surface area for reaction with other mud additives. These nanoparticles are also readily available and cheap, have low toxicity and are biodegradable [25,31,54,65]. Similarly, MWCNTs improve the strength of materials even when added in small quantities and they possess high thermal stability and chemical stability. MWCNTs are also able to enhance the thermal conductivity of the mud such that the heat being generated is dissipated, consequently reducing the mud temperature and allowing the mud to maintain its fluidity [24,38,44,76,91]. The large specific surface area also improves the interaction of the tubes with other mud additives [27] and the high aspect ratios enables the long tubes to interact with each other so that smaller quantities of the tubes are required [3,61].

The properties discussed informed the decision to use FeO₄ and MWCNTs for this study.

Although FeO₄ nanoparticles have been tested for their ability to modify the rheological and filtration properties of WBMs, the application has mostly been at low pressure and low temperature (LPLT) conditions. Studies at higher temperatures, have been limited to temperatures below 125°C with the highest temperature for filtration tests on WBMs with FeO₄ being 121°C by Vryzas et al. Vryzas et al., [86–88] and by Mahmoud and Nasr-El-Din [62]. Hence, the need to investigate the performance of FeO₄ at higher temperatures and pressures to help determine the suitability of these nanoparticles for application under such conditions. Additionally, most of the base muds that were used for FeO₄ nano-mud systems in past studies were water/bentonite muds, which are usually applicable at lower temperature and pressure conditions. Therefore, this study investigates the performance of FeO₄ in a polymer WBM system at elevated temperatures and pressures to determine the suitability of these nanoparticles in the chosen base mud in under these conditions.

The current investigation is based on a previous study [28] conducted by our research group on the use of CuO and ZnO nanoparticles for modification of the properties of a WBM system. In the previous study, ZnO and CuO nanoparticles were added to the base mud at 0.1, 0.3, 0.5, 0.8 and 1.0 wt% concentrations and rheological tests were conducted at 25°C, 50°C and 80°C while filtration tests were conducted at 100°C and 500 psi. The results of the tests showed improved performance due to the addition of the nanomaterials. For the filtration properties, the mud samples formulated with 0.8 wt% concentrations of both CuO and ZnO had the least filtrate volume, mudcake thickness and permeability though the performance of CuO was generally better than that of ZnO for all concentrations. The SEM analysis of the mudcake samples for the base-mud and the nano-mud samples with 0.8 wt% concentrations of both CuO and ZnO showed the mudcake with CuO to be less porous and permeable with less agglomeration compared to the sample with ZnO and the base mud. For the rheological properties, all the mud samples exhibited shear-thinning behaviour with the nano-mud samples showing lower shear stresses, PV, AV,YP and gel strength values compared to the base mud. To extend the application of the mud used in the previous study (BM) to meet the current challenges of drilling higher temperature wells, the performance of the base mud (BM) was investigated at higher temperatures (125 and 150°C) in the current study. Thereafter, a modified base mud (MBM) was formulated, aged and tested at 100, 125 and 150°C and the results compared with the results obtained from BM. The test results indicated that the modified base mud (MBM) had better rheological and filtration properties than the old base mud (BM) from the previous study. The MBM formulation was used as the base to prepare the nano-mud samples with 3 different concentrations of MWCNT.
or Fe₃O₄ and the samples were aged and tested at 150°C. Thus, exploring the feasibility of using the MBM at elevated temperature and pressure conditions, extending the application of nano-Fe₃O₄ to higher temperature and pressure conditions and testing the performance of the two nanomaterials in the MBM system.

2. Experimental equipment and procedures

2.1. Materials

Deionised water (pH 6.1–7.0) was used as the base fluid for the formulation of drilling fluids. Deionized water was chosen as the base fluid due to its purity and absence of dissolved minerals found in of tap water. Bentonite, Xanthan gum (Duovis), PAC (AquaFLO LV), barite (MI Wate) and calcium carbonate (SAFE-CARB 10 and SAFE-CARB 20) were kindly provided by MI Swaco, Aberdeen. Bentonite was used as the main viscosifier while xanthan gum and PAC were used as secondary viscosifiers and fluid loss control agents. Barite and calcium carbonate were used to increase the mud density and calcium carbonate also served as a bridging agent. Sodium hydroxide (NaOH) was supplied by Fisher Scientific UK; it was used to raise and maintain the pH of the mud and to aid preservation of the mud against bacterial degradation. KCl was used as an inhibitive agent to stabilize water sensitive shales by inhibiting shale hydration, swelling and disintegration. Formaldehyde was supplied by Sigma-Aldrich UK and was used to inhibit mud degradation due to bacterial action. Iron II, III oxide (Fe₃O₄) nanoparticles, black in colour with spherical shapes (purity of greater than 97 % as specified by the manufacturers) were supplied by Sigma Aldrich, UK while MWCNTs black in colour with a purity of 95%, were supplied by Iolitec, Germany. Both nanomaterials were used to modify the rheological and filtration properties of the mud. The properties and functions of all the materials used for the formulation of the mud samples used in this study are listed in Table 1.

2.2. Equipment

The equipment utilised for formulation and testing of drilling fluids and characterisation of the mudcake and filtrate are briefly discussed in this section.

![Fig. 1. OFITE HPHT filter press assembly with OFITE ageing cell.](image-url)

Table 1

<table>
<thead>
<tr>
<th>Additive</th>
<th>Physical Appearance</th>
<th>Average Particle Size</th>
<th>Molecular Weight (g/mol)</th>
<th>Function(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deionised Water</td>
<td>Colourless liquid</td>
<td>-</td>
<td>18.02</td>
<td>Base Fluid</td>
</tr>
<tr>
<td>Bentonite (Berkbent GS™)</td>
<td>Cream to greyish</td>
<td>1–100µm particle size</td>
<td>422.29</td>
<td>Main viscosifier</td>
</tr>
<tr>
<td>Xanthan Gum (DUO-VIS™)</td>
<td>Cream – tan powder</td>
<td>NA</td>
<td>NA</td>
<td>Rheology modifier</td>
</tr>
<tr>
<td>PAC (AquaFLO™ LV)</td>
<td>Off-white powder</td>
<td>NA</td>
<td>NA</td>
<td>Fluid loss control additive and rheology modifier</td>
</tr>
<tr>
<td>MWCNTs</td>
<td>Black powder</td>
<td>OD: 10–20 nm, L: 1–2 µm</td>
<td>12.01</td>
<td>Fluid loss control additive and rheology modifier</td>
</tr>
<tr>
<td>Fe₃O₄</td>
<td>Black spherical</td>
<td>50–100 nm particle size (SEM)</td>
<td>231.53</td>
<td>Fluid loss control additive and rheology modifier</td>
</tr>
<tr>
<td>Barite (M4 WATE™)</td>
<td>Tan-grey powder</td>
<td>NA</td>
<td>NA</td>
<td>Weighting agent</td>
</tr>
<tr>
<td>Sodium Hydroxide (NaOH)</td>
<td>White pellets</td>
<td>NA</td>
<td>74.55</td>
<td>Inhibitive agent</td>
</tr>
<tr>
<td>Potassium Chloride (KCI)</td>
<td>Off-white powder</td>
<td>63–100µm particle size</td>
<td>30.03</td>
<td>Bactericide</td>
</tr>
<tr>
<td>Biocide (Formaldehyde)</td>
<td>Colourless liquid</td>
<td>-</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
cell has a filter area of 3.5 in.² and a capacity of the of 175 mL while the filtrate receiver can hold up to 15 mL of filtrate at one time. A 500 mL OFITE ageing cell is shown beside the filter press. The ageing cell holds the mud sample that is heated in a stationary oven prior to conducting the tests. Fig. 2 shows the Fisherbrand static oven with the ageing cell holding the mud. A Fisherbrand static oven was employed to heat the mud samples to simulate downhole conditions of elevated temperatures and pressures when the mud is not in circulation. Static ageing was applied because higher temperatures are associated with mud that is left at the lower part of the wellbore while tripping leading to higher additive degradation compared to when circulation is in progress. Hence, static ageing is more representative of conditions in the wellbore (Bég, Espinoza et al., 2018).

A digital balance (Ohaus AV313C Adventurer Pro Digital Balance) was used to weigh the additives and a Hamilton Beach mixer was used for mixing and shearing the additives to produce a homogenous drilling fluid. An OFITE Atmospheric Mud Balance was used for measuring the density of the drilling fluids before and after aging while a pH meter (Themo Scientific – EUTECH pH5+) was used to determine the pH of the drilling fluids. An OFITE Model 800 Viscometer was used for the determination of the rheological properties of the mud.

2.3. Experimental procedures

2.3.1. Formulation and ageing of base mud samples

The sequence of experimental procedure is illustrated by the flowchart in Fig. 3. The experiments commenced with the preparation and testing of two base mud samples. The samples were formulated with deionised water as the base fluid and conventional drilling fluid additives (without nanomaterials) and designated as Base Mud (BM) and Modified Base Mud (MBM). The BM was formulated by adding bentonite, xanthan gum, PAC, potassium chloride, formaldehyde and sodium hydroxide chronologically to deionised water and mixing each additive for five minutes at the medium speed using a Hamilton Beach mixer. The total mixing time for each sample was 30 minutes.

To improve on BM and make the mud suitable for elevated temperature applications, an improved base mud (designated as Modified Base Mud - MBM) was formulated by adding bentonite, xanthan gum, PAC, barite, calcium carbonate and sodium hydroxide chronologically to deionised water and mixing each additive for five minutes at the medium speed using a Hamilton Beach mixer. Barite and calcium carbonate were introduced in the MBM to improve the filtration properties and to increase the mud density, as higher mud densities are required to supress high formation pressures encountered in deeper wells which usually have elevated to higher temperatures and pressures.

The base mud samples were placed in the OFITE ageing cell, pressurised to 100 psi and aged for 16 hours at three different temperatures (100, 125 and 150 ºC) in a static oven. Thereafter, filtration tests were conducted at the corresponding aging temperatures and rheological tests were conducted at ambient conditions for all samples. The results obtained for the filtrate volume, mudcake thickness and permeability as well as the shear stress/shear rate curves, 10⁰, 10¹, 10², 10³,YP and PV of the different samples were compared to determine the best base mud to use for the preparation of the nano-mud samples for the next stage of the investigation. The additives used for the formulation of the Base Mud and Modified Base Mud are listed in Table 2 with the corresponding quantities used in each formulation.

2.3.2. Formulation and ageing of nano-mud samples

The nano-mud samples were prepared by adding bentonite, xanthan gum, PAC, FeO₄, or MWCNT, barite, calcium carbonate and sodium hydroxide to deionised water chronologically and the additives were mixed for a total mixing time of 30 minutes to obtain a homogenous mixture. The additives used for the formulation of the nano-muds with the corresponding concentration are shown in Table 3.

All the additives were properly incorporated into the different mud samples by scraping off the additives that adhered to the sides of the mixing vessel or the mixing shaft into the vortex. The formation of ‘fish-eyes’ was also prevented by adding of the polymer and nanomaterials slowly into the vortex ([23], White, Needs 2016). The mud samples were placed in an OFITE aging cell and pressurised to 100 psi using CO₂ then aged for 16 hours in a static oven. After aging, the samples were left to cool down to room temperature before depressurising and opening the aging cell. Thereafter, the mud was scraped for 5 minutes with the Hamilton Beach mixer before the pH, mud weight, rheological and filtration tests were conducted. All the prepared samples had pH range of 9–10 and a density of approximately 10.6 ppg. The pH, density, 10⁰, 10¹, 10², 10³,YP and PV values are presented in Table 4.

2.3.3. Drilling fluid filtration tests

Mud HPHT filtration test was conducted according to API recommendations for field testing of WBM as outlined in API RP RB 13B-1 using the equipment outlined previously.

The filtration tests for the base mud samples were conducted at 100, 125 and 150 ºC to understand the effect of temperature variations on the drilling mud properties. Thereafter, the nano-mud samples were tested at 150 ºC for three different concentrations (0.2, 0.5 and 0.8 %) of each of the nanoparticles. In a standard run, approximately 130 mL of the mud sample was placed in the cell, covered with the filter paper and sealed. Thereafter, the cell was placed in the heating jacket, pressurised to 100 psi and left to heat up to the test temperature for one hour before starting the test. The tests were conducted with 500 psi differential pressure (600 psi top pressure and 100 psi bottom pressure) supplied by CO₂ cartridges across the filter paper. The filtrate was collected at 0.5, 1,
5, 7.5, 15 and 30 minutes, and the volume was recorded as the fluid loss (mL). The filtrate volume obtained from the experiment was multiplied by 2 because the size of the filtration area is half of that of the API standard filtration equipment (22.9 sq.cm and of 45.81 sq.cm respectively). Thereafter the cell was cooled to ambient temperature, depressurised and opened, and the filter paper was retrieved and the deposited mud was slightly washed under running water to obtain the filter cake. The filter cake thickness was measured using a Vernier calliper and recorded in millimetres and the filter cake was dried at
ambient conditions to prepare it for the SEM analysis.

2.4. Determination of filter cake permeability

The mudcake permeability (K) was investigated to get more insight into the filtration mechanism of the drilling fluid samples and to understand the relationship between the permeability of the mudcake and filter loss. The model developed by Engelhardt [35] was used to determine the permeability as shown in Eq. 1.

\[
k = \frac{Q_i l_i \mu}{2 P Ft}
\]  

Where:
- \( k \) is the mudcake permeability in Darcy (D)
- \( Q_i \) is the quantity of filtrate volume in cm² separated after time \( t \)
- \( l_i \) is thickness of filter cake in centimetre (cm)
- \( \mu \) is viscosity of filtrate in centipoise (cP)
- \( P \) is the filtration pressure in atm
- \( t \) is time in seconds (s)
- \( F \) is effective filter surface in square centimetre (cm²)

The filtrate viscosity is assumed to be 1 cP at 25°C for WBMs [15,19, 28].

2.5. SEM analysis of dried mudcake samples

The mudcake samples obtained after the filtration experiments were air dried at ambient conditions and SEM analysis of the samples was conducted using a Scanning Electron Microscope. The analysis was carried out to characterize the samples so that a better understanding of the morphology of the mudcake could be obtained. The analysis was conducted for the surface of the filter cake at (100, 150-, 200-, 350- and 900-times).

2.6. Drilling fluid rheological tests

Rheological tests were conducted to determine the flow properties of the mud samples using a rotational viscometer. Mud tests were conducted according to API recommendations for field testing water-based muds as outlined in API RP RB 13B-1 using the Model 800 8-Speed Electronic Oilfield Viscometer from OFITE.

The tests were conducted at ambient temperature and pressure where approximately 250 mL of the sample was placed in the test cup of the viscometer and the rotor was lowered into the mud to the appropriate level indicated on the sleeve. The sample was stirred for 10 seconds on the ‘stir’ setting of the viscometer. Subsequently, the dial readings were taken from the highest to lowest values at fixed speeds of 600, 300, 200, 100, 60, 30, 6 and 3RPM. The dial readings obtained were used to calculate the shear stress (\( \tau \)) and Plastic Viscosity (PV) according to Eqs. (1) and (2) while the fixed speeds of the viscometer at 600, 300, 200, 100, 60, 30, 6, 3 rpm were multiplied by the Shear Rate Constant, \( K \) (1.7023 sec⁻¹ per RPM for Shear Rate Range R1B1) to obtain the shear rates as shown in Eq. 3.

\[
\tau = \text{Dial reading} \times 1.067 \frac{Lbf}{100 \text{ft}^2}
\]  (2)
\[
PV = 600 \text{rpm reading} - 300 \text{rpm reading} (cP)
\]  (3)
\[
\gamma = \text{RPM reading} \times 1.7023 \text{sec}^{-1}
\]  (4)

The Yield Point (YP) which is the shear stress at zero shear rate was obtained from the intercept of the shear stress/shear rate curve on the Y-axis for the Herschel-Bulkley rheological model [5,23,49,67,77].

The gel strength readings were obtained directly from the equipment by stirring the mud for 10 seconds, then setting the speed selector knob at 3 rpm, switching off the equipment and allowing the mud to rest for 10 seconds and ten minutes as required. After the required time had elapsed, the equipment was started again and the maximum deflection was measured and recorded as the 10-second and 10-minute gel strength, respectively.

The dial readings obtained for the RPM readings were multiplied by a constant (1.067Lbf/100 ft²) to obtain the shear stress for the respective RPM readings where 1 degree of deflection of the bob gives rise to a dial reading equal to 1.067Lbf/100 ft². The fixed speeds of the viscometer at 600, 300, 200, 100, 60, 30, 6, 3 rpm were multiplied by the Shear Rate Constant, \( K \) (1.7023 sec⁻¹ per RPM for Shear Rate Range R1B1) to obtain the Newtonian shear rates of 1021.38, 510.67, 340.46, 170.23, 102.14, 51.069, 10.21 and 5.11 s⁻¹, respectively. The values of 1.7023 sec⁻¹ and 1.067Lbf/100 ft² were obtained from API 13RP-1. The results obtained from the calculations of the shear stresses and shear rates were used to plot the shear stress/shear rate curves for the different samples [5,48,49,67,77].

3. Results and discussions

The results obtained from the filtration and rheological tests for the base mud and nano-mud samples are discussed in this section. The first part discusses the results for the BM and MBM and compares the results obtained in terms of the changes observed due to the changes in the test temperature. The second section presents and discusses the nano-samples and the changes that occur in terms of filtrate volume, mudcake thickness and permeability of the mudcake samples as well as changes in the plastic viscosity, yield point and gel strengths due to the addition of different concentrations of the nanomaterials to the mud.

3.1. The effects of temperature on the rheological properties of the base mud samples

This section discusses the results of the rheological tests conducted at ambient temperature and pressure for the base mud samples (BM and MBM). The rheological properties of the drilling fluid indicate the fluid’s ability to clean the wellbore while drilling the well and its capacity to keep the cuttings suspended when drilling ceases [1,64,71]. Rheological tests were conducted for the mud samples at ambient temperature and pressure according to API RP 13B-1 after ageing at 100, 125 and 150°C. The properties investigated and presented include the shear stress/shear rate relationship, plastic viscosity (PV), yield point (YP) and 10-second and 10-minute gel strengths (10” and 10’ GS). The Gel strength and yield point are indicators of the attractive or electro-chemical forces in the drilling fluid. The yield point is a measure of the attractive or electro-chemical forces in a fluid under flow conditions while the gel

<table>
<thead>
<tr>
<th>Sample</th>
<th>pH</th>
<th>density</th>
<th>PV</th>
<th>YP</th>
<th>10’ GS</th>
<th>10” GS</th>
<th>Rheological Model</th>
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</thead>
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<tr>
<td>BM</td>
<td>9.26</td>
<td>8.73</td>
<td>7</td>
<td>72.6</td>
<td>63</td>
<td>67</td>
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<td>MBM</td>
<td>9.54</td>
<td>10.58</td>
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<td>10.7</td>
<td>10.0</td>
<td>22.0</td>
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<tr>
<td>MBM +</td>
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<td>10.623</td>
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<td>10.7</td>
<td>8.0</td>
<td>20.0</td>
<td>HB</td>
</tr>
<tr>
<td>MBM +</td>
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<td>10.639</td>
<td>22.0</td>
<td>8.5</td>
<td>8.0</td>
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<td>0.5 wt% FeOₓ</td>
<td>9.48</td>
<td>10.595</td>
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<td>7.5</td>
<td>7.0</td>
<td>12.0</td>
<td>HB</td>
</tr>
<tr>
<td>MBM +</td>
<td>9.62</td>
<td>10.614</td>
<td>25.0</td>
<td>6.4</td>
<td>6.0</td>
<td>16.0</td>
<td>HB</td>
</tr>
<tr>
<td>0.8 wt% FeOₓ</td>
<td>9.5</td>
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<td>6.0</td>
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<td>MBM +</td>
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</tbody>
</table>
strength is a measure of the attractive forces under static conditions. These forces are due to positive and negative charges located near or on the surfaces of the particles dispersed in mud, which cause attraction or repulsion between the particles leading to corresponding increase or decrease in the yield point and gel strength. Usually, gel strengths and yield point have a direct relationship and such the gel strength decreases as the yield point decreases. The gel strength is important for the suspension of weighting materials and drilled solids when the well is not being circulated while adequate yield point is required for hole cleaning while drilling is in progress [1,10,46,71,77]. High yield points lead to a reduction in the penetration rate, high pump pressure loss, and increase in surge and swab pressures, though a very low yield point may cause barite and drill cutting sag [73]. Hence, the gel strength and yield point should be high enough to ensure that the gel structure required to provide the suspension of the weight material and ensure removal of cuttings from the wellbore is maintained and low enough to minimise annular pressure losses and Equivalent Circulating Densities (ECDs) ([10,13,49,53,77]).

The plastic viscosity (PV) is the resistance to flow due to mechanical friction. It is dependent on the solid concentration, size and shape of solids and the viscosity of the fluid phase among other factors. Usually, an increase in the volume percent of solids leads to an increase in the plastic viscosity of the sample. In addition, smaller particles have higher surface to volume ratio, which causes increased frictional drag, increasing the PV. The plastic viscosity should be kept as low as practically possible to optimize penetration rates, enhance hole cleaning by increasing the flow in the annulus and to minimise wearing away of the equipment due to friction [10,77].

As shown in Fig. 4 which illustrates the shear stress/shear rate curves for the BM and MBM, the shear stress increased with increase in the shear rate which indicates that the mud samples exhibit shear–thinning behaviour. The shapes of the curves also indicate that the mud samples follow the Herschel Bulkley (HB) fluid model where the curves exhibit a yield stress at zero shear rate [18,36,77]. The need for shear-thinning behaviour in drilling fluids is reflected in the different sections of the wellbore during drilling operations. Shear thinning properties enable the mud to exhibit low viscosities at high shear rates (high velocities) in the drill string and through the bit, decreasing the circulating pressure and pressure losses. The mud also exhibits higher viscosity that aids in hole cleaning at the lower shear rates (lower velocities) in the annulus and develops gel strength that aids in suspending weight material and cuttings at ultra-low velocity where the mud has its highest viscosity while under static conditions [46,77,82].

The rheograms for the mud samples aged at 100, 125 and 150°C show that increasing the aging temperature of the samples from 100 to 125 and 150°C caused a downwards shift in the rheological profiles for all the mud samples as shown in the Fig. 4. At 150°C, BM exhibited a rheological profile that is almost flat indicative of breakdown of mud and loss of shear thinning properties of the sample indicating that the grid structure of the sample had been destroyed at 150°C (J. [58]; J.-P. [59]) whereas MBM maintained a profile indicative of a shear-thinning fluid which indicates that the sample was able to withstand the effects of elevated temperature ageing and testing.

Fig. 5 depicts the 10° GS, 10' GS, YPs and while Fig. 6 depicts the PVs for the BM and MBM at 100, 125 and 150°C. A decrease in the gel strengths, YPs and PVs occurred for all the samples as the temperature increased. The YPs and gel strengths of MBM are lower than that of BM at all temperatures while the PVs of the MBM are higher than those of BM. Overall, there was a reduction in the rheological properties of both samples with increase in temperature. High temperature changes the structure of the clay platelets, reducing the negative charge on the surface of bentonite and compressing the hydrational layer around the bentonite platelets in the drilling fluid. A reduction in the negatives charges reduces the magnitude of the electrostatic repulsion between the particles in the mud making the particles susceptible to coalescence.
as the particles collide due to random Brownian motion. Hence the particles coalesce forming larger particles with a face-to-face association (aggregates). The number of particles in the mud system is reduced and the gel structure of the mud is broken down, reducing the rheological properties of the mud as reflected in the reduction in the magnitude of the rheological parameters \([7,14,41,43,56]; [58]; J.-P. [59,83,85]; J. R. [95]). The difference in the rheological properties of BM and MBM occurred due to the variation in the additives used for the formulation of the samples. MBM showed better performance than BM just as obtained in the filtration tests.

3.2. The effects of temperature on the filtration properties of the base mud samples

The filtration tests were conducted for the BM and MBM samples at the corresponding ageing temperatures (100, 125 and 150°C) and the results obtained for the different samples were analysed and discussed to highlight the effects of elevated temperatures on the properties of the mud samples. The results obtained for the two base mud samples were compared and the sample that had better results was chosen as the base mud for the preparation of the nano-mud samples.

The parameters investigated were the filtrate volume, mudcake thickness and mudcake permeability. The 30-minute filtrate volumes, mudcake thicknesses and permeabilities for the BM and MBM are illustrated in Figs. 7, 8 and 9. The filtrate volumes obtained for the BM were 12.8 mL, 17.2 mL and 40.1 mL at 100, 125 and 150°C respectively while the filtrate volumes obtained for the MBM were 12.2 mL, 14.8 mL and 30.4 mL at 100, 125 and 150°C respectively. For both samples, the filtrate volume increased with increase in temperature. This is a normal phenomenon because higher temperatures affect the properties of the mud leading to higher filtrate loss as a result of reduction in filtrate viscosity of the liquid phase of the mud. This decrease in filtrate viscosity makes it easier for the filtrate to flow through the pores which may lead to increase in the filtrate volume as it has been empirically proven that the filtrate volume at 100°C can be 1.88 times as large as the filtrate volume at 20°C. Secondly, chemical breakdown of mud components at high temperatures can also affect filtration properties of the mud negatively because at temperatures higher than 100°C, many organic filtration control additives begin to break down and lose their properties and the degradation rate increases as the temperature increases. High temperature also changes the structure of the bentonite platelets, reducing the negative charge on the surface of bentonite and compressing the hydrational layer around the bentonite platelets in the drilling fluid. A reduction in the negatives charges reduces the
mudcake is usually desirable because thick mudcakes can lead to excessive torque and drag as well as differential pressure sticking ([2,37, 47]; [60,63,96]). For the mudcake permeability, the results obtained for the MBM were lower than that of the BM at all temperatures. This is expected because the permeability of the mud is a function of the filtrate volume and filter cake thickness. Hence with lower filtrate volumes and mudcake thicknesses, lower permeability values should be expected for the MBM. The results show that the MBM was able to form a thin, low permeability mudcake which impeded the flow of filtrate thus reducing the filtrate volume. MBM had lower filtrate volumes than BM because a higher concentration of fluid loss control agent (PAC) was used for the formulation of MBM and the use of CaCO₃ in the BM formulation also helped to reduce the filtrate [28,39,77,84]. Hence, MBM was chosen as the base mud for the formulation of the nano-mud samples for the investigation of the effects of the chosen nanomaterials (Fe₃O₄ and MWCNT) at elevated temperature and pressure.

### 3.3. The effects of nanoparticles on the rheological properties of the nano-mud samples

This section discusses the results of the rheological tests conducted at ambient temperature and pressure for the nano-mud samples. Fig. 10 shows the rheograms for MBM2 and the nano-mud with different concentrations of MWCNTs and Fe₃O₄. All the samples exhibited shear-thinning properties with yield stress. From the plot, we observe that the shear stresses for all the nano-mud samples reduced relative to that of the base mud. The change in the rheological parameters of the mud was caused by a combination of the repulsive forces between the nanomaterials and the negative charge on basal surface of clay platelets as well as the charges on the edges of clay platelets which are pH dependent. Montmorillonite clay platelet edges have points of zero charge (PZC) at pH 6.5 such that the edges are positively charged above the pH of 6.5 and negatively charged below the pH of 6.5. Hence the edges of the clay platelets in MBM are negatively charged since the pH of all the mud samples in this investigation ranges between 9.0 and 10. Additionally, both nanomaterials used also have their points of zero charge below the pH of the mud so they also exhibit different magnitudes of negative charges within the mud system. On adding the nanomaterials to the mud, the negative surface charges between the particles increase the interparticle repulsive forces resulting in the higher degree of dispersion manifested by the general reduction in the mud rheological properties relative to the base mud [4,55,66,80,92].

For the nano-mud samples with MWCNTs, the change in the shear stress with increase in nanoparticle concentration was low as observed from the rheograms. The rheograms for the samples with 0.2 and 0.5 wt% are shown to overlap at most of the points because the values of the RPM readings for the two samples were similar while a slight decrease in the magnitude of the shear stress was obtained at 0.8 wt% concentration as depicted by the slight change in the magnitude of the rheogram. This indicates that the change in the concentration of the MWCNTs did not have appreciable effects on the shear stresses.

However, the nano-mud samples with Fe₃O₄ had a slightly different trend. After the decrease in the shear stress relative to the MBM at a concentration of 0.2 wt%, increasing the concentration to 0.5 wt% caused an increase in shear stress and then another drop occurred when the concentration was increased to 0.8 wt%. The upward shift in the magnitude of the rheogram at 0.5 wt% occurred due to flocculation which occurred as the concentration of nanoparticles increased giving rise to face-to-edge and edge-to-face orientations of the clay platelets. The reduction in the inter-particle distance caused by the increase in nanoparticle concentration and the random movement of the nanoparticles due to Brownian motion of the nanoparticles led to higher attraction and rate of collision between the particles in the mud leading to flocculation which was accompanied by an increase in mud rheology and an upward shift in the magnitude of the rheogram. Conversely, with further increase in nanoparticle concentration to 0.8 wt%, the
nanoparticles penetrated the flocs formed, reducing the edge-to-edge and face-to-edge interactions between the bentonite platelets and breaking the floc structure leading to a reduction in mud rheology which caused a downwards shift in the magnitude of the rheogram ([20,36,52]; J. [58,87,89]).

Fig. 11 illustrates the 10'GS, 10'G and YP while Fig. 12 shows the PV of all the samples. From the figures, the base fluid has the highest values for all the rheological parameters. The addition of 0.2 wt% MWCNT reduced the 10'GS to 7 lb/100 ft.² and a further reduction occurred on increasing the concentration to 0.5 wt%. However, a further increase to 0.8 wt% did not lead to any change in the 10'GS as both 0.5 and 0.8 wt% had the same value of 6 lb/100 ft.². For the 10'G the addition of 0.2 wt% MWCNTs led to a reduction from 22 lb/100 ft.² for the base-mud to 15 lb/100 ft.² and the same value was also maintained at 0.5 wt% concentration but then reduced to 13 lb/100 ft.² at 0.8 wt% concentration of the nanomaterials. The addition of 0.2 wt% MWCNT reduced the YP from 10.7 lb/100 ft.² for the base-mud to 7.5 lb/100 ft.² and the same value was also maintained at 0.5 wt% concentration but then reduced to 6.4 lb/100 ft.². The YP exhibited the same trend as that of the 10'GS and the values of the two parameters are close. For the PV of MWCNTs, the trend was similar to that of the 10'GS where the value decreased from 25 cP for the base-mud to 24 cP at 0.2 and 0.5 wt% then dropped to 23 cP at 0.8 wt% concentration.

For the samples with Fe₃O₄, the 10'GS reduced to 8 lb/100 ft.² upon adding 0.2 wt% of the nanomaterial and remained constant without changing as the concentrations increased to 0.5 and 0.8 wt%. The 10'G decreased from 19 lb/100 ft.² for the base-mud to 19 lb/100 ft.² upon the addition of 0.2 wt% Fe₃O₄. Subsequently, the value increased to 100 ft.². The YP exhibited the same trend as that of the 10'GS and the values of the two parameters are close. For the PV of MWCNTs, the trend was similar to that of the 10'GS where the value decreased from 25 cP for the base-mud to 24 cP at 0.2 and 0.5 wt% then dropped to 23 cP at 0.8 wt% concentration.
20 lb/100 ft.² at 0.5 wt% and decreased again to 16 lb/100 ft.² at 0.8 wt% concentration. The YP of the mud reduced to 9.6 lb/100 ft.² at 0.2 wt% concentration of Fe₃O₄ and increasing the concentration to 0.5 wt% led to an increase to 10.1 lb/100 ft.². However, the value dropped to 8.5 lb/100 ft.² at 0.8 wt% concentration. The trends for the 10°GS and YP are similar as both parameters increase between 0.2 and 0.5 wt% concentration of the nanomaterial. This increase occurred as a result of flocculation due to the addition of the nanoparticles in the system leading to increase in mud rheology and increase in the 10°GS and YP. With further increase in the concentration to 0.8 wt%, the reduction in the gel strength occurred due to breakdown of the flocs structure as the nanoparticles penetrated the flocs formed, reducing the edge-to-edge and face-to-edge interactions between the bentonite platelets [14,19,89]. The reduction in the gel strength of the mud upon the addition of Fe₃O₄ is consistent with the result reported by Vryzas et al. where they obtained a reduction in the gel strength relative to the base-mud upon the addition of 0.5 wt% custom made Fe₃O₄ [88]. A reduction in PV relative to the base-mud was observed with the addition of Fe₃O₄. The sample with 0.2 wt% Fe₃O₄ had the least plastic viscosity (18 cP), after which the PV increased to 22 cP at 0.5 wt% and reduced to 21 cP at 0.8 wt%. This is consistent with the result obtained by Dhiman et al. where they reported a reduction in PV relative to the base-mud after adding 0.5 wt% of 20 nm Fe₃O₄ nanoparticles [29]. Furthermore, increasing the concentration of Fe₃O₄ from 0.2 to 0.5 wt%, caused an increase in the PV due to the increase in the number of particles present in the mud and flocculation. However, the value of the PV reduced at 0.8 wt% due to breakdown of the flocs and formation of aggregates with further increase in the concentration of nanomaterials to form larger plates, reducing the number of particles in the mud causing a slight reduction in the PV [14,41,51,73,89].

3.4. The effects of nanoparticles on the filtration properties of the nano-mud samples

This section analyses and discusses the filtration properties of the mud samples in terms of filtration volume and mudcake thickness for tests conducted at a temperature of 150°C and a differential pressure of 500 psi using an OFITE HPHT filter press after aging the mud samples at 150°C for 16 hours in a static oven. Thereafter, the results obtained from the permeability calculations and the micrographs obtained from the SEM analysis of the mudcake samples from the MBM and nano-mud samples with 0.8 wt% MWCNT and 0.2 wt% Fe₃O₄ are also discussed to highlight the effects of the nanomaterials on the mudcake characteristics.

Figs. 13 and 14 illustrate the filtrate volumes and mudcake thicknesses respectively for the MBM and nano-mud (NM) samples with three different concentrations (0.2, 0.5 and 0.8 wt%) of Fe₃O₄. The results indicate a slight reduction in filtrate volume at 0.2 wt% (3.29 %) and 0.8 wt% (1.32 %) compared to the base fluid. However, an increase in filtrate volume occurred with the addition of 0.5 wt% (23.68 %) of the nanoparticles. For the mudcake thickness, the addition of 0.2 wt% and 0.8 wt% of the nanoparticles gave a reduction of 15.56 % and 24.44 % respectively but the addition of 0.5 wt% gave a thicker filter cake with an increase of 4.44 % in the cake thickness. The decrease in filtrate volume and mudcake thicknesses relative to the base mud upon addition of 0.2 wt% Fe₃O₄ occurred due to the dispersion of the mud particles as a result of electrostatic repulsion between the particles as well as the plugging effect of the nanoparticles as the smaller particles migrate and deposit themselves within the main mudcake formed by the larger particles in the mud. However, the increase in filtrate volume and filter cake thickness at 0.5 wt% occurred due to flocculation which occurred as the concentration of nanoparticles increased. The reduction in the inter-particle distance caused by the increase in nanoparticle concentration and the random movement of the nanoparticles due to Brownian motion of the nanoparticles leads to higher attraction and rate of collision between the particles in the mud. Hence, flocculation occurs when the basal surfaces of the bentonite platelets and edges of the platelets assume face - to - edge and edge – to - edge orientations to form flocs which are porous and permeable increasing the filter cake thickness and permeability leading to increase in filtration. With further increase in
concentration to 0.8 wt%, the nanoparticles penetrated the porous filter cake formed due to flocculation, reducing the edge-to-edge and face-to-edge interactions between the bentonite platelets and breaking the floc structure. Thus, the nanoparticles plugged the pore spaces and reduced the effective size of the flocs leading to the formation of a thin, compact mudcake with lower permeability and reduction in filtrate loss. ([20,36,52]; [58,62,87,89]).

Figs. 15 and 16 show the filtration volumes and mudcake thickness for the MBM and samples with different concentrations of MWCNTs. The base mud had the highest filtration volume and mudcake thickness. The addition of MWCNTs reduced the fluid loss at all concentrations relative to the base mud. The filtration volume reduced by 14.47 %, 12.83 % and 15.79 % for the samples with 0.2 wt%, 0.5 wt% and 0.8 wt% respectively. The highest reduction in filtration volume was obtained upon the addition of 0.8 wt% of MWCNT while 0.5 wt%. gave the least reduction relative to the base mud. This result is consistent with the result obtained by Aftab et al., [6] who reported a 6.25 wt% reduction in the HPHT filtrate volume upon the addition of 0.1ppb MWCNT relative to the base mud at 121 °C and 500 psi. The trend for the filtration volume is similar to the result obtained with Fe₃O₄ where the filtrate volume increased at 0.5 wt% concentration then reduced again when the concentration was increased. As for the mudcake thickness, the addition of the nanoparticles led to a reduction in the thickness of the mudcake samples by 20 %, 26.67 % and 28.89 % as the concentration of the nanomaterial was increased. The MWCNTs adsorb on the surface of bentonite platelets, increasing the steric hindrance between the platelets to prevent coalescence, to form a thin and compact mudcake thus reducing filtrate loss as the MWCNTs filled up the pore spaces between the macro and micro particles ([32]; [59]). The deposition of a thin and compact mudcake is important because it limits the volume of filtrate that seeps into the formation, thereby helping to minimise formation damage. It also reduces the chances of the drill pipe sticking to the wellbore wall [2, 42,88].

Figs. 17 and 18 show the filtration volumes and mudcake thicknesses for the MBM and nano-muds with different concentrations of the two nanomaterials. It was observed that the filtrate volumes and filter cake thicknesses for all the samples except for 0.5 wt% Fe₃O₄ were lower than that of the base mud. The reductions obtained indicate that the nanoparticles enhanced the performance of the mud. The decrease in filtrate volume and mudcake thickness due to the addition of Fe₃O₄ was less
and a more compact mudcake. This observation was made by Tien [26, 27] who posited that in a mud that is made of different particle sizes, the smaller particles migrate and deposit themselves within the main filter cake. The lower filtrate volumes from MWCNT could also be due to the ability of MWCNTs to form stronger bonds between the particles in the mud, holding the particles closer together to reduce filtrate loss and mudcake thickness.

Another factor could be agglomeration in the samples formulated with Fe₃O₄ which are magnetic in nature and possess high surface charges which promotes the formation of large clumps which produce loose and thick filter cakes thus, higher filtrate volumes [78]. The results obtained for the nano-mud samples formulated with 0.2, 0.5 and 0.8 wt % MWCNTs were 26, 26.5 and 25.6 mL respectively. These values are close to the standard value of 25 mL quoted from SCOMI Drilling Fluids. Katende et al. [51] where they showed that the acceptable filtrate volume for HPHT applications should be less than 25 mL. However, the MBM and the nano-mud samples formulated with Fe₃O₄ had values above 25 mL. Katende et al. also reported that the acceptable mudcake thickness at HPHT should be less than 10/32 in. (7.9 mm). The results for the mudcake thickness for all the samples in this study are below the 7.9 mm standard with the highest mudcake thickness being that of the nano-mud sample with 0.5 wt% Fe₃O₄ (4.7 mm). Hence, the mudcake thickness for MBM and all nano-mud samples are acceptable.

After obtaining the filtrate volumes and filter cake thicknesses from the filtration experiment, the permeability of the mudcake samples for the base mud and the nano-muds with the different concentrations of MWCNT and Fe₃O₄ was calculated using Eq. 4 adapted from Engelhardt [35]. Scanning Electron Microscopy was employed to analyse some of the filter cake samples to detect the changes that occurred in the microstructure of the filter cake due to the addition of the nanomaterials. The results of the SEM analysis of the three (3) mudcake samples chosen for the analyses are discussed after the filter cake permeability so that the relationship between the 2 parameters can be highlighted.

The permeability values obtained are shown in Fig. 19 and are a direct reflection of the filtration properties (filtrate volume and filter cake thickness) as mentioned earlier. The permeability values of mudcake samples with Fe₃O₄ and MWCNTs indicate a direct relationship with the filter cake thickness as the mudcake permeability values increased or decreased just as the filter cake thickness did. The lowest mudcake permeability was that of the sample with 0.8 wt% MWCNT, which also had the lowest filtrate volume and lowest mudcake thickness while the highest mudcake permeability value was that of the sample with 0.5 wt% Fe₃O₄ which also had the highest filtrate volume and mudcake thickness. Generally, the samples with MWCNT displayed the lowest mudcake permeabilities compared to the samples with Fe₃O₄ and the base mud. The MWCNTs adsorb on the surface of the bentonite platelets to prevent the agglomeration of the platelets and effectively plug the pores to reduce the permeability, hence the reduction in filtrate volume and production of a more compact mudcake with low thickness at elevated temperature and pressure ([(59)]. It is important to obtain thin mudcakes as the formation of thick mudcakes is undesirable because it can lead to different operational problems ranging from drill string sticking, high surge and swab pressures and excessive torque and drag while high filtration causes formation damage and impairment of the reservoir leading to increase in downtime and total well costs. [(2, 33,50); (60,96)].

Scanning Electron Microscopy was employed to analyse the changes in the microstructure of the filter cake due to the addition of the nanomaterials to get a better understanding of the effect of addition of the nanomaterials on morphology of the filter cake. The mudcake from the MBM and the nano-mud samples that recorded the least filtrate volumes (0.2 wt% Fe₃O₄ and 0.8 wt% MWCNT) were chosen and analysed at 900X magnification. Fig. 20 is the SEM micrograph of the mudcake of the MBM while Figs. 21 and 22 are the micrographs of the mudcakes for the nano-mud samples with 0.2 wt% Fe₃O₄ and with 0.8 wt% MWCNT respectively.

On a close observation of the surfaces of the three samples, a difference in the morphology of the surfaces is noticed. Fig. 20 for the MBM has more and larger pores and a rougher surface compared to Figs. 21 and 22. In the same vein, Fig. 21 has more pores than Fig. 22 which has the fewest and smallest pores accompanied by the smoothest appearance.
with the least noticeable grains. The reduction in the sizes and number of pores in Fig. 21 compared to 20 indicates that Fe₃O₄ nanoparticles filled the pores and reduced the sizes leading to the reduction in the mudcake permeability as shown in Fig. 19, thus a decrease in the filtrate volume. An increase in cake compaction also occurred which led to a decrease in the mudcake thickness of the nano-mud sample with 0.2 wt% Fe₃O₄ relative to that of the MBM. The smoothness of the surface of the mudcake in Fig. 22 and the absence of large pores also indicate that the MWCNTs filled up the pore spaces forming a thread-like structure which bonded the macro additives leading to a more compact and thinner mudcake with lower permeability and lower filtrate volume.

Fig. 23 is a graphical presentation of the filtrate volume data obtained from other studies and the results obtained for 0.2 wt% Fe₃O₄ and 0.8 wt% MWCNTs from this study. The results presented here are the best obtained from each study. The result for the poly (sodium p-styrene sulfonate)-modified Fe₃O₄ nanoparticles had highest value of 37 mL while the samples with 2 % nano-laponite and 0.2 wt% Fe₃O₄ were in the same range with values of 30.4 and 29.7 mL respectively. The result for the samples with 1 % ADMOS and 2.625ppb TiO₂ were also within the same range with values of 28 and 26.6 mL respectively. The samples with 0.8 wt% MWCNTs from this study and 0.5 wt% Fe₃O₄/PAA composite exhibited the least filtrate volumes with values of 15.6 and 25.5 mL respectively.

4. Conclusions

An experimental study was conducted with the aim of improving the filtration and rheological properties of a WBM system to make the mud suitable for elevated temperature and pressure applications by adding different concentrations of MWCNTs and nano-Fe₃O₄ to the WBM systems and investigating the effects of the nanomaterials on the filtrate volume, mudcake thickness and permeability, gel strength, YP and PV of the nano-mud systems.

The addition of 0.2, 0.5 and 0.8 wt% of MWCNTs and Fe₃O₄ to the MBM and testing at 500 psi and 150°C showed improvements relative to the MBM for all concentrations of the nanomaterials added to the mud except 0.5 wt% Fe₃O₄ which recorded higher values than the MBM for all the parameters investigated. The samples formulated with 0.2 wt% Fe₃O₄ and 0.8 wt% MWCNT gave the best improvement in the filtration properties of the samples investigated. For the optimal concentration of 0.8 wt% MWCNT, a reduction of 15.79 %, 28.89 % and 39.98 % was achieved for the filtrate volume, cake thickness and cake permeability respectively while the filtrate volume, cake thickness and permeability reduced by 3.29 %, 24.44 % and 25.18 % respectively relative to the base mud for the sample with 0.2 wt% Fe₃O₄.

The micrographs from the SEM of the mudcake samples obtained for the MBM showed the surface of the mudcake from the MBM with several large pores while the cake from the sample with 0.2 wt% Fe₃O₄ had fewer and smaller pores than the MBM. Conversely, the cake sample with 0.8 wt% of MWCNTs had the smoothest surface with fewer, small pores indicating the formation of a thin and compact mud cake which impeded fluid flow and reduced filtrate loss.

The rheological tests revealed that all samples exhibited shear-thinning properties with yield stress. The addition of nanomaterials generally reduced the shear stresses compared to the base mud, attributed to increased interparticle repulsive forces due to negative charges on the nanomaterials and clay platelet surfaces. For MWCNTs, changes in concentration had minimal impact on shear stress, while Fe₃O₄ samples showed an initial drop at 0.2 wt%, an increase at 0.5 wt% due to flocculation, and a decrease at 0.8 wt% due to floe breakdown. Analysis of gel strengths (10⁻⁰'GS and 10⁻⁰'GS) and yield point (YP) indicated the highest values for the base fluid, with MWCNT addition leading to reductions and Fe₃O₄ addition resulting in a complex pattern of reduction and increase, linked to flocculation dynamics. Plastic viscosity (PV) trends also showed reductions relative to the base mud upon adding both nanomaterials, with specific variations due to concentration.
changes and flocculation dynamics. These findings align with previous studies, highlighting the intricate interplay between nanoparticle concentration and mud filtration and rheology, underscoring the importance of tailored nanomaterial applications in drilling fluid formulations.

The results obtained support the suitability of MWCNTs as additives for the development of WBMs for elevated temperature and pressure applications.

CRediT authorship contribution statement

Mehmet Huseyin Yildirim: Methodology, Investigation, Formal analysis. Afzal Waheed: Writing – review & editing, Supervision, Project administration, Methodology, Investigation, Conceptualization. Zahrah Zanna Ibrahim: Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation. Hossein Hamidi: Writing – review & editing, Supervision, Resources, Project administration, Methodology, Investigation, Conceptualization.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Zahrah Zanna Ibrahim reports financial support was provided by Petroleum Technology Development Fund. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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