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Experimental Investigation of the Effect of using Nanoparticles for Improved Oil Recovery

Mahmood Amani*, Mohamed Idris, Nikolayevich Dela Rosa, Mohamed Al Balushi, Arnel Carvero, Rommel Yrac

Texas A&M University at Qatar

Corresponding Author:** ***Mahmood Amani, *Texas A&M University at Qatar*

Abstract: *Oil and gas companies are racing towards increasing production in their fields. As the well's first stage of production through natural flow is completed, much of the effort is focused towards improved oil recovery. This is the application of diversified techniques with the purpose of improving the recovery factor of hydrocarbons. As varied techniques are readily available, we employ a novel method towards enhance oil recovery by utilizing nanoparticle materials that can complement current techniques used in the field.*

The use of nanoparticle material homogenously mixed with surfactants alters the properties of hydrocarbons sweeping from pore throats of the reservoir. This mechanism greatly affects interfacial tension, wettability through the contact angle and the capillary pressure of hydrocarbons. Any rise in the recovery can obviously increase production rates, recover additional reserves and ultimately substantial economic gains. This research demonstrated that employing nanoparticles to complement EOR operation can be a promising method to be further studied and developed and later introduced to the oil industry.

Keywords: *Nanoparticles, EOR Methods, Improved Oil Recovery, Oil Production, Mature Oil Fields*

1. INTRODUCTION

The everlasting emphasis on enhancing the extent of extracted quantities of oil and gas stored inside rock formation pores stems from the oil and gas industry's desire to improve recovery from established hydrocarbon-rich reservoirs. The concept of utilizing nanoparticles in the petroleum industry is one that is technically designed to supplement the current methodologies of surfactant flooding being implemented in order to effectively sweep hydrocarbon volumes within that reservoir, those ones that are unattainable with conventional primary methods of oil recoveries. This unattainability is due to strong capillary forces trapping the hydrocarbons within the pores of the rock. These forces are also referred to as capillary pressure. This pressure decreases with the reduction in the interfacial tension at the contact between the oil and rock and increases in the oil-rock contact angle.

Naturally occurring elements such as Calcium, Potassium, Silicon and Aluminum are just a few that form the prospective nanoparticles. These elements compound with oxygen or chlorine to form solid nanoparticles that can be homogenously mixed with ethanol, brine or diesel. The resulting mixtures function as injected surfactants in Enhanced Oil Recovery applications; altering key parameters such as interfacial tension, wettability through the contact angle and ultimately influencing the capillary pressure.

The essential mechanism by which the nanoparticles aid in sweeping hydrocarbons is arguably best exemplified in the below schematic. These infinitesimally small molecules are dissolved in the surfactants comprising the continuous fluid phase to assemble and accumulate at the fringes of the oil-rock contact location, thus creating a wedging effect between the crude oil and the rock illustrated in **Fig1.** below. The disjoining pressure mechanism exerted on the compact wedge film composed of nanoparticles reduces the required capillary pressure so that otherwise unrecoverable oil quantities are swept from the pore throats of the reservoir.

Not only does the presence of nanoparticles in fluids pose an effect on the disjoining pressure, but also serves to affect a range of reservoir fluid properties, such as to change the oil viscosity and density of the present oil droplets (effective in crude oil configurations) in favor of increasing the amount of hydrocarbons flowing.

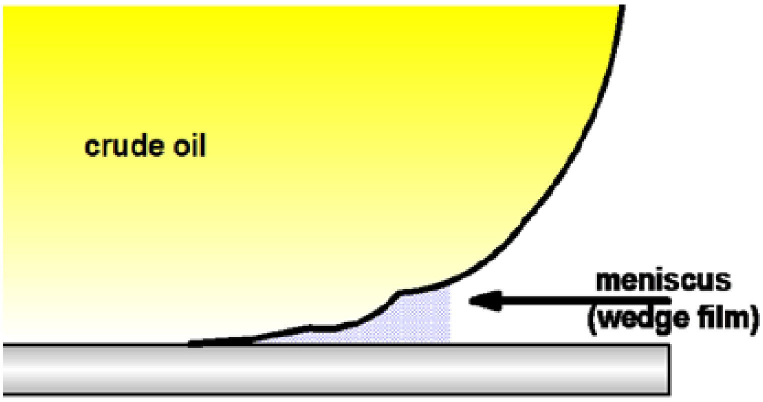

Figure1. *Wedging Effect of Nanoparticles on continuous phase (McElfresh, P.M. et al. 2012)*

While common practices of surfactant flooding have reported back progress with oil and water emulsions, the interventionist-driven call by El-Diasty and Aly (2015) for pursuit of nanotechnology is based on a distinction in the spatial property they possess, particularly their conveniently small size. Boasting a large specific surface area, those particles create wedge film of large area at the surface, which enhances the energy at which the fluids encompassing them operate [2]. Recent measures involving nanoparticle-fluid injection have not only reduced the capillary pressure required to acquire additional extracts of oil and gas but has proven monumental in reducing residual oil saturation to substantial lengths, boosting the process's high efficiency in enhancing oil recovery [3].

Through their reliance on the displacement efficiency expression which incorporates the initial oil saturation, Ogolo, Olafuyi and Onyekonwu (2012) replicated atmospheric states from previously conducted experiments so as to foster an identical working environment. Their experiments utilized loaded sand packs containing set quantities of distilled water, ethanol, low-salinity brine and diesel. They then soaked the sands in the nanoparticle-fluids for as long as 60 days before adding a 40% pore volume of oil in order to monitor the cleansing effect of the nanoparticle-fluids. Following an extensive research analysis of several nanoparticle-fluid combinations, they concluded that Aluminum Oxide (Al_2O_3) provided the best recovery rates particularly in diesel, with a whopping 30% recovered as a direct result of the harnessed nanoparticle-fluid mixture. Out of all surfactants, distilled water encountered issues with permeability and projected restrained outcomes of enhanced recovery, deemed to have the least effect on flushing out the oil investigated in this case under surface conditions [4].

Examining the compositions of oil-wet, water-wet, and intermediate-wet rock formations is essential to the study of the contact angle among other variables. McElfresh also noted how important it is to recall that an increase in salinity and temperature levels impact the nanoparticle colloids, destabilizing them and causing agglomeration [5].

This investigation comprised of two primary tests: contact angle measurement and core flooding analysis. The first phase utilized a purchased Rame-Hart goniometer, which allows for determination of wettability through the measurable contact angle of an oil droplet lowered into the surface of a rock. The higher the contact angle for the oil droplet on a rock surface captured with the goniometer, the poorer the oil wettability and adhesiveness of the mixture. The changes in the droplet's contact angle value before and after the application of nanoparticle-surfactant mixtures were recorded and examined. The abilities to decrease the oil wettability of the rocks determined which mixtures are eliminated from further analysis in the second phase. The second phase of the experiment will take the best performing nanoparticle-surfactant combinations from phase 1 and utilize them in a core flooding experiment that assesses these combinations' effects on oil permeability.

1.1. Objectives

The aim of this study is to examine the potential of nanoparticles in improving the efficiency of the enhanced oil recovery (EOR) method of surfactant flooding. The effects of nanoparticles in conjunction with commonly used surfactants in EOR on the wettability and oil permeability of rocks were to be measured and analyzed. The nanoparticles considered were Aluminum Oxide, Silica, and Copper Oxide. The surfactants used were distilled water, ethanol, or diesel. Each nanoparticle was mixed with each surfactant, forming a total of twelve distinct nanoparticle-surfactant mixtures (a complete list and full specifications of the mixture are shown in the **Procedure**). These mixtures bring up another objective of this study – to determine which of these combinations would best improve surfactant-flooding performance. Hence, it was set as a goal to determine and compare the oil wettability reducing abilities of each combination through contact angle measurements by a goniometer. In addition, it was important to measure and compare the effects of each combination on the oil permeability of rocks through core flooding tests.

1.2. Significance

Conventional primary productions were heavily devised in the early decades of soaring production, generating autonomous hydrocarbon quantities from free flowing, naturally driven wells in rich permeable reservoirs with limited complexity. In those times, there seemed to be no need at all for highly efficient production and enhanced oil recovery techniques. But that is seldom the case any longer. It is now more challenging to produce oil and gas from the ground as wells are getting deeper and the reservoirs from which oil and gas are produced become increasingly complicated. These led to rising demands to deviate constructed wells in order to establish larger contact with slightly less impermeable formations, drill deeper, and economically achieve a significantly greater yield of retrieved petroleum resources through more effective EOR methods. Its worth noting that even the slightest of percentage increases in the recovery factor can significantly boost production rates, recovered reserves and generate sizeable financial gains for the operating company that owns the field or is leasing it.

Therefore, it is no surprise that substantial sums and portions of budget have been allocated in recent times to research investigating the practical viability of novel methods of enhancing quantities of recovered oil, in an invigorated effort to amplify hydrocarbon extraction and recuperation, achieve larger profit sums, and enhance the commercial potential of discovered fields and reservoirs. As a result, the promising novel approach of supplementing nanoparticles to surfactant flooding for improved sweeping performance should not be looked over in research efforts.

2. PROCEDURE

As mentioned, the course of this investigation is divided into two phases – Phase 1: *Comparisons of Nanoparticle-Surfactant Mixtures' Effects on Oil Wettability through Contact Angle Measurements* and Phase 2: *Analysis of Nanoparticle-Surfactant Mixtures' Effects on Oil Permeability through Core Flooding Analysis*. The nanoparticles and nanoparticle-surfactant mixtures used in the experiments are shown in Table 1 and Table 2.

Table1. *The nanoparticles used in this research and their sizes.*

Nanoparticle	Particle Size (nm)
Aluminum Oxide	30-60
Copper Oxide	10-50
Silica	2

Table2. *The nanoparticle-surfactant mixtures used in this research and their compositions*

Nanoparticle + Surfactant Mixture No.	Nanoparticle	Surfactant	Weight of nanoparticles/weight of mixture (%)
1	Aluminum Oxide	Water	0.4
2		Brine (20 g salt/L)	0.4
3		Diesel	0.4
4		Ethanol	0.4
5	Copper Oxide	Water	0.4
6		Brine (20 g salt/L)	0.4
7		Diesel	0.4
8		Ethanol	0.4
9	Silica	Water	0.4
10		Brine (20 g salt/L)	0.4
11		Diesel	0.4
12		Ethanol	0.4

2.1. Phase 1

The first portion of the investigation attempts to examine the effects of the twelve nanoparticle-surfactant mixtures on oil wettability. Also, the three combinations that best decrease the oil wettability in Phase 1 will be further tested in Phase 2 for an analysis of their ability to improve oil permeability. The ability to reduce the oil wettability of a nanoparticle-surfactant mixture will be gauged through its observed effect on the contact angle of a placed droplet of crude oil on a rock surface. To facilitate such contact angle measurements, a Rame-Hart Goniometer, pictured in **Fig. 2**, along with its accompanying software was utilized.

Figure2. *Rame-Hart Goniometer (Rame-Hart Instrument Company)*

The rock samples used in this phase were obtained by cutting a whole carbonate core sample into circular slices 2 inches in diameter and 0.5 inches thick. In order to ensure adequate exposure to the nanoparticle-surfactant mixture one slice of core sample was submerged in 250 mL of each mixture for 24 hours. A picture of the twelve 250 mL of each the mixtures used for this phase of the experiments is shown **Fig. 3**, and twelve different slices of the core sample were wetted with the different mixtures. In order to mix the nanoparticle and surfactants a magnetic stirrer was used. Each mixture was stirred for a period of 2 minutes.

Figure3. *All 12 nanoparticle-surfactant mixtures created and used for phase 1*

After being submerged for 24 hours, the core samples were removed from their respective mixture, placed on the goniometer and the contact angle of each droplet of crude oil placed on the coated rock surface was measured. Before being submerged into the nanoparticle-surfactant mixtures, each core sample was preliminarily marked on the side that would be laying against the bottom surface of the beaker containing the nanoparticle-surfactant mixture in order to ensure that the other side - the side more exposed to the mixture - was the one on which the contact angle was measured. In addition, the contact angle of a droplet of oil dropped on a dry slice of a rock sample was recorded to establish a comparison with the contact angles of the rocks measured after they were submerged in the nanoparticle-surfactant mixtures. Since oil is being used to measure contact angle, the ideal effect of the nanoparticle-surfactant exposure should be an increase in the contact angle. This result would indicate the nanoparticle-surfactant mixture application results in a less oil-wet surface, which is favorable in oil extraction. An example of the computer program interface of the Rame-Hart software being used to measure the contact angle can be found in **Fig. 4**.

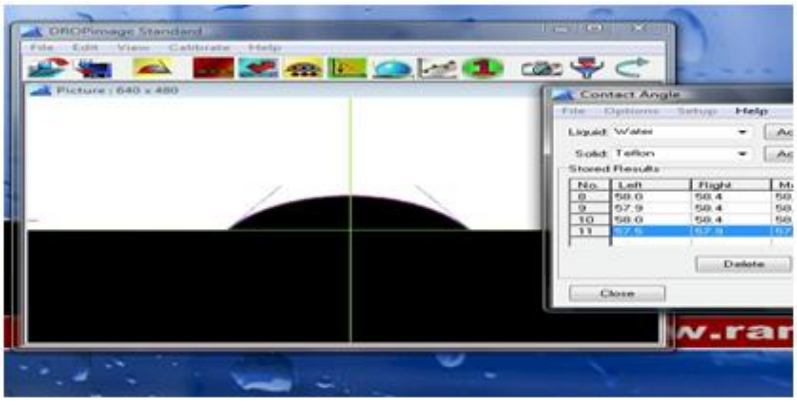

Figure4. *Interface of the Rame-Hart software used to measure the contact angle*

2.2. Phase 2

Once the best three nanoparticle-surfactant combinations were identified from phase 1, these mixtures were then studied in phase 2 for their ability to enhance oil permeability. This was executed by examining the oil permeability before and after nanoparticle-surfactant application. Time constraints as a result of delay in delivery of the much-needed nanoparticles resulted to testing only the best three rather than the other more mixtures. In order to measure oil permeability, the core is flooded with oil using the core flooding apparatus shown in **Figure 5**.

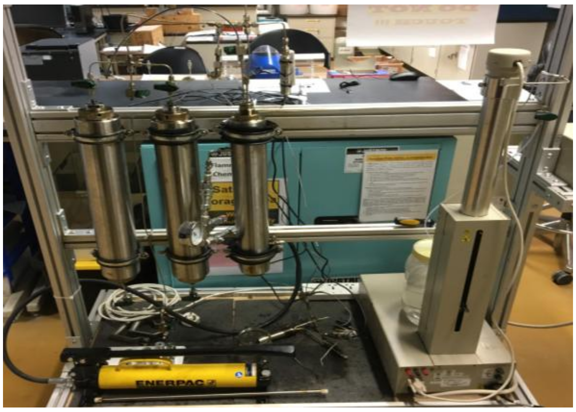

Figure5. *The core flooding apparatus used for Phase2 oil flooding of cores to obtain oil permeability values*

During the oil flooding of a core sample, the inlet and outlet pressures were monitored and recorded over time at oil flow rates of 1, 2, 3, and 4 mL/min. For a specific constant flow rate, the inlet pressure increases steadily and then stabilizes. The outlet pressure remains the same, as there is no build-up in pressure because the fluids are diverted out from the outlet and into a fluid sink. From the stabilized pressure drop (inlet pressure – outlet pressure) values and their corresponding flow rates, and the diameter (cm) and length (cm) of the core, a plot of 4 points of the flow rate divided by flow-cross-sectional core area (Q(mL/s = cm^3/s) /A(cm^2)) vs. pressure drop (inlet pressure – outlet pressure) divided by the core length (dP(atm)/L(cm)) is obtained.

A line was then fit through the 4 Q/A-dP/L points using the MS Excel software and the slope for this line was recorded. The slope value here indicates the oil permeability (Darcy) per oil viscosity (cp) for a core sample with connate water (**explanation for this connate water below)**. This is how the oil permeability of the cores before and after nanoparticle-surfactant application is obtained. Given the fact that the same oil was utilized for all Phase 2 core floorings, oil permeability readings obtained this way can be compared reliably with one another for the purpose of examining and comparing the effects of three different nanoparticle-surfactant mixtures on oil permeability.

As with Phase 1, carbonate rock core samples were used. These cores samples were first flooded with brine since reservoir rocks have connate water. The core samples after brine flooding are displayed in **Fig. 6**.

Figure6. *Phase 2 core samples after flooding with brine for imitating reservoir rocks that usually have connate water.*

The cores were then flooded with oil to ascertain the measures of their initial oil permeability values. Next, the three nanoparticle-surfactant mixtures were applied to the core samples. The application of a nanoparticle-surfactant mixture was carried out by submerging the core in such mixture for a day and at a confined pressure of 100 psi. Finally, oil is flooded again into the cores and the new oil permeability after the nanoparticle-surfactant applications can be found and compared to the initial oil permeability. The rock core samples after nanoparticle-surfactant applications and oil re-flooding are shown in **Figure 7**.

Figure7. *Phase 2 core samples after nanoparticle-surfactant application and oil re-flooding.*

3. Results And Discussion

3.1. Phase1 Results

For the first phase of the experiment, it was necessary to re-stir the nanoparticle-surfactant mixtures at 80 rpm (rotational speed for the magnetic stirrer), 40^{o} C (temperature of the stirrer plate), and for two minutes before submerging the rocks in them. The reason for this was that the rock samples were only made available a day after the mixtures were prepared, so the emulsion of the mixtures were destabilized and needed to be stabilized again by re-stirring them. Then, after taking the initial contact angle measurement of a dry rock with the Rame-Hart goniometer, the rock samples were immersed in the mixtures. Afterwards, their respective contact angles were measured. The obtained measurements and contact angle percent increases due to the mixtures are presented in **Table 3**.

Table3. *Experimental results for Phase 1*

Rock Sample	Immersed in solution of	Initial Contact Angle (degrees)	Contact Angle after immersion (degrees)	Percent Increase (%)
1	Aluminum Oxide and distilled water	5	10.75	115
2	Copper Oxide and distilled water	5	29.6	492
3	Silica and distilled water	5	10	100
4	Aluminum Oxide and brine	5	10.5	110
5	Copper Oxide and brine	5	5	0
6	Silica and brine	5	11.1	122
7	Aluminum Oxide and Ethanol	5	12.5	150
8	Copper Oxide and Ethanol	5	28.4	468

9	Silica and Ethanol	5	81	1520
10	Aluminum Oxide and Diesel	5	15.6	212
11	Copper Oxide and Diesel	5	15.3	206
12	Silica and Diesel	5	5	0

As seen from the table above, immersing the rock sample in a solution of Silica and Ethanol resulted in a contact angle increase of 1520%, the highest increase. Hence, so far, the Silica and Ethanol mixture shows the most promise in reducing oil wettability. Conversely, rock samples immersed in Copper Oxide-brine and Silica-Diesel expressed almost no contact angle increase at all. The rest of the nanoparticle + surfactant mixtures at least doubled the contact angles, which indicates satisfactory effectiveness in reducing oil wettability.

Aluminum Oxide was most effective in increasing contact angle with Diesel, validating the findings in “Enhanced Oil Recovery Using Nanoparticles” by Ogalo et. al., 2012, regarding the effectiveness of Aluminum Oxide in reducing oil wettability. As this experiment illustrates, the Aluminum Oxide nanoparticle has increased the contact angle tenfold, and with another finding confirming it as an effective agent in improving oil recovery, such nanoparticle may have promising applications in EOR. Consequently, it merits being involved in future investigations on using nanoparticles for EOR surfactant flooding.

Keeping in mind that oil was used in the contact angle measurements, a contact angle less than 90^o indicates oil-wet condition and that the lower the contact angle, the more oil-wet. Thus all rock samples remained oil-wet, although their oil wettability levels decreased considerably after submersion in the mixtures except for those immersed in Copper Oxide- brine and Silica-Diesel.

Finally, it was observed earlier that the destabilization of the nanoparticle-surfactant mixtures as the rocks were immersed in them for a day during Phase 1 would undoubtedly impact the effectiveness of such mixtures in altering contact angles and oil wettabilities, thus causing errors in the contact angle measurements.

3.2. Phase2 Results

From the results of Phase 1, it was decided that the Silica-Ethanol, Copper Oxide-Distilled Water, and Copper Oxide-Ethanol are the best three out of the twelve nanoparticle-surfactant mixtures in reducing oil wettability. Consequently, those mixtures were selected for further examination in Phase 2 to monitor their effects on oil permeability. Two trials were made for each three mixtures. The results for both trials are illustrated in **Table 4** and **Table 5**.

Table4. *Trial 1 experimental result for Phase 2*

Nanoparticle + Surfactant (N+S) Mixture	Oil Permeability (mD/oil viscosity (cp))		% Increase
	Before N+S application	After N+S application	
Silica + Ethanol	5.4	6.1	13.0
Copper Oxide + Distilled Water	6.6	6.2	-6.1
Copper Oxide + Ethanol	6.7	5.1	-23.9

Table5. *Trial 2 experimental results for Phase 2*

Nanoparticle + Surfactant (N+S) Mixture	Oil Permeability (mD/oil viscosity (cp))		% Increase
	Before N+S application (mD/oil viscosity (cp))	After N+S application (mD/oil viscosity (cp))	
Silica + Ethanol	3.9	3.6	-7.7
Copper Oxide + Distilled Water	9.4	8.5	-9.6
Copper Oxide + Ethanol	5.9	5.2	-11.9

As seen from **Table 4** and **Table 5**, the best nanoparticle-surfactant mixtures did not necessarily improve the oil permeability of the carbonate core samples. With the exception of the first trial for Silica-Ethanol improving the oil permeability by 13%, all others resulted in a permeability reduction. There is however some consistency with the findings in Phase 1 and Phase 2. In each trial for Phase 2, the decrease in oil permeability due to a mixture decreases as the mixture’s ability to reduce oil wettability (as determined in Phase 1) increases. In other words, in each trial, the best mixture – Silica-Ethanol – from Phase 1 caused the least decrease in oil permeability for Phase 2, the 2nd best mixture – Copper Oxide-Distilled Water – from Phase 1 caused the 2nd least decrease in oil permeability for Phase 2, and so on.

Such decreases in oil permeability can be explained by the fact that the nanoparticle-surfactant mixtures were unstable. This mixture instability caused nanoparticles to aggregate. Thus, instead of all the nanoparticles entering the pores of the rock separately as tiny particles, some entered the pores as larger aggregates and thereby effectively plugging the pores. This led to a notable decrease in oil permeability. This is turn meant that the plugging by larger aggregates overcame any increase in oil permeability caused by the mixtures reducing oil wettability and the smaller nanoparticles forming the aforementioned wedge films to decrease capillary entry pressures. On the other hand, in Trial 1 for this phase’s use of Silica-Ethanol and the only trial where oil permeability improved, the opposite outcome occurred. Due to the extremely small size of the Silica nanoparticles (about 2 nm), it was possible for the oil wettability and capillary pressure reducing abilities of the silica-ethanol mixture to overcome the pore plugging by Silica nanoparticle aggregates. Furthermore, these aggregates would have been much smaller than the Copper Oxide nanoparticle aggregates, which affected permeability decreases for Phase 2 trials of Copper Oxide containing mixtures.

4. Conclusion And Outlook

During Phase 1, an area of improvement for the procedure was made evident. The issue involves the instability of the nanoparticle-surfactant mixture. It was found that even after a couple of hours the nanoparticle solids would aggregate and accumulate at the bottom of the mixture, leaving the mixture not compositionally homogenous. After consulting a chemist and an expert of mixtures, two suggestions were made. The first was to use an ultrasonic machine to more effectively stir the mixture. Ultrasonication is apparently the more ideal way of thoroughly dispersing the nanoparticle solids within the mixture than mixing with a magnetic stirrer. This is because ultrasonication can cause more agitation than magnetic stirrers and thereby more effective mixing. Secondly, it was suggested that an acid or a base be added to the mixture in order to move the overall pH of the solution below or above the isoelectric point of the nanoparticles in the mixture, thus charging the particles and preventing them from aggregating thus stabilizing the mixture. Mixing by ultrasonication along with having a pH above or below isoelectric points of the nanoparticles will ensure the mixtures remain stable for the entire time span of core sample submersion.

Another apparent issue in the methodology of phase 1 is the porosity of the core sample used. When conducting contact angle measurements, the droplets of oil seemed to be losing volume to the pores on the rock sample, a factor which undoubtedly added irregularities in the obtained data. This will not change significantly the validity of the current Phase 1 experimental results in any way as the focus is on the surface and surface properties of the rock sample, the matrix structure and pores within it is irrelevant in this case. Hence, for the purpose of less discrepancy and_more accurate contact angle measurements, it is crucial to use low porosity rock samples.

Results of the Phase 1 experiment revealed the Silica-Ethanol mixture to be most effective in reducing oil wettability, while the Copper Oxide-Brine and Silica-Diesel mixtures had almost no effect at all. While adding Silica to Ethanol surfactant flooding seems a promising prospect, Copper Oxide and Silica additions to Brine surfactant flooding and by the same token Diesel surfactant flooding may be both a waste of time and nanoparticles’ quantities.

It was also pointed out that it may be worth involving Aluminum Oxide in future studies for application of nanoparticles in EOR methods since another result, pertained in (Ogolo et. al., 2012) provides an endorsement to its ability in reducing oil wettability and improving oil recovery. Along with the Silica-Ethanol mixture, Copper Oxide-Distilled Water and Copper Oxide-Ethanol mixtures were found to be the three best performing mixtures and further analyzed in Phase 2.

As is often the case in petroleum engineering and engineering in general, proposed methods to improve efficiency of operations may not always produce the desired outcome. In this research, almost all the results of Phase 2 indicated that the three best mixtures decreased oil permeability. These decreases can be attributed to the fact that the mixtures were unstable by 24 hours after mixing. The nanoparticles were forming large aggregates that plugged the pores of the rock and reduced oil permeability. Hence, as mentioned in Phase 1 observations and further reinforced through results in Phase 2, it is essential to ensure that when nanoparticles are applied to surfactant flooding, the nanoparticle-surfactant mixture is made stable. The mixture can be stabilized by more sufficient mixing by ultrasonication and through the addition of acids or bases. The mixture stability must also be guaranteed for research purposes involving nanoparticle-surfactant mixtures.

Despite the unexpected results of Phase 2, both Phase 1 and 2 consistently indicate Silica-Ethanol to be a better mixture than Copper Oxide-Distilled Water, with the latter more effective than Copper Oxide-Ethanol in terms of their potential to enhance the effectiveness of current surfactant flooding approaches.

In one trial of Phase 2, the Silica-Ethanol increased oil permeability by 13%. This shows potential for Silica-Ethanol surfactant flooding to enhance oil recoveries. The Silica-Ethanol combination should then be considered in future studies of nanoparticle applications in surfactant flooding and possibly in actual field EOR flooding applications.

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AUTHORS' BIOGRAPHY

Dr. Mahmood Amani, He is an Associate Professor of the Petroleum Engineering at Texas A&M University at Qatar (TAMUQ). Dr. Amani holds a BS degree in Mechanical Engineering, MS degree in Natural Gas Engineering and a PhD in Petroleum Engineering.

Mohamed Idris, Has received a BS degree in Petroleum Engineering from Texas A&M University at Qatar and currently is working towards a post-graduate degree at the University of Calgary in Canada

Nikolayevich Dela Rosa, He is a senior Petroleum Engineering student at Texas A&M University at Qatar.

Mohamed Al Balushi, Earned a BS degree in Petroleum Engineering fromTexas A&M University at Qatar and is currently working towards a post-graduate degree at French Institute of Petroleum (IFP) in France.

Arnel V. Carvero, Completed a degree of Bachelor of Science in Chemistry in the Philippines, worked for over 10 years in the industry before joining Petroleum Engineering at Texas A&M University at Qatar. He is a Research Associate at Texas A&M University at Qatar.

Rommel Duave Yrac, Earned his BS degree in Chemical Engineering in the Philippines, spent over 20 years in the Oil and Gas specializing in drilling operationsprior to joining academia at Texas A&M University at Qatar. He is a Technical Laboratory Coordinator at Texas A&M University at Qatar.

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