Research Article

Applicability of Nanoparticle Flooding Process in a Carbonate Rock of Kurdistan Region: Experimental Investigation of Interfacial Tension and Wettability

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Abstract

Enhanced oil recovery (EOR) has long proven to be a good method to mobilize the residual oil that is by passed and capillary trapped by secondary recovery methods. Chemical EOR methods enhance the microscopic and macroscopic efficiency, and ultimately the overall oil recovery is improved. However, the adsorption rate of the surfactant, low resistance to high temperature and salinity are some of the factors that would turn chemical flooding impractical and uneconomic in many cases. Lately, the application of nanotechnology in enhanced oil recovery has showcased some good and prolific results in terms of incremental oil recovery. In this study, the applicability of Nanoparticle flooding in carbonate rocks of Pilaspi formation was probed through a series of tests such as thin section analysis, x-ray diffraction, x-ray fluorescence, interfacial tension and contact angle measurements. The results showed that the composition of the carbonate rocks is predominantly calcite ($CaCO_3$) with minor traces of quartz and dolomite. From the interfacial tension (IFT) measurements, it was figured out that the silica and alumina Nanofluids lowered the IFT by 27% and 42% with the light oil, and 43% and 49% with the heavy oil, respectively. The contact angle measurements revealed that the Alumina Nano-fluid at 0.25 wt. % reduced the contact angle on the surface of the light and heavy oil aged thin sections from 169° and 115° to nearly 119° and 78°. On the other hand, the silica nanoparticle at 0.25 wt. % reduced the contact angles on both thin section types to around 129° and 80°, respectively.

Keywords: Interfacial Tension, Wettability, Carbonate Reservoirs, Nano-Fluid Flooding, Enhanced Oil Recovery.

1. Introduction

Petroleum Industry has always been highly expected to meet energy demand as it has predominantly been one of the most common sources of energy for many decades. Primary and secondary recovery methods of petroleum industry have shown to be tremendously limited in producing and draining the reservoirs efficiently. As a result, a huge portion of the oil would usually be capillary trapped or untouched. As the demand for oil increases, the need to develop new recovery techniques and strategies also rises. Enhanced oil recovery (EOR) has proven to be an effective technology that can be properly used to ramp up production. EOR is usually defined as any method that aims to recover the residual and remaining oil that has been trapped and left behind by primary and secondary recovery methods (Donaldson et al, 1985; Mashat et al, 2018). The restrictions imposed on most of chemical flooding projects necessitate the urge to develop and investigate the applicability of other techniques. The implementation of nanotechnology in petroleum industry has been

investigated for nearly over a decade, and it has shown very good and promising results (El-Diasty and Ali, 2015; Nourafkan et al, 2018). Silica Nanoparticles (SiO_2 NPs) are the most prevalent nanomaterials utilized in EOR applications. Moreover, they are less costly and can be easily obtained. Nanoparticles (NP) as nanomaterials have a 3D dimension with a size ranging from 1-100 nm possess higher ratio of surface area to volume and therefore have superior thermal, chemical, physical and electrical properties than their bulk counterparts. The most important characteristics of nanoparticles for EOR projects are revolving around large surface to volume ratio (because of their small size) along with charge confinement, chemically altered surfaces, and modification of the structure of the material. Additionally, nanoparticles improve the surfactant's solution used in EOR applications, lower IFT, change the fluid rheology (mainly when used with polymer), and alter wettability (Marwan and Nageh, 2019). This project aims to experimentally investigate the performance of nanoparticle flooding in carbonate reservoirs of Pilaspi Formation in Kurdistan Region. The two mechanisms of wettability alteration and IFT reduction are going to be investigated to understand the effectiveness of nanoparticles (alumina and silica) as EOR agents.

1.1 Study Area

The study area of this work is around Pirmam that is in Northeast of Erbil. The latitude and longitude of the area are 36°21'39.35" N and 44°11'12.30" E respectively. The rock samples were collected from this area, and the location of the collection can be seen through a satellite image in Figure 1. This project aims to experimentally investigate the performance of nanoparticle flooding in carbonate reservoirs of Pilaspi Formation in Kurdistan Region. The two mechanisms of wettability alteration and IFT reduction are going to be investigated to understand the effectiveness of nanoparticles (alumina and silica) as EOR agents.

Figure 1. Satellite Image of Pirmam Area (ESSRI, 2013).

1.2 Literature Review

Various nanoparticles have already been utilized as EOR agents to understand how efficient they can be in modifying wettability towards water-wet state and reducing interfacial tension (IFT). Onyekonwu and Ogolo (2010) targeted the ability of three polysilicon Nanoparticles (PSNP) to enhance oil recovery in water wet formations. The NPs used were hydrophobic and lipophilic PSNP (HLPN), lipophobic and hydrophilic PSNP (LHPN), and neutrally wet PSNP (NWPN) while the dispersing phases used were brine and ethanol. The results achieved showed that HLPN and NWPN dispersed inn ethanol can be considered as good EOR agents in water wet formations since they improved the recovery.

Moreover, Ogolo et al (2012) selected nine nanoparticles (oxides of Aluminum, Zinc, Tin, Iron, magnesium, Nickel, Zirconium, Silane treated Silicon Oxide and Hydrophobic Silicon Oxide) with three different dispersing fluids (distilled water, brine, and ethanol) and investigated their impact on oil recovery. They concluded that the Aluminum Oxide and Silicon Oxides resulted in a very good increase in oil recovery compared to the usage of the dispersing fluids alone. All the nanoparticles gained a rise in oil recovery once dispersed in distilled water except hydrophobic silicon oxide. In addition, Roustaei et al (2012) compared between the effectiveness of a modified silica nanoparticle in improving oil recovery from intermediate and light oil reservoirs in Iran. They used wettability alteration and interfacial tension (IFT) reduction as the main mechanism to mobilize the additional residual oil. It was observed that the nanoparticles altered the wettability and reduce the IFT to a good extent. Also, Bayat et al (2014) practiced and explained the impacts of three

various metal oxides nanoparticles named aluminum oxide, silicon oxide and titanium oxide on intermediate wet limestone samples for EOR purposes at different temperatures of 26˚, 40˚, 50˚ and 60° C. Their results showed that the nanoparticles did a good job in lowering IFT and changing wettability towards water wet at most of the temperature ranges. In this paper, a higher temperature value (80◦C) is used to grasp whether the nanoparticles (Alumina and Silica) can impact the IFT and wettability intensively.

2 Materials

In this section, the main materials needed for the research are explained in details. The type of rock, core plugs made from the rocks, the thin sections made from the core plugs, the synthesis of the brine , and the properties of the oil samples used are all vital parts of the meaterials section.

2.1 Rock Sample Collection and Core Plug Preparation

Rock samples from the outcrop of Pilaspi Formation (Fgure 2a and 2b) were collected. Pilaspi formation is one of the carbonate reservoir formations in Kurdistan Region, and all the carbonate rocks used within this paper are from this formation. Then, the samples were taken to the laboratory to prepare core plugs (Figure 2d) that will later be used in the experimentations. Core plug machine (Drill Press) as seen in (Figure 2c) was used to make the core plugs. Later, the plugs were put in an oven for nearly 12 hours at a temperature of 100 C to be dried off.

Figure 2a. The Pilaspi Formation.

Figure 2b. Closer View of the Pilaspi Formation.

Figure 2c. Drill Press (core plugging machine).

Figure 2d. Core Plugs.

Figure 2. a) The Pilaspi Formation, b) Closer View of the Pilaspi Formation, c) Drill Press (core plugging machine), and d) Core Plugs.

2.2 Thin Section Preparation, XRF and XRD

Thin sections (Figure 3a) were made from the core plugs using thin section device. Once the thin sections are made, they are properly trimmed on both sides and polished so that the analysis under microscope will be handy and easy to understand. The thin section process was ended by putting the thin sections into an oven (Figure 3b) for nearly 24 hours under a temperature of 80° C. Additionally, aside from the microscopic thin section analysis, x-ray diffraction (XRD) and x-ray fluorescence (XRF) tests were also conducted so that the composition of the rock would be accurately determined. The XRF test was conducted using Spectro XRF with XEPOS model.

Figure 3a. Thin Section.

Figure 3b. Thin Sections Inside the Oven.

Figure 3. a) Thin Section, and b) Thin Sections Inside the Oven.

2.3 Brine Synthesis and Oil Samples

The reservoir brine used throughout this study was basically synthesized in the laboratory. Tables 1 and 2 elaborate on the composition of the reservoir brine for the two oil samples used. The concentrations of the salts as indicated in Table 1 and 2 were first found, and all the comprising salts were later added to 1 liter of deionized water. Finally, the jar containing the salts and 1 liter of deionized water was placed on a homogenizer with an RPM of 1000 for 24 hours, and the reservoir brine was obtained. The total dissolved solids (TDS) of the brine of the light oil reservoir were nearly 34461.6 ppm while the one of the heavy oil reservoir was approximately 16178.1 ppm. Table 3 shows some properties of the oil samples. The density of the oil & water and the oil viscosity were measured by pycnometer and MCR300 respectively.

Table 1. Ions and Salts Concentrations of Light Oil Reservoir.

Ion	Concentration (mg)	Concentration (mole)	Salt	Concentration (gr)
Cl	16200	0.456942995		
SO_4	1141	0.011877993	Na_2SO_4	1.704
S	2750	0.085763293	Na_2S	6.712
HCO_3	1600	0.026222286	$NaHCO_3$	2.184
Ba	67	0.000487887	$BaCl_2$	0.104
Na	10789	0.469295079	NaCl	14.481
K	1610	0.041178261	KCl	3.057
Cu	171	0.002690964	$CuSO_4$	0.479
Mg	380	0.015634643	$MgCl_2$	1.523
Ca	1520	0.037926044	$CaCl_2$	4.217

Table 2. Ions and Salts Concentrations of Heavy Oil Reservoir.

Ion	Concentration (mg)	Concentration (mole)	Salt	Concentration (gr)

Cl	9000	0.254		
SO_4	1266	0.013	Na_2SO_4	1.847
S	489	0.015	Na_2S	1.171
HCO_3	950	0.016	$NaHCO_3$	1.344
Na	4468	0.194	NaCl	7.150
K	358	0.009	KCl	0.671
Mg	160	0.007	$MgCl_2$	0.666
Ca	1192	0.030	$CaCl_2$	3.329

Table 3. Oil Samples' Properties.

Properties (Unit)	Light Oil	Heavy Oil
API°	36.6	14.3
Density (Ambient) (gm/cc)	0.745	0.862
Density (80C) (gm/cc)	0.8	0.911
Viscosity (cp)	9.5	150

2.4 Thin Section Aging

The thin sections made earlier need to be restored to an oil wet state. Once the reservoir brine was synthesized and prepared, it would be poured into aging cells and the thin sections are put & immersed inside and sealed tightly. Initially, the thin sections-filled containers are placed inside the oven for 5 days at a temperature of 80° C until the thin sections are aged with water. Then, the water aged thin sections are placed in another jar filled with the oil samples (heavy and light separately) for nearly 20 days inside the oven at a temperature of 80° C.

3 Experimental Work

The main experimental tests conducted throughout this work are: Interfacial Tension (IFT) measurement and contact angle measurement.

3.1 Interfacial Tension Measurement

The IFTs were measured using high pressure high temperature (HP-HT) pendant drop apparatus- model (VIT-ES20) (Figure 4). Aluminium oxide and silicon dioxide nanoparticles were used throughout this paper. Table 4 shows the two nanoparticles along with some of their properties. The nanoparticles were provided by the same German Company that had also provided the surfactants. The nanoparticle dispersions were made by ultrasound wave maker apparatus (Figure 5). The nanoparticle was placed in a small jar as seen, and 100 cc of formation brine (of both oil reservoirs individually) was added to the jar. The mixture was then placed on the device, and the probe completely went inside the jar, so that enough energy was transferred to the solution. The process was finished once the solution was totally homogenous (no insoluble particles existing). For the IFT measurements, different concentrations of both nanoparticles at 0.1, 0.25, 0.5 and 1 wt. % were added to both synthesized brines, and the IFTs were measured with both heavy and light oil samples at 80° C. The IFT of each Nano-fluid with both oil samples was measured.

Table 4. The Properties of Alumina and Silica Nanoparticles.

Properties	Alumina Nanoparticle	Silica Nanoparticle
Weight	25 g	25 g
Purity	99%	99.9%
APS	20 mm	30 mm
SSA	<200 m^2/g	>150 m^2/g
Appearance	White powder	White powder

Figure 4. HP-HT Pendant Drop Apparatus.

Figure 5. Ultrasound Wave Maker.

3.2 Contact Angle Measurement

To show the ability of the solutions to alter the wettability of the carbonate rock thin sections, contact angle method was used. The high pressure-high temperature (HPHT) pendant drop apparatus that was used for IFT measurement was also used to measure the contact angle between the oil wet thin sections and a drop of the solution.

The contact angle between each Nano-fluid and the oil wet thin sections (aged with heavy and light oil) were made at nanoparticle concentrations of 0.1 and 0.25 wt. %. The contact angle measurement at this stage lasted 480 hours at a temperature of 80° C.

4 Results and Discussion

The main results and discussion of this work are broken down into: Thin section, XRF, and XRD results, the wettability alteration by nano-fluids, and IFT reduction by nano-fluids.

4.1 Thin Section, XRF and XRD Results

According to the microscopic analysis of the thin sections (Figure 6a), the studied samples were concluded to be microcrystalline limestone. This rock is part of carbonate rocks' group, and sparry calcite composes the main constituent of the samples. The crystal size ranges between 10 to 270 microns however the crystal size is predominantly revolving around 50 microns. Moreover, the analysis showed that the sample is of high purity, and free of fossils and detrimental minerals. In addition, very few traces of dolomite can be hardly seen within the specimen.

Additionally, x-ray diffraction (XRD) and x-ray fluorescence (XRF) tests were also conducted so that the composition of the rock samples is accurately determined. As it can be seen from the XRF results (table 5), the rock composition is

mainly calcite by nearly 49.44% followed by very few traces of quartz (SiO_3) and a low 4.23% of $Mg(CO_3)_2$. It is important to understand that a big portion of the samples was lost on ignition (L.O.I) since it was at a temperature of nearly 750 °C for an hour. Lastly, the composition of the samples was further verified to be mainly calcite through XRD (Figure 6b). It was indicated that the samples are highly composed of calcite by nearly 99%, and the remaining 1% was made of quartz. The calcite section has shown the highest peak in the result. It can be concluded from all three tests that the composition of the core plugs is mainly calcite along with small traces of quartz and dolomite.

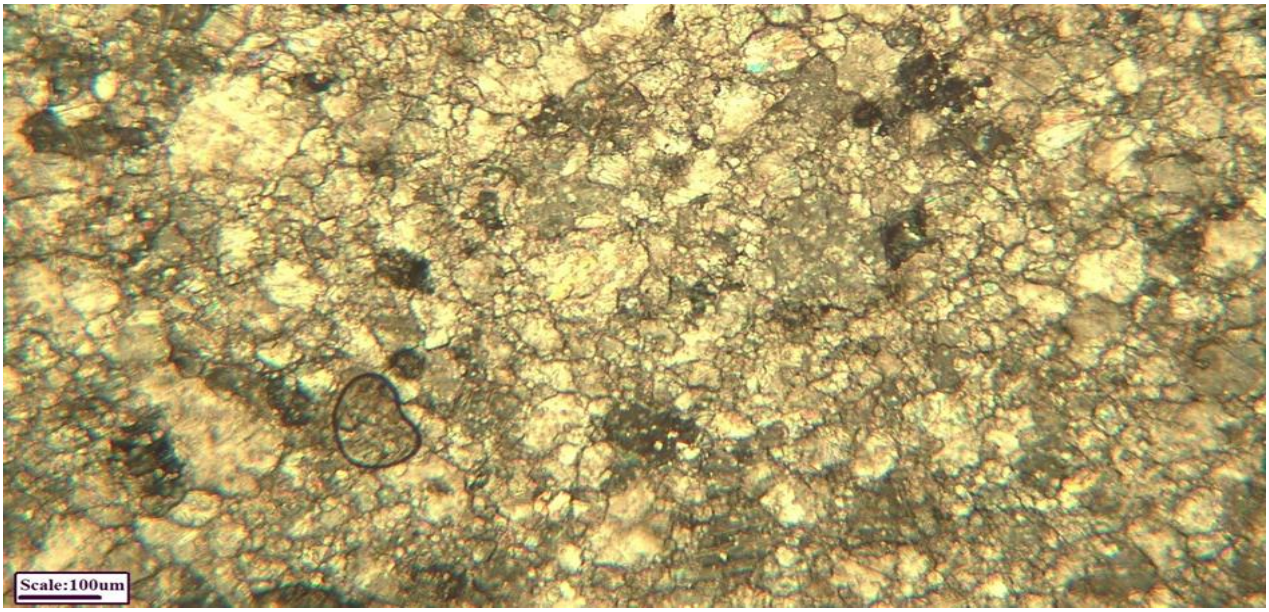

Figure 6a. Microscopic Image of Thin Sections (Suramairy et al, 2021).

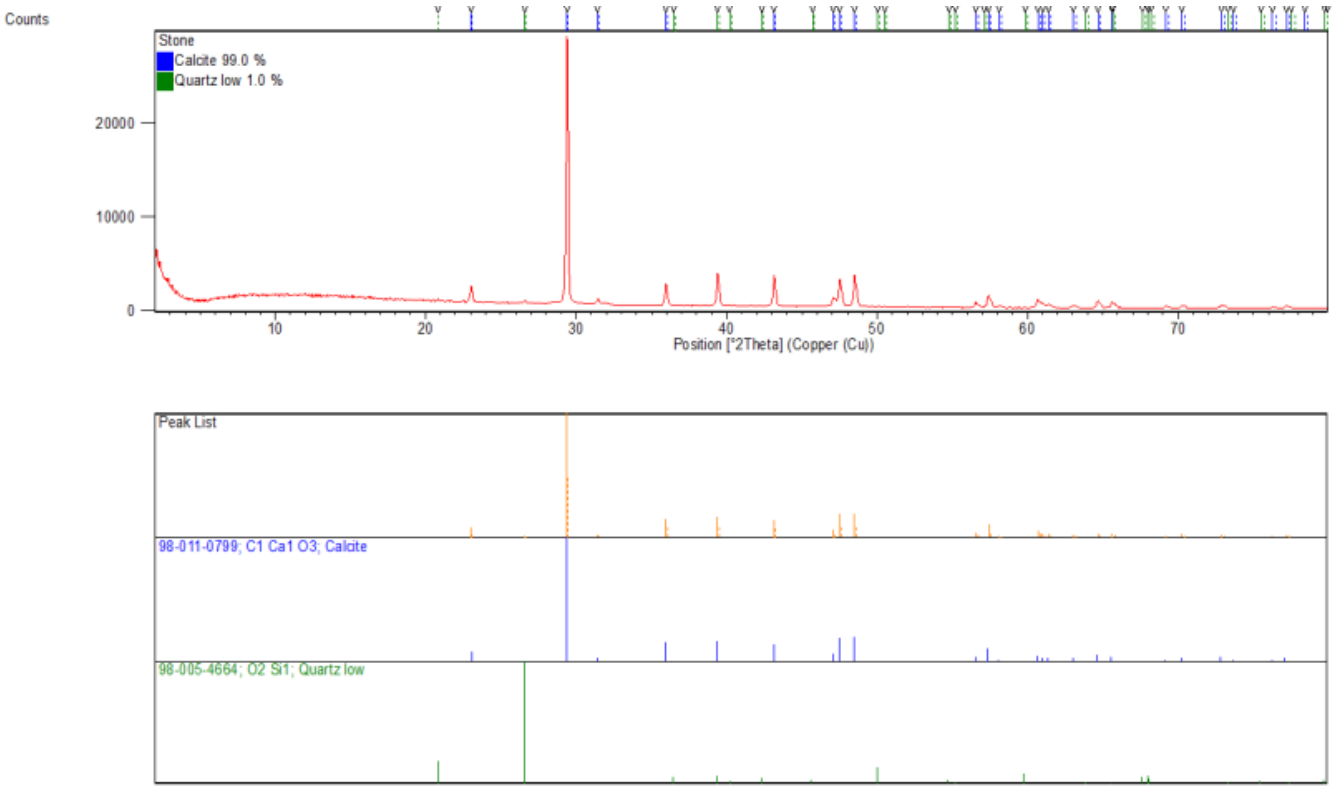

Figure 6b. XRD Results (Suramairy et al, 2021).

Figure 6. a) Microscopic Image of Thin Sections, and b) XRD Results.

Table 5. XRF Results.

Component	%
(Calcite) $CaCO_3$	49.44
(Quartz) SiO_2	1.16
(Dolomite) $Mg(CO_3)_2$	4.23
L.O.I	45.17

4.2 The Wettability Alteration by Nano-Fluid

The contact angles of the thin sections after being aged by light and heavy oil samples for 480 hours were measured to be nearly 169° and 115° respectively, and these show that the thin sections are in an oil-wetting state. To alter the wettability towards water-wet, the thin sections were treated by nanoparticles (alumina and silica NP) at a temperature of 80° C.

First, upon the treatment of the light oil aged thin sections with formation water, the contact angle for different time periods and up to 240 hours was measured to be nearly 129° (Figure 7). The treatment only took 240 hours because the contact angle values stabilized at that period. This shows that the synthesized water is not effective in altering the wettability of the thin sections from oil-wet to water wet. Moreover, the contact angle was reduced from nearly 115° to 90° upon treating the heavy oil aged thin sections with formation water.

To investigate the impact of the Nano-fluids on the thin sections' wettability, two concentrations of both SNP and alumina NP at 0.1 and 0.25 wt. % were used. These two concentrations were selected based on the IFT results of the Nano-fluids. The treated contact angles on the surface of both thin section types were reduced as the concentration increased. Alumina Nano-fluid at 0.25 wt. % reduced the contact angle on the surface of the light and heavy oil aged thin sections from 169° and 115° to nearly 119 ° and 78° respectively (Figure 8a, 8b, 8c, 8d). On the other hand, the silica NP at 0.25 wt. % reduced the contact angles on both thin section types to around 129° and 80° respectively (Figure 9a, 9b, 9c , and 9d). As it can be viewed from Tables (6 and 7), both Nano-fluids are able to reduce the contact angle, but the alteration is not very sharp, and the reason can be attributed to the effect of ionic strength that would make the alteration process slow. The mechanism of the wettability alteration by the Nano-fluids is strongly related to the adsorption of the nanoparticles on the surface of the thin sections. This would subsequently modify the free surface energy and alters the wettability towards more water-wet (Roustaei et al, 2012).

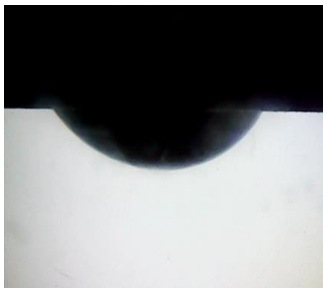

Figure 7. FW 240 hr (129°).

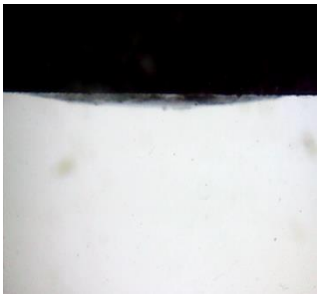

Figure 8a. Alumina NP 48 hr (166°).
Light Oil

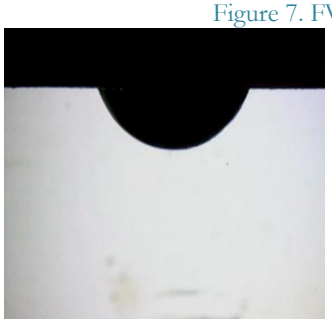

Figure 8b. Alumina NP 480 hr (119°).
Light Oil

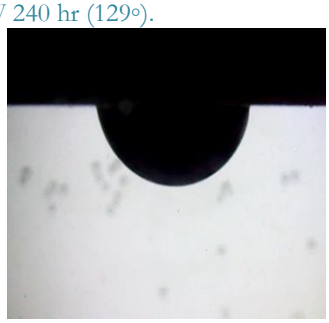

Figure 8c. Alumina NP 48 hr (100°).
Heavy Oil

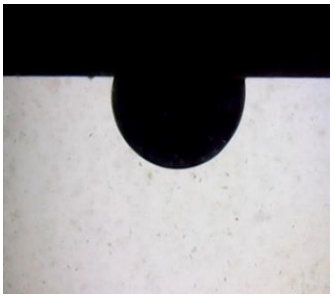

Figure 8d. Alumina NP 480 hr (78°).
Heavy Oil

Figure 8. a)Alumina NP 48 hr 166◦ (light oil) b)480hr (light oil) 119◦ , c) 48hr 100◦ (Heavy Oil), and d) 480 hr 78◦ (Heavy Oil).

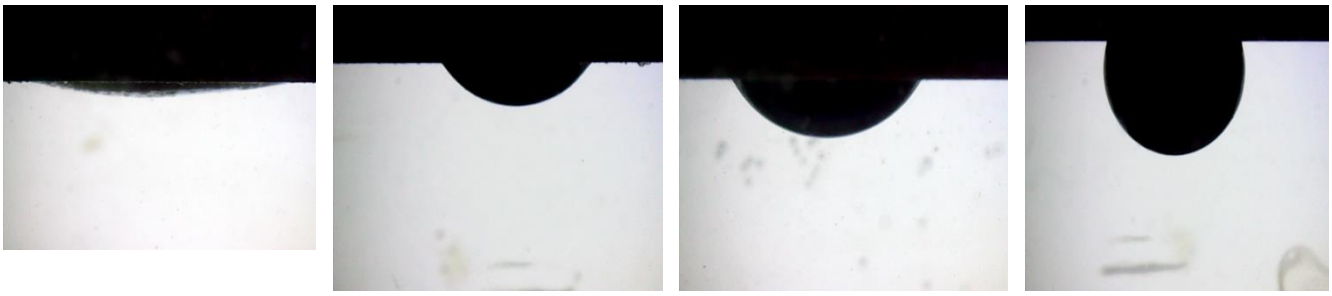

Figure 9a. Silica NP 48 hr (165◦). Light Oil

Figure 9b. Silica NP 480 hr (129◦). Light Oil

Figure 9c. Silica NP 48 hr (103◦). Heavy Oil

Figure 9d. Silica NP 480 hr (80◦). Heavy Oil

Figure 9. a)Silica NP 48 hr 165◦ (light oil), b) 48 hr 129◦ (light oil), c) 48 hr 103◦ (Heavy Oil), and d) 480 hr 80◦ (Heavy Oil).

Table 6. Contact Angle Results of the Nanofluids (Light Oil Thin Sections).

Alumina 0.1%					
Contact angle		48hr	120hr	240hr	480hr
		163	156	148	139
		162	145	143	143
		164	152	146	141
		163	154	146	142
		161	160	148	137
		163	153	146	145
Time (hr)	0	48	120	240	480
Average C.A.	170	163	153	146	141

Alumina 0.25%					
Contact Angle		48hr	120hr	240hr	480hr
		168	150	149	120
		163	151	152	120
		168	151	154	115
		164	152	152	122
		169	153	153	119
		162	150	150	120
Time (hr)	0	48	120	240	480
Average C.A.	169	166	152	151	119

Silica 0.1%					
Contact Angle		48hr	120hr	240hr	480hr
		154	148	143	139
		158	143	143	136
		156	152	141	138
		159	148	148	138
		154	149	137	139
		155	144	149	138
Time (hr)	0	48	120	240	480
Average C.A.	169	156	147	144	138

Silica 0.25%					
Contact Angle		48hr	120hr	240hr	480hr
		164	156	140	129
		166	153	139	132
		164	154	140	128
		167	156	137	129
		164	154	140	127
		165	149	138	128
Time (hr)	0	48	120	240	480
Average C.A.	168	165	154	139	129

Table 7. Contact Angle Results of the Nanofluids (Heavy Oil Thin Sections).

Alumina 0.1%					
Contact Angle		48hr	120hr	240hr	480hr
		111	101	84	75
		102	98	82	70
		110	100	85	73
		101	96	81	70

Alumina 0.25%					
Contact Angle		48hr	120hr	240hr	480hr
		103	97	92	81
		100	92	90	75
		102	95	93	82
		98	90	87	78

		109	101	84	74			102	95	91	79
		101	96	82	71			98	91	89	74
Time (hr)	**0**	**48**	**120**	**240**	**480**		**Time (hr)**	**0**	**48**	**120**	**240**	**480**
Average C.A.	**118**	**105**	**99**	**83**	**72**		**Average C.A.**	**116**	**100**	**93**	**90**	**78**
Silica 0.1%							**Silica 0.25%**					
Contact Angle		**48hr**	**120hr**	**240hr**	480hr		**Contact Angle**		**48hr**	**120hr**	**240hr**	480hr
		104	96	89	68				106	96	88	83
		97	90	82	78				102	93	86	79
		101	92	88	72				104	95	89	81
		93	89	83	77				102	94	84	80
		99	91	87	72				106	95	87	80
		95	89	81	79				100	92	83	78
Time (hr)	**0**	**48**	**120**	**240**	**480**		**Time (hr)**	**0**	**48**	**120**	**240**	**480**
Average C.A.	**113**	**98**	**91**	**85**	**74**		**Average C.A.**	**115**	**103**	**94**	**86**	**80**

4.3 IFT Reduction by the Nanofluids

In this part of the experimental work, the IFT between water and oil through Nano-fluids was investigated to perceive how effective the nanofluids can be. Both alumina and silica nanoparticles with various concentrations at 0.1, 0.25. 0.5, and 1 wt. % were prepared, and their impact on IFT was examined with both light and heavy oil samples at a temperature of 80°C and a pressure of 100 psi. The IFT measurement experimentations showed that both Nano-fluids can reduce the IFT, but with a slight difference of the alumina Nano-fluid being better. The results showed that both Nano-fluids at 0.1 wt. % resulted in the biggest IFT reduction with both oil types. Alumina Nano-fluid reduced the IFT between the water and light oil to 7.23 mN/m while the silica Nano-fluid lowered the IFT to 9.13 mN/m (Figure 10a). Moreover, the Alumina Nano-fluid–heavy oil system resulted in an IFT of 4.82 mN/m whereas the silica Nano-fluid –heavy oil system obtained an IFT of 5.35 mN/m (Figure 10b). Furthermore, any increase of nanoparticle concentration only increased the IFT (Figure 10a and 10b). The mechanism behind the IFT reduction by the Nano-fluids can be related to the interaction between the water phase/Nano-fluid and oil phase/Nano-fluid. Additionally, the adsorption of the Nano particles onto the interface can be another explanation regarding the mechanism (Roustaei et al, 2012). IFT reduction by Nano-fluids has been proven to be one of the mechanisms that would result in improving oil recovery (Onyekonwu and Ogolo, 2010; Shahrabadi et al, 2012; Zaid et al, 2014; Roustaei et., 2012; Hendraningrat and Torsaeter, 2014).

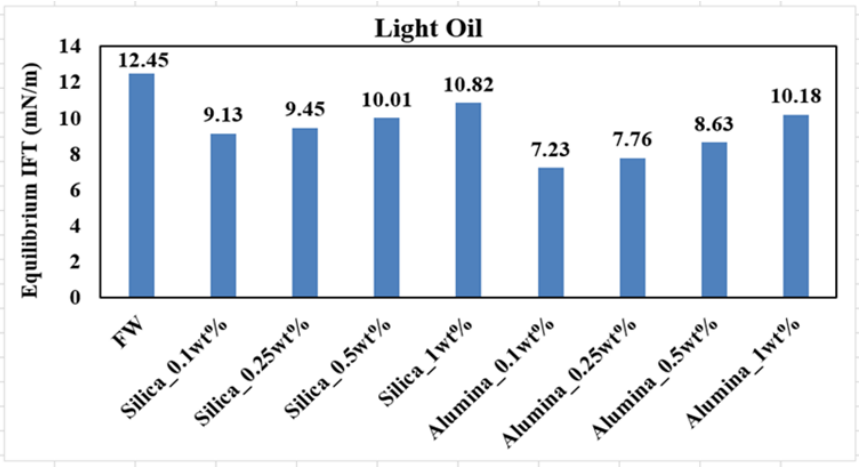

Figure 10a. Various Concentrations of Nano-fluids vs. IFT (Light Oil).

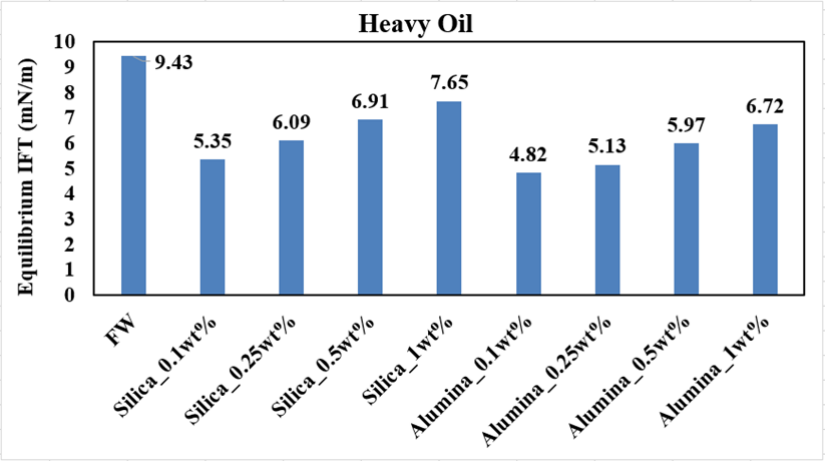

Figure 10b. Various Concentrations of Nano-fluids vs. IFT (Heavy Oil).

Figure 10. Various Concentrations of Nano-fluids vs. IFT a) Light Oil , and b) Heavy Oil.

5 Conclusions and Recommendations

In this section, the main concluding points of the research including the most important results along with a recommendation are pinpointed.

5.1 Conclusions

The following are the main concluding points of the experimental investigation of the impact of nanoparticle flooding, as an EOR, on oil recovery in carbonate rock of Pilaspi formation in Kurdistan Region.

1) Microscopic analysis, XRD and XRF results were all nearly in-line with each other and showed that the composition of the rock is predominantly calcite (CaCO3) along with minor traces of quartz and dolomite.
2) The silica Nanofluid at 0.1 wt. % lowered the IFT between the aqueous phase and the light oil to 9.13 mN/m while the IFT between the aqueous phase and the heavy oil was reduced to 5.35 mN/m at the same concentration. However, the alumina Nanofluid at 0.1 wt. % shrunk the IFT between the water and the light/heavy oil to 7.23 mN/m and 4.82 mN/m, respectively.
3) The contact angle of the oil wet thin sections upon treating by the formation water was altered from nearly 169◦ and 115◦ to 129◦ and 90◦ for the light oil and heavy oil, respectively.
4) Alumina Nano-fluid at 0.25 wt. % reduced the contact angle on the surface of the light and heavy oil aged thin sections from 169◦ and 115◦ to nearly 119 ◦ and 78◦ respectively while 0.25 wt. % silica Nano-fluid lowered the contact angles to 129◦ and 80◦ for the light and heavy oil samples respectively.

5.2 Recommendations

The following point is recommended for further research in this specific era of EOR in Pilaspi formation carbonate rocks: Investigating the applicability of other Nanoparticles such as the oxides of zinc, nickel, tin, iron, magnesium, and zirconium to understand how imperative they can be as EOR agents in Carbonate rocks of Pilaspi.

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