

Improvement in Fluid Loss Control and Viscosity of Water-based drilling Mud under High Temperature and Sodium Chloride Salt Conditions using Nanohydroxyapatite

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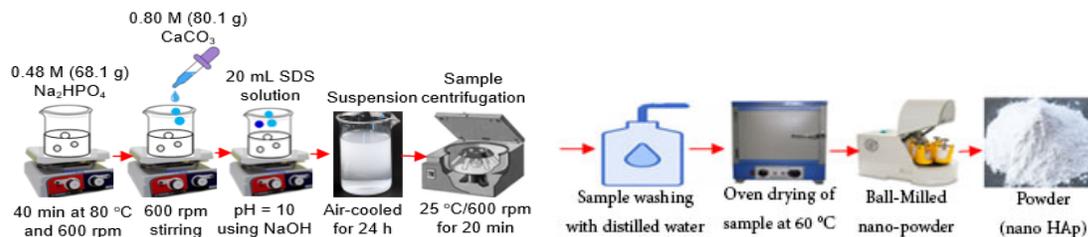
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ABSTRACT: It is difficult to drill efficiently with bentonite (BN)-based mud (BN-WBM) or water-based muds (WBMs) in high-salt electrolytes and deep wells. This is because the fluid's rheological parameters and filtration properties change in undesirable ways, affecting the well's production efficiency. To fix this, a high-salt and high-temperature-resistant nanohydroxyapatite (nanoHAp) additive was designed using sodium dodecyl sulphate (SDS). 0.1 to 0.5 wt% nanoHAp was added to WBMs, and a salt-resistant BN-WBM with nanoHAp was formulated with 4.8 wt% BN, 5.0 wt% sodium chloride (NaCl), and 0.5 wt% nanoHAp. At 25, 150, 180, and 210° C, the filtration and rheological characteristics of the drilling muds were evaluated. The findings revealed that between 25 and 210° C, nanoHAp increased the viscosity of the WBM by 15–139% at a 1021 s⁻¹ shear rate. It also controlled the fluid loss of the WBM from 12.1–44.6 mL to 6.7–21.8 mL at all temperatures. It serves as an anti-salt agent by decreasing the NaCl-contaminated BN's viscosity by 57% compared to the reference value of 20.8 mPa. s at a shear rate of 1021 s⁻¹. Further, it reduced the fluid loss by 56.8%, from 169 mL to 73 mL at 210° C. The nanoHAp surface has anionic sulphate head groups of SDS that efficiently attach to the BN surface. This keeps the Na⁺ ions from attacking the plate-like structure of the BN. This study reveals that nanoHAp has the capacity to inhibit BN coalescence and flocculation under saturated Na⁺ solutions and at high temperatures.

Keywords: Bentonite; Fluid loss; High temperature; NanoHAp; Viscosity; Water-based mud.

تحسين التحكم في فقدان السوائل ولزوجة الطين المائي القائم على الماء تحت ظروف درجات الحرارة العالية وكوريد الصوديوم باستخدام نانو هيدروكسي أباتيت

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الملخص: يُعد الحفر الفعال في المحاليل الملحية عالية التركيز والآبار العميقة باستخدام طين الحفر القائم على البنتونيت (BN-WBM) أو الطين المائي القائم على الماء (WBMs) تحديًا بسبب التغيرات غير المرغوبة في الخصائص الريولوجية والتصفوية للسائل، مما يؤثر على كفاءة إنتاج البئر. لمعالجة هذه المشكلة، تم تطوير مادة مضافة من نانو هيدروكسي أباتيت (nanoHAp) مقاومة للملح ودرجات الحرارة العالية باستخدام سلفات دوديسيل الصوديوم (SDS). تم إضافة nanoHAp إلى WBMs بتركيز تتراوح بين 0.1 إلى 0.5 وزن %، وتم تكوين BN-WBM مقاوم للملح يحتوي على 4.8 وزن % BN، 5.0 وزن % كلوريد الصوديوم (NaCl)، و 0.5 وزن % nanoHAp. تم تقييم الخصائص الريولوجية والتصفوية لطين الحفر عند درجات حرارة 25، 150، 180، و 210 درجة مئوية. أظهرت النتائج أن nanoHAp زاد من لزوجة WBM بنسبة تتراوح بين 15-139% عند معدل قص 1021 ثانية⁻¹ عبر نطاق درجات الحرارة. كما قلل من فقدان السوائل من 12.1-44.6 مل إلى 6.7-21.8 مل عند جميع درجات الحرارة المختبرة. كما عمل مضاد للملح، قلل nanoHAp من لزوجة BN الملوثة بـ NaCl بنسبة 57% مقارنة بالقيمة المرجعية البالغة 20.8 م.باسكال. ثانية عند معدل قص 1021 ثانية⁻¹ وقلل من فقدان السوائل بنسبة 56.8%، من 169 مل إلى 73 مل عند 210 درجة مئوية. تلتصق مجموعات الرأس الكبريتية الأيونية لـ SDS على سطح nanoHAp بفعالية بسطح BN، مما يمنع أيونات Na⁺ من مهاجمة الهيكل الصفيفي لـ BN. توضح هذه الدراسة أن nanoHAp يمكن أن يمنع تكتل وتفكك BN في المحاليل المشبعة بـ Na⁺ وعند درجات الحرارة العالية.

الكلمات المفتاحية: بنتونيت؛ فقدان السوائل؛ درجات الحرارة العالية؛ نانو هيدروكسي أباتيت (NanoHAp)؛ اللزوجة؛ طين مائي القائم على الماء.

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NOMENCLATURE

API	American Petroleum Institute
AV	Apparent viscosity
BN	Bentonite
BN/HAp	nanoHAp treated mud
BN/Na	Salt polluted bentonite mud
BN/Na/HAp	Salt-polluted mud treated by HAp
BN-WBM	Bentonite-based water solution
Ca/P	Calcium to phosphorus ratio
CaCO ₃	Calcium carbonate
CO ₃ ²⁻	Carbonate groups
DTG	Derivative thermogravimetry
FCT	Cake thickness
FLAPI	Fluid loss at ambient temperature
FLPHT	Fluid loss at high temperatures
HPHT	High-pressure high-temperature
HT POLY SAL	High-temperature polysaccharide
Na ₂ CO ₃	Soda ash
Na ₂ HPO ₄	Disodium hydrogen phosphate
NaCl	Sodium chloride
nanoHAp	Hydroxyapatite nanoparticles
NaOH	Caustic soda
NPs	Nanoparticles
OBMs	Oil-based muds
-OH	Hydroxyl groups
PO ₃ ²⁻	Phosphate groups
PSD	Particle size distribution
SDS	Sodium dodecyl sulphate
SO ₄ ²⁻	Hydrophilic sulphate head groups
TEM	Transmission electron microscope
TGA	Thermogravimetric analysis
WBM	Water-based mud
XC-polymer	Xanthan gum polymer
XRD	X-ray diffraction pattern
ZP	Zeta potential

1. INTRODUCTION

Recently found petroleum resources are mostly located in deeper wellbore sections, often seen as one of the future oilfield's viable locations. This is due to the rapid global expansion of the petroleum industry and the continued increase in demand for gas and oil resources (Al Jaber et al., 2021; Liu et al., 2020). In addition to improving the efficiency of drilling fluids, it is crucial to monitor the drilling fluid effectively to avoid the infiltration of contaminants that might change the fluid's characteristics. Severe alterations in fluid characteristics suggest fluid contamination (Hassiba and Amani, 2013). For an optimal drilling fluid system, parameters like filtration, viscosity, and density of the drilling fluid should remain constant throughout the drilling process. Nevertheless, this rarely happens in reality because unstable wellbore systems stem from complex geological environments often characterized by high salinity and high temperatures (Ali et al., 2013). In addition, drilling fluid characteristics in the field may vary over time, even

when the hole conditions are stable (Abduo et al., 2016; Velden and Jahwari, 2013; Ali et al., 2013; Baris et al., 2007).

Drilling mud has limited applicability in complicated geological environments, such as deep formations with high temperatures and high salt levels. This is due to the fact that most polymers in the drilling mud system degrade at high temperatures and salinities, resulting in fast ageing and significant polymer cross-linking. These geological circumstances are very detrimental to drilling mud treatment chemicals because they alter how the designed drilling muds flow, making them less successful in controlling fluid loss (Zhu and Zheng, 2021). In many drilling areas, both onshore and offshore, salt domes or beds with salt cations like calcium cations (Ca²⁺), magnesium cations (Mg²⁺), or sodium cations (Na⁺) are penetrated. These salts may contaminate the drilling mud that is used in drilling operations, either directly or indirectly (Li et al., 2020). Furthermore, the probability of hitting such a layer during drilling operations is higher in deep wells. In addition, salts may be present by design, such as when salts are introduced to the drilling mud system to produce salt-saturated mud or in offshore operations when seawater is used to prepare drilling mud. When the formation salt comes into contact with an unsaturated liquid, it dissolves, causing hole expansion and excessive rheological property changes (Raheem and Vipulanandan, 2020).

Bentonite (BN) clays in freshwater reduce filtrate loss by forming a filter cake along the wellbore walls (Akhtar et al., 2013). However, when there is a lot of salt in the wellbore and the temperature is high, the charge distribution on the BN clay surface in the drilling mud changes. It is also harder for BN clay particles to absorb water on their surfaces, which leads to a lower zeta potential (ZP), agglomerated BN particles, bigger particles, and unstable drilling mud (Hassiba and Amani, 2013). When BN particles flocculate, they change the rheological properties, pH, and density a lot. They also lose control over filtration, which makes a thick but poor filter cake (Dong et al., 2019; Li et al., 2015; Hassiba and Amani, 2013). According to Liu et al. (2020), adding a high salt content of NaCl damaged the BN clay particle structure at a temperature of 180° C under salt conditions. Raheem and Vipulanandan (2020) also found that drilling salt gypsum strata containing NaCl increases the drilling obstacles proportionally with borehole depth, demanding more from the drilling mud in terms of performance and salt control. This implies that the drilling mud should have high-temperature and high-salt-resistance additives that can function optimally in salty solutions, making it more resistant to salt and more stable at high temperatures (Li et al., 2020; Li et al., 2015).

Furthermore, the dispersion and hydration capacity of BN clays must be maintained under high salinity and high-temperature conditions to enhance the colloidal stability of the drilling mud. This is very important for improving fluid loss control, keeping the wellbore stable, lowering drilling costs, protecting oil and gas reservoirs

from damage, making drilling safer, increasing the efficiency of drilling mud, and encouraging deep gas and oil advancement (Zhu and Zheng, 2021; Li et al., 2020; Al Kindi et al., 2017; Li et al., 2015; Ali et al., 2013). Therefore, utilizing thermal-resistant and salinity-enduring additives can preserve the rheology and colloidal strength of the BN clay mud system under conditions of increased temperature and high salinity, minimizing the risk of drilling difficulties and ensuring the success of drilling operations (Zhu and Zheng, 2021; Wilson et al., 2003).

Water-based muds (WBMs) are increasingly used in areas where oil-based muds (OBMs) were formerly recommended, such as high-temperature, complicated, and water-sensitive shale formations, due to their ecological benignity, operational efficiency, and ease of preparation (Oseh et al., 2023a; Yahya et al., 2023; Salam et al., 2022; Abdo et al., 2017). To improve the rheological and filtration properties of WBM, the muds are primarily prepared to consist of high-functional polymers and BN clay particles. This is to improve mud rheology and lubricity while decreasing water loss from the drilling mud (Al-Yasiri et al., 2019; Pervez et al., 2012). However, WBMs made for the oilfield industry usually break down at temperatures of up to 150–180 °C because they do not hold up well at high temperatures. The only exceptions are synthetic polymers and multi-polymers made with vinyl monomers or sulfonic acid groups. These polymers, such as N-vinylpyrrolidone and acrylamides, have the propensity to resist high salt content and high temperatures (Li et al., 2020). Even though synthetic polymers are better and more stable at high temperatures, ultra-deep wells with high salinity ($\geq 100,000$ ppm) and temperature (≥ 200 °C) still make it hard for the polymers to self-assembly, cross-link, form molecular chains, and stay stable when they break down (Zhu and Zheng, 2021).

NPs have unique physical and chemical properties that can enhance the properties of the drilling muds (Perween et al., 2018). These include superior fluid loss control, lubricity, shale pore plugging, rheology, high-temperature stability, and high salt resistance (Perween et al., 2018). NPs have also been found to diminish the polymer chain mobility at high temperatures, thereby making polymers more heat-resistant (Yang et al., 2017). As a result, hydroxyapatite nanoparticles (nanoHAp) with a chemical formula are designed to resist salt contamination and improve the drilling of high-temperature wells. NanoHAp is non-toxic, affordable, and has fascinating bioactivity and biocompatibility (Chandrasekar et al., 2013). The important features of nanoHAp include an inherent functional group (CO_3^{2-} , PO_4^{3-} , and $-\text{OH}$) suitable for modifying its particles (Córdova-Udaeta et al., 2021). It also possesses exceptional thermal stability, having survived 600 °C without deteriorating (Bulina et al., 2021).

Adding an anionic surfactant like sodium dodecyl sulphate (SDS) to the preparation of drilling fluid additives can make them better in some ways. The anionic SDS is known as a particle modifier that has the potential

to enhance surface chemistry, promote particle dispersion and adsorption, and increase fluid stability (Koroleva et al., 2020). NanoHAp prepared using in situ chemical precipitation with SDS functionalities can inhibit Na^+ from the BN surface under high temperatures and high salinity through the SDS hydrophilic sulphate head group (SO_4^{2-}) interactions with $-\text{OH}$ groups (Ngouangna et al., 2022a; Ngouangna et al., 2022b). This activity will help preserve fluid viscosity and control fluid loss in complex geological conditions.

Chemical precipitation is a commonly used technique for synthesizing NPs that depends on chemical processes. This approach is simple and cost-effective for NP preparation. This technique generates material that is pure and uniform (Ngouangna et al., 2022b). This approach offers some advantages, including a low synthesis temperature (<100 °C) and non-toxic by-products. This approach also enhances material stability by minimizing degradation (Koroleva et al., 2020). To design high-performing nanoHAp particles for potential application in drilling mud systems to inhibit NaCl salt-cations in high salinity and high-temperature environments, an in situ chemical precipitation method was adopted to effectively attach SDS to the nanoHAp surface. The other goal of this method is to make nanoHAp-based drilling fluid systems disperse and stay stable in the wellbore for a long time while improving the drilling mud's rheological and filtration properties.

So, the goal of this study is to create high-performance WBM systems using nanoHAp that was made with SDS through in situ chemical precipitation methods. The drilling muds were specifically designed to function well in environments with high salt and high temperatures. The rheology and filtration characteristics of the nanoHAp in WBM were evaluated at temperatures of 25, 150, 180, and 210 °C. Additionally, nanoHAp's ability to resist salt was tested both before and after it was aged for 16 hours at 210 °C in a BN-water solution.

2. METHODOLOGY

2.1. Test items

Disodium phosphate (Na_2HPO_4), calcium carbonate (CaCO_3), SDS, and sodium hydroxide (NaOH) were used to synthesize nanoHAp particles. Barite, bentonite, high-temperature polysaccharide (POLY SA HT), xanthan gum (XC-polymer), and sodium carbonate (Na_2CO_3) were used to formulate the conventional WBM system, while NaCl was used as a source of contamination for BN-based water solutions.

2.2. Synthesis of nanoHAp particles

Figure 1 shows the in situ chemical precipitation route for making nanoHAp particles, which is similar to previous methods used (Koroleva et al., 2020; Chandrasekar et al., 2013). According to Figure 1, 68.1 g of Na_2HPO_4 powder was mixed in 1000 mL of distilled water at 80 °C/600 rpm for 30 minutes using a magnetic stirrer (Model 05-15, 2000 rpm, 20L). Under 600 rpm stirring, a graduated

burette was used to slowly add 80.1 g of CaCO_3 solution. This ensured that the CaCO_3 dissolved properly. CaCO_3 was slowly added to the Na_2HPO_4 solution to stop calcite from forming. 200 mL of SDS solution (300 mL of distilled water containing 10 g of SDS) was added to the mixture and stirred at 600 rpm, which is the magnetic stirrer's selected stirring speed. The pH was kept at 10 with 0.625 M NaOH, and the solution was stirred at 600 rpm to make a white suspension. With a pH of 10, the solution prevented low calcium formation. To avoid heat shock on the nanoHAp surface, the sample was cooled using a natural air-cooling system. After the precipitates had cooled, they were separated at room temperature for 20 minutes at 6000 rpm using a centrifuged machine (Eppendorf 5810 R centrifuge) and then rinsed three times with distilled water to get rid of any impurities. The sample was made dry using an air-drying kiln method for 1440 minutes at 60 °C and air-cooled naturally, powdered, and characterized.

In this synthesis, SDS may develop complexities with Ca^{2+} ions, causing changes to the behaviour of the nanoHAp and potentially affecting its properties. According to Bricha et al. (2012), complexes of SDS sulphate anions and intermediate Ca^{2+} cations are produced when the precursors are mixed during nanoHAp production. This might result in the partial production of calcium-dodecyl sulphate (Bricha et al., 2012), which would promote the development of nanoHAp in calcium-rich domains (Tang et al., 2008). In this situation, the micelle structure could be retained while nanoHAp production continued on the micelle surface, which functioned as a matrix. Some of the SDS will be integrated into the developing particles, thus the sulphate phase will remain in the final product (Koroleva et al., 2020). Furthermore, the calcium-rich domains will cause nanoHAp particles to form quickly when in contact with phosphate (PO_4^{3-}) ions in the aqueous phase (Koroleva et al., 2020).

According to Tari et al. (2011), the electrostatic interaction effect of SDS molecules causes the positional stabilization of Ca^{2+} ions inside the nanoHAp structure, which benefits the development of ordered nanoHAp crystals. These authors emphasized that the kind of surfactant applied has a significant impact on the shape of synthesized nanoHAp structures. Prasad et al. (2023) highlighted that the presence of SDS sulphate anions had a significant impact on the morphology of the nanoHAp product owing to a strong interaction between SDS sulphate groups and Ca^{2+} ions in the solution and on the surface of nanoHAp particles. This interaction often results in a highly ordered nanoHAp structure. Thus, in this synthesis, the interaction between Na_2HPO_4 and CaCO_3 is thought to result in the rapid production of nanoHAp particles upon contact with SDS sulphate anions in the aqueous phase due to the localized Ca^{2+} concentration effect (Prasad et al., 2023; Tari et al., 2011).

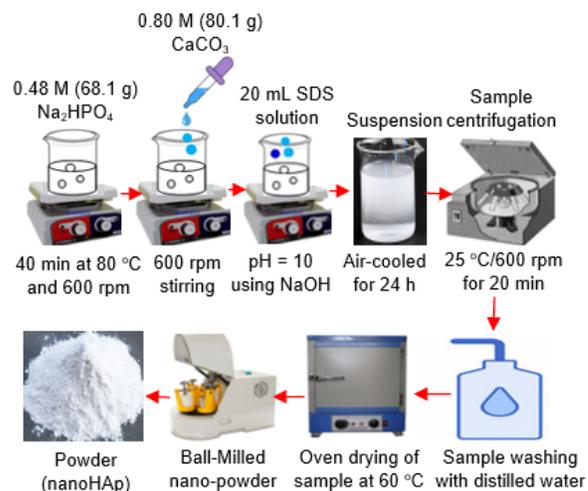


Figure 1. NanoHAp powder synthesis technique with an SDS acting as a particle modifier.

2.3. Characterization of nanoHAp

Different methods were used to characterize the nanoHAp to understand its morphology, crystallinity, and thermal stability. Malvern ZetaSizer (version 7.11, Malvern Instruments, UK) was used to assess the ZP of the nanoHAp and SDS samples in water at different pHs using HCl and NaOH titrations at room temperature. The diameter of the nanoHAp particles (PSD) was analyzed using a particle size analyzer obtained through a light scattering device (ZetaSizer version 7.11, Malvern Instruments). A thermogravimetric analyzer (TGA) (Model TGA Q5000, USA) measured the thermal stability from 20 to 1000° C at 10° C/minutes, while the microstructure was examined using a High-resolution transmission electron microscope (HR-TEM 120.0 kV, Hitachi HT7700, Japan). The elements and crystal pattern of nanoHAp were measured on an X-ray diffraction (XRD) (Bruker D8 Advance, AXS, Germany) apparatus over an angle (scan) range of 20–60° for 2θ .

2.4. Preparation of WBM and BN suspension

Two drilling muds, namely conventional WBM and bentonite-water solution (BN or BN-WBM), were prepared. To formulate a conventional WBM according to API RP 13B-2. (2023) recommended standards, 350 mL of deionized water was mixed with 7% BN, 0.08% Na_2CO_3 , 0.07% NaOH, 0.12% POLY SAL HT, 0.1% XC-polymer, and 21% barite by weight. At 25° C, high-speed mud mixers mixed the slurry for 30 minutes at 10,000 rpm. To evaluate high-temperature resistance, nanoHAp-based drilling muds were formulated by adding concentrations of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% (by weight) to WBM. The study examined nanoHAp's salt-inhibition performance at a concentration of 0.5 wt% in a BN-NaCl-saturated solution, taking into account the potential contamination of drilling mud in deep and very deep wellbores by Na^+ . For the formulation of BN-WBM, the procedures described by Li et al. (2020) were used as follows: In a stainless-steel beaker, 4.8 wt% BN was added to 350 mL of deionized water (pH 9.0, salinity 210 ppm)

and mixed at 10,000 rpm for 60 minutes using a high-speed mud mixer. BN/Na, BN/HAp, and BN/HAp/Na drilling mud systems were prepared by adding 5 wt% NaCl to BN, 0.5 wt% nanoHAp to BN, and 5 wt% NaCl to BN/HAp, respectively. The BN-based suspensions were kept for 24 hours in an airtight container. Thereafter, the rheology and filtration properties were measured.

2.5. Measurement of viscosity

This study used the shear rate-viscosity relationship to evaluate the viscosity of all prepared drilling muds to meet API offshore drilling criteria for the oilfield industry (API RP, 13B-1, 2023). A 12-speed electric modern oilfield Fann rheometer (Model 35A/SR 12) described the drilling fluid viscosities. The rheometer directly measures Newtonian and non-Newtonian fluid rheology. The 35A/SR 12 viscometer's precision, digital display, pre-programmed API tests, and 12-preset speed range allow for greater estimation. This rheometer measured the viscosity of all the prepared drilling muds at 25° C and after 16 hours of ageing in an oven chamber at 150, 180, and 210° C. Equations (1), (2), and (3) were used to describe the viscosities of drilling muds by taking 12 dial readings (600, 300, 200, 180, 100, 90, 6, 30, 6, 3, 1.5, and 0.9 rpm) equivalent to the shear rates (1021, 511, 340, 306, 170, 153, 102, 51.1, 10.2, 5.1, 3.1, and 1.5 s⁻¹), respectively.

$$\text{Shear rate (s}^{-1}\text{)} = 1.7032\omega \quad (1)$$

$$\text{Shear stress (Pa)} = 0.511\phi \quad (2)$$

$$\text{Apparent viscosity (mPa} \cdot \text{s)} = 300 \times \phi / \omega \quad (3)$$

ϕ = 12-speed viscometer readings (°) and ω = rotor speed (rpm).

2.6. API and HPHT filtration tests

The prepared mud samples were placed on an API filter meter (Fann API filter press, Series 300, USA) to examine the API fluid loss (FLAPI) at an applied pressure of 100 psi for 30 minutes. The fluid losses under HPHT conditions (FLHPHT) of each mud sample were measured using an HPHT filter press API filter meter (Fann HPHT filter press, Series 3000, USA). A thermostat-controlled heating jacket heated each mud sample to the selected temperatures of 150, 180, and 210 °C in the mud cell (pressure difference of 500 psi) for 30 minutes. Following disassembly and cleaning, the thickness of the formed filter paper's cake (API and HPHT FCT) was measured using a vernier calliper.

3. RESULTS AND DISCUSSIONS

3.1. Material characterization data

The study's analysis is based on primary data collected from characterization tests and experimental work carried out using the proposed nanoHAp. The characterization data of nanoHAp powder are presented

(Figures 2-6). Highly dispersed NPs have a ZP index of ≤ -30 mV or $\geq +30$ mV. In the stable region, pure SDS powder (without nanoHAp) and the synthesized nanoHAp powder have a higher ZP at pH > 10. During the synthesis, NaOH was employed to attune nanoHAp solution pH to 10, in which the nanoHAp ZP absolute value is 43.2 mV, while that of SDS is 55.6 mV, indicating electrostatic colloidal stabilization according to the colloidal stability indicator (Elochukwu et al., 2017). At a maximum pH of 11.7, the absolute ZP magnitudes of SDS and nanoHAp are 71.7 and 47.6, respectively (Figure 2). These ZP magnitudes demonstrate that nanoHAp has the stable dispersion properties desired for drilling fluid systems (Elochukwu et al., 2017) (Figure 2). NanoHAp particles are negatively charged even without SDS because the reaction system's electrolytes and reaction products form an electrical double layer (Koroleva et al., 2020). When SDS was added, a surfactant bilayer formed on the surface of the nanoHAp. This bilayer protected the growing crystals' surface, lowering the ZP to a stable region in an alkaline medium.

Figure 3 shows that nanoHAp is predominantly 70–600 nm in diameter, with a peak frequency of 105.7 nm. The mean particle size is 110.4 nm, while the distribution standard deviation is 38.3 nm. D10, D50, and D90 diameters are, respectively, 22.2 nm, 91nm, and 189.8 nm.

Introducing nanoHAp particles to WBM can enhance the interaction between drilling muds and cuttings. The reason for this is that the nanoHAp particles are much smaller in size compared to particles of micro or macro dimensions. To enhance the transport of drilled cuttings to the surface, one possible approach is to enhance the colloidal contacts between nanoHAp particles and the cuttings. This may result in a notable increase in cutting transport efficiency with WBM containing nanoHAp particles (Abbas, 2021; Boyou et al., 2019).

SDS inhibited the growth of crystals on the surface of nanoHAp, resulting in smaller particles ranging in size from 70 to 600 nm (Koroleva et al., 2020). In Koroleva et al.'s (2020) HAp synthesis with SDS, sodium phosphate and calcium nitrate were added to make rod-shaped HAp NPs. Adding a 1 M NH₄OH solution maintained a constant pH of 9.0 ± 0.2 in the reaction medium and formed an SDS bilayer on the HAp surface, protecting the developing crystals and reducing the nanorod size. Using a TEM micrograph, the authors measured the rod-like HAp particle sizes. The measured particles had lengths of 35–400 ± 5 nm and a diameter of 20–30 nm. The HAp did not agglomerate because of electrostatic repulsion, indicating that HAp suspensions are stable with respect to sedimentation (Koroleva et al., 2020). This pH range and nanorod size are similar to the pH of 10 that was used in the current study to make nanoHAp with SDS. As shown in Figure 4, the nanoHAp rods' lengths were measured by TEM and are between 20 and 160 nm, with a mean rod length of 80 nm. Their diameters are between 10 and 30 nm. These values are similar to those reported by

Koroleva et al. (2020) for the HAp rod lengths and diameters.

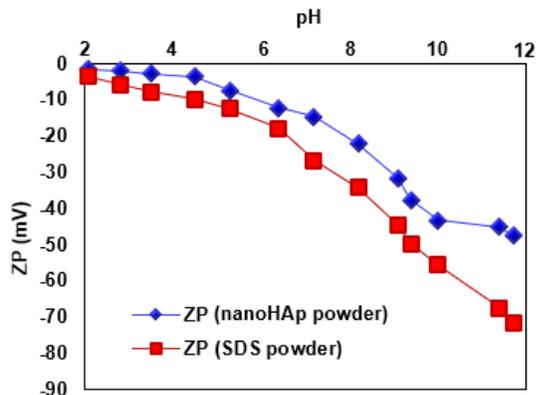


Figure 2. ZP magnitudes of nanoHAp and SDS.

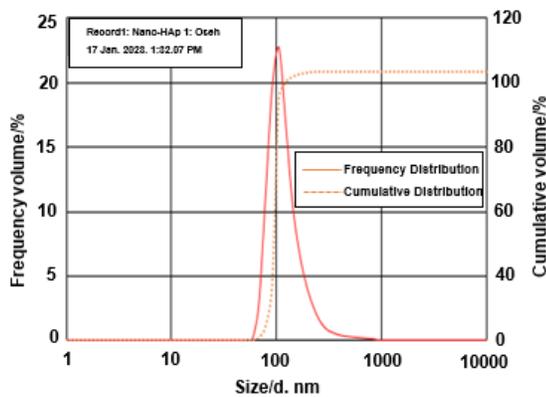


Figure 3. PSD of nanoHAp.

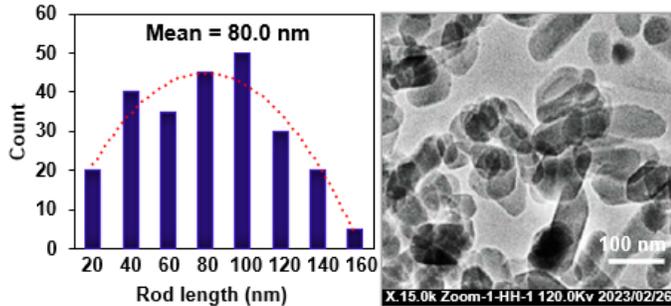


Figure 4. TEM micrograph of nanoHAp.

So, the nanoHAp TEM micrograph shows rod-shaped particles distributed in a way that matches PSD data in the nanometric range (Figure 4).

This structure is often desired in drilling operations to aid in reducing fluid loss and plugging shale pores (Perween et al., 2018; Zarei et al., 2018). Given their nano size, the nanoHAp particles can make a filter cake that is thin, firm, and low-permeable, which is applicable to other small-sized particles in the nanometric domain (Perween et al., 2018; Shakib et al., 2016). The nanoHAp particles can fill in the spaces between bigger particles, such as bentonite (bridging material) or fluid loss-reducing polymeric agent in a drilling solution to build a strong and dense protective barrier (filter cakes) that protects the wellbore wall, thereby retarding the flow of

drilling fluid into the drilled formation (Yahya et al., 2023; Shakib et al., 2016). In addition, the nanoHAp particles owing to their sized dimension can penetrate the shale micro and nanopores to plug the narrow openings in the pores, thereby hindering the mud's quick penetration into the shale, which will help reduce the rate of pore pressure transmission in shale; hence, hindering the capacity of the clay in the shale to swell (Zarei et al., 2018).

Figure 5 reveals that nanoHAp is very thermally stable up to 750° C, retaining 73% (i.e., 27% deterioration) of its original form. This makes it suited to drilling deeper formations. The three peaks on the derivative weight curve (DTG) indicate that the SDS-supported nanoHAp breaks down in three different ways. The stability of the particles dropped at 850° C. These results are consistent with previous studies conducted by Bulina et al. (2021) and Koroleva et al. (2020). This finding also provides confirmation that SDS molecules are present in the nanoHAp structure. The observed peak at 70 °C is attributed to the desorption of water molecules that were previously adsorbed onto the sample. The temperature at which the peak occurs, namely around 270° C, is attributed to the comprehensive breakdown of -OH groups as well as the alkyl and sulphate chains of SDS. At 850° C, the PO₄³⁻ and CO₃²⁻ parts of the particles that make up the SDS-supported nanoHAp layer completely break down. This means that the nanoHAp particles are no longer stable up to 850° C.

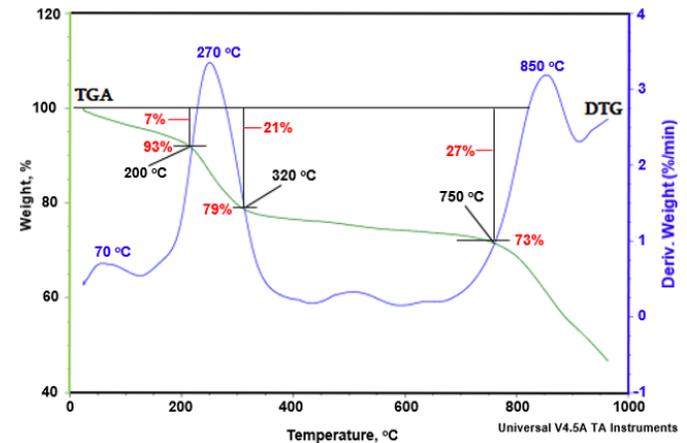


Figure 5. TGA of nanoHAp.

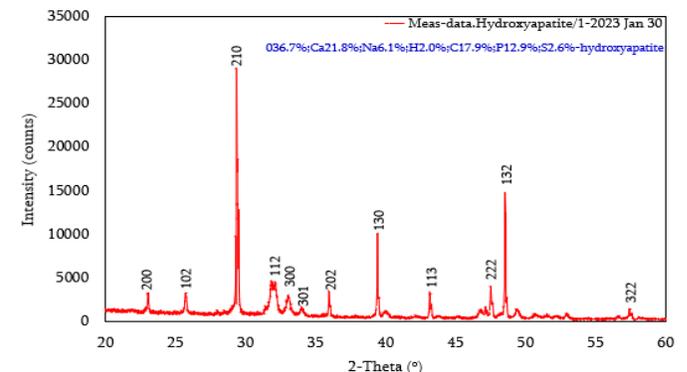


Figure 6. XRD pattern of nanoHAp particles.

NanoHAp has strong and weak diffraction peaks that match the stoichiometric HAp values from ICDD® 9-0432 and other sources (Koroleva et al., 2020). NanoHAp consists of Ca/P and SDS parts (blue label), and its Ca/P ratio is almost the same as that of stoichiometric HAp (1.664 ± 0.005). This shows that the particles were made correctly. This finding also provides confirmation that SDS molecules are present in the nanoHAp structure.

3.2. Viscosity and filtration properties of nanoHAp at high temperatures

This section contains the experimental data collected in the laboratory, as well as their interpretations. At ambient temperature (25° C), the viscosity of WBM with nanoHAp concentrations was first determined. Afterwards, WBMs were aged at 150, 180, and 210° C for 16 hours with 0.1, 0.2, 0.3, 0.4, and 0.5 wt% nanoHAp to test their viscosity variation with temperature (Figure 7).

At 25° C, the effective viscosities of the drilling solutions are 48.8, 56.1, 62.1, 67.2, 73.8, and 80.4 mPa. s for 0.0 wt.% (WBM), 0.1, 0.2, 0.3, 0.4, and 0.5 wt.% concentrations of nanoHAp, respectively, at 1021 s⁻¹ shear rate (Figure 7a). After ageing for 16 hours, drilling mud viscosity decreased with increasing temperature. The viscosity curves further demonstrate that the viscosity rises with increasing nanoHAp content and decreases with increasing shear rate. The mud particles' intermolecular interactions and associations are what cause the shear viscosity to rise and the thermal stability to increase at high temperatures (Liu et al., 2020). However, at higher temperatures, mud molecules experience increased energy and detach from their mutual traction, resulting in reduced viscosity and increased fluid loss volume (Table 1).

The viscosity curves show that the changes in viscosity with temperature are different for different amounts of nanoHAp at a shear rate of 1021 s⁻¹ (Figures 7e and f). The nanoHAp mud systems, between 0.1 and 0.5 wt%, significantly increased the viscosity of the base mud at all

temperatures and shear rates. At 25 and 150° C, nanoHAp recorded a viscosity increment of 14.9–64.8% and 23.5–85.1%, respectively. Additionally, nanoHAp increased the viscosity of the base mud by 40.5–116% at 180° C and 30.1–139% at 210° C, both with a shear rate of 1021 s⁻¹ (Figures 7a–d).

As the temperature rises, the viscosity of the base mud decreases the most, compared to that of nanoHAp drilling solutions. The nanoHAp mud system decreases with increasing temperature (Figure 7e); however, the percentage decrease is less in an increasing nanoHAp concentration based on the control temperature (T = 25° C) (Figure 7f). This could be because, with an increase in temperature, the drilling solutions retain more of their active colloidal particles in mud systems with higher nanoHAp concentrations (Liu et al., 2020; Mao et al., 2015).

The viscosity of WBM decreased significantly at different shear rates of 1021 s⁻¹ after 16 hours of ageing at temperatures of 150, 180, and 210° C. Specifically, the viscosity decreased by 14.6%, 31.1%, and 41%, respectively, compared to the control temperature of 25° C (Figure 7f). When the nanoHAp mud systems were heated from 25 to 210° C, their viscosities dropped by 8.19 to 26.6% (0.1 wt%), 5.31 to 20.5% (0.2 wt%), 5.65 to 18.8% (0.3 wt%), 6.78 to 18.3% (0.4 wt%), and 3.98 to 14.4% (0.5 wt%) compared to 25° C (Figure 7f). Increasing temperatures have a significant impact on drilling solutions due to the rise in their average kinetic energy. This leads to molecules surpassing the intermolecular attractive forces, resulting in a reduction in the viscosity of the drilling mud (Elochukwu et al., 2017). As previously mentioned in Figures 7e and f, the nanoHAp particles demonstrated superior thermal stability in comparison to the base mud. This finding is further supported by the TGA results presented in Figure 5.

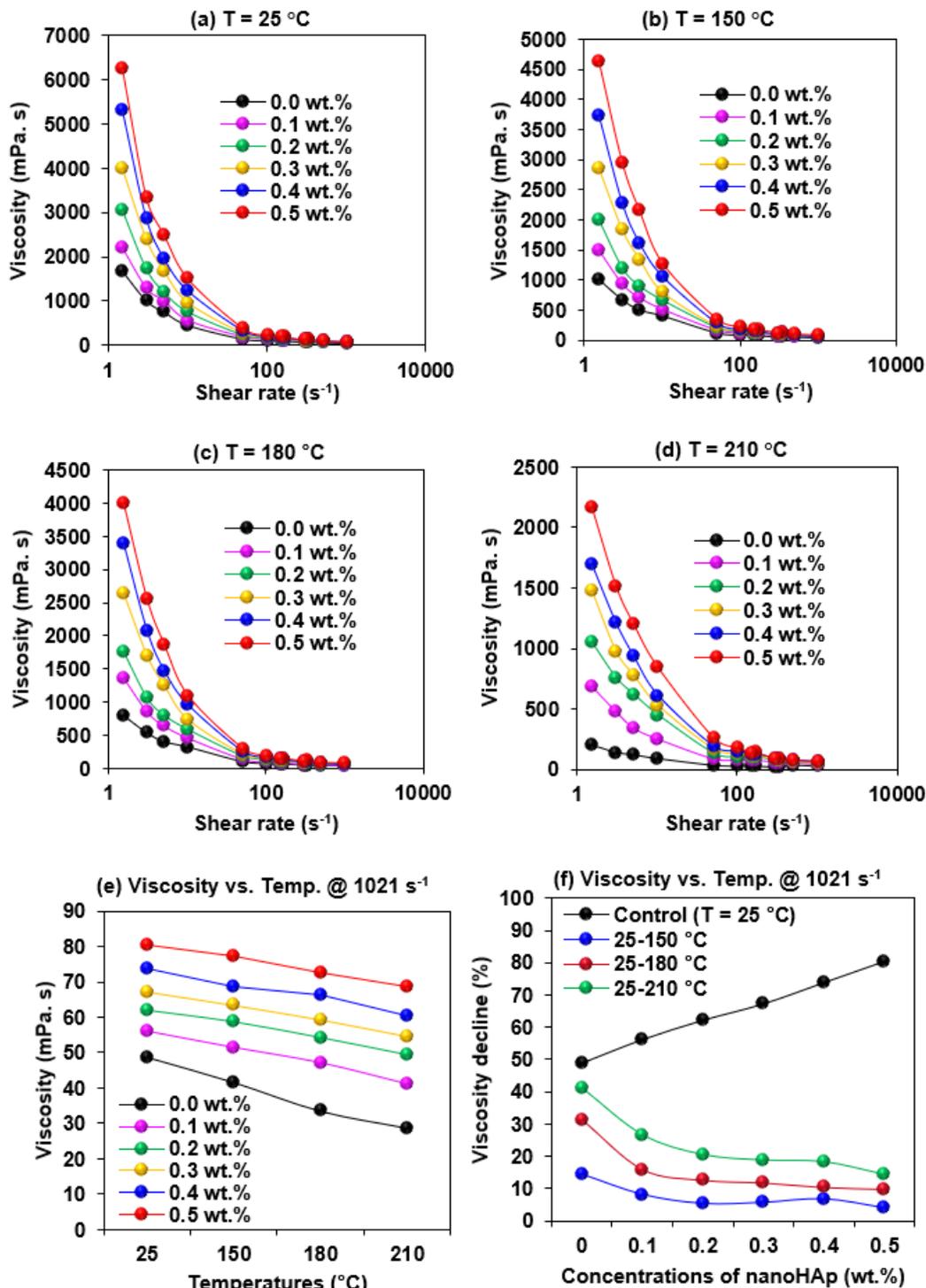


Figure 7. Viscosity curves of nanoHAp-based drilling muds: (a) 25° C, (b) 150° C, (c) 180° C, (d) 210° C, (e) changes in viscosity against temperature at 1021 s⁻¹ shear rate, and (f) percentage decline in viscosity with temperatures at 1021 s⁻¹ shear rate.

The findings recorded in Figures 7a–f reveal that nanoHAp can survive temperatures up to 210 °C, which is important for ultra-deep well drilling (Liu et al., 2020). The nanoHAp particles containing SDS particles are very good at adsorbing onto polymer and clay surfaces, which lets them participate in polymer-clay adsorption sites in the WBM. This effect makes it less likely for the particles to separate while strengthening the mud's polymer-clay matrix (Oseh et al., 2023a; Oseh et al., 2023b).

Furthermore, the internal friction between the mud particles in suspension became stronger with nanoHAp particles compared with the base mud. This phenomenon led to a much slower decrease in the viscosity of the nanoHAp mud systems (Yang et al., 2017).

This finding is consistent with several studies on the viscosity of NP-based drilling fluids. Graphene nanoplatelets (Yahya et al., 2023), zinc titanate NP (Perween et al., 2018), nano silica (Al-Yasiri et al., 2019;

Elochukwu et al., 2017), and functionalized cellulose nanocrystals (CFCs) (Li et al., 2020) have all been studied. These authors believed that decreasing particle size resulted in increased particle viscosity. Increasing the weight or volume concentration of NPs in drilling muds can lead to increased viscosity owing to enhanced molecular interaction with the base mud. However, the viscosity ratio could rise nonlinearly with increasing NP concentration (Perween et al., 2018). At different shear rates, it was also revealed that the viscosity of NPs varies with temperature in the mud system. At higher concentrations, viscosity decreases with increasing temperature, while at lower concentrations, temperature has no significant influence on viscosity (Abduo et al., 2016). Overall, the viscosity properties of various NP-based fluids were found to be stable and favourable for enhanced drilling operations; however, they should be continuously monitored and improved throughout the drilling process.

Tables 1 and 2 summarize the filtration properties of nanoHAp wt% concentrations applied to base mud. As demonstrated in Table 1, increasing the concentration of nanoHAp increased the WBM's capacity to decrease fluid loss, resulting in a reduction in both FLAPI and FLHPHT. As the temperature increased, the amount of fluid loss in all the mud systems increased. Between 25 and 210° C, WBM fluid loss ranged from 12.1–44.6 mL. However, at nanoHAp doses (0.1–0.5 wt%), it drastically decreased. For example, between 0.1 and 0.5 wt%, following 16-hour jacket heating at 210° C, nanoHAp decreased the FLHPHT from 44.6 mL to between 37.2–21.8 mL as its concentration increased.

Increasing temperatures also increased the FCTAPI of the mud samples, but the FCTHPHT values were within the recommended API acceptable limits for high temperatures (Shakib et al., 2016) (Table 2). Moreover, nanoHAp showed excellent fluid stability at high temperatures. For example, at 210° C, adding 0.1 to 0.5 wt% of nanoHAp improved the base mud's ability to control fluid loss by 16.6 to 51.1% as the concentration increased (Table 1). The developed nanoHAp provides coating properties to the WBM, which covers the filter paper's pores, preventing water from passing through. Due to their high viscosity, nanoHAp particles effectively form a protective barrier on the borehole wall, thereby preventing water leakage into the reservoir. NanoHAp-based drilling muds have rheological and filtrate loss control properties that can effectively mitigate differential pipe sticking, enhance hole cleanliness, and improve the drilling mud's performance.

Table 1. Fluid loss data of nanoHAp after 30-minutes

Samples	FLAPI (mL)		FLHPHT (mL)		Δ FL @ 210
	25° C	150° C	180° C	210° C	
0.0 wt.%	12.1	19.7	26.8	44.6	-
0.1 wt.%	10.9	17.2	21.3	37.2	-16.6
0.2 wt.%	9.2	15.7	18.2	31.3	-29.8
0.3 wt.%	8.7	13.6	16.6	26.3	-41.0
0.4 wt.%	7.9	12.7	15.1	24.4	-45.3
0.5 wt.%	6.7	11.8	13.2	21.8	-51.1

Table 2. Cake thickness of nanoHAp after 30-minutes.

Samples	FCTAPI (mm)		FCTHPHT (mm)	
	25° C	150° C	180° C	210° C
0.0 wt.%	1.83	2.79	3.64	4.10
0.1 wt.%	1.62	2.66	3.18	3.96
0.2 wt.%	1.53	2.44	3.11	3.82
0.3 wt.%	1.49	2.34	3.08	3.74
0.4 wt.%	1.35	2.28	2.93	3.67
0.5 wt.%	1.32	2.23	2.81	3.52

3.4. Salt resistance investigation of BN suspension

0.5 wt% nanoHAp was introduced into 4.8 wt% BN with 5 wt% NaCl to test nanoHAp's salt resistance in BN-WBM. NanoHAp in BN-WBMs with Na⁺ at 25° C and after 16 hours of ageing at 210° C are shown in Figure 8. The graphs show the viscosity, FLAPI, FLHPHT, FCTAPI, and FCTHPHT. As shear rates increased, the viscosities decreased. At a 1021 s⁻¹ shear rate, the BN has a viscosity of 5.5 mPa. s, and it increased unduly to 113.1 mPa. s with a NaCl addition of 5.0 wt% at 25° C. The viscosity of BN/Na changed from 113.1 mPa. s to 40.9 mPa. s when nanoHAp was added to the mud system (Figure 8a). Furthermore, at 210° C, the BN viscosity increased from 1.55 mPa. s to 20.8 mPa. s under NaCl conditions, rising 13 times (1242%). However, when 0.5 wt% nanoHAp was added, it increased from 6.43 mPa. s (about 4-fold) by 315% to 8.91 mPa. s (about 5.7-fold) by 475% (Figure 8b). With nanoHAp, BN HPHTFL at 210° C reduced from 71 mL to 33 mL, indicating a 53.5% drop. However, when Na⁺ was added, it increased to 169 mL, indicating a 138% rise (Figure 8c). Also, with 0.5 wt% nanoHAp, the FLHPHT of a BN-NaCl-contaminated solution of 169 mL decreased by 56.8% to 73 mL. For all mud systems, the fluid losses are much lower at API conditions. The FLAPI of the BN was 22 mL, and it decreased substantially to 10.8 mL with 0.5 wt% nanoHAp. However, with 5.0 wt% NaCl in the BN-WBM, the FLAPI increased notably to 56 mL. When the nanoHAp salt-treating agent was added to the BN/Na mud system, the FLAPI dropped by 52%, from 37.2 mL to 26.8 mL. In general, based on these results, BN properties can be considered undesirable for drilling operations when a high concentration of NaCl is present, which means that it might not be a good additive for suspending and moving cuttings (Zhu and Zheng, 2021). It may also cause circulation loss, mud loss, stress, and formation fracture (Oseh et al., 2023b; Yang et al., 2017). NanoHAp made the BN-salt-contaminated solution less thick because the counter-ion hydration shell was partially broken, prompting the recurrence of less counter-ion hydration (Raheem and Vipulanandan, 2020).

Figure 8d shows that NaCl increased the FCTAPI and FCTHPHT of the BN mud. When nanoHAp was present, the FCT decreased at both temperatures, demonstrating that nanoHAp can prevent pipe sticking (Oseh et al., 2023c; Perween et al., 2018). 5.0 wt% of NaCl thickens the filter cake more, making it more porous and permeable, which reduces the cake's water-blocking capacity (Al Jaber et al., 2021). With nanoHAp, the BN-based solution

filter cake was thin, smooth, and compact without notable porosity or agglomeration, proving that nanoHAp can enhance BN filter cake quality. NanoHAp kept its great ability to stick to the BN surface after being aged at 210° C. This prevented the particles from sticking together and flocculating, making the BN-based suspension more resistant to NaCl contamination while still forming high-quality filter cake at high temperatures.

Figure 9 shows the use of nanoHAp to screen NaCl from the BN surface. When NaCl was added to the BN suspension, the BN mechanism transitioned from exfoliation (Figure 9a) to flocculation (Figure 9b). The cross-linking agent Na⁺ (monovalent cations) interacted with the BN platelets "edge-to-face" so that they could penetrate the BN microstructure and cause flocculation. The viscosity of the BN-water solution rose considerably due to the NaCl-induced platelet flocculation. BN particle flocculation and coalescence at high temperatures significantly decreased the BN platelets' filtration barrier property (Liu et al., 2020), increasing the filtrate loss and thickening the filter cake. NanoHAp particles stuck to the edges of BN particles through their SO₄²⁻ hydrophilic head groups. They blocked Na⁺ from reaching the BN-ion-susceptible spots (Figure 9c). As a result, the addition of nanoHAp particles inhibited BN particle flocculation and promoted fluid dispersion stability, preserving the BN platelet's plate-like structure (Figure 9d). Subsequently, the viscosity and fluid loss in the BN/Na fluid system decreased substantially.

Figure 9b demonstrates that when the Na⁺ in the drilling mud system dissolves, it causes BN-WBM to flocculate. NaCl's positive charge attracts and binds to the BN clay particles' negative charge. This makes the particles bigger, which then causes them to settle out of the mud in which they were suspended (Akpan et al., 2020). In the BN/Na-contaminated mud, the nanoHAp anti-salt agent is a sticky gel-like substance that does not include clay. The drilling fluid's tolerance to Na⁺ dissolution was enhanced, and the number of reactive particles in the solution was reduced due to the nanoHAp layer that adsorbed on the BN/Na mud system, which was caused by the sulphate groups (SO₄²⁻) of nanoHAp. As a result, the BN/Na mud system was able to reduce the impact of the Na⁺, making the drilling mud more resistant to saturated NaCl solution.

In summary, at a high temperature of 210° C, nanoHAp increased the rheology of drilling fluids, decreased the fluid loss volume at deeper depths, and resisted Na⁺ contamination. It optimizes BN colloidal suspension stability and controls the fluid properties of BN under Na⁺ assault at both low and high temperatures, which could be advantageous for drilling high-temperature wells. Using nanoHAp to decrease the amount of NaCl in BN-WBM would also improve the mud's viscosity, lubrication, water loss management, and other properties. It could also protect drilling equipment better, making the process more efficient. Additionally, it will make the mud more effective in removing cuttings and other debris from the hole.

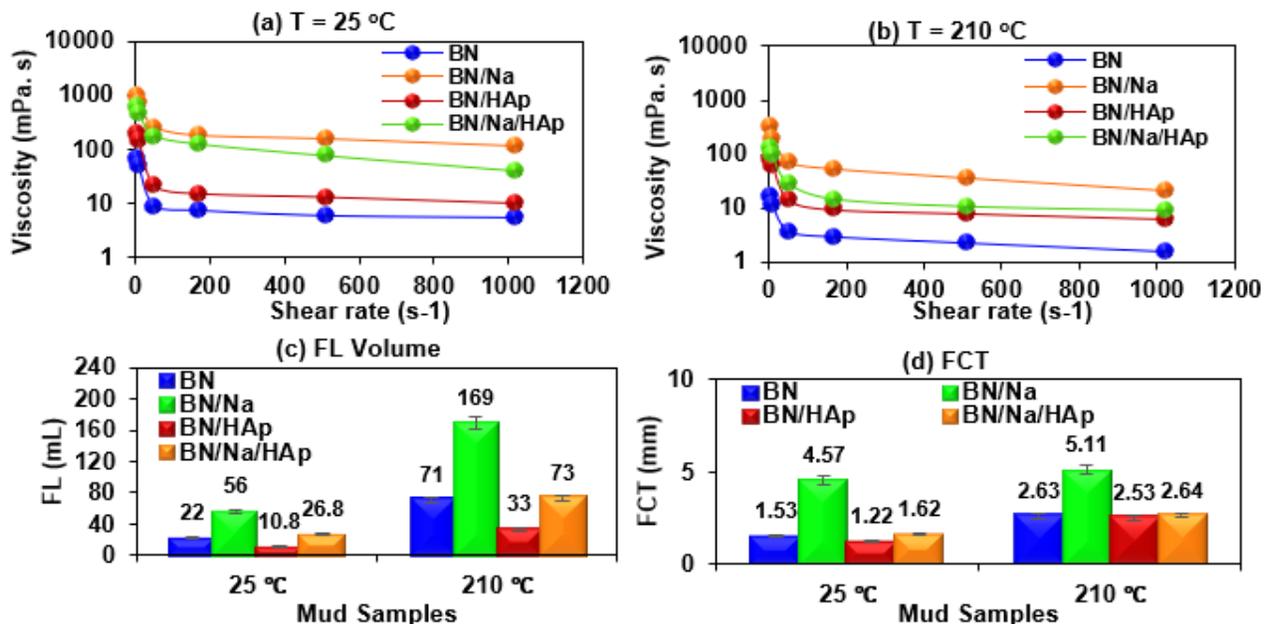


Figure 8. BN suspension with NaCl and nanoHAp (a) viscosity at 25° C (b) viscosity at 210° C (C) FL, and (d) FCT.

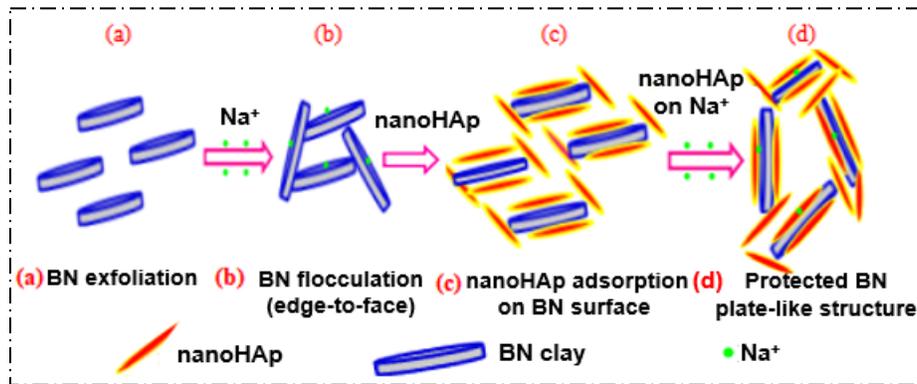


Figure 9. Salt-screening process: (a) exfoliated BN microstructure, (b) BN plate-like structure damage by sodium ion, (c) nanoHAp attachment, and (d) nanoHAp screening of sodium ion.

4. CONCLUSIONS

In this study, a salt-resistant, high-temperature viscosities and fluid loss reducer additive termed nanoHAp was produced. The salt-resistant and high-temperature performance of nanoHAp in WBM and BN-water solutions at temperatures of 25, 150, 180, and 210° C was examined. A summary of the key findings is itemized as follows:

1. PSD, ZP, XRD, and TEM authenticated the nanoHAp production. TGA/DTG indicated nanoHAp's excellent thermal stability, while ZP confirmed its good colloidal stability.
2. Filtration and rheological tests showed that the presence of different concentrations of nanoHAp significantly reduced the fluid loss volume and increased the apparent viscosity of the base mud at an increasing concentration.
3. The optimal nanoHAp concentration of 0.5 wt% stayed stable for 16 hours at 210 °C and kept 85.6% of its original viscosity of 80.4 mPa. s, which was measured at 25 °C and 1021 s⁻¹ shear rate. In contrast, the base mud only kept 59% of its original viscosity of 48.8 mPa. s, which was measured at the same conditions.
4. Furthermore, the salt screening data demonstrated that the interactions between nanoHAp particles and BN clays were not completely destroyed in the inhibition of NaCl assault at 210 °C.
5. After 16 hours of ageing at 210 °C, the saturated salt filtration of a BN/Na suspension of 169 mL was reduced by 56.8% with nanoHAp.
6. The combined action of CO₃²⁻, PO₄³⁻, SO₄²⁻, and intermolecular interactions through the hydrophobic alkyl side chains made the nanoHAp more stable at high temperatures when the salt levels were high. The SO₄²⁻ head groups adsorb on the Na⁺ ions and protect the normal plate-like structure of BN.
7. The findings of this study reveal that nanoHAp has excellent viscosity and filtration control under Na⁺ assault and can withstand ultra-high temperatures of

210° C after 16 hours of ageing, making it suitable for drilling deep and ultra-deep formations.

5. LIMITATIONS AND FUTURE SCOPE

There have been several efforts to produce nanoHAp, but chemical precipitation synthesis stands out since it is simple to use, has a low synthesis temperature, is inexpensive, produces smaller particles, and is very pure (Prasad et al., 2023). Furthermore, most nanoHAp particles are synthesized in surfactant-containing solutions to slow crystal growth and prevent particle aggregation. However, varying surfactant concentrations during nanoHAp production will have a considerable impact on the size and shape of the resulting particles. This will help to determine the structure and size of the nanoHAp particles that are created. Also, buffers should be used to keep the pH of the synthesis solution the same while monitoring changes in the pH of the experimental medium when controlled chemical precipitation is used to make nanoHAp. This is due to the fact that pH plays an essential role in nanoHAp chemical processes by changing the ionization state of the particles. This alters the reaction rates and general chemical behaviour of the nanoHAp particles that are produced. The authors believe that nanoHAp's in situ chemical precipitation synthesis technique with different surfactant concentrations, the detection of pH changes, and the addition of a buffer to the synthesis solution provide several prospects for further study. These findings may lead to positive advances in the design of nanoHAp additives for fast-tracking nanofluid enhancements.

CONFLICT OF INTEREST

All authors have reviewed and endorsed this work, confirming the absence of any competing interests.

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