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An Experimental Investigation of Low Salinity Oil Recovery in Carbonate and Sandstone Formation

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Abstract: *Water flooding has for a long time been employed to improve oil recovery in many oil fields. Formation damage due to water injection was the main issue of water flooding design process for many years and oil companies conducted different compatibility tests between injection water and formation water to eliminate any possibility of formation damage. In recent years, the results of extensive research work demonstrated that alteration of water salinity concentration and composition improves significantly the ultimate oil recovery of water flooding. Up to date there is no universal agreement among the researchers on the mechanism of low salinity flooding. Different mechanisms are proposed in the literature such as wettabiliy modification, fine migration, interfacial reduction, emulsion, and ionic exchange. In this paper an experimental investigation on the possible mechanism of low salinity flooding was conducted. Contact angle changes as function of time, and low salinity water flood experiments using limestone and sand stone rocks for various injection brines were performed. Sand stone and carbonate rocks were obtained from actual Libyan field. High and low salinity waters (223,000 ppm and 20,000 ppm), sea water (49,000 ppm), and water with different sulfate concentrations were employed in this investigation. The results from this work indicated that low salinity flooding can improve the oil recovery of carbonate formation and its performance is function of carbonate type.*

1. Introduction

Water injection to improve the oil recovery has been employed for many years. The effect of injection brine composition and concentration on the displacement efficiency has been ignored in the design of water flooding in the past. Historically avoiding formation damage by making sure no interaction between injected brine and indigenous brine will take place during water flooding was the main design parameter of water flooding. Morrow et al. (1996) concluded that oil recovery optimization during water flooding requires alteration of injection water brine composition. Tang and Morrow 1999; and McGuire et al. 2005 concluded that decreasing brine salinity results in an improvement of oil recovery. Jerauld et al. 2008 indicated that low salinity flooding of more than 20 sandstone cores at reservoir conditions in secondary and tertiary modes had been conducted as reported in the literature. They also reported an improvement of recovery efficiency of 5 % to 38% and 3% to 17% reduction of residual oil saturation as a result of low salinity flooding. Nasralla et al. 2011, made a comparison between sea water and de-ionized water flooding in a secondary mode and demonstrated that using deionizer water produced a significant improvement in oil recovery over seawater. Tang and Morrow 1999 stated that fine migration during low salinity water floods of Brea sandstones cores is the main mechanism responsible to the improvement of oil recovery. They indicated that the exposure of rock surface as a result of fine migration is the mechanism behind alteration of the system wettability. On the other hand, high salinity water floods does not react with clays and as a result of that the reservoir rock maintains its wettability condition. Detachment of clay particles from the rock surfaces and reduction of permeability associated with low salinity flooding (less than 1550 TDS) were also reported Tnag and Morrow. Valdya and Fogler 1992 revealed both release of fine and high pH in low-salinity flooding. They had noticed a significant change in permeability of the system at a pH higher than 9 which indicated a formation damage caused by fine migration. Bazin and Labrid 1991 reported that cation exchange capacity (CEC) of clay sandstones plays a major rule on fine migration. Khilar et al. 1990 concluded that the reduction of formation permeability during water flooding of sandstone is mainly due highcation exchange capacity. Khilar et al. 1990 and Kia, S.F 1987, indicated that the permeability reduction will take placers if the ionic strength of the injected water is equal to or less than, the critical flocculation concentration (CFC). The CFC is strongly dependent on the relative concentration of divalent cations such as Ca^{2+} and Mg^{2+}. Divalent cations lower the Zeta potential resulting in the lowering of the repulsive force and that leads to clay stabilization. Tang and Morrow 1999 indicated that low salinity water flooding can result in fine migration. On the other hand, BP reported a number of corefloods experiments using its *LoSal*™ EOR technology have demonstrated improved oil recovery, with no fine migration or permeability reduction. Some laboratory work indicated a rise of pH of produced water as function of pore volume injected (Lager et al 2006), see Fig. 1.

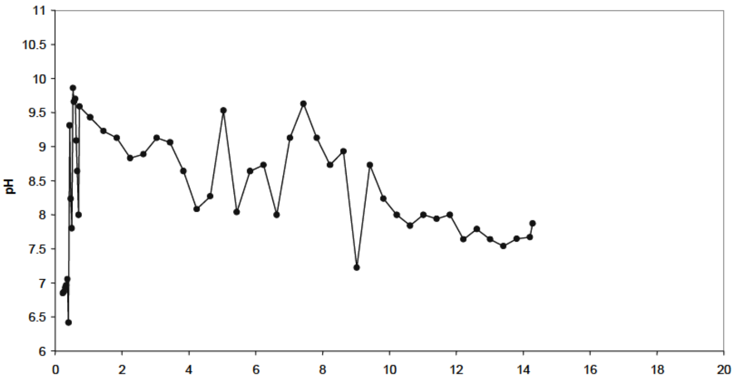

Figure 1. *pH variation during a LoSal waterflood, lrwf North Sea Reservoir System(Lager et al 2006).*

This increase in pH is developed as a result of carbonate dissolution and cation exchange. The dissolution of calcite and/or dolomite results in the increase of OH^- and cation exchange takes place between clay minerals and the injected brine. The dissolution reactions are function of the amount of calcite and/or dolomite present in the rock (Lager et al 2006).

$$CaCO_3 \rightleftharpoons Ca^{2+} + CO_3^{2-} \qquad (1)$$

$$CO_3^{2-} + H_2O \rightleftharpoons HCO_3^- + OH^- \qquad (2)$$

On the other hand, cation exchange occurring on the clay minerals, the mineral surface will exchange H+ present in the brine with cations previously adsorbed. This will result in a reduction of H^+ concentration in the brine resulting in a rise of the pH, Lager et al 2006. Increasing the pH of the system above 9 would make the brine flooding behave similar to alkaline waterflood. Alkaline water flooding applied to acidic oil can reduce oil and water interfacial forces, change the wettability of the system, and formation of water in oil emulsion (Jensen & Radke 1988). Lager et al concluded that cation exchange between the mineral surface and the invading brine to be the primary mechanism underlying the improved waterflood recovery observed with *LoSal* low salinity waterflooding. They stated that this mechanism explains why *LoSal*technologydoes not seem to work on carbonate reservoirs.

A number of research work has been published indicated that calcium ion (Ca^{+2}), magnesium ion (Mg^{+2}), and sulfate ion (SO_4^{-2}) are the responsible ions for the alteration of wettability in brine injection process (RezaeiDoust et al. 2009). The alteration activity of these ions increases with increasing the temperature above 100 ℃. Zhang et al. 2007 have studied the impact of Ca^{+2}, Mg^{+2}, SO_4^{-2}, and T on the oil recovery from chalky limestone of low water wetness in a spontaneous imbibitions process, see Fig. 2. The results clearly demonstrated that increasing SO_4^{-2} in the presence of Mg^{+2} at higher temperatures improves the oil recovery significantly. As shown in Fig. 2 no significant improvement in oil recovery was observed at both 70 and 100 ℃ in the presence of NaCl, therefore they concluded that sulfate could not change the wettability to improve the spontaneous imbibitions at low temperature (Zhang et al. 2007). On the other hand in the presence of Ca^{+2} and/or Mg^{+2} with sulfate significant imprudent in the imbibitions of water was observed and attributed that improvement to change of wettability of the system to more water wet. Zhang et al. proposed a chemical mechanism for alteration of wettability as illustrated in Fig. 3.

They suggest that if injected water contains Ca^{+2} and SO_4^{-2}, sulfate ions will adsorb onto the positively charged chalk surface, and a reduction of the positive surface charge will prevail. The electrostatic repulsion will decrease in this case and more of Ca^{+2} can be attracted to the surface (RezaeiDoust et al., 2009). RezaeiDoust et al. suggested that Mg^{+2} is able to displace the Ca^{+2}, which is connected to the carboxylic group, in the same way as Mg^{+2} is able to displace other Ca^{+2} ions from the surface lattice of the chalk.

Frontiers BP, 2009, presented a hypothesis for low salinity effect in the presence of clay, see Fig. 4. They suggested that the negatively charged clay particles produce a diffuse double layer, where in the aqueous phase in the vicinity of clay is positively charged.

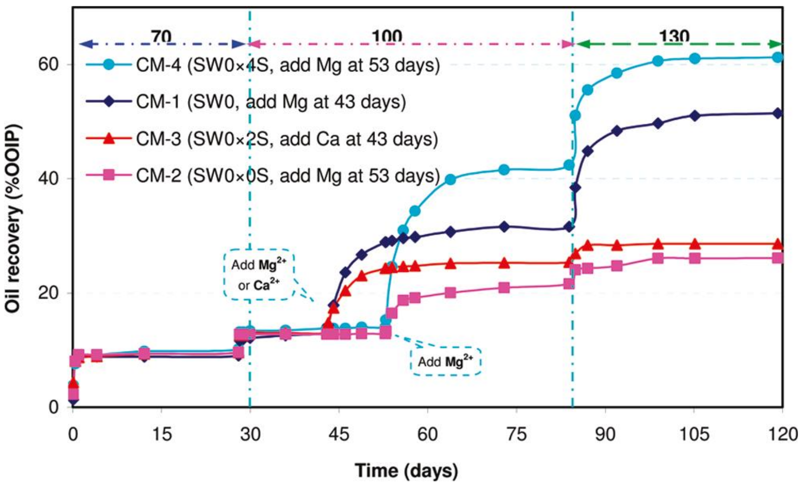

Figure 2. *Spontaneous imbibition tests on chalk cores suing different SO_4^{-2}, (Zhang et al. 2007).*

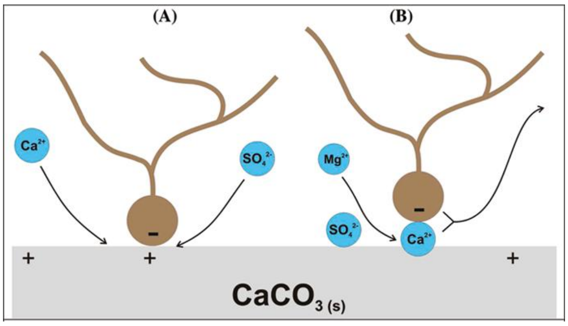

Figure 3. *Schematic model of mechanism for the wettability alteration, (Zhang et al. 2007).*

The thickness of the double layer increases with decreasing salinity. Water molecules within the double layer are rigid or "quasi-crystalline" and that will result in an increase of oil phase relative permeability as medium becomes more water wet. On the other hand if hardness (Ca^{+2} and/or Mg^{+2}) present in the system, negatively charged oil surface can bind with the clays via an intermediate, such as divalent ion calcium. Berg et al. 2009 provided direct experimental evidence indicated that wettability modification of clay surfaces was a microscopic mechanism for low salinity flooding. They ruled out emulsification, interfacial tension reduction, fines migration and selective plugging of water-bearing pores via clay swelling as most relevant mechanisms. They have confirmed wettability modification as the relevant mechanism, and they have indicated that they are trying to distinguish between double layer expansion and cation exchange or if a layer of clay detaches together with each oil droplet. They stated that oil has been released in low salinity system where also clay de-flocculation and formation damage has taken place, and at least for Montmorillonite clays there was a range of salinity where oil can be removed with no damage.

McGuire et al. 2005 reported BP experience with low salinity flood. They indicated that BP tested four areas using water injection salinity ranges between 1500 to 3000 ppm and the benefits of its *LoSal* EOR ranged from 6 to 12% OOIP, resulting in an increase in waterflood recovery of 8 to 19%. It well known that low salinity flood has the following advantages: high EOR potential, environmentally friendly, and combination with other recovery methods possible (such as polymers, alkaline, surfactant ...etc.). Robertson, 2007 showed, using data obtained from three oilfields, oil recovery increases as the salinity ratio of the waterflood decreases see Figure 5. The injection water and field water salinities are presented in Table 1.

Kumar et al 2010 conculded that oil recovery from Berea by traditional flooding is accompanied by fine migration in quantities sufficient to have some bearing on oil-brine interfacial stability, and that low salinity flooding increases this tendency. Wideroee et al. 2010 employed NMR relaxation/diffusion measurements and CryoESEM Imaging for detecting wettability changes during low salinity flooding of sandstone cores, they concluded that the responses in the data from low salinity flooding experiments may beattributed to wettability changes.

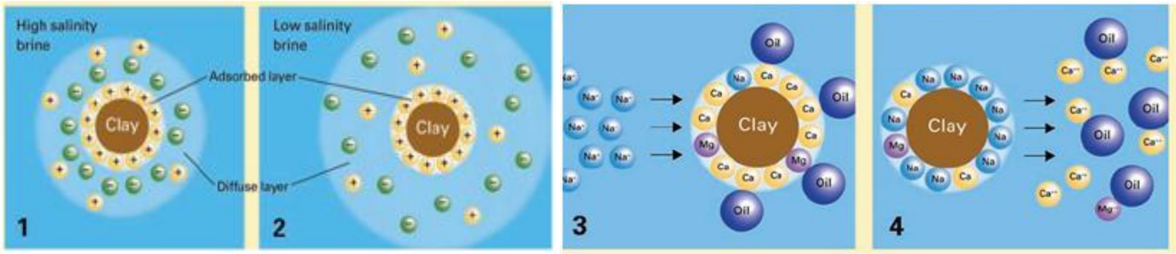

Figure 4. *Formation of double layer by negatively charged clays, Frontier BP.*

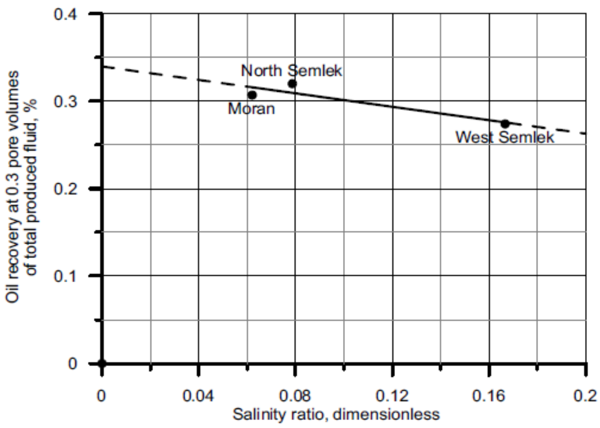

Figure 5. *Oil recovery and salinity ratio for three Minnelusa waterfloods, Robertson 2007.*

Table 1. *Injection and formation salinity for three reservoirs, Robertson 2007.*

Field	Formation	Injection	Ratio
West Semlek	60,000	10,000	0.166
N. Semlek	42,000	3,304	0.0787
Moran	128,000	7,948	0.0621

LSW flooding involves injecting brine with a lower salt content or ionic strength. The latter is typically in the range of 500–3,000 parts per million of total dissolved solids (TDS), and no more than 5,000 ppm (parts per million). This can be compared with salinities for seawater or formation water, which are about 30,000 ppm and 60,000 ppm respectively. The introduction of LSW in an equilibrium system of high salinity appears to cause a shift to a new system equilibrium, which tends to favorimproved oil recovery (IOR).

2. Description of Experiments

Apparatus and Materials

Contact Angle Measurements. To determine effect of salinity on wettability, contact angles are measured using the sessile drop method. The device consists of a box made of Pyrex with dimensions of 10 cm × 10 cm × 13.5 cm. A circular limestone disk (Diameter 3.6 cm) is placed on the top of the open side of the table as shown in the schematic diagram, Fig. 6. The box is filled with the specified saline solution. Then a small drop of oil is placed at the bottom of the limestone disk and given a dynamic water receding condition. The changes in the drop size as function of time are monitored using a digital camera. Different runs were performed to assess the effect of salinity on the contact angle of the studied system. A photo of the oil drop as function of time of the studied systems is taken every mint. The change in wettability of crude oil with time in the presence of brine solutions is measured. These runs were analyzed for contact angle determination using Sigma Scan Pro. Image analysis software.

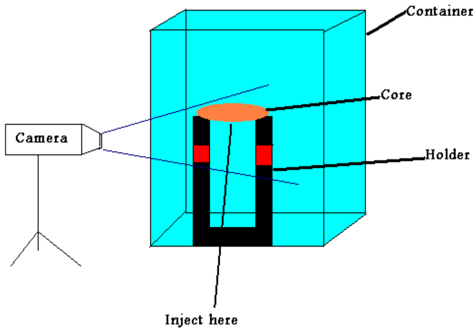

Figure 6. *Schematic of the device to measure contact angles.*

Core Flooding Apparatus. The core flooding apparatus consist of fluid accumulators which connected to a variable rate injection pump. The core holder is placed in a variable temperature oven. Pressure and temperature transducers are connected at both ends of the core inside the core holder. A chart recorder and a digital pressure recorder are connected to the temperature transducer and pressure transducer respectively.

Oil Sample. The oil used in the flooding of limestone and sandstone cores obtained from an oil field, well-A. The chemical composition of this crude oil is presnted in Table 2 and properties of the used crude oil are presented in Table 3.

Experimental Procedure

Contact angle for sea water (50,000 ppm, sulfate 4048 ppm), saline water with salinity similar to sea water with no sulfate concentration and formation brine (140,00 ppm, sulfate 378 ppm) were measured for limestone and chalky lime cores. Then contact angles for different water samples prepared in the lab containing 0, 2500, and 6500 ppm of sulfate representing no sulfate, low sulfate and high sulfate concentrations. Limestone cores were dried at 80°C for 72 hours after cleaning. Each core was evacuated for almost 12 hour and fully saturated with actual filtrated formation brine. The clean and dry core weights were measured and used to calculate pore volume and porosity. The cores were flooded with same formation brine until a steady-state flow condition of water was well-established. The variation of pressure drop along the core was used with other rock and flow parameters to calculate the rock permeability using Darcy's Law.The third step was to flood the cores continuously with oil to displace all of the displaceable water then each core sample then cores were flooded again with the specified brine and measured the oil recovery after brine flood.Some physical properties of the crude oil used are presented in Table 3. The oil has a kinematic viscosity of 6.3 cSt at 40 ºC, and has a low content of asphaltenes of 0.2 weight %. The oil has a slightly lower content of C_6-C_8 cuts and higher content of the n-C_9 to n-C_{22} cuts. The brine employed in this project collected at surface conditions from the selected field, well-B.

Table 2. *Oil composition used in this study*

Component	%	Component	%	Component	%	Component	%
C6	0.020	C14	4.315	C22	2.074	C30	0.332
C7	0.045	C15	4.096	C23	1.581	C31	0.216
C8	0.285	C16	3.622	C24	1.413	C32	0.198
C9	1.586	C17	3.299	C25	1.056		
C10	3.448	C18	2.987	C26	0.975		
C11	4.687	C19	2.610	C27	0.733		
C12	5.418	C20	2.287	C28	0.598		
C13	4.837	C21	2.130	C29	0.418		

Table 3. *Physical properties of the used crude oil.*

Condition	Value	ASTM method used
Specific gravity at 20 °C	0.8672	D-287
API gravity at 15 °C	31.67 degree	D-287
Kinematic viscosity at 40°C	6.30 cSt	D-445
Total Acid Number	0.9537 mg KOH/gm oil	D-974
Asphaltene Content, wt %	0.20	D-6560

3. Results and Discussion

Effect of Salinity on the contact Angle

The contact angle measurement should provide a clue about the possible wettabilty of the system. In this project we used the contact angle of different brine to compare between them with respect to the possible alteration of the system wettability as a result of alteration of brine composition and concentrations. The contact angle between formation water (140, 00, sulfate 378 ppm), saline water (50,000, sulfate 0.0ppm), sea water (49000, sulfate 4048 ppm) and actual crude oil using a chalk limestone disks were measured as function of time. Table 4 presents the measured contact angle of different studied systems as function of time. A value of contact angle below 90 degree represent more likely a water wet system or the system moving toward water wetness, and the system with a contact angle above 90 ° indicates an oil wet system. The results presented in Table 4 clearly demonstrated that decreasing the salinity of the system and increasing the amount of sulfate concentration of the system moves the system toward more water wet system. The data shown in Fig. 7 indicated that the salinity is not the critical factor in the process but the composition plays a major rule in wettability alteration in chalky limestone media. The system with relatively low salinity (salinity similar to the sea water) produced a contact angle of 140 degree which indicates oil wet system on the other hand the same salinity system, i.e. sea water that contains sulfate concentration of 4048 ppm produced a contact angle for the same oil and shaky limestone rock of 67 degree which translated to a switch of salinity to a water wet system.

Table 4. *Contact angle for different waters, Chalky limestone*

Formation Water Chalk		Saline Water Chalk		Sea Water Chalk	
Time, min	Contact Angle, deg	Time, min	Contact Angle, deg	Time, min	Contact Angle, deg
0	46.034	0	98.039	0	55.981
1	69.73	1	118.529	1	65.854
30	70.92	30	141.839	30	73.041
180	85.796	180	143.132	180	70.388

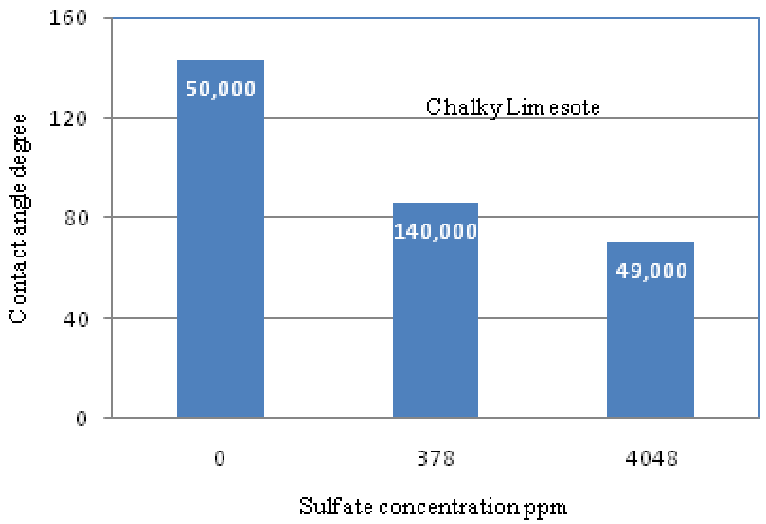

Figure 7. *Contact angle as function of sulfate concentration, Chalky LS.*

Meanwhile, high salinity water (140,000) still produced a system that can be considered a water wet system due to the presence of sulfate of concentration of 378 ppm. Therefore, the previous argument can lead to the fact that presence of small amount of sulfate in the water might shift the wettability toward water wetness to some degree in chalky limestone media. Anderson 1986 indicated that at a pH below 9.5 carbonate surfaces are positively charged. It is well known that the clay content of the carbonates is very small and can be ignored. Electrostatic attraction forces will facilitate the adsorption of the negatively charged oil droplets onto the positively charged carbonate rock surfaces at this relatively low pH environment, Ligthelm et al. 2009. Carbonate rock surfaces are positively charged have ionic exchange capacity and anions such as sulfate if exist might adsorb to these surfaces. As discussed in the introduction, as sulfate adsorb onto the surfaces of carbonate with excess calcium close to the carbonate that will facilitate the substitution of adsorbed hydrocarbon by the sulfate. The results of contact angle measurements are consistence with the previously results published by Austad 2008. He stated that sulfate containing fluids such as sea water can alter the wettability of carbonate rocks to more water wet state.

Contact angles as function of time using different waters for reservoir limestone rocks were measured and the collected data are presented in Table 5.Results indicated that in the case of limestone rocks the concentration of salinity plays a major rule in contact angle modification, i.e. possible change of wettability. High salinity systems will not change significantly the wettability of the system in the presence of low sulfate concentrationas indicated in Fig. 8.Therefore, the pore size distribution and mineralogy of the system affects the wettability alteration mechanism of sulfate.

Ligthelmet al. 2009 stated that there is no need for increased electrostatic repulsive forces by expansion of electrical double layers and hence the low electrolyte content is not required to modify the wettability in carbonate media. The process of wettability modification works very well and significant alteration in the presence of sulfate was observed, as shown in Fig.8. Results indicates the alteration of wettability in microcrystalline limestone's may require relatively low salinity and relatively high sulfate concentration. Each system should investigated separately to arrive at a definite conclusion and the results of this work give an indication of the possible alteration only due to the limited data obtained from the project.

Table 5. *Measured contact angle for different saline solution.*

Formation Water		Saline Water		Sea Water	
Time, min	Contact Angle, deg	Time, min	Contact Angle, deg	Time, min	Contact Angle, deg
0	49.568	0	91.999	0	67.131
1	104.268	1	116.565	1	78.626
30	131.055	30	141.278	30	79.702
180	170.776	180	180	180	89.338

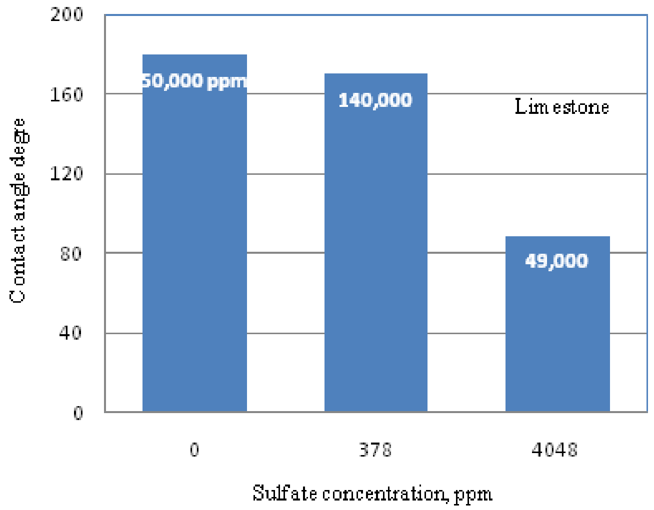

Figure 8. *Contact angle as function of sulfate concentration, reservoir LS.*

Core Flooding Chalky Limestone. The results of coreflooding experiments using brines of different sulfate concentrations for same initial wettability conditions are shown in Fig. 9**.** It shows a comparison of oil recoveries for injecting brines of five different sulfate concentrations employing cores with similar property (porosity and permeability) and wettability conditions. In general, the oil recovery increaseswith increasing of the sulfate concentration of the injected brine. The sulfate concentration wasranged from 0% to 6500 ppm as indicated in Figure 9. Experimental work results indicated that the oil recovery can be improved by94% as a result of increasing sulfate concentration from 0 to 6500 ppm. The oil recovery for 0 and 6500 ppm of sulfate concentration were 34% OOIP and 68% respectively. For the studied system and range of sulfate used, a linear trend between sulfate concentration and oil recovery is observed and the following equation obtained based on the plotted trend:

Oil recovery = 0.0044 Cs + 35.618 $R^2 = 0.9512$

The result does not mean that there is no limit to the trend of oil recovery and sulfate concentration of chalky LS. More experimental work is needed to complete the investigation of the relationship between sulfate concentration and oil recovery, keeping in mind that there are other variables that have a significant impact in the process and they should be taken in consideration.

Core Flooding of Sand Stone Cores. Six sandstone cores obtained from Upper Sarir formation of a Libyan oil reservoir were used to study the effect of salinity on the oil recovery. Table 6presents conventional core analysis results of the employed cores. Low salinity (20,000 ppm) and high salinity (223000 ppm) brines were used to study the effect of salinity on the oil recovery. The water injection rate was kept constant (0.5 cc/mint) for all runs except for sample 85 which was flooded with both brines at a rate of 1 cc/min. Fig. 10 presentsthe results of the experimental work and the results indicates using cores of low permeability produced an improvement in oil recovery for low and high salinity floods except for ultra low permeability of 0.45 md and the lithology probably plays a major rule in the high recovery associated with the core of 2.64 md permeability.

As shown in Table 6 core no. 85 (k = 2.64 md) is the only sample that contains dolomite sand i.e. contains a certain percentage of dolomite (Ca^{+2} and Mg^{+2}) and that tends to improve the ionic exchange process whereby sodium can place the calcium ions and magnesium ions in the process which leads to freeing the oil droplet from the surface of the rock and that resulted in improvement in of the oil recovery. Flooding core no. 85 with low salinity brine resulted in the recovery of 80% of original oil in place compared to 70% of OOIP in case of high salinity brine, i.e. addition of 10% of OOIP due to alteration of salinity. This improvement is quite significant, keeping in mind the application of carbon dioxide flooding on the average if the right conditions for that application exist can add on the average around 8-10% of OOIP in most optimistic cases. Although, the speed of the flood for core no. 85 (1cc/min) is different than the others (0.5 cc/min) but that cannot be a factor in the observed high recovery simply due to the fact that high velocity will decrease the ionic exchange process becuase ionic exchange is a function of time. The results of this work demonstrate without any doubts that low salinity flood does work in the sandstone of one of the Libya oil reservoirs and a significant amount of oil can be added to our reserves. The presence of dolomite in the sand stone reservoir can lead to huge improvement of low salinity flooding as presented in Fig. 10.Cores that contains sandstone and silt stone only produced on the average around 50% of OOIP compared with 80% for the core that contained dolomite sand. An improvement in the recovery of 30% of OOIP can be considered by the company as a new discovery. The results of this work is preliminary in nature due to limited data and more work is required to shed more light on the mechanism of the process and how much more oil can be obtained from the low salinity flood. Process optimization in the lab also should be conducted before moving any further in the project. Process optimization means running more experiments using different concentrations and compositions to come up with the best system for the selected field taking in consideration the effect of litho logy of the rock which required studying the lithological variation within the field, porosity, permeability and pore size distribution, flood rate, and reservoir brine salinity. The most important finding of this work is the process does work in sandstone environment without the presence of clay. Many researchers previously indicated that low salinity flood requires the presence of clay to produce significant improvement in the oil recovery but the results of this project proved otherwise.

Table 6. *Core analysis results of the sand stone cores.*

Sample	Depth int.	Permeability	Porosity	Density		
#	ft	kh { mD	{ % }	Bulk	Grain	Lithology (grain size)
8	14107.5	162.00	10.83	2.56	2.75	Sst, md to crs, Sil Cmt Patches
21	14120.6	649.06	11.94	2.45	2.65	Sst, md to md/crs, Sil Cmt
22	14121.5	388.56	13.59	2.43	2.65	Sst, f to md/f, Sil Cmt
65	14177.4	2.05	9.46	2.49	2.65	Sst, f/md to vcrs, Sil Cmt, bit
68	14181.5	10.43	11.98	2.45	2.65	Sst, crs, Sil Cmt, bit spots
85	14199.45	6.16	10.66	2.47	2.65	Sst, md to md/crs, Sil Cmt, DOS/Bit

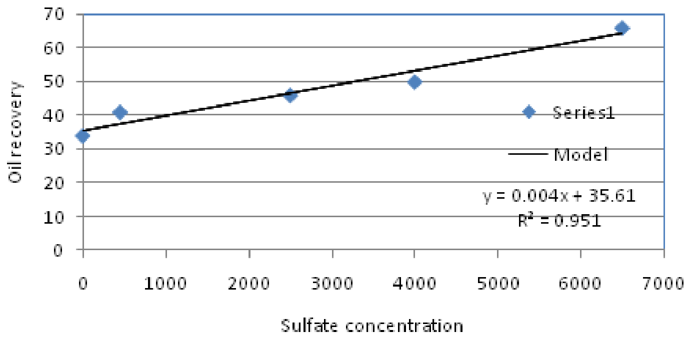

Figure 9. *Oil recovery versus sulfate concentration, chalky LS.*

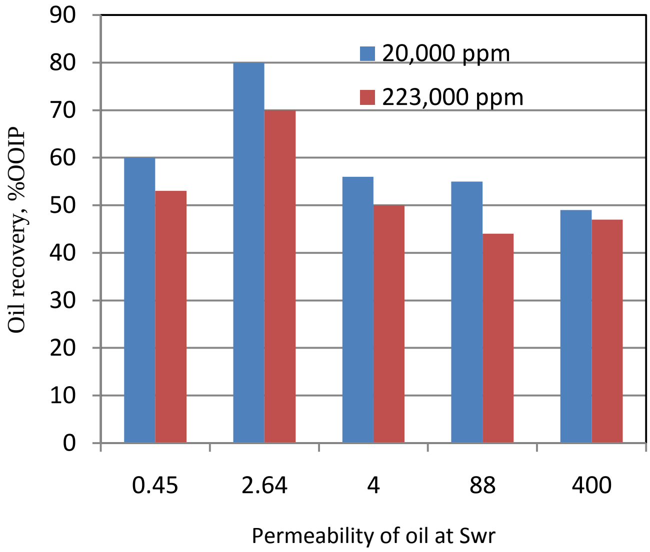

Figure 10. *Oil recovery versus permeability.*

4. CONCLUSIONS

Based on the experimental work conducted in this project the following conclusions are drawn:

1. *LoSal™*low salinity flooding can be applied to carbonate rocks (limestone), and its performance is function of carbonate type, chalky or microcrystalline limestone.
2. Wettability alteration most likely is the mechanism responsible for the improved oil recovery by *LoSal*technololgy in carbonate formation.
3. Increasing the sulfate concentration increases the waterwettnes of the chalky and microcrystalline limestone.
4. *LoSal* low salinity flooding can perform quite well in a sandstone reservoir with no clay present.
5. The performance of the *LoSal* technology improves significantly in a sandstone strata contains dolomite sand.

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