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The effect of hydroxyapatite nanoparticles on wettability and brine-oil interfacial tension as enhance oil recovery mechanisms

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ABSTRACT

A novel concept of utilizing nanoparticles (NPs) to boost oil recovery and reduce entrapped oil in hydrocarbon reservoirs is being explored. The use of nanofluids (NFs) flooding to change wettability and reduce interfacial tension (IFT) between oil and water has been shown to be highly effective in experiments. Preparation and modification methods influence the performance of NPs. The application of hydroxyapatite NPs (HAP) in EOR has yet to be investigated. HAP was synthesized in this study using the co-precipitation method and in-situ surface functionalization with Sodium Dodecyl Sulphate SDS to visualize it effect on IFT reduction and wettability alteration under different salinity and temperature settings. To confirm the synthesis of HAP, zeta potential (ZP), Fourier transform infrared spectroscopy (FTIR), Particle size analysis (PSA), Transmission electron microscopy (TEM), and Energy dispersive X–ray (EDX) spectra were used respectively. According to the results, HAP was produced, and the particles were finely distributed and stable in aqueous solution. When the pH was altered from 1 to 13, the particles' surface charge rose from -5mV to −27mV, showing long-term stability in EOR processes. At salinity range from 5000 ppm to 30,000 ppm, and under temperature range from 25°C to 80°C, the HAP NFs changed the wettability of sandstone core from oil–wet at 111.7° to water–wet at 9.0°. In addition, at 0.1 wt% HAP concentration, the IFT was lowered to 3 mN/m. As a result, the HAP NF proved very effective in reducing IFT and altering wettability under both low and high salinity environments, and it is thus suggested for EOR operations.

1. Introduction

Crude oil is one of the most important existing fuel sources, accounting for more than 33% of total global energy consumption (Gielen et al., 2019). Indeed, it is expected that global energy demand will increase by 50% over the next 30 years, compared to its current level (Yarima et al., 2022). Due to the current scenario, it is more important than ever to enhance oil output. Primary oil recovery technologies have increased oil production by allowing crude oil to migrate towards the production well without being stimulated by external factors (Nikolova and Gutierrez, 2020). In a greenfield development approach with good reservoir characteristics, 20–40% of hydrocarbon (H–C) recovery is achieved through primary and supplementary procedures, while 60–70% of oil remains in the reservoir (Babadagli, 2007; Nwidee et al., 2016). and can only be recovered through enhanced oil recovery (EOR), a tertiary oil recovery technology (Negi et al., 2021).

EOR entails integrating cutting-edge technologies including chemical, gas, microbial improved oil recovery, and thermal energy to further improve H–C recovery. (Yernazarova et al., 2016; Nwidee et al., 2016; Nikolova and Gutierrez, 2020). Through rock-fluid contact, EOR causes a reduction in interfacial tension (IFT) between displacing and displaced fluids, wettability changes, and improved drive water viscosity and

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mobility-control, as well as oil swelling and viscosity reduction (Manshad et al., 2017; Gbadamosi et al., 2019; Ngouangna et al., 2020). EOR agents have the ability to alter reservoir dynamics and achieve more favorable oil recovery (Mogghadom et al., 2015; Negi et al., 2021; Yakasai et al., 2021a). EOR procedures are frequently used when an oil reservoir's natural energy or (secondary) water injection cannot successfully generate oil (Farajzadeh et al., 2021).

Chemical enhanced oil recovery (CEOR) has been studied as a successful oil recovery technology for the recovery of bypassed oil and residual oil retained in the reservoir. The chemicals used in EOR include foam, alkali, surfactants, polymers, and NFs (Gbadamosi et al., 2019). They can be used individually, but in most circumstances, combining two or three has proven to be more effective due to synergy. Chemical injections are used in this EOR method to help in oil recovery. It's either capillary forces capture the oil (residual oil) or the oil is bypassed in some other way (Yang et al., 2021). The bypassed oil is caused by heterogeneity in the reservoir or a poor mobility ratio between the aqueous and oleic phases. In contrast, residual oil is made up of discrete ganglia that occur when an oleic mass protrusion forms a narrow neck as a result of the combined effects of local pressure gradient and interfacial stress (Gbadamosi et al., 2019). The question is how to extend the life of the field by using cost-effective and environmentally acceptable enhanced oil recovery (EOR) methods. Alkali–surfactant (AS), surfactant/polymer (SP), alkaline/polymer (AP), and alkaline/surfactant/polymer (ASP) are chemical EOR (CEOR) slugs. In oil wells, the synergy of the combined conventional chemicals boosted efficiency. Recent studies have demonstrated that foam enhanced with surfactants and polymers for greater stability and mobility control can improve oil recovery (Gbadamosi et al., 2019) In the oil business, polymers especially polyacrylamide (PAM) is frequently utilized, notably for increased oil recovery. However, due to its poor salt tolerance and thermal stability, the use of this water-soluble polymer in highly salinized and high temperature oil reservoirs is restricted (Al-Anssari et al., 2021). Nevertheless, under simulated high temperature and salinity oil reservoir conditions, Zhu et al. (2013) evaluated the rheological characteristics of partially hydrolyzed polyacrylamide/amidobetaine surfactant hybrid and core flooding experiments. The hybrid samples showed better salt tolerance and long-term thermal stability, according to the data. Surfactants also called surface-active agents, are absorbable at low concentrations. They can improve oil recovery by converting hydrophobic rock matrix to hydrophilic and decreasing interfacial tension (IFT), which displaces trapped crude oil in microscopic pore spaces with water. This mechanism of pore-scale recovery is connected to sweep efficiency, which is controlled by viscous and capillary forces and defined by IFT and contact angle (wettability measurements) (Ghasem et al., 2020). However, the emergence of nanotechnology creates an unprecedented opportunity to alter the oil and gas business in the upstream, downstream, and midstream sectors.

The Nano-solution acts as a property modifier, lowering the IFT and easing the transition from oily to watery wettability, as well as modifying the rheology of formation fluid in situ. (Negi et al., 2021). As fossil-based energy technologies are developed over the next 30 years, nanotechnology is predicted to completely revolutionize the way oil and gas resources are exploited (Mohanty et al., 2021). In comparison to their large-scale counterparts, the uses of NPs in the petroleum industry are primarily owing to their nanoscale dimensions, which reveal fascinating qualities and enable the modification and control of numerous aspects (Yarima et al., 2022; Yakasai et al., 2021a). Furthermore, NPs have a reasonable resistance to deterioration at high salinity and temperature in oil and gas reserves. Surfactant solutions with NPs as NFs have also been explored in harsh reservoir settings to boost oil recovery. In contrast to traditional CEOR compounds, NPs have shown significant benefits in EOR applications (Sharma et al., 2016., Olayiwola and Dejam, 2019). Because of their small size, they can be forced into pores that larger materials cannot access without becoming caught (Khan et al., 2017). Their high surface energy and reactivity can alter fluid and rock characteristics, causing oil displacement to increase (Yuanhao et al., 2021). Adil et al. (2020) studied the influence of NPs concentration on electromagnetic-assisted oil recovery using ZnO nanofluids and reported that the minimum IFT value, 10.02 mN.m-1, and the smallest three-phase contact angle of 42.47 were achieved by 0.1 wt % ZnO@500NPs, which is proportional to the additional recovery of 13.08% OOIP The average hydrodynamic size (240.9 nm) of 0.1 wt% ZnO@500NPs, which is directly linked with the change in IFT and contact angle, is related to the surface energy of the NPs. NPs can, in fact, be adapted to perform a variety of tasks. These occurrences are linked to variables such reservoir rock wettability modification, spontaneous emulsion formation, IFT reduction between reservoir fluids, and porous media flow characteristics, all of which aid oil recovery (Cheraghian et al., 2020). NPs have recently emerged as a viable option for producing stable foam under difficult reservoir conditions, either by adding them to the foaming surfactant solution or by modifying the surface wettability (Gbadamosi et al., 2019).

IFT is a measure of the energy required to keep the two-phase interface stable during separation. When IFT is reduced, the two-phase interface can be easily broken (Raya et al., 2020). According to studies, NPs can efficiently minimize IFT between crude oil and water and combine both phases to form a microemulsion phase (Massarweh and Abushaikha, 2020). The IFT between the oil and water phases in EOR processes is a critical component that impacts oil recovery (Ragab and Mansour, 2021). For CO2 geo-storage and EOR processes, several studies have suggested employing surfactants, polymers, and conventional nanoparticles to minimize IFT. Injecting these chemicals into oil wells has the potential to impact shallow water zones on land and marine environments offshore due to the probability of oil spills and injection fluid leaks. Green nanocomposites are secure in marine conditions and shallow water and can increase oil recovery (Nazarahari et al., 2021). Wettability, on the other hand, is a measure of a rock's affinity for oil and water. The rock favours oil over water in an oil-wet formation (Chen et al., 2018) and prefers water to oil in a water-wet formation (Alhosani et al., 2020). After waterflooding, a substantial volume of oil sticks to the surface of the rock and creates residual oil (Ngouangna et al., 2020). The interior of pores and pore throats are filled with oil (Hu et al., 2015; Zhang et al., 2018; Hao et al., 2021). The conditions are more difficult in intermediate-wet or mixed-wet formations (Kumar et al., 2010). By removing or covering the rock surface with different materials, the wettability of rocks can be altered (Alhosani et al., 2020). Oil composition, rock mineralogy, rock surface shape, formation temperature and pressure, and formation fluid pH are all factors that influence wettability (Gbadamosi et al., 2019; Song et al., 2020). The impact of IFT and wettability on oil recovery is important. Reducing the IFT is the most practical way to improve the capillary number by orders and make oil flow easier (Guo et al., 2017). Distinct CEOR materials, on the other hand, have different effects on IFT and wettability (Gbadamosi et al., 2019). The wettability of rocks has repeatedly been reported to be greatly influenced by salinity (Alotaibi et al., 2011; Hosseini et al., 2020, 2021). Ionic species have equally been reported to be very beneficial for oil recovery by low salinity water injection (Jha et al., 2020a; Jha et al., 2020b). NPs also change wettability while modifying IFT marginally (Cheraghian et al., 2020; Ngouangna et al., 2020; Yakasai et al., 2021b). As a result, we may utilize the impact on IFT and wettability as EOR material selection criteria if we know the optimal IFT value and wettability conditions.

Hydroxyapatite NPs (HAP) is an inorganic mineral with the standard apatite lattice structure (Fig. 1) ($A_{10}(BO_4)6C_2$), where Ca, P, and OH define A, B, and C, respectively. Pure HAP has a Ca/P mole ratio of 1,67 due to the fact that it contains 39.68% calcium and 18% phosphorus by weight (Habibah et al., 2022). HAP is widely employed as an implant material for bone tissue regeneration (osteogenesis) and as a drug carrier for drug and gene delivery systems due to its bioactive and biocompatible qualities (Chocholataet al., 2019). Not only in biomedicine, but equally in other fields of science and technology and an increasing

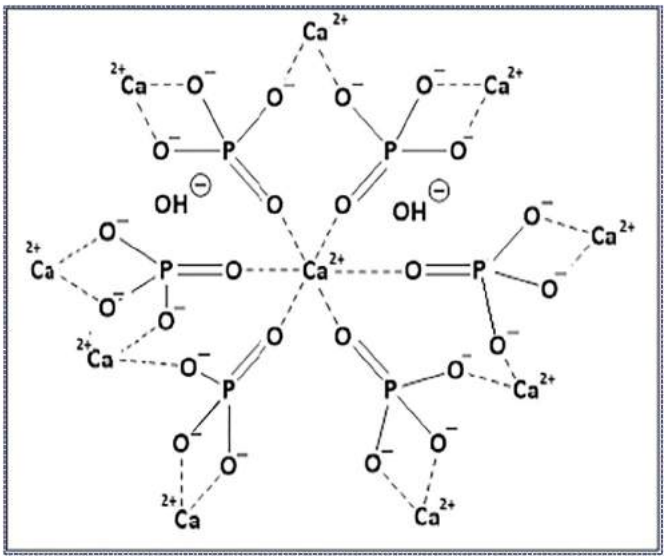

Fig. 1. Chemical structure of HAP

number of academics are expanding study into its uses and applications (Callahan et al., 2020; Vomero and Schiavone, 2021). For HAP and its potential function in sectors such as magnetic resonance, controlled therapeutic drug delivery, and cellular drug delivery, new synthesis methodologies, characterization, and functionalization approaches are being investigated (Domenico et al., 2019). HAP has sparked a lot of interest in the field of biomedicine for a variety of applications, including implants or prosthesis in orthopaedics, maxillofacial surgery, and dentistry, with the goal of mending or removing hard tissue. This is due to its exceptional consistency, biocompatibility, and bioactivity, owing to the chemical analogy of human bone mineral compounds and dental hard tissues, as previously described. Nano HAP has been used in bioimaging as well as therapeutics. However, little or no attention has been paid to this material in the oil and gas sector, hence the objective of this work. In this study, the effects of HAP functionalized in-situ with Sodium dodecyl Sulphate (SDS) on wettability alteration and IFT reduction in a variety of salinity and temperature settings was carried out. The contact angle (CA) was used to investigate the overall wettability change of a sandstone core plug treated with NPs. Second, the IFT between oil and water was determined. The outcomes of these two approaches were thoroughly examined. The majority of NP-based IFT and wettability tests have been performed at low salinity levels, despite the fact that most reservoirs operate at distinct ionic strengths.

This research is therefore unique in various ways. First, this is the first quantitative investigation of HAP's ability to affect wettability and IFT, and second, it is the first study of HAP's use in EOR applications at moderate to high salinity and varied temperature conditions.

2. Materials and methods

2.1. Materials

The following chemicals were used throughout the investigation: Sodium dihydrogen phosphate (Na2HPO4), molar mass: 141.96 g/mol, melting point: 250 °C, and density: 500 kg/m^3, Sodium dodecyl sulphate (SDS), molar mass: 288.372 g/mol, density: 1.01 g/cm3, Calcium Nitrate 4-Hydrate, molar mass: 288.372 g/mol, melting point: 250 °C (NO3) 164.088 g/mol 2 M mass, CAS number: 13,477-34-4, all supplied by Tay Scientific Instruments Sdn. Bhd Malaysia were used without further purification. The crude oil used in this project came from Malaysia's Sarawak oil field. It was a medium crude oil with 33.3 °API and a viscosity of 142.68 cP at 27 °C and 73.10 wt % saturates, 19.23 wt % aromatics, 7.48 wt % resins, and 0.19 wt % asphaltenes. Merck Group (Sdn. Bhd., Selangor, Malaysia) provided sodium chloride (NaCl), hydrochloric acid (HCl), and caustic soda (NaOH). A 3 cm × 7 cm sandstone core from Sarawak oil Field Malaysia with porosity of 20.1% and porosity and 161.1 mD permeability. Fig. 1 shows the chemical formula of HAP.

2.2. Methods

2.2.1. Synthesis of HAP

All equipments, were washed with distilled water in order to ensure they are impurities free before use. The experimental setup is further elaborated in Figure (2). 200 mL of 0.06 M Na2HPO4 was introduced into a 1 L beaker with the use of a measuring cylinder, followed by 200 mL of SDS into the solution. The mixture was heated at 60 °C for 30 min while stirring with the use of a magnetic stirrer at 350 rpm 20 mL of 0.1 M $Ca(NO_3)_2$ solution at 1mL/Min was added into the mixture using a graduated burette under continuous stirring at 350 rpm. The pH of the solution was adjusted and maintained at 9.0 with the use of aqueous solutions of HCl or NaOH. Aging of the solution for 24 h at 25 °C under continuous stirring to ensure complete precipitation. Centrifugation at 300 rpm for 30 min followed by repeated filtration of precipitate with the use of a filter paper and washing with distilled water then oven drying for 2 h at 100 °C was carried out. Calcination at 800 °C for 3 h in a furnace and then crushing in order to obtain powder HAP. The experiment was repeated in order to ensure accuracy. The controlled precipitation was used to synthesize HAP according to the following scheme:

$$10Ca(NO3)2 + 6NaHPO4 + 8NaOH = Ca10(PO4)6(OH)2 + 20NaNO3 + 6H2O \quad (1)$$

2.2.2. Characterizations

HITACHI (Model: HT 7700) 120 kV high resolution transmission electron microscopy (HR-TEM) with energy dispersive x-ray (EDX) was used to examine the morphology, particle size of and elemental composition of HAP. The functional groups of the NPs were evaluated using a Shimadzu IR Tracer-100 FTIR with a scanning range of 370–4000 cm-1. A light scattering device (Malvern Instruments, Serial Number: MAL1098100, Zetasizer Ver. 7.11) for particle size analysis (PSA) was used to measure the surface charge and zeta potential (ZP) with the average hydrodynamic sizes of HAP. An omega cuvette was used to conduct the analysis after the HAP had been dispersed in deionized water and sonicated for 10 min. The count rate was 241.5 (kcps) and the temperature was 25 °C at varied pH range.

2.2.3. Fluid preparation

In brine solutions ranging from 5000 to 30,000 ppm, NFs with concentrations of 0.05–0.1 wt % were produced. This range of concentration was chosen based on the optimum concentration of NFs reported in literature. Concentrations higher than 0.1 wt% may lead to fast agglomeration, pore channel obstruction, formation damage and permeability impairment (Predoi et al., 2018). The dried NPs were collected, weighed, and dispersed into a beaker containing brine, where they were agitated for 5 min at 28 °C and under atmospheric pressure using a magnetic stirrer. Subsequently to get a homogenous solution, the prepared solution was sonicated for 15 min in an ultrasonic bath (Branson DHA-1000-E, 100 W, 40 kHz, Danbury) in order to minimize agglomeration.

2.2.4. IFT measurements

The (IFT) between crude oil and the NFs was measured using the Plate method. The measurement technique included cleaning the platinum plate with acetone and a high flame to guarantee pollutants free. Submerging the plate 3 mm deep into the aqueous phase, adding oil without disrupting the aqueous phase, and then measuring the force

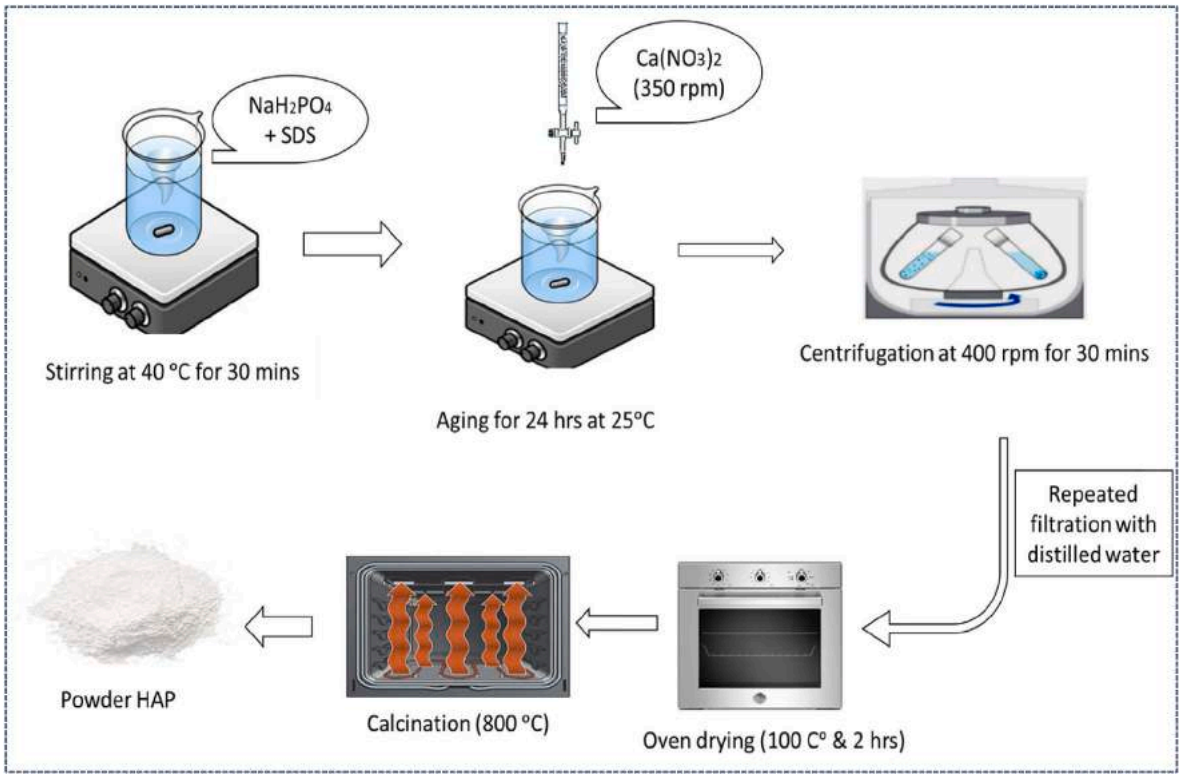

Fig. 2. Synthesis of HAP using Co-Precipitation in the presence of SDS.

necessary to lift the plate out of the dense phase. Particle concentration (0.05–0.1 wt %), brine concentration (5000 ppm - 30,000 ppm), and temperature (26–80 °C) were all altered in the trials. The surface tension of distilled water was determine to be 71.84 mN/m at 26 °C under atmospheric pressure. Each data point was tested three times to ensure accuracy, and the average result recorded.

2.2.5. Contact angle measurements

To test the NFs' capability to influence rock wettability, CA measurements were performed using the sessile drop method. The CA measurement setup is shown in Figure (4). The CA between the water droplet and the rock surface is measured when a droplet of water is put onto the core sample. To investigate changes in wettability after oil saturation, CA measurements were done before and after treatment with the NFs. Each saturated core sample was treated at different levels of NFs (0.05–0.1 wt%), brine concentration (5000 ppm - 30,000 ppm), and temperature (26–80 °C) respectively. Prior to the CA measurements, sandstone cores were repeatedly washed with toluene and acetone to remove oil and other impurities, aged in crude oil for 48 h to ensure oil saturation, cleaned and dried with air at room temperature for CA to be measured. The cores were then aged again in NFs for another 48 h to ensure wettability alteration and the C A measurement repeated. Prior to the temperature variation, the NF solution was heated overnight at the desired temperature for the wettability to be altered before CA measurement.

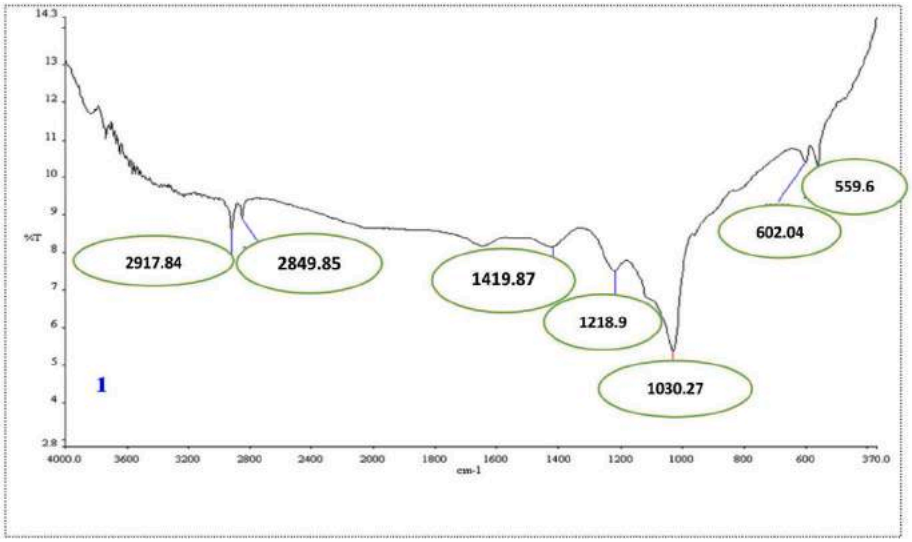

Fig. 3. Ftir spectra for HAP

3. Results and discussions

3.1. FTIR

FTIR is a useful method for determining the chemical properties of powder samples. The phosphate group ($PO4^{3-}$) and hydroxyl group (OH^{-}) are the most distinctive functional groups in the FTIR spectrum of produced hydroxyapatite (HAP). Figure (3) shows the results of the HAP FTIR spectrum that was evaluated. The bands at 2849.85 and 2917.84 cm^{-1} are associated with the stretching modes of hydroxyl groups in adsorbed water molecules on HAP (Fern and Salimi, 2021). These findings indicated that from the aqueous solution, a small amount of water molecules and a large amount of structural OH^{-} groups were adsorbed onto the surface of HAP. The band at 1030.27 represents the symmetric stretching mode of (P–O), whereas the bands at 602.04 and 55,960 represent the asymmetric bending vibrations of (P–O) bonds attributed to $PO4^{3-}$ (Predoi et al., 2018).

3.2. Particle Size Distribution Analysis (PSA)

The distribution of HAP was measured using Malvern Zeta Sizer version 7.11. The results show that the particles are predominantly nanosized ranging from 250 to 400 nm though have few particles in the micrometer range. The average hydrodynamic diameter 329.5 nm (Fig. 4), shows that the particles are within the nm range (Ngouangna et al., 2020) and can be recommended for use in EOR. The sizes are greater as compared to the TEM analysis which could be attributed to two factors: Either the sizes increased by growth, due to the absorption of water molecules onto the surface of the NPs during dispersion in water (Szałaj et al., 2019), or either by agglomeration (Babakhani, 2019).

3.3. Zeta potential analysis (ZP)

The most frequent stabilization test used to measure the dispersion stability of compounded NFs is sedimentation (Ngouangna et al., 2020). However, because this method is time-consuming and produces insignificant findings, it is ineffective for evaluating HAP dispersion stability. In order to analyze the dispersion stability of the produced NFs, ZP measurements were carried out to quantify the surface charge. The ZP as a function pH was done using a Malvern Zeta Sizer version 7.11. The pH level at which the maximum hydroxyl ions are placed on the surface of the HAP was determined using several pH ranges (Fig. 5). The SDS attachment was able to neutralize and replace the positive charges of HAP with negative ions of SDS at a pH of 1.69 (acidic). The results show that the ZP ranges from −5.69 mV to −27.2 mV at pH values of 1.69 and 12.8 respectively, indicating that the particles are more stable in a basic medium. The results are consistent with those reported by Varadarajan et al. (2020). ZP values of NFs with magnitudes less than 5 (±5.0 mV) indicate unstable suspensions and likely caused by agglomeration (Oseh et al., 2020). The mean ZP value suggests that the insitu-modified HAP are very stable. The magnitude reveals the colloidal system's potential stability. If all of the particles in suspension have a large negative or positive ZP, they will repel one another, and there will be no tendency for the particles to stick together. Based on HAP's colloidal stability and dispersion with a ZP of −27.2 mV, it indicates long-term fluid stability and as such, can be suggested for applications in EOR.

3.4. Particle size, morphology and elemental composition

The composition, size, and morphology of NPs synthesized in the presence of a surfactant, often depend on the order of component mixing. In this work, a Na_2HPO_4 solution was added to a reactor containing an SDS solution with a 0.5 M concentration according to the procedure described in Section 2.2.1. Rod-like particles with crystalline structures were formed during this process with lengths ranging from 20 to 140 nm and mean length at 45.3 nm (Fig. (6 a), consisting of NPs having diameters between 5 and 15 nm according to the HR-TEM image. This finding is consistent to the one reported by Koroleva et al. (2020). The NPs lengths were measured using image-J software and the distribution analyzed using origin software (Fig. 6 c). The elemental composition presented by the EDX spectrum (Fig. 8b), indicates the presence P, Ca and O all attributed to HAP, confirming its formation. The calculated Ca/P molar ratio results from the elemental composition gave 1.69 which is below 2 and equally within the range of acceptable HAP values. Standard HAP ratio for Calcium and Phosphorus is supposed to be 1.67 (Ebadipour et al., 2021). The sizes (rod-lengths) of NPs are within the standard sizes of NPs used in EOR applications (Sircar et al., 2021), since the reservoir pore sizes are within the micrometer range. This therefore confirms that HAP is suitable for EOR applications (Zhang et al., 2016).

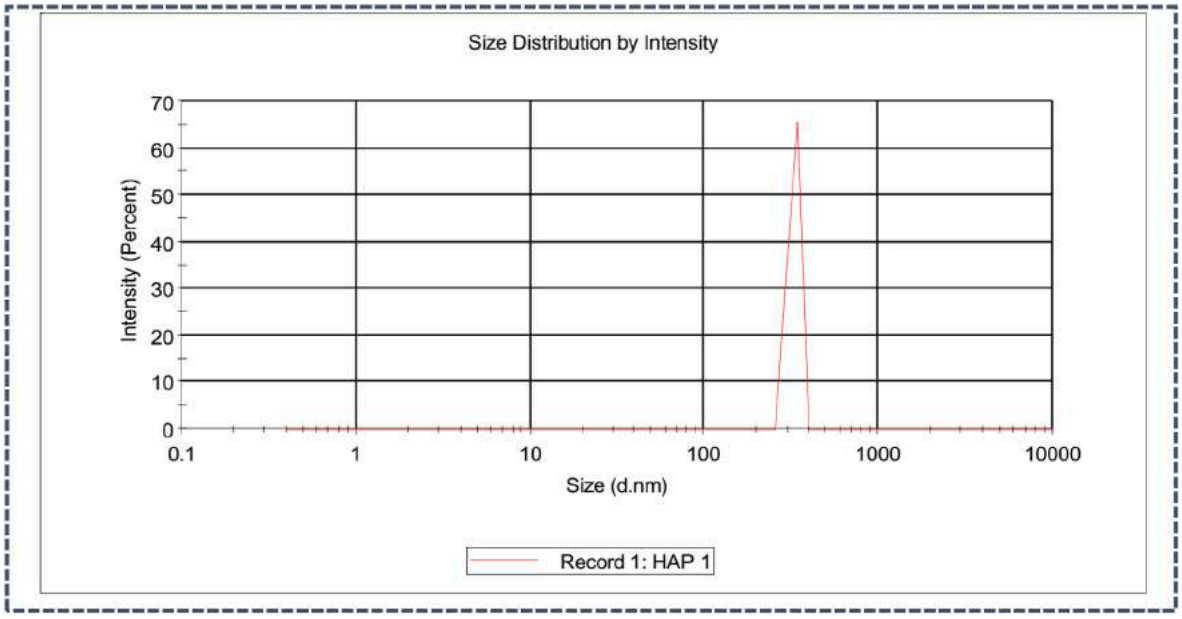

Fig. 4. Particle size distribution analysis.

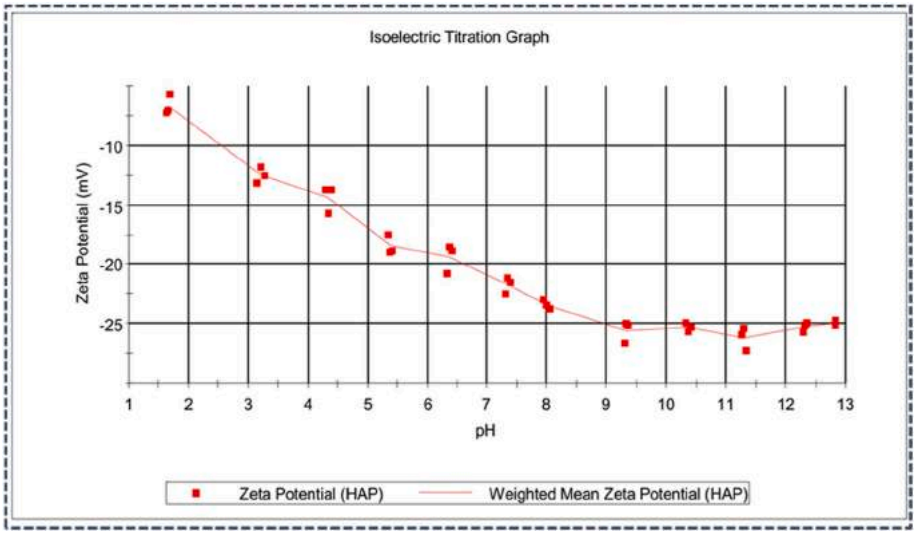

Fig. 5. The ZP of HAP as a Function of pH.

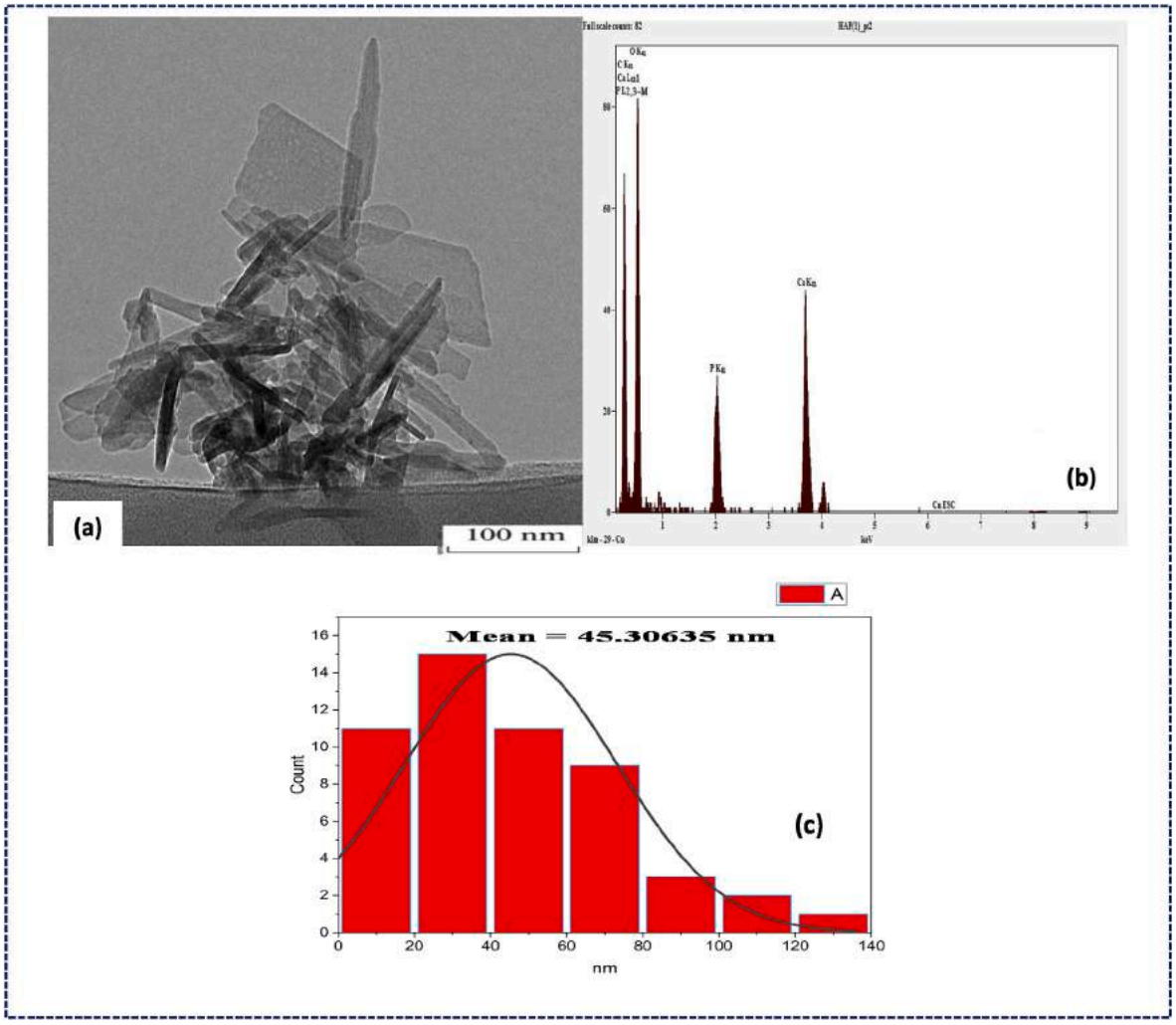

Fig. 6. (a) HR-TEM micrographs of HAP Synthesized with SDS and zoomed at 100 nm (b) EDX Spectrum and (c) Rod lengths Distributions.

3.5. IFT

The energy at the interface of two immiscible fluids is measured by IFT, which is an important metric in EOR processes. Surfactants, polymers, and low-salinity water are examples of successful EOR agents that reduce the IFT between oil and the displacing fluid. When the IFT

between oil and water drops, trapped oil droplets distort and spread easily across the porous medium. NP surface energy, brine concentration, oil composition, NP concentration, and temperature are just a few of the variables that might affect IFT. As a result, the impact of particle concentration, salinity, and temperature on the HAP's IFT process was investigated in this section.

3.5.1. *Influence of HAP concentration on IFT*

The impact of NPs concentration on IFT reduction has long been a contentious issue. Despite the fact that some studies have revealed that NPs have little or no influence on IFT, others have discovered that NPs significantly lower IFT (Yakasai et al., 2021a,b). As a result, IFT measurements with varied HAP concentrations (0.02–0.1 wt %) were performed to look into the possibility of HAP NF IFT reduction (Figs. 7a, 8b and 8c). The findings show that IFT is affected by particle concentration at varied salinity and temperature settings. The reference value for the IFT of oil/water systems was 26.54 mN/m. At 25 °C, 50 °C, and 80 °C, and at 5000 ppm, 10,000 ppm, 20,000 ppm and 30,000 ppm NaCl brine concentration, the highest IFT reduction was 3 mN/m. This value was achieved at 0.1 wt% HAP concentration, 25 °C and 30,000 ppm brine respectively. The trend showed that increasing the concentration of NFs decreased IFT because the NPs were positioned at the oil-water interface, reducing the free surface energy and causing disturbance between the oil and water molecules. However, due to particle sedimentation, the IFT of HAP increased slightly over the optimal concentration. A spare monolayer form at low concentrations, and as concentrations rise, more

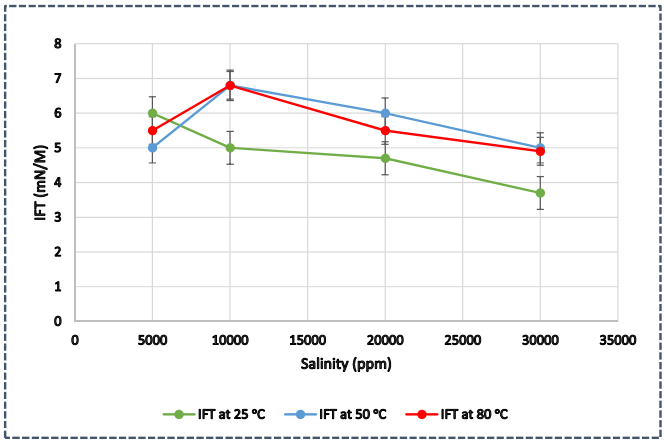

Fig. 8. Influence of Salinity on IFT at varied Temperatures.

NPs are pushed to the oil-water interface, resulting in a compact monolayer. NPs have no appreciable adsorption affinity for the oil-water interface, according to the literature. Overall, the optimum concentration for all Salinities and temperatures corresponding to the best IFT reduction was derived from the 3 figures (7a, 9b, and 9c) at 0.08 wt % accordingly. The lowest value of IFT (3 mN/m) achieved using HAP was quite low as compared to other NPs reported in literature (Aurand et al.,

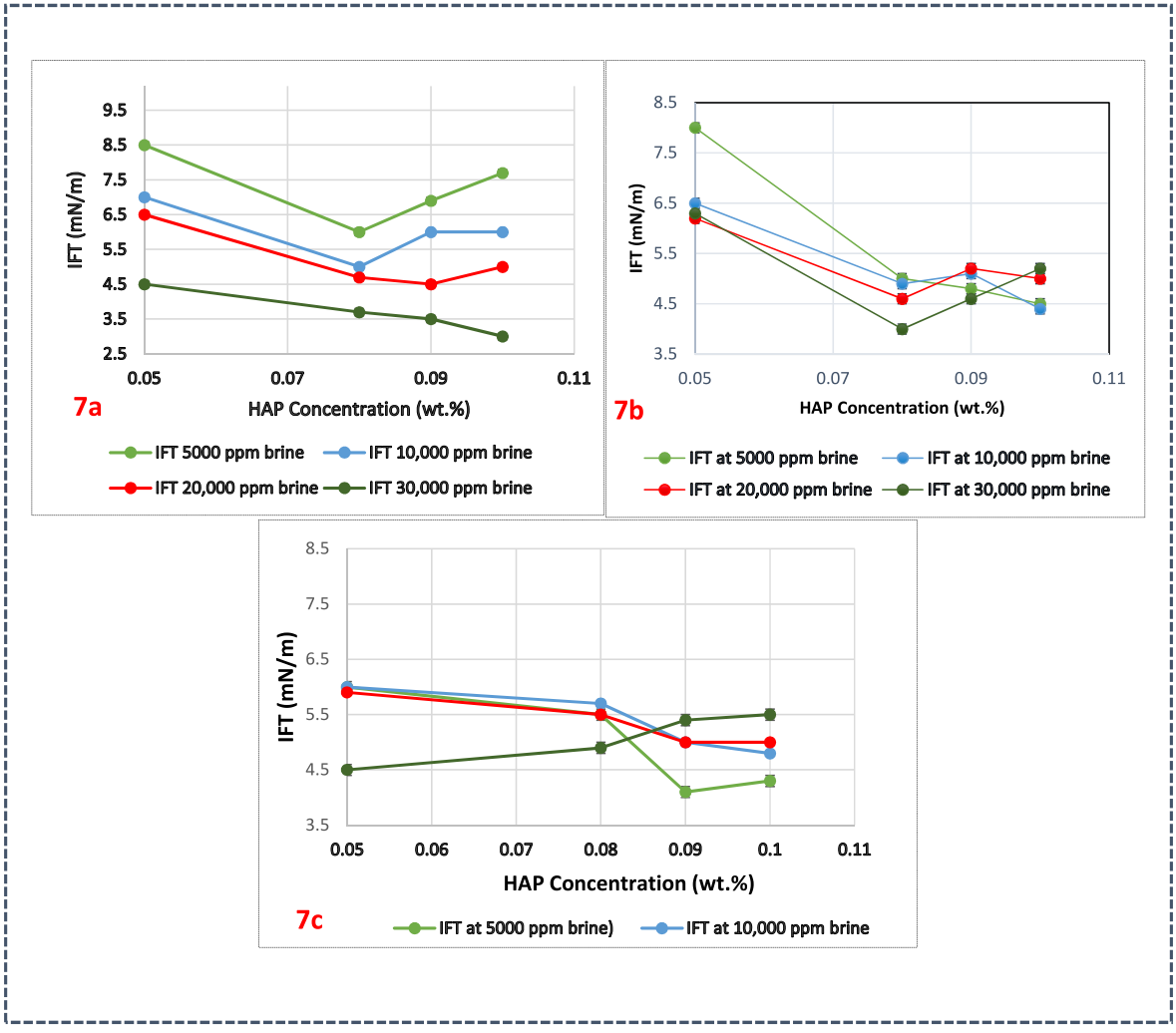

Fig. 7. Effect of HAP concentration on IFT at varied salinities and temperature of (a) 25°C, (b) 50 °C and 80 °C.

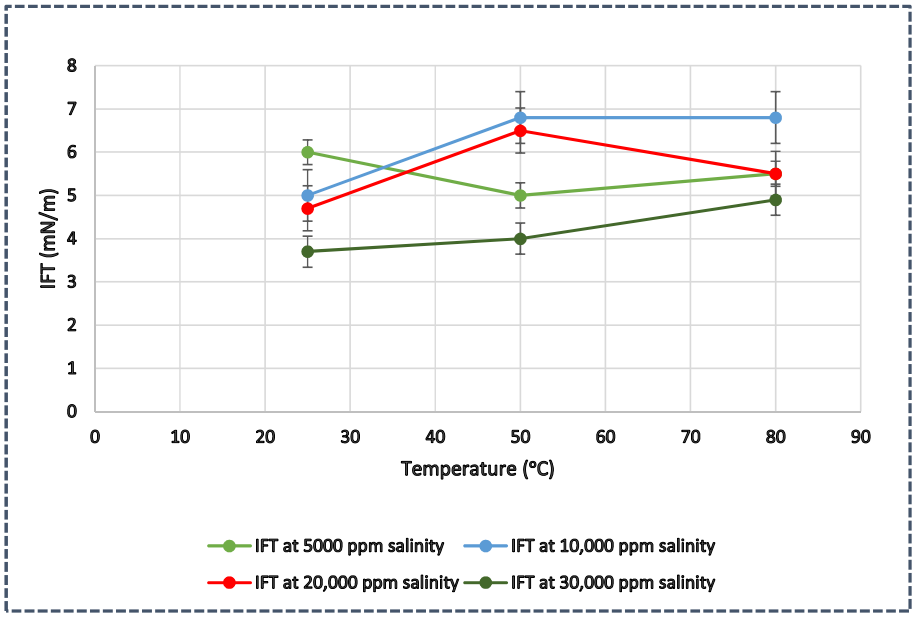

Fig. 9. Effect of temperature on IFT at varied concentrations of electrolyte.

2014; Emadi et al., 2017; Ali, 2018). This suggests HAP as a good IFT reducing agent for EOR applications. However, the low IFT reduction could not only be influenced by HAP alone but the presence of SDS as well since surfactants have higher capacities to reduce IFT to ultra-low values.

3.5.2. *Effect of ionic strength on IFT*

Particle surface properties and placement at the oil-water interface are hampered by electrolyte. Consequently, some researchers have found that they provide a significant contribution to IFT reduction (Sulaiman et al., 2015), whereas others have found completely opposite tendencies. Figure (8) depicts the NPs' dynamic IFT behaviour at a fixed bulk concentration with different ionic strengths. At different temperatures of 25 °C, 50 °C, and 80 °C respectively. The equilibrium IFT values for 0.08 wt % NPs concentration at different ionic strengths were observed. At all three temperatures, the IFT drops steadily as the NaCl content rises. The negative charge at the contact forms an electric double layer on the NPs' surface. At very low NaCl concentrations (5000 ppm), the ions (Na^+ and Cl^-) in the bulk aqueous phase are stable and do not approach the oil–water contact, allowing the structure of water at the interface to remain unchanged. The ions become closer to the oil–water interface as the concentration of NaCl rises. When NaCl is introduced, the IFT drops, owing to greater contact between the NPs and the aqueous solution. The sodium cation (Na^+) interacts more readily with the NPs' partially negative charge. The opposing charge of Na^+ screens the negative charges on the surface of HAP. This reduces the double layer thickness, which results in a larger density of NPs at the interface, lowering the IFT value. Cl^- ions, on the other hand, are present in the double layer as counter ions and are thought to adsorb to the oil–water interface as well. Ferdous et al. (2012) reported a similar result using gold NPs. Furthermore, a molecular dynamics simulation by Tian and Shen (2009) demonstrated that Cl^- can adsorb at hydrophobic material–water interactions. Because of their attraction to Na^+, more NPs are adsorbed to the interface as the NaCl concentration rises. The IFT is reduced as the adsorption of NPs at the contact increases. As a result, salt lowers the IFT at the oil–water interface, indicating that as ionic strength increases, so does the number of particles at the interface. Salts screen NPs' surface charges, reducing repulsive interactions between modified particles and accelerating NP aggregation in NF. The clusters created are larger than individual NPs and remained adsorped at the oil-water interface thus reducing IFT (Al-Anssari et al., 2018).

3.5.3. *Effect of Temperature on IFT reduction at varied ionic strengths*

At four different salinities (5000 ppm, 10,000 ppm, 20,000 ppm, and 30,000 ppm) and 0.08 wt. HAP, the effect of increased temperature on the IFT of the NF-oil system was evaluated (Fig. 9). The achieved IFT values range from 6 to 3.7 mN/m depending on salinity, with the lowest (3.7 mN/m) recorded at 30,000 ppm. The maximum achievable temperature chosen for this experiment was 80 °C based on the temperature limitation of the equipment used. The results show that the measured IFT values increased as the system's temperature rose. This could be due to particle agglomeration and aggregation as a result of van der Waals attraction between NPs at high-temperature conditions, losing their colloidal stability (Li et al., 2020) and, subsequently, the sedimentation of the NPs leading a decrease in the NPs adsorption at the oil-water interface. Despite the fact that the IFT increased with temperature, the highest value obtained was 6.8 mN/m, which is within the range of incremental oil recovery for NPs published in literature. Hence, the influence of temperature leading to the agglomeration and aggregation processes were modest on the IFT increase using HAP.

3.6. *Wettability*

To release oil capillary entrapment or boost water imbibition and oil counter-current production, any rock's wettability can be converted from oil-wet to water-wet. The water layer that separates crude oil from the mineral surface is not necessarily permanent, and disturbing it could cause the rock's natural water wetness to change. As a result, the disjoining pressure must be addressed by altering the wettability of the aqueous film in order to destabilize and rupture it (Katende and Sagala, 2019). CA, which can provide information on how reservoir rocks wet, was used to evaluate the effect of HAP on wettability modification of sandstone cores. Water–wet rocks have a CA of less than 70°, intermediate–wet rocks have a CA of less than 110° but larger than 70°, and oil–wet rocks have a CA of greater than 110°. (Ngouangna et al., 2020). The Wilhelmy plate method was utilized prior to the CA measurements. This method was prioritized due to its higher precision than optical methods and the use of a sensitive microbalance, higher reproducibility due to the broad scanning area of substrates, and reliable analysis of the dynamic effect during wetting (Huminic et al., 2021). The influence of HAP concentration, salinity, and temperature on the wettability of oil-wet sandstone cores was studied (Fig. 10). The wettability change of the NFs is classified using the value of the water droplet CA on the rock

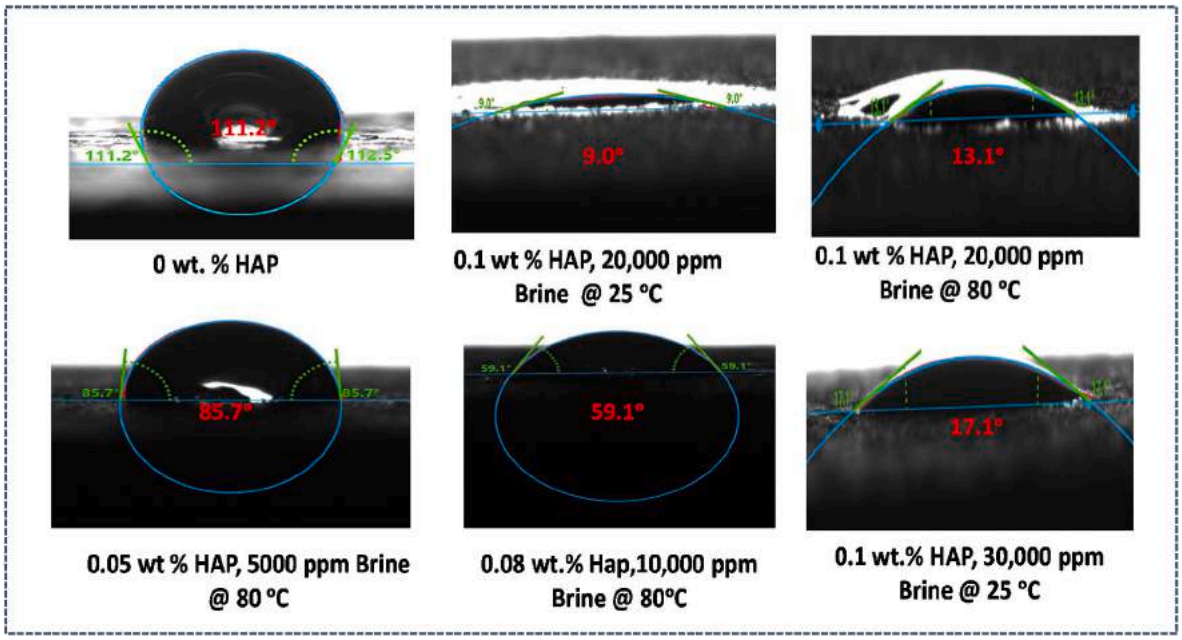

Fig. 10. Contact Angle Images of Sandstone treated with HAP NF.

surface (Equation (2)).

$$\sigma\_os - \sigma\_ws = \sigma\_ow \cos\theta \quad (2)$$

Where σ_os(dyne/cm) is the interfacial energy between rock and oil, σ_ws(dyne/cm) the interfacial energy between rock and water, σ_ow(dyne/cm) is interfacial energy between water and oil and $\cos\theta$ (^{oo}) is CA at oil - rock - water interface measured through the water.

3.6.1. *Effect of HAP concentration on wettability alteration*

The concentration of NPs is one of the most important elements in NF flooding applications due to the fact that NPs become more efficient as concentration increases. Researchers focused on defining NP concentration ranges as well as their effect on wettability change in various studies. The impact of NPs on wettability alteration has been explored at a variety of concentrations. Most studies found that as NP concentration increases rock wettability improves due to repulsion forces (Eltoum et al., 2021). Excessive concentrations, on the other hand, may cause pore plugging and increase operational costs. As a result, optimal concentration for wettability modification is always desired. Fig. 11a, b, c, and d demonstrate water droplet contact angle measurements on sandstone cores treated with HAP NFs at various NP concentrations (0.05–0.01 wt %), salinities of 5000 (11 a), 10,000 (11 b), 20,000 (11c) and 30,000 (11 d) ppm, and temperatures of 25 and 80 °C, respectively. The results show that increasing the concentration of NPs causes a decrease in CA, which is consistent across all salinities. The optimum concentration is 0.1 wt %. This result is consistent to that reported by Li et al. (2019). The lowest CA was observed at 20,000 ppm brine, indicating that as the salinity increases further, so does the CA. At any given HAP concentration and salinity, it was also discovered that the CA is significantly reduced at lower temperatures. All the measured C A fall below 70°, showing a strong indication that HAP NF can efficiently alter wettability from oil-wet to water-wet since water-wet rocks fall within this range (Ngouangna et al., 2020). The decrease in CA with increase concentration of NPs is linked to two possible mechanisms: NP adsorption at the rock's surface and disjoining pressure. The ability of NPs to segregate the oil phase from oil-wet deposits is improved by their wedge-like structures. As a result, an increase in NF concentration raises structural disjoining pressure, causing wettability to shift toward water-wetness (Rezk and Allam, 2019; Razavirad et al., 2022). Brownian motion and electrostatic repulsion between NPs cause structural disjoining pressure, which increases the spreading and wetting of NFs on solid rock surfaces, resulting in wettability changes (Humic et al., 2021). The role of structural disjoining pressure in the wetting and spreading of NFs on a solid surface has been studied theoretically, and it appears that these phenomena can be regulated by adjusting NP size and concentration, as well as IFT (Wasan et al., 2011). The type of oil and the surface charge of NPs, on the other hand, are important elements in the disjoining force that causes the oil to be displaced from the rock surface. Consequently, more Np adsorption on the surface generates a larger structural disjoining force, allowing oil to detach more easily from the surface.

3.6.2. *Effect of electrolyte on wettability*

It is common knowledge that oil reservoirs typically have relatively high salt concentrations. The salinity of a fluid can significantly impacts on its properties (Sun et al., 2018). So, it is critical to investigate the performance of HAP NFs in different salinity environments. According to CA tests, comparing crude oil and oil–wet sandstone cores substrates aged in the base fluid, the lowest CA measured corresponds to the 20, 000 ppm brine solution. However, above this optimum salinity (20,000 ppm), the CA value increases marginally with salinity, (Fig. 12). The result is consistent with that reported by Razavirad et al. (2022) on functionalized iron-carbon nanohybrid. The adherence of NPs and oil dissolution may have been aided by electrostatic interactions between NA^{+}, Cl^{-} and the sandstone surface. Thus, the wettability alteration in low concentrations of salinity is improved (Hou et al., 2015). The oppositely charged anions and cations of NaCl, on the other hand, reduced the electrostatic repulsive forces of the NPs, resulting in lower disjoining pressure, faster aggregation, and sedimentation. As a result, the spreading and interaction of NPs with sandstone cores is reduced (Rezk and Allam, 2019). This is consistent with research that show the thickness and expansion of the electric double layer has a dominant influence in the detachment of oil from the rock surface, and that the larger the magnitude of the electrostatic forces, the greater the magnitude of the disjoining pressure (Mehana and Fahes, 2018; Xie et al., 2019). It is worth noting that, even at high NaCl concentrations, all of the NFs have a strong ability to change the wettability of sandstone surfaces to a water-wet state. This finding suggests that HAP has a high potential for use in EOR where high salinities are common.

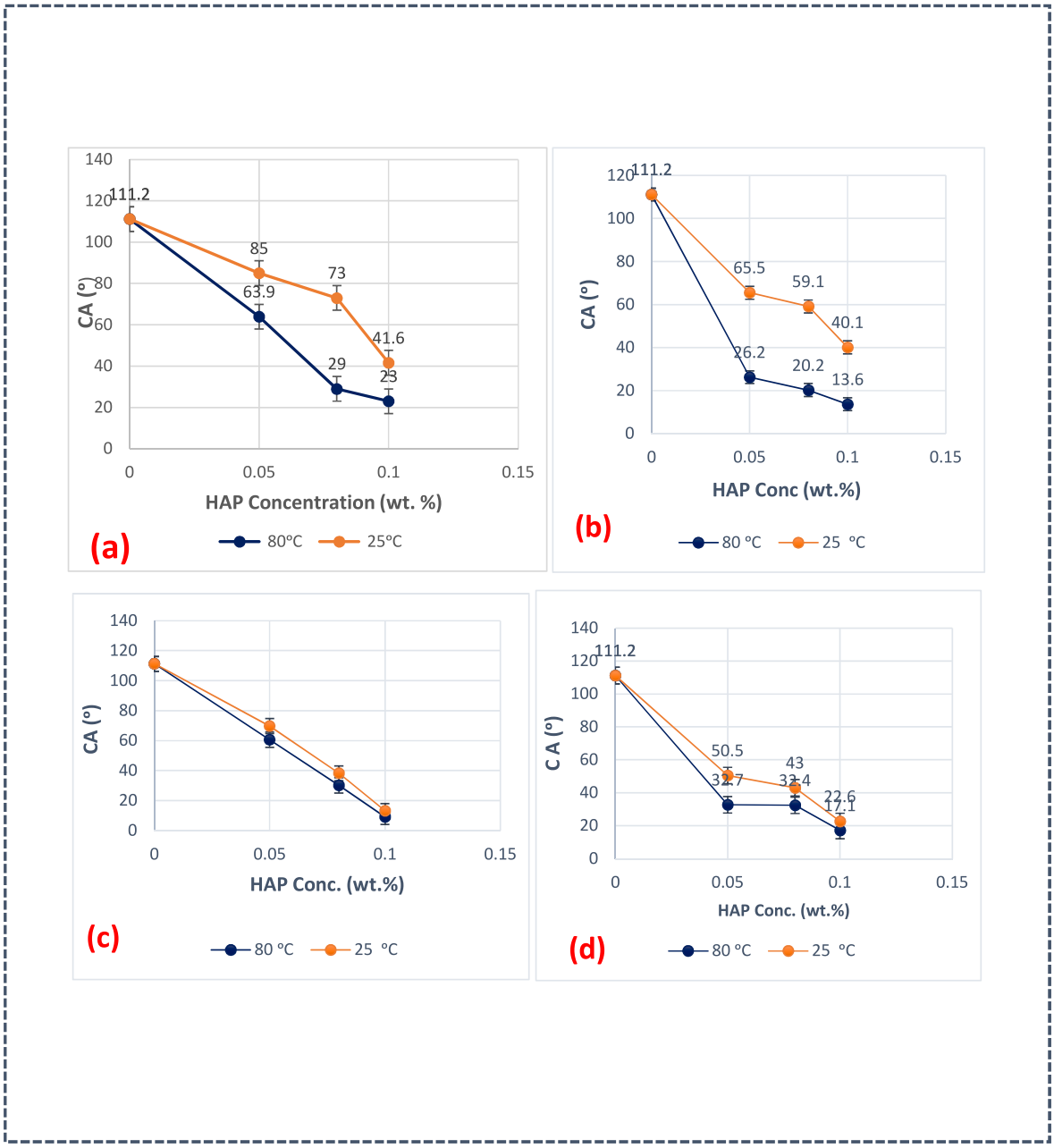

Fig. 11. Effect of HAP Concentration on C A alteration at (a) 5000 ppm Brine, (b) 10,000 ppm Brine, (c) 20,000 ppm Brine and (d) 30,000 ppm Brine.

3.6.3. *Effect of Temperature on wettability alteration*

The advancing and receding CAs were measured as a function of temperature for 0.1 wt % HAP concentration and various salinities. The results show that CA decreases with temperature, indicating that the sandstone surface becomes more water-wet as temperature increases (Fig. 13). Many authors have reported that at high temperatures, the wettability of mineral surfaces shifts toward water-wet (Hamouda and Rezaei Gomari, 2006). A variety of factors, including ions in the brine, ionic composition, pH, viscosity, relative permeability, oil–water IFT, thermal expansion, density, buoyancy effect, thin-film stability, and so on, influence the temperature dependence of wettability alterations (rock/oil/water three-phase interfacial interactions) (Sakthivel, 2021). The most well-studied phenomenon in the literature is the temperature dependency of the oil–water relative permeability in porous surfaces (Hamouda et al., 2008; Sakthivel, 2021). As the temperature of the system rises, the relative permeability of the system shifts to the right side. It demonstrates that the water-phase relative permeability in porous media increases as the temperature rises (Zhang et al., 2017). Furthermore, raising the temperature while lowering the fluid's viscosity (intramolecular hydrogen bond reduction) may increase fluid permeability, particularly water relative permeability, which will invade the pore and thin layer at the three-phase interface. Furthermore, when the system temperature rises, the oil–water IFT decreases, indicating that the capillary force reduces water resistance to flow in both the three-phase interface (at the porous media surface) and the porous media (Sakthivel, 2021). As the temperature rises, the dynamic motion of the increased particle collision may also increase, leading to a higher increase in the fluid's (water) pressure, resulting in disjoining pressure. In addition, the rock surface becomes less positive or negative, rejecting oil drops (organic components) and favouring water-wet behaviour due to increase in temperature. Enhanced water wetness is preferred over lower temperature due to the combined effect of relative permeability, oil–water interfacial tension, increased particle collision and diffusion into the thin layer, and surface charges or potential temperature change

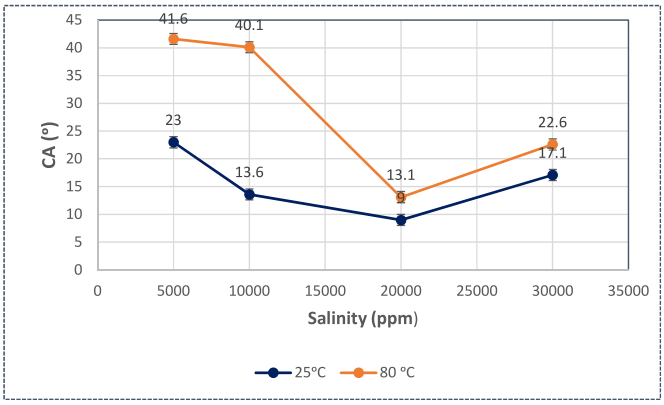

Fig. 12. Influence of Salinity on Wettability Alteration at 0.1 wt% HAP and 25 °C and 80 °C.

(Mahani et al., 2017). Overall, increasing the temperature enhances the oil's cohesive nature (Sivabalan, 2021), which reduces the crude oil's adhesive propensity on rock surfaces greatly.

Based on low IFT values of 3 mN/m, CA change from 111-9°, HAP modified with SDS is good for EOR as compared to bare NPs reported in numerous literatures. HAP is equally efficient in low and high conditions whereas most NPs are efficient in low salinity reservoirs. However, much work needs to be done to thoroughly access the ability of HAP-modified SDS in other areas of EOR such as foam and emulsion stabilization, viscosity enhancement, mobility control and asphaltene precipitation.

4. Conclusion

The effect of dispersion stability of insitu functionalized HAP with SDS on wettability modification of sandstone and IFT decrease between brine and oil was investigated in this work. At various salinities and temperatures, HAP lowered IFT and changed the wettability of sandstone rock from oil-wet to water-wet. The following are the most important conclusions drawn from the findings of this study.

1. The synthesis of HAP was confirmed by TEM and EDX results, as well as the free OH^{-}, and $PO4^{3-}$, groups on the FTIR spectra, which influenced the properties performance.
2. ZP measurements at various pH levels were used to explain HAP interactions, improving awareness of IFT reduction and dynamic wettability alteration. HAP-based EOR improved wettability and reduced IFT. Capillary pressure reduction, disjoining pressure enhancement, and particle adsorption contribute to the HAP's good influence on wettability, whereas NaCl helps reduce IFT by increasing NP-aqueous solution interaction.
3. Salinity influenced both IFT and wettability, with 20,000 ppm being the optimum salinity. At 30,000 ppm, however, both IFT and CA increased slightly. The slight rise was still within the literature's range of IFT and CA impacted by NPs. As a result, HAP appears to be appropriate for use in high-salinity reservoirs during EOR operations.
4. The IFT increased as the temperature rose which could be due to particle aggregation and agglomeration caused by van der Waals attraction between NPs at high temperatures, causing colloidal stability to be lost. CA, on the other hand, decreased when the temperature increased. As the temperature of the oil rises, so does its cohesive nature, and consequently, the crude oil's adhesive propensity on rock surfaces decreases dramatically.
5. The EOR mechanisms in this work rely on both IFT reduction and wettability modification. The change in wettability actually causes oil droplets to detach from the rock, while the low values of IFT eases the flow of oil. Therefore, IFT and wettability both play major roles.
6. Surfactants are well known for their ability to reduce IFT between oil and water to ultra-low values, implying that SDS is the primary factor governing the low achieved IFT values. However, wettability is primarily influenced by NP adsorption on rock surfaces.

Credit to author statement

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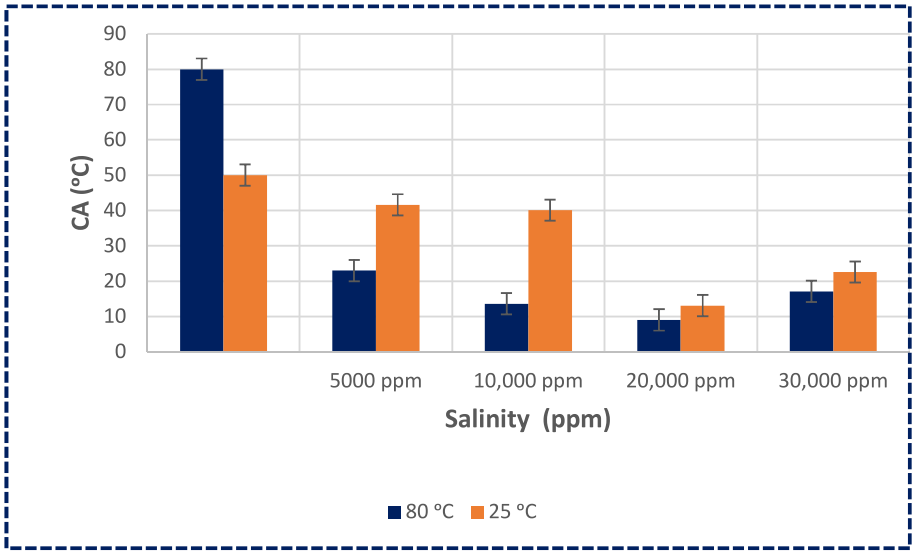

Fig. 13. Effect of temperature on Contact Angle at Varied Salinities and constant HAP concentration of 0.1 w. %.

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Declaration of competing interest

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

Data availability

The data that has been used is confidential.

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