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The Effect of a Novel Surface Modified Nanoparticles on Wettability Alteration and Enhanced Oil Recovery

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ABSTRACT

Recently, Nano-fluids (N-fluids) have emerged as cost-effective and efficient materials for enhanced oil recovery (EOR). In this context, N-fluids have shown significant potential in altering the wettability of carbonate rocks and reducing the interfacial tension between oil and water. In this study, titanium dioxide was synthesized and surface-modified using a specific approach. The synthetic solution was then prepared synergistically with engineered low-salinity water. Generally, in previous studies, changes in wettability and interfacial tension in the presence of synthetic N-fluids did not result in substantial changes in contact angle and interfacial tension. However, in this study, the greatest change in contact angle and interfacial tension was achieved through an innovative surface modification approach, indicating the presence of effective compounds within the N-fluid structure. TEM, XRD, and FT-IR analyses were employed to confirm the structure of the surface-modified N-particles. To investigate the effect of synthetic N-fluids on oil recovery, contact angle measurements, interfacial tension evaluations, and core flooding tests were conducted. In the presence of synthetic N-fluid, the interfacial tension (IFT) decreased from 19.88 mN/m (reference value) to 2.8 mN/m, and the contact angle reduced from the initial value to 26°. The results demonstrated a significant hydrophilic wettability change for the core rock.

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1. Introduction

Carbonate reservoirs hold a significant share of the world's hydrocarbon reserves and have wet/mixed-wet oil surfaces with heterogeneous pore structures [1-3]. Recently, there has been considerable interest in utilizing N-particles and low salinity water to improve oil recovery [4-6]. While it has been noted that a low concentration of N-particles can modify the wettability of rock pore surfaces when used with low salinity water [7-9]. Low salinity water flooding has garnered significant attention due to its reduced environmental effect. This method enhances oil recovery by utilizing fluid interactions and geochemical processes within the porous media [10]. Although low salinity water flooding has demonstrated effectiveness on its own, its combination with Nano-technology has led to the emergence of hybrid enhanced oil recovery procedures that gain even higher efficiency. The incorporation of N-particles into low salinity water flooding introduces additional mechanisms for enhancing oil recovery [11].

Generally, N-particles play a significant role in diminishing interfacial tension within oil-water systems, which in turn reduces capillary pressure and improves the efficiency of oil displacement [12]. It also replaces carboxyl anions on these surfaces by forming a wedge-like layer that helps to separate oil droplets from rock surfaces, changing the wettability to a water-wet state [13]. This mechanism, alongside various physicochemical factors, has been observed to contribute to the cleansing dynamics of oil-impacted soils when N-fluids are utilized [14]. Additionally, the interaction of the contact line in solid/N-fluid/oil phase leads to the detachment of oil droplets is linked to the molecular diffusion of water molecules between the solid substrate and the oil droplet [15]. The infiltration of water molecules into organic materials facilitates solvation through the formation of a water structure around hydrophobic components via hydrogen bonding. Cations present in the aqueous phase can disrupt this water structure, which reduces the solubility of organic materials. Consequently, lower salinity can enhance solubility if it falls below a certain critical ionic strength [16].

Furthermore, N-particles interact with both monovalent and divalent ions in brine, further altering the properties of the rock surface [17]. For example, carbon nanostructures possess distinctive physicochemical characteristics and are crucial in advanced technologies [18]. Carbon Nano dots are a specific type of carbon particle that measures under 20 nm [19]. Generally, the application of low salinity water, along with N-particles—whether individually or in combination—has recently attracted considerable interest from researchers due to its cost-effectiveness and efficacy [20,21]. N-particles can also mitigate fines migration and are frequently injected alongside low salinity water during increased oil recovery efforts [22]. The use of N-particles in enhanced oil recovery has shown favorable outcomes, such as interfacial tension reduction [23-25], and wettability alteration [26-29]. On the other hand, as inhibitors, they can prevent asphaltene accumulation in reservoir conditions [30, 31]. Also, polymer flooding is a widely used oil extraction method and an alternative to conventional flooding methods [28]. Moreover, utilizing N-particles is an effective way to increase the efficiency of polymer flooding in oil reservoirs [32]. However, for field-scale applications, the N-particle synthesis method should be easy and cost effective [33].

Khajeh Kulak et al. (2024) [34] introduced a hybrid enhanced oil recovery method that combines a low-salinity water/N-particles (silica and gamma alumina)/surfactant (Gum Arabic). The interfacial tension showed that the presence of cations in salinity water reduces interfacial tension values. Also, the micromodel flooding results showed that the oil recovery for γ-Al_2O_3/SiO_2 modified with surfactant and dispersed in low salinity water was reported 60.34%. Ali et al. (2019) [35] produced a novel Nano-composite (N-composite) with low-salinity water to increase oil recovery. In this regard, at a concentration of 2000ppm, the lowest interfacial tension and the highest stability were achieved. Also, the wettability of the rock reached hydrophilic conditions with a change in contact angle from 137° to 34°. The oil recovery achieved was about 19.28% of the original oil in place. Habibi et al. (2020) [36] designed interfacial tension, stability, and contact angle measurements and core flooding experiments to explore the impact of the composition smart water/50 ppm modified N-composite on enhanced oil recovery processes. The results indicated that by enhancing the concentrations of calcium and sulfate ions in smart water, oil production is improved, compared to seawater. Also, the findings revealed that adding N-fluids to the solutions enhanced the oil recovery by 3% to 7%. Habibi et al. (2020) [37] investigated enhanced oil recovery using a functionalized N-composite. These N-composites were characterized by FTIR, SEM, and XRD. Stability, contact angle, and interfacial tension analyses were done. The findings revealed that the surface modification process can considerably improve wettability. Also, the results revealed that the N-composites are able to enhance the oil production.

In the past, most studies have focused on sandstone reservoirs; however, due to the complexity of oil recovery mechanisms in carbonate reservoirs, ongoing investigations are necessary. In this regard, the mechanisms that effect wettability are complex and not fully understood, so it is necessary to formulate the most efficient compounds to understand them. On the other hand, the expense associated with producing N-fluids to alter rock wettability has been considerable. Generally, this manuscript goals to systematically examine the effect of N-fluid on wettability alterations over a wide range of experimental conditions. Therefore, this study introduces a novel combination and offers an innovative and effective method to address economic concerns while establishing a robust water-wet condition for oil-wet carbonate rock. Thus, this is the first time this new synthetic N-fluid has been used to enhance oil recovery. In this study, structural properties of synthetically modified N-particles were determined using FT-IR, XRD, and TEM analyses. In general, in this study, tests such as IFT, contact angle, zeta potential, and core flooding were performed.

2. Materials and methods

2.1. Materials

Tetraethyl orthosilicate, sodium hydroxide, 2-propanol, ethanol, ethylenediamine, urea, carboxymethyl cellulose, citric acid, glycerol, and nitric acid was purchased from Merk. In this regard, crude oil has characteristics such as API (38.7), viscosity (15.41cP), and density (15.41gr/cm^3).

2.2. Synthesis of surface-modified N-particles

In this study, three steps were used to synthesize surface-modified N-particles. In this regard, the carbon Nano dots were synthesized using a domestic microwave-assisted procedure. In this process, 0.2g of citric acid and 4.02g of urea served as the precursors for the carbon Nano dots. In this regard, the mixture was stirred vigorously for approximately 12 minutes before being placed in a domestic microwave for 5 minutes. After allowing it to cool naturally, the product was dissolved in deionized water, followed by centrifugation at 7000rpm for 25 minutes to separate the carbon Nano dots solution from any by-products. Then, the supernatant containing carbon Nano dots was dried. Next, a soft and light powder was obtained. Subsequently, after that, the titanium dioxide N-particles was surface modified using carbon Nano dots. In this step, 25ml of distilled water was mixed with 6mL of ethanol, followed by the addition of 390 mg of tetraethyl orthosilicate and 3.5mL of the carbon Nano dots dispersed in ethanol. Also, a small amount of carboxymethyl cellulose were added to the solution. The reaction mixture was stirred for 25 minutes at room temperature to ensure uniformity before being transferred into an autoclave, where it was heated at 135°C for 4 hours. The resulting functionalized titanium dioxide N-particles was then washed with deionized water, collected through centrifugation, and dried at 95°C. Finally, the surface-modified N-particle was synthesized.

2.3. Nanofluid preparation

In this study, low salinity water (NaCl (25.450 Wt.%), KCl (1.244 Wt.%), $CaCl_22H_2O$ (1.650 Wt.%), $MgCl_26H_2O$ (12.109 Wt.%), Na_2SO_4 (6.823 Wt.%), and $NaHCO_3$ (0.341)) with was used to prepare synergistic N-fluids. The N-fluids with various concentrations (100, 200, 300, 400 and 500 ppm) were prepared applying an ultrasonic bath.

2.4. Assessment stability

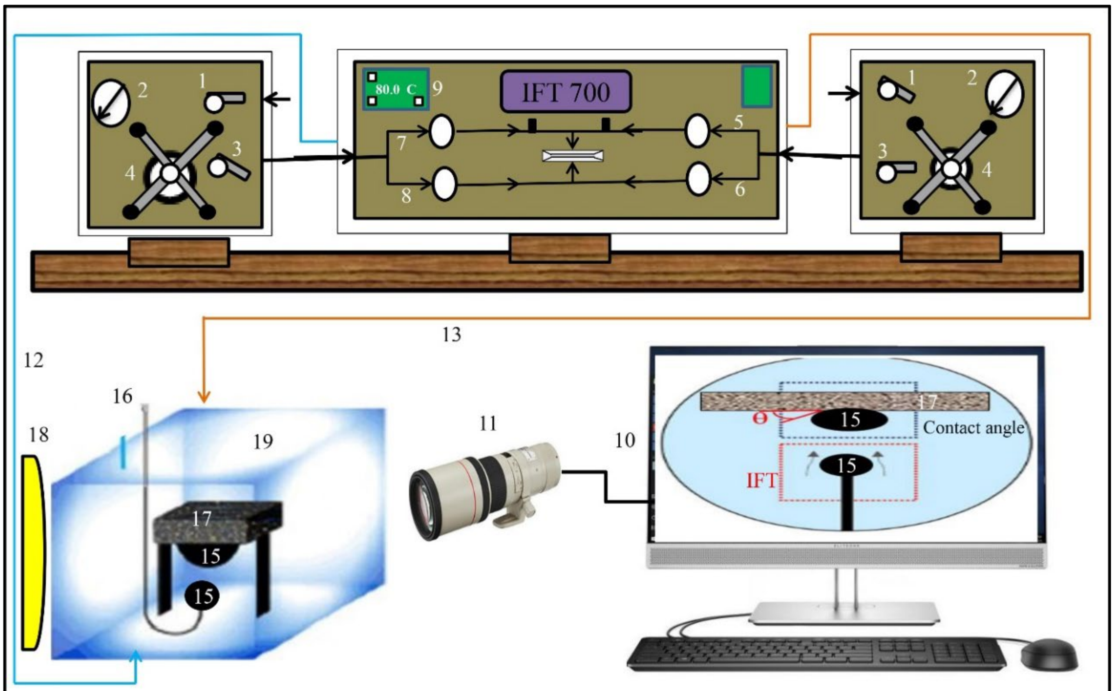
The stability of the N-fluids solution was evaluated using visual observations. After preparation, the N-fluids were transferred to transparent containers and examined for changes in dispersion stability over time [38]. The prepared solutions were stirred with an ultrasonic. Zeta-sizer Nano Z device was used for these measurements. So, the 200ppm N-fluids illustrated good stability. To observe the ionic forms of solutions, InoLab® cond 7110 conductivity measurement method was used.

2.5. Contact angle and interfacial tension measurement

The contact angle test was used to check the wettability of core rock. In this study, the physical properties of the core samples include porosity (%) (22.613.121), pore volume (ml) (13.05), Diameter (cm) (3,800), permeability (md) (0.483), and (Length (cm) (5.11)). The aging process was considered for carbonate substrates to set the strongly oil-wet condition using the reservoir crude oil. The substrates were placed in a glass jar containing crude oil and kept in an oven at 80°C for 8 weeks to allow the crude oil and substrates to equilibrate. Generally, the result accuracy and reproducibility of the pendant drop/sessile drop technique is dependent upon the extreme cleanliness of apparatus. For this reason, the cell or and the needle from which the drop was suspended were washed with gasoline and dried by air pump [39]. Thus, in this regard, the aged substrates were put into the top of the window cell. Reservoir crude oil was added to the bottom of the window cell and then it was pressurized by solutions. After that, the reservoir temperature (80°C) was considered, and once the temperature was fixed, the pressure was increased to reservoir condition. Then, the equilibrated oil droplet was put on the carbonate substrates at the same volume during each contact angle test. The visual observation technique was considered via a camera, and the average of contact angles of oil droplets was measured. Also, to measure the oil/water IFT, the sessile drop method was used. Generally, the software recorded images of oil droplets using camera. Therefore, in the end, the IFT was determined. Fig. 1 depicts the schematic representation of the experimental set-up used for the IFT/contact angle tests.

2.6. Core flooding experiments

In this section, in order to conduct the experiment, the containers are each filled with low salinity water, N-fluids, and crude oil. A pump is utilized to inject water from a bottle through pipes, which drives the piston plate situated within the cylinders. To ensure cleanliness of the line before introducing a different fluid, a bypass flow line is incorporated. Additionally, a hand pump is employed to fill the annulus between the rubber sleeve and the core holder with water, reaching the required pressure to prevent fluid leakage from the area between the core and the rubber sleeve. A precision pressure gauge records the differential pressure across both sides of the core holder, while an accumulator measures the effluent from the core. The pore volume and porosity of the core were determined by measuring the amount of low salinity water retained within it. To assess absolute water permeability, the core plugs were subjected to brine flooding at three distinct flow rates: 0.2, 0.6, and 0.8 $mL.min^{-1}$.



1. Cylinder inlet valves (for importing fluid (oil)); 2. Pressure indicators; 3. Cylinder outlet valves; 4. Valves connected pistons; 5. Oil inlet valve to the top of the container; 6. Nanofluid inlet valve to the top of the container; 7. Nanofluid inlet valve to the bottom of the container; 8. Oil line to the bottom of the container; 9. Thermometer; 10. Data acquisition; 11. CCD camera; 12. Nanofluid line to the top of the container; 13. Oil line to the bottom of the container; 14. Container; 15. Oil droplet; 16. Neddle; 17. Carbonate rock surface ; 18. LED light; 19. Nanofluid.

Fig. 1. Schematic illustration of the IFT/contact angle experiment

Subsequently, crude oil was introduced into the core at a flow rate of 0.1 mL/min until no additional low salinity water was expelled. The volume of oil present in the core was equivalent to the volume of low salinity water that was produced. Additionally, calculations for oil saturation and water saturation percentages were performed. Measurements of core porosity and permeability were also conducted, along with assessments of core dimensions and it dry weights.

Next, a core flood test was performed. In this regard, Low salinity water was injected at a consistent rate of 0.2 mL/min for about 2 pore volumes. The volumes of the resulting effluents, which included both oil and water, were measured every 5 minutes. This low salinity water flooding, serving as secondary recovery, continued until it was confirmed that no additional oil was being produced. Following this phase, the injection of N-fluids was initiated at the same constant rate of 0.2 mL/min for approximately 3–4 PV as part of the tertiary recovery process. The flooding with N-fluids was carried out using various concentrations until oil production ceased. The total oil recovery was then calculated, allowing for the determination of the ratio of oil recovered after N-fluids flooding (at different concentrations) to the overall oil recovery. Generally, Fig. 2 depicts the schematic representation of the experimental set-up used for the core flooding tests.

In general, in this study, the following assumptions were considered. In this regard, it is assumed that the petro physical properties of the rock, including the porosity and permeability of the reservoir rock, are constant. The effect of Nano fluids on the wettability of the rock is also investigated. The contact angles of oil droplets on the surfaces of carbonate core sample sections are measured and monitored to determine the wettability. Additionally, flooding is employed in specific scenarios in a controlled manner, with defined core and injection parameters, including porosity, absolute permeability, constant injection flow rate, and the recording of pressure differences, core dimensions, and initial saturations of in-situ oil and water.

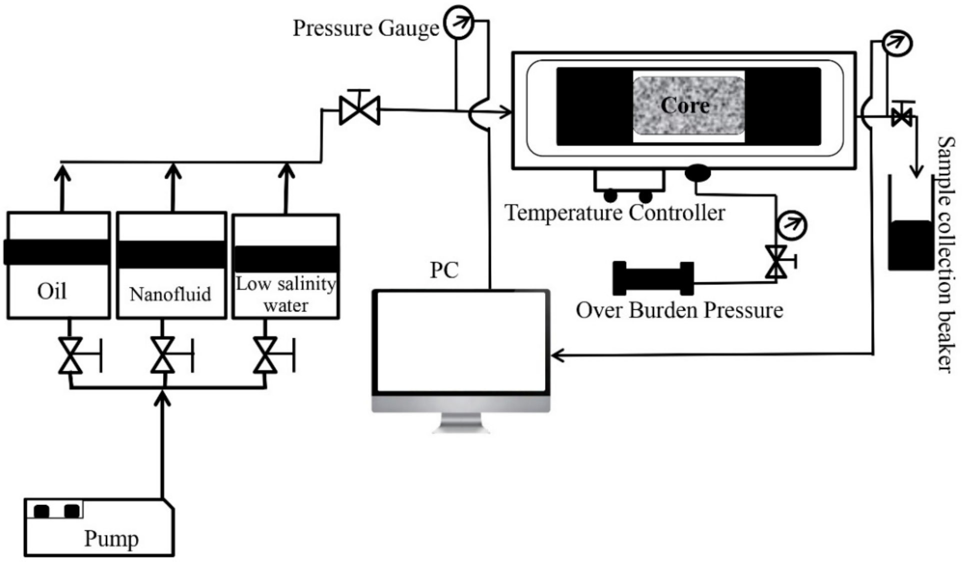


Fig. 2. Schematic representation of the experimental set-up used for the core flooding tests

3. Results and discussion

3.1. Structural and morphological properties of functionalized titanium dioxide N-particles

To confirm the structure of the synthesized carbon Nano dots, titanium dioxide, and functionalized titanium dioxide, XRD and FT-IR spectra were performed (Fig. 3, 4). Also, to investigate the morphology of the functionalized titanium dioxide N-particles, transmission electron microscopy was employed. The TEM images (see Fig. 5) revealed the presence of N-particles with an average diameter of less than 20 nm. The results of the TEM analysis are illustrated in Fig. 5a and 5b, indicating that the carbon Nano dots are uniformly distributed on the surface of the titanium dioxide N-particles.

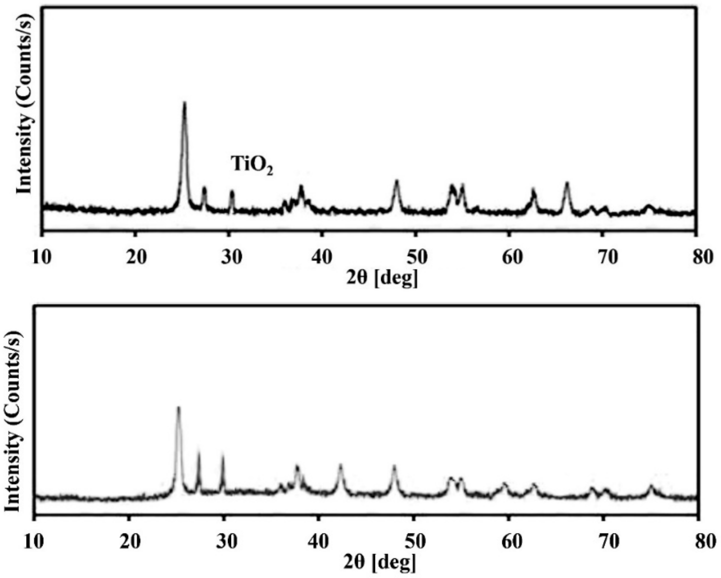


Fig. 3. XRD diffraction patterns for TiO_2 and functionalized titanium dioxide N-particles

3.2. Stability N-fluids

In this section, the pH levels of various N-fluids at concentrations of 100, 200, 300, 400, and 500 ppm were assessed. The zeta potential values provide insights into the electric potential at the slipping plane within the bulk liquid, away from the contact surface [1].

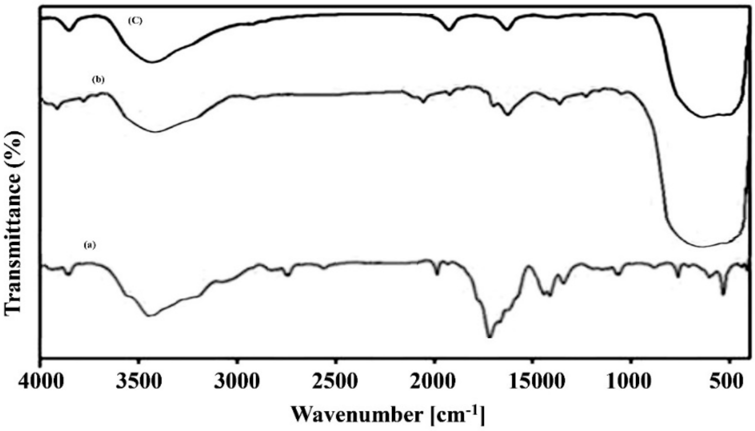


Fig. 4. FT-IR spectrum for carbon nanodots (a), titanium dioxide N-particles (b), and functionalized titanium dioxide N-particles (c)

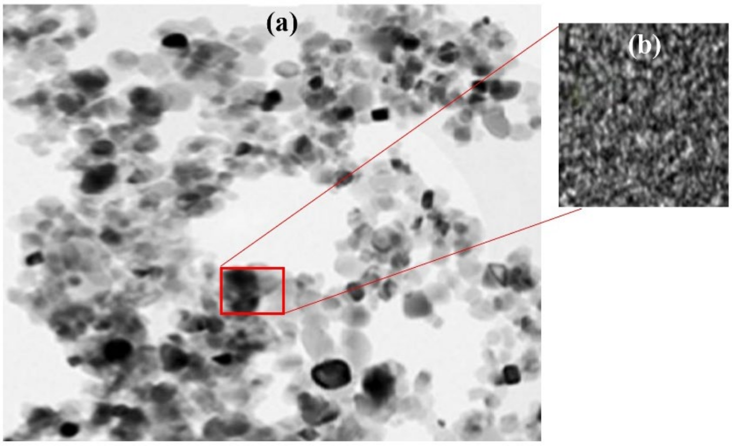


Fig 5. TEM image of functionalized titanium dioxide N-particles at (a) 100 nm, and (b) 10nm

3.2. Stability N-fluids

In this section, the pH levels of various N-fluids at concentrations of 100, 200, 300, 400, and 500 ppm were assessed. The zeta potential values provide insights into the electric potential at the slipping plane within the bulk liquid, away from the contact surface [1]. The stability of N-fluids is influenced by the surface charge of N-particles and the salinity of the base fluid. Specifically, a higher surface charge on N-particles leads to increased stability of the solution, while greater salinity results in decreased stability of the N-fluid [40,41]. Generally, zeta potential values around -30 mV are indicative of effective stability [42]. As depicted in Fig. 6, the N-fluids at a concentration of 200 ppm exhibited significant stability.

3.3. Impact of concentration N-fluids on interfacial tension and contact angle

In this section, the electrical conductivity of N-fluids at different concentrations was measured. As shown in Fig. 7, an unusual change was observed at the 200 ppm concentration, suggesting a dependence on ion release and properties of the Electrical Double Layer [43]. This indicates that strong interactions occur at this concentration. Consequently, the N-fluids at 200 ppm exhibited a pronounced negative charge, as confirmed by both electrical conductivity and zeta potential measurements. Furthermore, in the presence of N-fluid (200 ppm), the IFT decreased. The decrease in IFT was from the reference value of 19.88mN/m to 2.8mN/m, as illustrated in Fig. 8. The uncertainty linked to the interfacial tension measurements is ±0.5mN/m, indicating that the obtained interfacial tension values are trustworthy.

Therefore, any variations observed among the measured interfacial tension values are not due to measurement uncertainty.

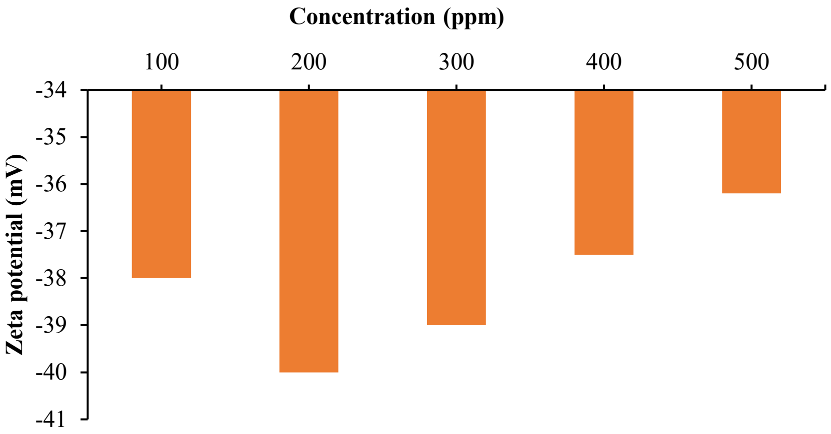


Fig. 6. Zeta potential values of the chosen N-fluid concentrations

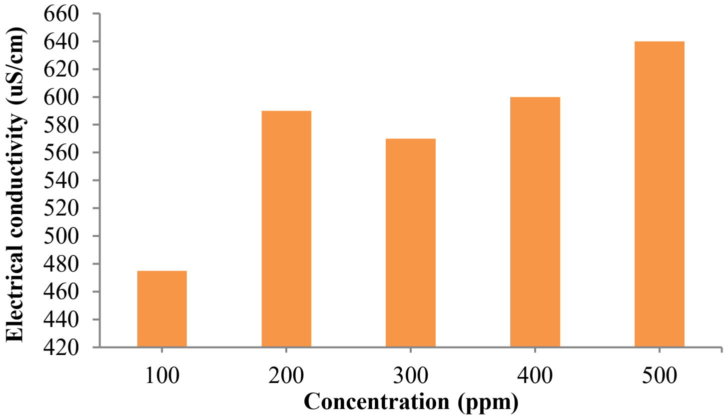


Fig. 7. Conductivity values of the chosen N-fluids concentrations

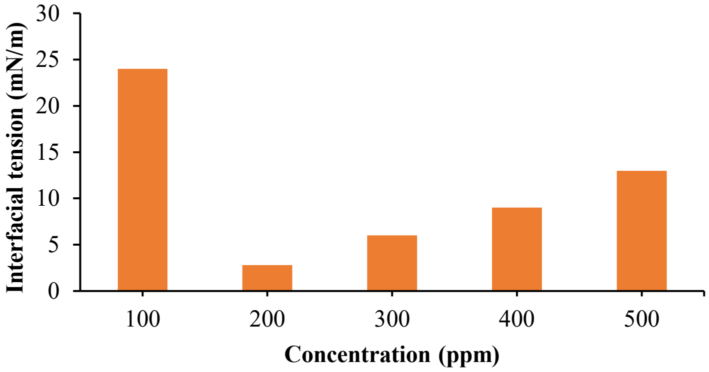


Fig. 8. IFT values of the chosen N-fluids concentrations

The initial contact angle of the rock indicated an oil-wet condition prior to treatment. Fig. 9 illustrates the oil contact angle measurements taken at various N-fluids concentrations (100, 200, 300, 400, and 500 ppm). Notably, the contact angle decreased from its original value to 26° (200 ppm) (Fig. 10), signifying a transition to a water-wet condition. To accurately compare data from different samples, operators, or methods, it is essential to comprehend the measurement uncertainty. Due to the uneven and non-uniform nature of the carbonate surfaces slice, uncertainties may arise during contact angle measurements. To mitigate this, the authors conducted at least three measurements at each point to determine the contact angle. Consequently, each reported contact angle value represents the average of a minimum of three independent measurements, with a maximum uncertainty of ±1° to 2°, as indicated by the statistical analysis.

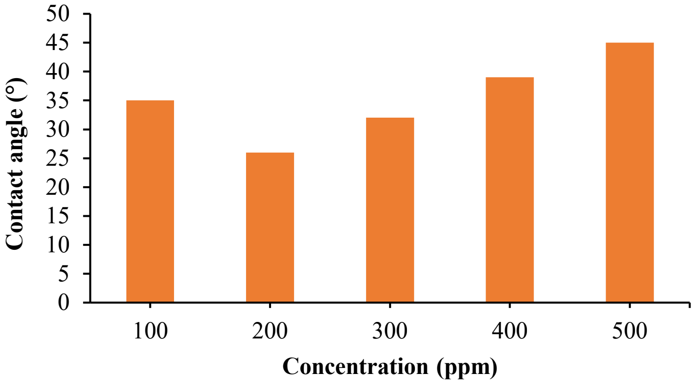


Fig. 9. Contact angle values at various concentrations of N-fluid

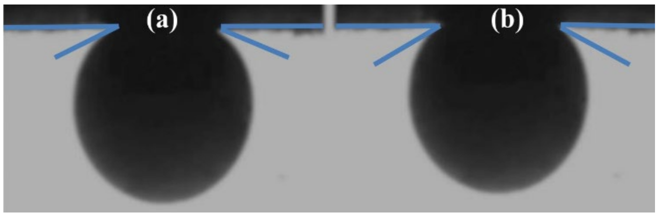


Fig. 10. Image of the contact angle measurement at 200 ppm (a) and 300 ppm (b)

3.4. Core flood tests

Core flooding tests revealed that the oil production at breakthrough was 51.5%. After injecting almost 5 pore volumes of the N-fluids dispersions, the overall oil production reached to 70.6%. The additional oil recovery attributed to the injection of 200 ppm N-fluids was found to be 19.1%. This results demonstrated that the N-fluid effectively displaces oil molecules from the oil-wet rock interface. The core flood test showed that at a concentration of 200 ppm, the oil recovery rate was 37%.

3.5. Displacement mechanism

In general, crude oil components, particularly negatively charged acids, tend to adhere to rock surfaces, resulting in oil-wet conditions. Thus, this study highlights the effectiveness of a formulated compound that incorporates N-particles, which enhance the viscosity of the aqueous phase while reducing the apparent viscosity of oil. The N-particles also create surface pressure at the oil-brine interface, promoting oil detachment from calcite surfaces through ion exchange and cation bridging. At a concentration of 200 ppm, the N-fluid behaves like an electrolyte due to its strong negative charge, and increasing its concentration raises system entropy. The positive surface charges of carbonate core rocks attract the negatively charged N-fluid, facilitating wettability changes. The interaction between hydroxyl ions in the N-fluid and the rock surface leads to a shift towards water-wet conditions, with the formation of a wedge film at the interface.

According to the model proposed by Wasan et al. [44], the N-particles in the oil/N-fluid/solid three-phase contact region tend to create a wedge film. Based on this theory, the modified N-particles on the solid surface cause a pressure gradient that shifts the oil-water interface, allowing the N-fluid to spread underneath the oil droplet. A layer of N-fluid forms between the oil and the rock surface, with modified N-particle molecules adhering to the rock due to π-π and n-π interactions. The presence of hydrophilic functional groups in the structure of the N-fluid alters the rock surface to a water-wet condition. At this point, a water film develops on the rock surface, facilitating the easy detachment of the oil droplet. When disjoining pressure exceeds the adhesion between oil droplets and the rock, oil separation occurs [45, 46]. These wettability changes result from various interactions, including electrostatic and van der Waals forces.

Furthermore, introducing ions such as SO_4^{2-}, Mg^{2+}, and Ca^{2+} into the N-fluids enhances the detachment of acids from rock surfaces [47], with SO_4^{2-} effectively neutralizing positive charges on carbonate rocks and releasing stearic acids. This process reduces oil-wet characteristics. The mobility of the N-fluid is influenced by factors such as the surface charge and stability of N-particles, as well as the rock's surface properties. The study suggests that when the surface charges of N-particles and core rocks are similar, the adsorption of N-fluids decreases, thereby enhancing mobility and improving oil recovery potential. Table 1 compares the results of this study with those of other research. In this regard, N-fluid (200 ppm) showed significant changes in terms of altering the wettability of the rock and reducing interfacial tension. The findings also indicate that N-fluid properties such as stability, mobility, and solubility of fluids in the reservoir can be enhanced by carbon Nano dots/carboxymethyl cellulose coating on the surface of the N-particles. Overall, the results of this study indicate that the synergistic N-fluid can simultaneously demonstrate significant efficiency in reducing interfacial tension, altering the wettability of the core rock, and improving oil recovery. Such an approach is rarely found in previous studies where a synthetic N-fluid can effectively reduce interfacial tension, alter wettability, and enhance oil recovery.

Table 1. Comparing the findings of this study with other studies

N-fluids	**IFT**	**Ultimate oil recovery (OOIP %)**	**References**
Functionalized titanium dioxide/Low salinity water	19.88 mN/m to 2.8 mN/m	70.6%	Current research
Green TiO_2/Quartz nanocomposite	36.4 mN/m to 3.5 mN/m,	54%	[45]
TiO_2 nanoparticles and polymer/salinity	24.5 mN/m to 9.5 mN/m	62%	[48]
TiO_2/polymer/brine	-	51.3%	[49]
TiO_2/GO nanocomposite with surfactant	39 mN/m to 10.5 mN/m	65%	[50]
Functionalized SiO_2 with β-cyclodextrin	35 mN/m to 22.5 mN	59.2%	[51]
MgO/surfactant	47.9 mN/m to 5.6 mN	-	[52]

4. Implications

This study highlights that employing N-particles at an optimal concentration of 200 ppm in low salinity water solutions significantly enhances oil recovery. This improvement is primarily due to changes in wettability towards a water-wet state, facilitated by the adsorption of nanoaggregate complexes and a reduction in interfacial tension between the oil and aqueous phases. Additionally, the migration of fines resulting from low salinity effects can be effectively managed with the use of N-particles. The implications of this research are significant for enhancing the performance of low salinity water injection projects.

5. Conclusion

As a material with superior performance (i.e. TiO_2), with super wettability, it exhibits remarkable properties in EOR. Wettability alteration and IFT are considered as the key petro physical properties that aid in the displacement of oil. Ensuring changes in such reservoir characteristics are the mainstream performances of the N-particles in the reservoirs. In this study, a surface-modified, stable, and cost-effective nanoparticle for oil recovery was synthesized using a multi-step process. Carbon Nano dots, known for their exceptional stability, were used as promising candidates in the synthesis process. The structural characteristics of the resulting samples were analyzed using X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FT-IR), and Transmission Electron Microscopy (TEM). Five different concentrations of N-fluids—100 ppm, 200 ppm, 300 ppm, 400 ppm and 500 ppm—were investigated. The XRD analysis indicated that the lack of diffraction peaks for carbon Nano dots was due to their tiny size and uniform distribution on the titanium dioxide surface. Furthermore, it was observed that fines migration driven by low salinity water effects is significantly mitigated when incorporating the N-fluids into the solution. The presence of compounds (carbon Nano dots/carboxymethyl cellulose/low salinity water) in the synthetic N-fluid structure provided high stability for the N-fluid. Among all tested samples, the formulation with 200 ppm of N-fluids achieved the highest oil recovery rate, reaching 37%. The results showed that in the presence of synthetic N-fluid, IFT decreases from 19.88 (reference value) to 2.8 (mN/m). Also, the contact angle decreased from the initial value to 26°. These results highlight the critical role of selecting suitable N-particles to optimize hybrid low salinity water flooding processes, offering practical insights for developing advanced enhanced oil recovery technologies for the future.

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