

A Feasibility Study on the Application of γ -Fe₂O₃ Nanoparticles Coupled with Low-salinity Water for Enhancing Oil Recovery in Tipam Sandstone Formation of the Hapjan Oil Field of Upper Assam Basin

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Abstract

It is observed that iron oxide (Fe₂O₃/Fe₃O₄) nanoparticles can substantially boost oil recovery in sandstone reservoirs by altering the rock wettability to a more water-wet state and decreasing the interfacial tension (IFT) and oil viscosity. According to recent research, using nanoparticles in conjunction with Low salinity waterflooding (LSW) can offer advantages from both LSW and nanoparticles. This study investigates the potential of using γ -Fe₂O₃ (Iron Oxide) nanoparticles in combination with LSW to improve oil recovery from the Tipam Sandstone formation of the Hapjan Oil Field, located in the Upper Assam Basin, India. Certain clay minerals, multivalent cations and polar compounds are evident in the analysis of the crude oil/brine/rock (COBR) system of the study area. These elements are necessary to successfully implement the low-salinity nanofluid Enhanced Oil Recovery (EOR).

Moreover, the high acid number (0.86) and resin-asphaltene ratio (25.35) in crude oil and the occurrence of mica, plagioclase feldspar and megahemite in the reservoir rock indicate that the area under study is a suitable candidate for applying low-salinity γ -Fe₂O₃ nanofluid EOR technique. The results of the study demonstrate that low-salinity γ -Fe₂O₃ nanofluid can modify the rock's wettability to become more water-wet and reduce the crude oil-nanofluid IFT. Moreover, the stability study shows that the iron oxide nanoparticles remain stable at specific concentrations in the low salinity environment. Therefore, using γ -Fe₂O₃ nanoparticles in combination with LSW could serve as an effective hybrid EOR technique for the study area.

Keywords: COBR, EOR, LSW, Nanoparticle, Wettability.

Introduction

Conventional oil recovery involves three main stages: primary, secondary and tertiary recovery processes. In the primary stage, oil is produced using the reservoir's own energy, typically recovering about 15-20 % of original oil in place (OOIP). As production continues, the reservoir

pressure diminishes, leading to a decline in oil recovery efficiency. To counteract this, the secondary recovery process is employed, which involves injecting gas or water into the reservoir to increase or maintain the reservoir pressure, thereby boosting oil recovery to around 30-50 % of OOIP. The tertiary recovery method, often referred to as enhanced oil recovery (EOR), involves the application of more expansive and exotic fluids in the reservoir, which extract additional oil of around 30-60 % of OOIP from the reservoir by favorably altering some reservoir properties^{13,53}. The main aim of any EOR technique is to lower the residual oil saturation (ROS) by increasing the overall displacement efficiency.

Different EOR techniques applied to improve oil recovery are thermal, chemical, miscible and others²⁶. Chemical EOR methods use various chemicals to increase flood efficiency in order to improve the oil recovery by increasing mobility ratio, decreasing interfacial tension (IFT), and/or altering rock wettability^{2,15,23,58}. However, there are some negative aspects to chemical EOR, including the degradation of chemicals under reservoir conditions, the need for a large chemical volume and high costs. The nanoparticles' application in the oil and gas field for enhancing oil recovery has emerged as a transformative force, capable of shifting rock wettability to more water wetness, improving mobility ratio, improving sand consolidation, reducing fine migration, decreasing oil-water interfacial tension (IFT) and reducing asphaltene precipitation.

Nanoparticles (NPs) are particles with sizes ranging between 1 to 100 nanometers²⁸. Compared to the injection fluids used in traditional EOR methods, such as chemicals, brine and gas, they have some useful properties as EOR agents. These small particles differ from bulk materials in that they possess unique electrical, optical, magnetic and chemical properties. Earlier studies have observed that nanoparticles can move into the pore spaces of the reservoir rock along with the injected fluid, which conventional recovery fluids cannot, resulting in better recovery¹³. In recent years, nanoparticles have gained significant attention, revealing their potential in EOR applications. Numerous studies over the past decades have highlighted the role of nanoparticles in optimizing EOR techniques^{6,7,36}.

Research has shown that nanoparticles are particularly effective in controlling fluid mobility, reducing water

production, increasing sweep efficiency and oil recovery factor^{52,56}. Additionally, they can decrease capillary forces, lower the relative permeability of water and modify the flow paths of water within the reservoir rock⁵². Moreover, nanoparticles have shown excellent resistance to degradation in high salinity and temperature conditions of reservoirs.

Silica nanoparticles (SiO_2 NP) are the most widely studied nanoparticles, which have demonstrated significant potential in EOR³⁸⁻⁴⁰. Recent studies have also observed that Aluminium Oxide (Al_2O_3), Iron Oxide ($\text{Fe}_2\text{O}_3/\text{Fe}_3\text{O}_4$), Copper Oxide (CuO), Magnesium Oxide (MgO), Nickel Oxide (Ni_2O_3), Zinc Oxide (ZnO) nanoparticles have great possibilities in EOR applications in addition to silica nanoparticles^{5,43}. Several mechanisms have been suggested for how nanoparticles improve oil recovery including disjoining pressure, prevention of asphaltene precipitation, pore channel plugging, increased viscosity of injection fluid, reduction of oil-nanofluid interfacial tension (IFT) and alteration of rock wettability⁵⁷.

Iron oxide nanoparticles (IONPs) are gaining significant interest in the field of EOR due to their distinctive properties. Magnetic nanoparticles (MNPs), including iron oxide, are increasingly being utilized in EOR, as evidenced by numerous research studies^{16,21,25,42,47}. Earlier studies have demonstrated that IONPs can adsorb more asphaltene molecules, which enhance the stability of the emulsions⁴⁷. Additionally, it is found that IONPs effectively remove asphaltene molecules, modify rock wettability and prevent scale formation, all of which can contribute to better flow assurance during nanofluid flooding in EOR applications⁴².

The magnetic properties of IONPs are the most important characteristics that make them attractive for the EOR technique. Literatures highlights IONPs as highly promising nanoparticles for oilfield applications due to their low production costs, low toxicity, easy synthesis and modification processes and ease of magnetic separation^{1,44,48}. Ferrofluids are suggested to be used for enhancing oil production by reducing the viscosity of oil, as the dipole moments within the particles cause reservoir fluid molecules to align, thereby lowering flow resistance²⁷. It is observed that Fe_3O_4 nanoparticles can enhance the viscosity of the nanofluid while simultaneously reducing the viscosity of oil, leading to an improved mobility ratio⁵⁵.

Studies such as sand pack flooding and spontaneous imbibition have demonstrated that Fe_3O_4 dispersed in deionized water holds significant potential for reducing residual oil saturation⁴¹. It has been observed that using iron oxide nanoparticles with particle sizes of 10-25 nm and 30-90 nm can lead to incremental oil recoveries of 38% and 27% respectively¹⁸. Previous studies have reported significant alteration in oil-nanofluid interfacial tension when using iron oxide nanoparticles^{11,42}. It has also been observed that iron oxide nanoparticles can create stable *in situ* emulsions within

the porous media, which can improve the recovery of oil²⁴. As mentioned above, the shifting of rock wettability state to a more water wetness is one of the major reasons for improving oil recovery during nanofluid flooding. It is found that the major mechanisms for the wettability modification by nanoparticles include the formation of ion-pair and the adsorption of nanoparticles onto the rock surface through hydrogen bonding and hydrophobic and electrostatic interactions. Therefore, the high surface energy of the hydrophilic iron oxide nanoparticles can shift the rock wettability in the direction of the more water-wetting state, which improves the oil recovery¹⁹.

Furthermore, low salinity waterflooding (LSW) is a new green enhanced oil recovery (GEOR) method that can improve the oil recovery efficiency through different mechanisms, including fine migration, multicomponent ion exchange (MIE), pH increase and electrical double layer (EDL) expansion³⁰. All these different mechanisms ultimately alter the rock wettability to a more water-wet state which improves the oil recovery. Earlier studies have shown that this GEOR technique can improve the oil recovery by 2% to 42% depending on the crude oil composition, brine chemistry and rock type¹⁴. According to recent research, using nanoparticles in conjunction with LSW can offer advantages from both LSW and nanoparticles.

In the present study, Fe_2O_3 (gamma) nanoparticles have been taken to study the feasibility of the application for enhancing oil recovery coupled with low-salinity water in the Tipam Sandstone formation of the Hapjan oil field. The Hapjan structure was identified in 1984 on the basis of the interpretation of seismic data. It is a part of the greater Hapjan oil field within Oil India Limited's (OIL's) operation area in the Upper Assam basin, India.

Material and Methods

For this study, an analysis of the reservoir rock, brine and crude oil has been made to study the composition of the COBR system of the area under study, which plays a pivotal role in the success of low-salinity nanofluid flooding. The reservoir rock samples were collected from a depth range of 2315.58 – 2329.50 meters from the study area. The brine and the crude oil were collected from the same Tipam Reservoir Sandstone of the Upper Assam basin. To study the effects of Iron Oxide NP coupled with low salinity water on oil recovery in the Tipam Reservoir Sandstone, ferric oxide (Gamma) NP ($\gamma\text{-Fe}_2\text{O}_3$) was purchased from the Sisco Research Laboratories Pvt. Ltd., Maharashtra, India, which has an assay of 99.9 % and average particle size of 20-50 nanometer. Additionally, Molychem in Mumbai was the source of the magnesium chloride, sodium chloride and calcium chloride needed to prepare the brine solution.

Reservoir Rock Analysis: The successful application of low salinity nanofluid EOR in a sandstone reservoir depends critically on the presence of certain clays, feldspar and mica in the rock became analyzing the mineralogical composition

of the rock is crucial^{20,34,37,45,46,50,51}. Here, the rock samples were examined using Thin Section, X-ray diffractometer and Scanning Electron Microscope (SEM). Figures 1-2, 3-4 and 5 show thin section photomicrographs, SEM images and X-ray diffraction (XRD) patterns respectively of a few rock samples. It is observed that Kaolinite, Smectite, Illite, Plagioclase Feldspar, Mica-Montmorillonite, Muscovite, Maghemite and Quartz are present in the rock matrix.

Analysis of the Formation of Brine and Crude Oil: Previous research has demonstrated that brine salinity can be crucial to the stability of the COBR system, which affects oil recovery^{4,50,51}. Also, for a successful nanoparticle-assisted LSW technique, Ca^{2+} and Mg^{2+} ions presence in the brine is very important^{3,30}. It is observed that the salinity (as NaCl) of the formation brine is 1000 mg/l, whereas the total dissolved solid (TDS) is 1241 mg/l. Moreover, the brine contains 56 mg/l Ca^{2+} and 28.8 mg/l Mg^{2+} ions respectively¹⁷.

It is observed that the asphaltene and resin (polar components) and high Acid Number (AN) of oil have a major effect on the oil recovery efficiency during low salinity nanofluid EOR^{10,29}. In order to ascertain the asphaltene content, resin content and acid number of crude oil, a study has been conducted utilizing the test methods IP 143/84, ASTM D 3279 and designation 139/98 (Under D974) respectively. It is observed that the acid number, resin and asphaltene present in the crude are 0.86, 10.14% (w/w) and 0.40 % (w/w) respectively. Moreover, the API gravity (at 60 °F), pour point and wax content of the crude Oil are 20.3, <9 and 12.55 % (w/w) respectively¹⁷.

Separation and Analysis of the Reservoir Magnetic Material: For this study, a produced sand sample was collected from one of the producing wells in the study area. The sand sample was dried in an oven at 80° C for around 40 minutes. A magnet was then used for the separation of the magnetic materials present in the sand sample. A total of 1.95% magnetic materials were observed in the collected produced sand sample. The separated magnetic materials were then studied under SEM and EDX (Energy Dispersive X-Ray Analyzer). The study confirms the presence of iron oxide in the produced sand sample (Figure 6).

Preparation of Iron Oxide Nanofluids: Earlier studies have shown that an injection brine with a salinity of 57% of formation brine can provide the highest recovery of oil in the Tipam and Barail reservoir sandstone of Upper Assam basin through the reduction of contact angle (CA) and oil-brine IFT^{32,33}. Therefore, the salinity (as NaCl) of the low-salinity brine was kept at 570 ppm, which is 57% of the formation brine salinity (1000 ppm) of the study area. The low salinity brine was prepared by mixing NaCl, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ and $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ in required proportions with distilled water. Seven Fe_2O_3 nanoparticle solutions were prepared with the low-salinity brine (570 ppm) for this study. The nanoparticle concentrations in the nanofluid range from 0.01 to 0.5 %

(w/w). The nanoparticles were mixed with the brine solution using an Ultrasonic Processor (Sonicator, Model EI-750UP).

Effects of Iron Oxide Nanoparticles on Rock Wettability: Previous studies show that wettability alteration occurs in the reservoir rock during the application of nanoparticles in sandstone reservoirs. As mentioned above, the mechanism behind this wettability alteration is forming a wedge film structure that desorbs the oil from the rock surfaces¹². To study the effects of low salinity Fe_2O_3 nanofluid on rock wettability, eight reservoir rock samples were cut (0.5-inch-thick) from the collected rock samples. The rock samples were cleaned and dried properly using 'Soxhlet Extraction Unit,' 'Ultrasonic Cleaner,' and 'Humidity Oven.' The different solvents used for cleaning purposes are toluene, methanol, chloroform and acetone. Chloroform has been found to be excellent for dissolving many Upper Assam Crudes and toluene has been found useful for asphaltic crudes and/or mixed base compounds⁸. The samples were dried in the humidity oven at 40% relative humidity and 63 °C Dry Bulb temperature.

The prepared rock samples were first saturated in brine for one day in vacuum desiccator. The samples were then saturated in crude oil for another day. The contact angles of the rock samples were then determined using 'Drop Shape Analyzer- DSA25' at atmospheric pressure and 70°C. The alteration of rock wettability (Contact Angle) with different concentrations of Fe_2O_3 nanoparticles is presented in table 1. The contact angle of the reservoir rock sample saturated with crude oil and reservoir brine (salinity 1000 ppm) was 52.96 degrees.

Effects of Iron Oxide Nanoparticles on Crude Oil-Nanofluid Interfacial Tension: As mentioned earlier, nanoparticles can reduce oil-nanofluid IFT through the adsorption of nanoparticles at the oil-brine interface. This IFT reduction greatly reduces the residual oil saturation in the pore spaces of the rock. In this work, a study has been made on the effects of Fe_2O_3 nanoparticles on crude oil-nanofluid IFT under a low salinity environment. The experiment was conducted in Drop Shape Analyzer -DSA25 at 70 °C. Table 1 shows the Crude Oil- Fe_2O_3 Nanofluid IFT against various concentrations of Fe_2O_3 nanoparticles in 570 ppm brine. The crude oil-formation brine IFT was found to be 33.14 mN/m.

Study on the Stability of Iron Oxide Nanoparticles in Nanofluid: The stability of the iron oxide nanoparticles in the nanofluid plays a crucial role in enhancing the oil recovery during nanofluid flooding. According to Stokes Law, viscous, buoyancy and gravity force affect the nanoparticle suspension in the base fluid. For this work, a study has been made on the stability of the Fe_2O_3 nanoparticles in low-salinity brine for a duration of ten days. Nanoparticles concentrations of 0.01 wt%, 0.05 wt%, 0.1 wt%, 0.2 wt%, 0.3 wt%, 0.4 wt% and 0.5 wt% were mixed with the 570 ppm brine using the Sonicator (Model EI-

750UP). The prepared nanofluids were kept in seven separate test tubes with stands. The samples were then observed for ten days. Care was taken to ensure no jerking in the test tubes throughout that period. It is observed that Fe_2O_3 nanoparticles progressively settle at the test tube bottom over time (Figure 7). However, after ten days, some nanoparticles are seen to be in suspension.

Zeta potential analysis is another essential tool for predicting the long-term stability of nanoparticles in solution. This technique measured the suspended nanoparticle's surface charge in a solution (colloid). Nanoparticles possess a surface charge that attracts a surrounding layer of oppositely charged ions, forming an electrical double layer (EDL). The electric potential at the edge of this EDL is referred to as the Zeta potential, which typically ranges from +100 mV to -100 mV. The Zeta potential value provides insights into colloidal stability. A colloidal system will have moderate stability when the zeta potential is from ± 10 to ± 50 mV and has excellent stability when the potential is greater than ± 50 mV⁵⁴.

It is also observed that, for good physical stability, the Zeta Potential values of nanoparticles should be higher than ± 30 mV³¹. However, for nonionic stabilizers like water, a colloidal nano-dispersed system remains stable when the Zeta potential values are greater than ± 20 mV⁴⁹.

To study the $\gamma\text{-Fe}_2\text{O}_3$ nanoparticle's stability in low salinity nanofluid in detail, the Zeta potential of the nanofluids was

determined after ten days. Here, the low salinity nanofluids were prepared by mixing the $\gamma\text{-Fe}_2\text{O}_3$ nanoparticles with diluted formation brine. The salinity of the formation brine of the study area was reduced to 570 ppm (as NaCl) by mixing distilled water in required proportion.

For this study, 'Litesizer™ 500' was used to measure the Zeta potential of the $\gamma\text{-Fe}_2\text{O}_3$ NPs in the nanofluids. The instrument can determine the Zeta potential by Electrophoretic light scattering (ELS). It measures the speed of the particles in the colloidal suspension in the presence of an electric field. The particle speed was determined by their surface charge or Zeta potential. The colloid becomes more stable as the Zeta potential increases. Table 2 shows the Zeta potential of the nanofluids after 10 days of nanofluid preparation.

Results and Discussion

Applications of enhanced oil recovery methods have gained much attention over the past decade since petroleum reserves have been declining. While conventional EOR techniques are still in use, emerging technologies such as nanotechnology are showing significant promise in this field. Nanoparticles (NPs) show better results in recovering the entrapped oil from the rock pore spaces compared to other conventional methods. As mentioned above, injection of iron oxide nanoparticles coupled with low salinity water into oil reservoirs can recover more oil than some other EOR methods.

Table 1
The alteration of Contact Angle and Crude Oil-Nanofluid IFT with various concentrations of Fe_2O_3 Nanoparticles

S.N.	NP Concentration (wt%)	Crude Oil-Nanofluid IFT (mN/m)	Contact Angle (Degree)
1	0	28.44	51.24
2	0.01	19.24	49.21
3	0.05	17.91	46.48
4	0.1	17.21	42.16
5	0.2	16.68	41.78
6	0.3	16.24	40.44
7	0.4	15.98	40.16
8	0.5	15.89	39.84

Table 2
The Zeta Potential of Low Salinity Fe_2O_3 Nanofluid after 10 days

S.N.	NP Concentration (wt%)	Zeta Potential (mV)
1	0.01	-31.2
2	0.05	-30.1
3	0.1	-29.8
4	0.2	-29.1
5	0.3	-27.6
6	0.4	-25.8
7	0.5	-23.4

The petrographic analysis of the reservoir rock shows the presence of Kaolinite, Smectite, Illite, Plagioclase Feldspar, Mica-Montmorillonite, Muscovite, Maghemite, Ferruginous Cement and Quartz (Figures 1-5). Earlier studies have shown that Kaolinite and Illite clay minerals can detach from the rock surfaces and migrate with the flowing fluid in a sandstone reservoir during LSW³⁷. It has already been proved that these clay minerals migrate in the Tipam reservoir sandstone of the Upper Assam basin during LSW³⁴. It is also found that Smectite swelling occurs during the injection of low salinity brine, which breaks the fines, attached to them. As a result, fines are migrated with the flowing fluid (Swelling-induced Migration)³⁷. This phenomenon can improve the sweep efficiency by changing the fluid flow direction towards the previously un-swept flooded volume, thus improving the oil recovery⁴⁶.

In addition to this, mica can contribute to the improvement of sweep efficiency by moving with the flowing fluid, which can further reduce the remaining oil saturation⁴⁵. It is observed that these migrated fines also improve the oil recovery efficiency by reducing the oil-brine IFT and contact angle⁵⁰. Further, the Plagioclase feldspar in the rock matrix shows that the area under study is favorable for LSW application²⁰. The presence of Maghemite (γ -Fe₂O₃) and Ferruginous cement in the reservoir rock confirms that the area is favorable for injecting γ -Fe₂O₃ nanoparticles for enhancing oil recovery as this already existing component will not affect the health of the reservoir. The iron oxide presence is also confirmed by the SEM and EDX analysis of the magnetic materials separated from the produced sand samples in the study area.

The reservoir brine analysis shows that it contains 56 mg/l Ca²⁺ and 28.8 mg/l Mg²⁺ ions. Earlier studies have proven that Ca²⁺ and Mg²⁺ ions play a crucial role in the 'Multicomponent Ion Exchange' mechanism during LSW which shifted the rock wettability towards more water wetness for enhancing oil recovery²⁹. The study also shows that the acid number, resin and asphaltene present in the crude oil are 0.86, 10.14% (w/w) and 0.40 % (w/w)

respectively, as mentioned above. The presence of resin and asphaltene, along with the acid number of oil greater than 0.2 plays a crucial role in altering the rock wettability in a favorable way during LSW^{9,10,22,29}.

Previous studies indicate that a resin/asphaltene ratio of less than three can lead to asphaltene precipitation, which increases the rock's oil-wetness, reduces rock permeability and increases residual oil saturation³⁵. In the current study, the resin/asphaltene ratio is 25.35, suggesting that asphaltene precipitation is unlikely to occur in the study area.

The study on the effects of γ -Fe₂O₃ nanoparticles on rock wettability shows that the nanoparticles have a pronounced effect on wettability. The contact angle of the reservoir rock sample saturated with crude oil and reservoir brine (salinity 1000 ppm) is 52.96°. However, this contact angle decreased to 51.24° when the rock sample was saturated with low salinity brine (570 ppm), which is 57% of formation brine. As mentioned above, an injection brine with a salinity of 57% of formation brine can provide the highest recovery of oil in the Tipam and Barail Reservoir Sandstone of Upper Assam basin with the highest wettability alteration towards more water wetness. With increasing the iron oxide nanoparticle concentrations in the low salinity brine (570 ppm) from 0.01 wt% to 0.5 wt%, the contact angle of the rock decreases from 49.21° to 39.84° (Table 1).

Although low salinity brine can reduce the contact angle by 01.72°, low salinity iron oxide nanofluid can alter the rock wettability to a great extent, up to 13.12°. This indicates that the iron oxide (γ -Fe₂O₃) nanoparticles coupled with the low salinity brine can shift the reservoir rock wettability to a more water-wet condition in the study area which can ultimately enhance the oil production. The study also shows that the decrease in contact angle is more when the γ -Fe₂O₃ nanoparticles concentration increased from 0.01 wt% to 0.2 wt%. However, the effect of γ -Fe₂O₃ nanoparticles on contact angle is low when its concentrations increase from 0.2 wt% to 0.5 wt%.



Figure 1: Photomicrograph showing Plagioclase Feldspar and Quartz.

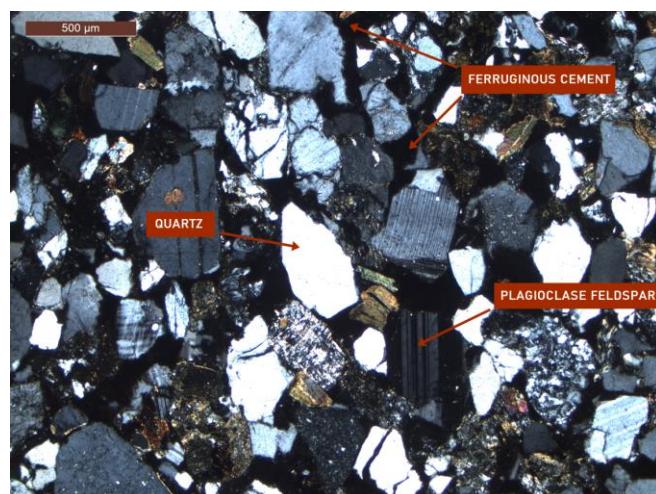


Figure 2: Photomicrograph showing Plagioclase Feldspar, Ferruginous Cement and Quartz.

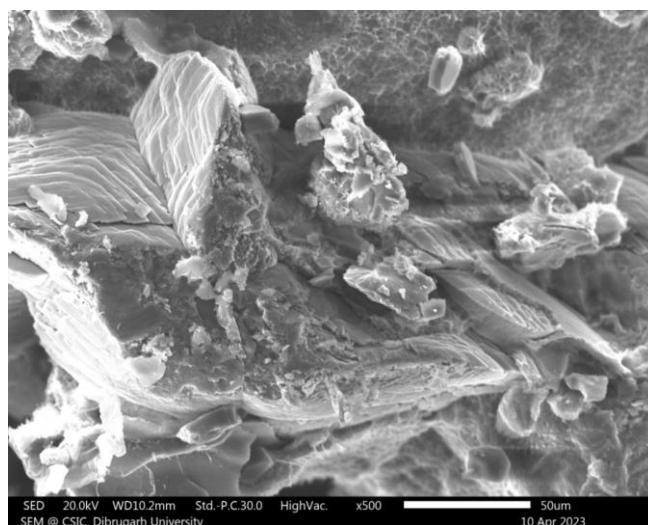


Figure 3: SEM Image showing Kaolinite, Smectite and Feldspar.

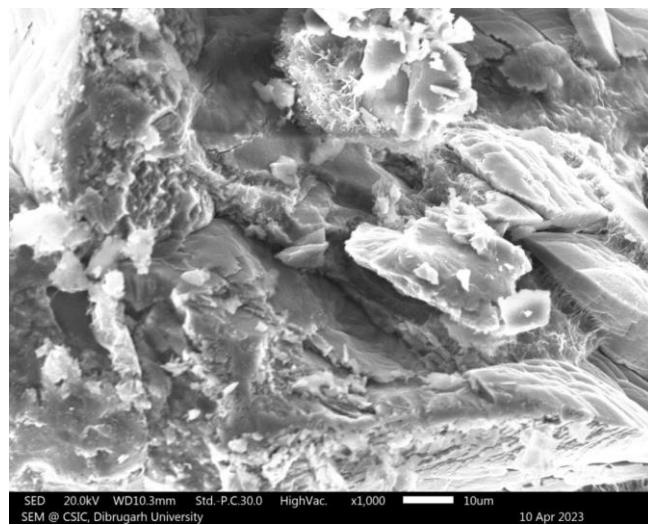


Figure 4: SEM Image showing Kaolinite, Ilite and Mica.

It is observed that the low salinity (570 ppm) brine can reduce the crude oil-brine IFT from 33.14 mN/m to 28.44 mN/m, where 33.14 mN/m is the IFT between the crude oil and reservoir brine of the study area. Increasing the γ -Fe₂O₃ nanoparticle concentrations from 0.01 wt% to 0.5 wt% in the

low salinity brine can decrease the crude oil-nanofluid IFT from 19.24 mN/m to 15.89 mN/m (Table 1). This indicates that γ -Fe₂O₃ nanoparticles can reduce the IFT more effectively than the low-salinity brine alone in the study area. The higher is the concentrations of γ -Fe₂O₃ nanoparticles in

the 570 ppm brine, the lower is the oil-nanofluid IFT. This low IFT can increase the oil production in the study area. However, it is observed that there is a rapid decrease of IFT up to 0.2 wt% γ -Fe₂O₃ NP concentration. Between 0.2 wt% and 0.5 wt%, the change in IFT values is comparatively low.

As mentioned above, the nanoparticle's stability in the nanofluid plays a major role in improving the oil recovery during nanofluid flooding. Therefore, a stability study has been made on γ -Fe₂O₃ nanoparticles in 570 ppm low salinity brine prepared by diluting the formation brine of the study area. The stability of the 0.01 wt%, 0.05 wt%, 0.1 wt%, 0.2 wt%, 0.3 wt%, 0.4 wt% and 0.5 wt% nanoparticles was observed for ten days, which shows that the nanoparticles progressively settle with time (Figure 7). The visual

observation also shows that the lower concentrations of nanoparticles are more stable in the colloidal solution during that period. The Zeta potential study shows that 0.01 wt% and 0.05 wt% γ -Fe₂O₃ nanoparticles are very stable with -31.2 mV and -30.1 mV respectively, even after ten days (Table 2). As mentioned above, for good physical stability, the Zeta potential values of nanoparticles should be higher than ± 30 mV³¹. The nanoparticle concentrations from 0.1 wt% to 0.5 wt% are also moderately stable, with Zeta potential values ranging from -29.8 mV to -23.4 mV, as suggested by Wakman⁵⁴. This indicates that lower concentrations of γ -Fe₂O₃ nanoparticles in 570 ppm brine are more stable in the nanofluid, which can result in higher oil recovery in the area under study.

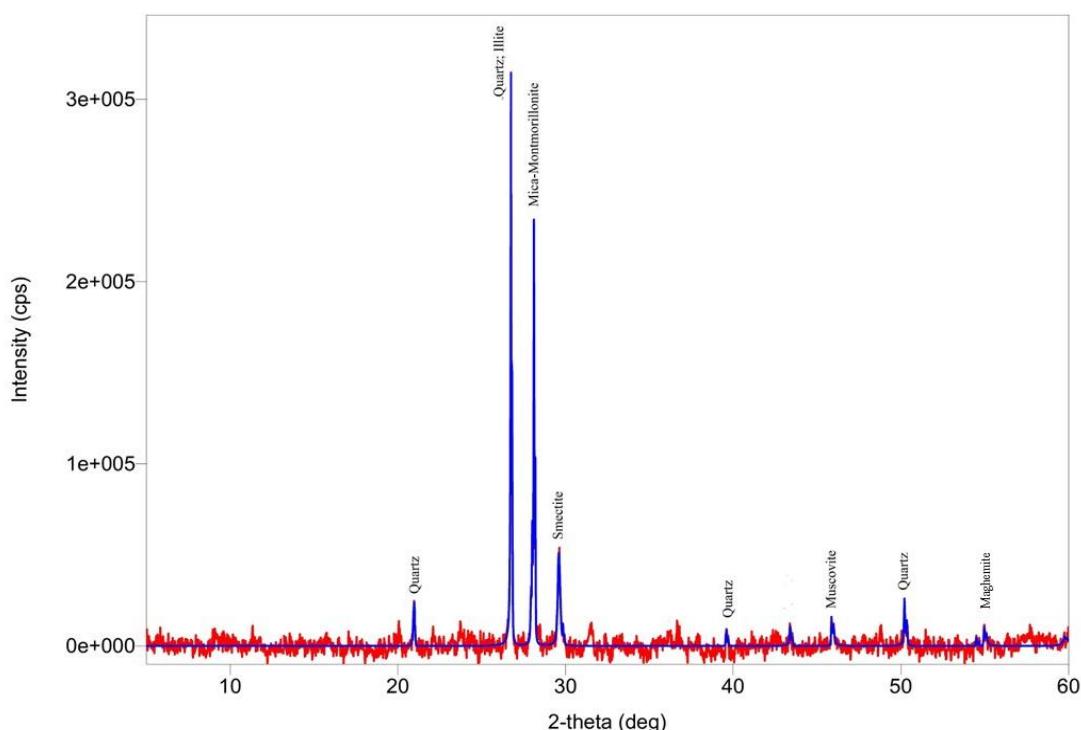


Figure 5: XRD patterns showing Smectite, Mica-Montmorillonite, Illite, Muscovite, Maghemite and Quartz.

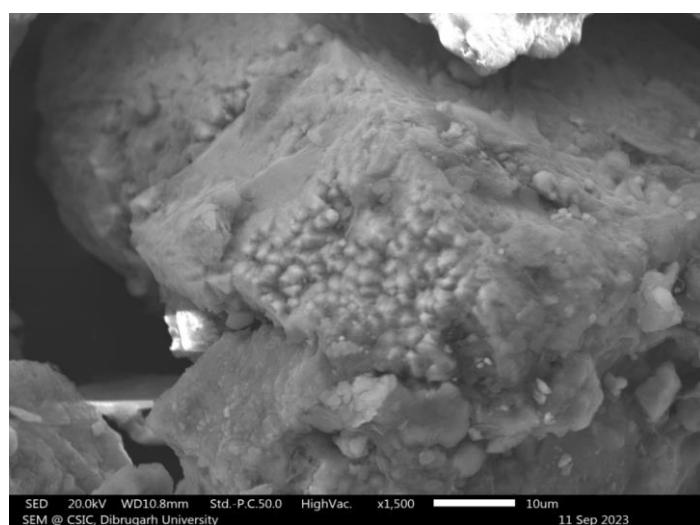


Figure 6: SEM Image showing Iron Oxide.

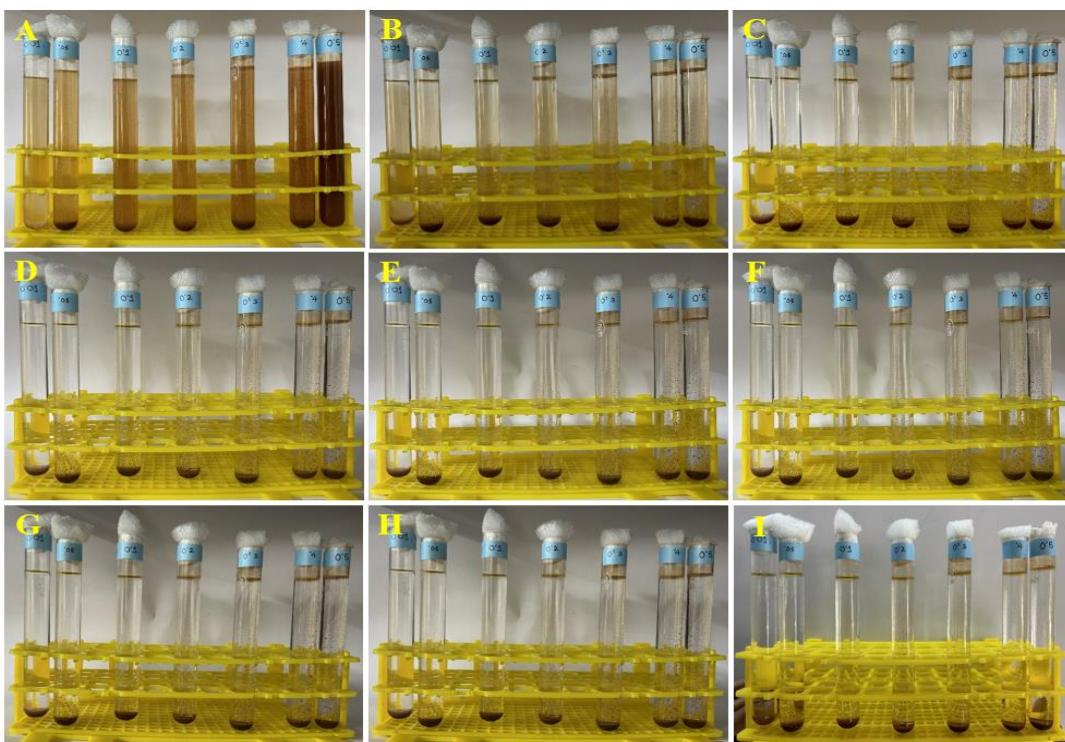


Figure 7: Stability of the Iron Oxide Nanoparticles in Low Salinity Water after (A) 00 Hour, (B) 01 Day, (C) 02 Day, (D) 03 Day, (E) 04 Day, (F) 05 Day, (G) 06 Day, (H) 08 Day and (I) Day 10.

Conclusion

The presence of clay minerals, Plagioclase feldspar, mica, iron oxide, ferruginous cement and maghemite in the reservoir rock, asphaltene and resin in the crude oil and Ca^{2+} and Mg^{2+} ions in the formation brine strongly indicates that the iron oxide ($\gamma\text{-Fe}_2\text{O}_3$) nanoparticles can be applied with low-salinity brine for enhancing oil recovery in the Tipam Sandstone formation of the Hapjan oil field. The $\gamma\text{-Fe}_2\text{O}_3$ low-salinity nanofluids with nanoparticle concentrations from 0.01 wt% to 0.5 wt% are effective in lowering the oil-brine IFT and shifting the rock wettability to a more water-wet state.

The stability state of the $\gamma\text{-Fe}_2\text{O}_3$ nanoparticles is from good to moderate in 570 ppm brine. Therefore, the application of $\gamma\text{-Fe}_2\text{O}_3$ nanoparticles coupled with 570 ppm low salinity brine can be a new green enhanced oil recovery (GEOR) technique in the study area.

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References

1. Adewunmi A.A., Kamal M.S. and Solling T.I., Application of magnetic nanoparticles in demulsification: A review on synthesis, performance, recyclability and challenges, *Journal of Petroleum Science & Engineering*, **196**, 107680 (2021)
2. Ahmadi M.A. and Shadizadeh S.R., Experimental investigation of adsorption of a new nonionic surfactant on carbonate minerals, *Fuel (London, England)*, **104**, 462-467 (2013)
3. Anderson W.G., Wettability literature survey-part 6: The effects of wettability on waterflooding, *Journal of Petroleum Technology*, **39(12)**, 1605-1622 (1987)
4. Boussour S., Cissokho M., Cordier P., Bertin H. and Hamon G., Oil recovery by low salinity brine injection: Laboratory results on outcrop and reservoir cores. SPE Annual Technical Conference and Exhibition, New Orleans, Louisiana, October 4-7 (2009)
5. Cheraghian G. and Hendraningrat L., A review on applications of nanotechnology in the enhanced oil recovery part A: effects of nanoparticles on interfacial tension, *International Nano Letters*, **6(2)**, 129- 138 (2016)
6. Cheraghian G. and Tardasti S., Improved oil recovery by the efficiency of nano-particle in imbibition mechanism. 74th EAGE Conference and Exhibition Incorporating EUROPEC, Copenhagen, Denmark (2012)
7. Cheraghian G., A new thermal method concept for IOR from oil reservoir using optimized *in-situ* combustion, 78th EAGE Conference and Exhibition, 1-3 (2016)

8. Chowdhury M.A., Scope of Implementation of IOR techniques based on Core Flood Studies with emphasis on the determination of Residual Oil Saturation for a part of Lakwa and Geleki oil fields of Upper Assam Basin, Ph.D. Thesis, Dibrugarh University (2013)

9. Ehrlich R. and Wygal R.J. Jr., Interrelation of crude oil and rock properties with the recovery of oil by caustic waterflooding, *Society of Petroleum Engineers Journal*, **17**(4), 263-270 (1977)

10. Ehrlich R., Hasiba H.H. and Raimondi P., Alkaline waterflooding for wettability alteration-evaluating a potential field application, *Journal of Petroleum Technology*, **26**(12), 1335-1343 (1974)

11. El-hoshoudy A.N., Gomaa S. and Taha M., Improving oil recovery using Fe_2O_3 nanoparticles flooding, *Petroleum and Coal*, **61**(4), 798-807 (2019)

12. Eltoum H., Yang Y.L. and Hou J.R., The effect of nanoparticles on reservoir wettability alteration: a critical review, *Petroleum Science*, **18**(1), 136-153 (2021)

13. Engeset B., The Potential of Hydrophilic Silica Nanoparticles for EOR Purposes: A litterateur review and an experimental study, Institutt for petroleumsteknologi og anvendt geofysikk, Retrieved from <https://ntuopen.ntnu.no/ntnu-xmliu/handle/11250/239771> (2012)

14. Gamage P. and Thyne G., Comparison of oil recovery by low salinity waterflooding in secondary and tertiary recovery modes, SPE Annual Technical Conference and Exhibition, Denver, Colorado, USA, October 30 (2011)

15. Glover P., Formation Evaluation, M.Sc. Course Notes, Aberdeen University, 19 (2001)

16. Gomaa S., Taha M. and El-hoshoudy A.N., Investigating the effect of different nanoparticles on the interfacial tension reduction, *Petroleum & Petrochemical Engineering Journal*, **2**(4), 1-7 (2018)

17. Goswami U. and Medhi N., Scope of implementation of low salinity nanofluid to improve oil recovery in a part of the hapjan oil field of upper Assam basin, *Res. J. Chem. Environ.*, **28**(2), 100-109 (2023)

18. Guan B.H., Khalid M.H.M., Matraji H.H., Chuan L.K. and Soleimani H., An evaluation of iron oxide nanofluids in enhanced oil recovery application, 3rd International Conference on Fundamental and Applied Sciences (ICFAS 2014): Innovative Research in Applied Sciences for a Sustainable Future, Kuala Lumpur, Malaysia, 3-5 June (2014)

19. Hou B.F., Wang Y.F. and Huang Y., Mechanism and influencing factors of wettability alteration of water-wet sandstone surface by CTAB, *Journal of Dispersion Science and Technology*, **36**(11), 1587-1594 (2015)

20. Hughes D., Law S. and Pitt G., Low Salinity EOR 'State of Play' Review, A12DEC056A, Department of Energy and Climate Change, Scotland (2012)

21. Izadi N., Koochi M.M., Amrollahi A. and Pourkhalil M., Investigation of functionalized polyelectrolyte polymer-coated Fe_3O_4 nanoparticles stabilized in high salinity brine at high temperatures as an EOR agent, *Journal of Petroleum Science & Engineering*, **178**, 1079-1091 (2019)

22. Jensen J.A. and Radke C.J., Chromatographic transport of alkaline buffers through reservoir rock, *SPE Reservoir Engineering*, **3**(3), 849-846 (1988)

23. Kamari A., Gharagheizi F., Shokrollahi A., Arabloo M. and Mohammadi A.H., Integrating a robust model for predicting surfactant-polymer flooding performance, *Journal of Petroleum Science & Engineering*, **137**, 87-96 (2016)

24. Kazemzadeh Y., Dehdari B., Etemadan Z., Riazi M. and Sharifi M., Experimental investigation into Fe_3O_4/SiO_2 nanoparticle performance and comparison with other nanofluids in enhanced oil recovery, *Petroleum Science*, **16**(3), 578-590 (2019)

25. Kazemzadeh Y., Sharifi M., Riazi M., Rezvani H. and Tabaei M., Potential effects of metal oxide/SiO₂ nanocomposites in EOR processes at different pressures, *Colloids and Surfaces, A, Physicochemical and Engineering Aspects*, **559**, 372-384 (2018)

26. Kokal S. and Al-Kaabi A., Enhanced Oil Recovery: Challenges & Opportunities, World Petroleum Council: Official Publication, <https://api.semanticscholar.org/CorpusID:16917044> (2010)

27. Kothari N., Raina B., Chandak K., Iyer V. and Mahajan H., Application of ferrofluid for enhanced Surfactant Flooding in EOR, SPE EUROPEC/EAGE Annual Conference and Exhibition, Barcelona, Spain, June 14 (2010)

28. Krishnamoorti R., Technology tomorrow: Extracting the benefits of nanotechnology for the oil industry, *Journal of Petroleum Technology*, **58**(11), 24-26 (2006)

29. Lager A., Collins I.R., Jerauld G.R., McGuire P.L. and Webb K., BP Low Salinity Patent Application (Hydrocarbon Recovery Process), PCT/GB2007/003337 - WO 2008/029124 A1 (2006-8)

30. Lager A., Webb K.J., Black C.J.J., Singleton M. and Sorbie K.S., Low Salinity Oil Recovery – An Experimental Investigation, International Symposium of the Society of Core Analysis, Trondheim, Norway, 12-16 September (2006)

31. Lee J.H. et al, Effective viscosities and thermal conductivities of aqueous nanofluids containing low volume concentrations of Al_2O_3 nanoparticles, *International Journal of Heat and Mass Transfer*, **51**(11-12), 2651-2656 (2008)

32. Medhi N. and Das M., An Experimental Study of Low Salinity Waterflooding in Secondary Recovery Mode in Geleki Oil Field of Assam-Arakan Basin, India, *Indian Journal of Natural Sciences*, **13**(71), 39633-39644 (2022)

33. Medhi N., A study on Low Salinity Waterflooding for Improving Oil Recovery with special reference to Geleki Oil Field of Upper Assam Basin, Ph.D. Thesis, Dibrugarh University (2016)

34. Medhi N., A Study on Fine's Migration during Low Salinity Waterflooding in Geleki Oilfield of Upper Assam Basin, *Journal of Applied Geochemistry*, **20**(3), 344-351 (2018)

35. Mehana M., Abraham J. and Fahes M., The impact of asphaltene deposition on fluid flow in sandstone, *Journal of Petroleum Science & Engineering*, **174**, 676-681 (2019)

36. Miranda C.R., de Lara L.S. and Tonetto B.C., Stability and mobility of functionalized silica nanoparticles for enhanced oil recovery applications, SPE International Oilfield Nanotechnology Conference and Exhibition, Noordwijk, The Netherlands, June 12 (2012)

37. Mohan K.K., Vaidya R.N., Reed M.G. and Fogler H.S., Water sensitivity of sandstones containing swelling and non-swelling clays, *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, **73**, 237-254 (1993)

38. Nasr M.S., Esmaeilnezhad E. and Choi H.J., Effect of silicon-based nanoparticles on enhanced oil recovery: Review, *Journal of the Taiwan Institute of Chemical Engineers*, **122**, 241-259 (2021)

39. Nasr M.S., Esmaeilnezhad E. and Choi H.J., Effect of carbon-based and metal-based nanoparticles on enhanced oil recovery: A review, *Journal of Molecular Liquids*, **338(116903)**, 116903 (2021)

40. Neginah Vijayasingh and Athiappan Murugan, Bio-transforming Syzygium Cumini Kernel into Microbial Metabolites: Towards enhancing Pharmacological Characterization, *Res. J. Biotech.*, **18(5)**, 31-44 (2023)

41. Ogolo N.A., Olafuyi O.A. and Onyekonwu M.O., Enhanced oil recovery using nanoparticles, SPE Saudi Arabia Section Technical Symposium and Exhibition, Al-Khobar, Saudi Arabia, April 8 (2012)

42. Pereira M.L. de O., Maia K.C.B., Silva W.C., Leite A.C., Francisco A.D. dos S., Vasconcelos T.L., Nascimento R.S.V. and Grasseschi D., Fe_3O_4 nanoparticles as surfactant carriers for enhanced oil recovery and scale prevention, *ACS Applied Nano Materials*, **3(6)**, 5762-5772 (2020)

43. Pérez-Gramatges A., Maia C.B., de Freitas F.A., Nascimento M.A.C. and Nascimento R.S.V., Silica particles as surfactant nanocarriers for enhanced oil recovery, In Springer Series in Materials Science, Computational Materials, Chemistry and Biochemistry: From Bold Initiatives to the Last Mile, Springer Series in Material Science, **284**, 503-519 (2021)

44. Punia P., Naagar M., Chalia S., Dhar R., Ravelo B., Thakur P. and Thakur A., Recent advances in synthesis, characterization and applications of nanoparticles for contaminated water treatment- A review, *Ceramics International*, **47(2)**, 1526-1550 (2021)

45. Rex R.W., Authigenic kaolinite and mica as evidence for phase equilibria at low temperatures, *Clays and Clay Minerals (National Conference on Clays and Clay Minerals)*, **13(1)**, 95-104 (1964)

46. RezaeiDoust A., Puntervold T., Strand S. and Austad T., Smart water as wettability modifier in carbonate and sandstone: A discussion of similarities/differences in the chemical mechanisms, *Energy & Fuels: An American Chemical Society Journal*, **23(9)**, 4479-4485 (2009)

47. Rezvani H., Riazi M., Tabaei M., Kazemzadeh Y. and Sharifi M., Experimental investigation of interfacial properties in the EOR mechanisms by the novel synthesized $\text{Fe}_3\text{O}_4@\text{Chitosan}$ nanocomposites, *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, **544**, 15-27 (2018)

48. Simonsen G., Strand M. and Øye G., Potential applications of magnetic nanoparticles within separation in the petroleum industry, *Journal of Petroleum Science & Engineering*, **165**, 488-495 (2018)

49. Souza I.D.L., Saez V. and Mansur C.R.E., Lipid nanoparticles containing coenzyme Q10 for topical applications: An overview of their characterization, *Colloids and Surfaces B: Biointerfaces*, **230**, 113491 (2023)

50. Tang G.Q. and Morrow N.R., Influence of brine composition and fines migration on crude oil/brine/rock interactions and oil recovery, *Journal of Petroleum Science & Engineering*, **24(2-4)**, 99-111 (1999)

51. Tang G.Q. and Morrow N.R., Oil Recovery by Waterflooding and Imbibition - Invading brine Cation Valency and Salinity, SCA-9911, <https://api.semanticscholar.org/CorpusID:208154674> (1999)

52. Tian Y., Wang L., Tang Y., Liu C., Ma C. and Wang T., Research and application of Nano polymer microspheres diversion technique of deep fluid, SPE International Oilfield Nanotechnology Conference and Exhibition, Noordwijk, The Netherlands, June 12 (2012)

53. Torsæter O., Application of nanoparticles for oil recovery, *Nanomaterials (Basel, Switzerland)*, **11(5)**, 1063 (2021)

54. Wakeman R., The influence of particle properties on filtration, *Separation and Purification Technology*, **58(2)**, 234-241 (2007)

55. Yakasai F., Jaafar M.Z., Bandyopadhyay S., Agi A. and Sidek M.A., Application of iron oxide nanoparticles in oil recovery – A critical review of the properties, formulation, recent advances and prospects, *Journal of Petroleum Science & Engineering*, **208**, 109438 (2022)

56. Yuan W., Liu X., Wei H., Liu J., Yang H., Hu S., Li Y. and Wang D., Research and application effect of polymeric microsphere in Wen-10 of Sinopec Zhongyuan Oil Field, Inner Mong, *Petrochem*, **12**, 122-126 (2010)

57. Zaid H.M., Ahmad Latiff N.R. and Yahya N., The effect of zinc oxide and aluminum oxide nanoparticles on interfacial tension and viscosity of nanofluids for enhanced oil recovery, *Advanced Materials Research*, **1024**, 56-59 (2014)

58. Zendehboudi S., Ahmadi M.A., Rajabzadeh A.R., Mahinpey N. and Chatzis I., Experimental study on adsorption of a new surfactant onto carbonate reservoir samples—application to EOR, *The Canadian Journal of Chemical Engineering*, **91(8)**, 1439-1449 (2013).

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