Low salinity water flooding in carbonate reservoirs – dissolution effect

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Abstract. One of the most significant research subjects in the industry of oil is the low salinity water injection techniques (LSWI). This is because there are possible benefits of raising recovery in oil as opposed to the conventional injection of seawater. Recent research has been done to find out the causes of less salinity with the injection of water in carbonated rocks presented significant findings. The primary mechanisms that are thought to lower salinity water injection are carbonate rocks dissolution, fines migration and alteration of wettability to more water wet. However, many researchers have elected to oppose the mechanism of dissolution and instead attributed high recovery of oil to be the primary wettability modification mechanism. Additionally, previous researchers provided no evidence characterizing about the rock and fluids produced in a detailed manner. This research work provides a detailed description of the underlying mechanisms, the core flood laboratory and the spontaneous imbibition in carbonate rocks. In the experiment, we carried out a series of steps using a limestone core with low permeability. This core was obtained from a nearby oilfield in Iraq and used it to study how dissolution and fines migration mechanisms take place. Moreover, in the process of carrying out the experiment and before its starting, SEM imaging analysis was used. In this case, as we carried out the experiment, we had to inject two-phase flow of sea water (SW), de-ionized water (DW) as well as soltrol-130. This was done in a sequential manner at 50 °C as well as 0.02 ml per minute. As the experiment progressed, the concentration of ions and pH value of the extracted water were measured in a continuous manner. The results obtained indicate that the rate of change in concentration of Ca²⁺ ions is directly proportional to the effluent pH value. By just balancing the mass, we were able to easily see the rate at which limestone dissolved as the experiment progressed. The maximum increase in pH was registered when fresh water was injected (pH=9.2). An increase in pH value can have a positive effect on altering wettability in that it can increase the alkalinity of flood water. To notice fines migration, the Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS) are used to scan the images of the inlet and outlet of the core plugs after completion the experiment. This method can indicate that dissolution can induce fines dislodgement for the surface of the rock. The two mechanisms of fine migration and carbonate rock dissolution can significantly increase recovery of oil and at the same time inject water low in salinity.
1. Introduction

One of the mechanisms of altering wettability in carbonate reservoirs, which has improved oil recovery techniques, is low salinity water injection (LSWI). Based on increased efficiency level in the displacement of medium and light gravity crude oils, it has become very popular. Other than for this reason, water availability and affordability, low capital and low cost have raised its popularity, which eventually leads to its favorability compared to other methods of IOR or EOR.

However, this technique has a significant challenge when it comes to water disposal and water sourcing.

According to Al-Shalabi et al. (2017) [1], other names that are used for this technique of water injection is LoSalTM by BP, Smart Water Flood used by Aramco Saudi Arabia, Shell’s Designer Waterflood and ExxonMobil’s Advanced Ion Management (AIMSM). Following the successful injection of low salinity water in carbonate rocks, research has been taken a notch higher, both in the laboratory and in the field scale. This technique has recently been implemented on sandstone rocks. Statistics have it that carbonate oil reservoirs are greater than 50% [2]. This has necessitated the investigation of the validity of LWSI in carbonate rocks. Previously, water-wetting of carbon rocks was done at the deposition age and oil-wetting applied to the aging oil. Moreover, approximately 90% of limestone is either intermediate in wetness or oil-wet [2]. Several factors have to be considered when a rock is being transformed from water-wet to oil-wet. These factors include mineralogy of the rock, base and acid number of the crude oil, the composition of brine in it and the pH value with the reservoir’s temperature being no exception.

Carbonate rocks share one unique characteristic, which is their low oil recovery in comparison to the sandstone rocks, which have a system that is oil-wet. This means that they have a lower permeability to oil compared to water-wet rocks. It is not possible to significantly increase oil content in a reservoir by injecting more water into the carbonate rock in a secondary recovery stage, not having undergone the initial treatments of pressure enhancement. This, therefore, shows how important the investigation of the effect of saline brine is. According to (Al-Shalabi et al., 2016) [3], several methods can be followed to lower the salinity of the water, and these are; brine dilution, a decrease in the concentration of cations and the elimination of non-active ions such as Cl- and Na+. Compared to other methods such as Enhanced Oil Recovery (EOR), reduced water salinity has several advantages and these are; first, it is readily available and affordable. Secondly, it is not difficult to inject water into reservoirs. Thirdly, the cost of operation and the capital required are also low. Fourthly, there is efficiency in the displacement of the medium to light gravity of oil. Fifthly, this technique can ultimately recover oil at a lower cost compared to the EOR method.

Finally, the approach can be easily applied at initial and later stages of the reservoir’s life. Morrow (1999) and Lager et al. (2006) [4] argued that the LSWI in limestone lacks validity, because limestone is low in terms of the amount of clay it contains. Additionally, heterogeneity of carbonate rocks makes it difficult to apply LSWI. According to Austad et al. (2005) [5], several studies have been conducted in the field as well as in the laboratory and the majority of these studies have confirmed and given positive feedback on low salinity injection. In fact, these findings have seen their translation in oil recovery in the secondary as well as the tertiary injection modes. In our laboratory, we presented in this work dissolution and fines migration. Moreover, core preparation, experimental procedure, setup and the results of two phase flow, including pH values, effluents concentration, oil recovery and pressure drop, are reported. SEM-images and the EDS-analysis are reported as the last part of the results, as discussed in the sections below:
2. Core Preparation

The core preparation is one of the most crucial parts of the research because cores are usually heterogeneous and have a low permeability of contents of dead crude oil reservoirs. In the cleaning section, we ensured that pumping solvent was avoided through the cores as it may lead to alteration of the interior structure of the pore of the cores as well as fines migration may occur courtesy of drag force emanating from the injected solvent. Therefore, to ensure that this trend does not recur, the Soxhlet method was utilized as a method of cleaning. To clean the core plugs, both Toluene and Methanol were used. So, the core plugs were cleaned in the regular technique with Toluene and Methanol. Anderson (1986) [6] stated that the effectiveness of Toluene is that it can remove hydrocarbons amongst them asphaltenes and other weak polar compounds. On the contrary, stronger polar methanol (CH$_3$OH) can eliminate the compounds responsible for the alteration of wettability. In this regard, we used Toluene before the emergence of transparent and clean solvent, which emerged in the apparatus that was used. We also combined the solvent with methanol up to such a point when another explicit solvent reappeared. This process was repeated for both methanol and toluene. The process of cleaning took close to one month as we had to ensure that the cores were cleaned and ready for the experiment even though the core had low permeability.

3. Procedure of the Experiment

From our Iraqi field of Rumali, it was obtained a core of heterogeneous limestone with an average of absolute permeability 0.86 mD as well as porosity of 15.67%. In the absence of clay, from the XRD and XRF results It was found out that the limestone contents were made up of 3.4% dolomite CaMg(CO$_3$)$_2$ and 96.6% Calcite CaCO$_3$ although these measurements are a deficit of clay. The core had original dimensions of 60.44 mm in length and 2.5 mm in its diameter. Because the sample length was limited regarding utilizing the Scanning Electron Microscope Instrument, the core was cut to half. The two plugs are used in the experiment. From most of the laboratory data available, it is not abnormal to find that the core has selective permeability. Table-1 list the core petrophysical properties are articulated after carrying out experiment. Both soltrol and fluid properties used during the experiment listing in tables 2 and 3.

Table-1, Limestone core plugs used in the experiment.

| Core Plug No. | Diameter (mm) | Length (mm) | Porosity (%) | Pore Volume (ml) | Absolute Sea Water Permeability (mD) | Swirr | Sor @ SW | Sor @ DW |
|--------------|---------------|-------------|--------------|-----------------|--------------------------------------|-------|----------|----------|
| 1            | 25.5          | 29.306      | 15.165%      | 2.261           | 0.86                                 | 0.31  | 0.318    | 0.195    |
| 2            | 25.5          | 31.136      | 16.172%      | 2.574           | 0.86                                 | 0.31  | 0.305    | 0.194    |
4. Scanning Electron Microscope (SEM)
Having completed the cleaning step, it was time to take the Scanning Electron Microscope images for the composite core’s inlet and outlet. The division of the interface took place realizing four quarters where each quarter had many SEM images selected for it. SEM images from before and after the experiment were compared to study the dissolution impact on the changing geometry of the rock. This can also help in the examination of the possibility of fines migration induced by the dissolution of limestone.

5. Experimental Setup
In the investigation of dissolution mechanism that results from low salinity water, de-ionised water and sea water were used. In a bid to achieve better and higher oil recovery; de-ionised water is used to be the best source of water based on its richness for magnesium, calcium and sulfate ions, which are potential determinants towards carbonated rocks. To obtain higher oil recovery such as wettability alteration, these ions are crucial although not for the dissolution mechanism. Therefore, we prepared sea water synthetically in the lab and utilized it to flood composite core and added de-ionized water (DW). The composition of seawater and de-ionized water are listed in table 3. Then limestone core plugs were saturated with sea water using a Desiccator and later left them in the sea water for 16 hours to allow for full saturation before the commencement of the experiment. Pore volume and porosity of the cores were measured by using the weight difference between wet and dry core. We conducted the experiment at 122 °F reservoir temperature with 0.02 cc/min rate of injection and confining pressure set at 300 psi. 44,580 mg/l total dissolved solids (TDS) of sea water was used in the experiment whereas the DW has zero TDS as shown in table 3. This research was carried out with the aim of examining the dissolution mechanism while the injection of two phase flow of Soltrol-130, high salinity and low salinity water are being tested. In our experiment, we ensured that at the outlet no filter was used in a bid to make sure that the core was the sole origin of the fines and ended up in the effluent container. Moreover, this was to ensure the

| Table-2, Oil properties used in the experiment |
|-----------------------------------------------|
| **Soltrol Properties**                        |
| Viscosity (cp) @ 25°C                          | 1.176 |
| Density (gm/cc) @ 25°C                         | 0.7608 |

| Table-3, Composition of SW and DW.            |
|-----------------------------------------------|
| **Ions**                                      |
| Brine (mg/l)                                  |
| Sea water (mg/l)                              |
| de-ionized water (mg/l)                       |
| KCL                                          | 860   | N/A   |
| MgCl₂                                        | 5,875 | N/A   |
| Na₂SO₄                                       | 4,880 | N/A   |
| CaCl₂                                        | 1,385 | N/A   |
| NaCl                                         | 31,580| N/A   |
| TDS                                          | 44,580| N/A   |
| pH                                           | 5.85  | 6.5   |
| Viscosity at 50°C (cp)                       | 0.634 | 0.548 |
| Density (km/m³) at 50°C                      | 1032.7| 987.683|
concentration of ions in the effluent, which typically happens while core flooding takes place. We then used a water pump to flood the core composite with a low flow rate of about (0.02 cc/min) because the core is less permeable. In ensuring that water vaporization was avoided, the experiment was carried out at a temperature of 50 °C and to incorporate the effect of temperature; the reservoir temperature was mimicked. There was a drop in pressure, and we ended up setting final pressure at 300 psig. The figure 1 shows how the experiment was set up. There are two pumps connected into the Hassler core holder and these pumps were controlled by valve in order to use one for the water injection and the other for soltrol injection. Initially sea water was injected into the core plugs which were mounted in a Hassler core holder at least 10 PV and also 20.16 psi pressure drop across the core. Then, the absolute permeability of the composite core was calculated by the pressure drop. Soltrol was then injected at 50 °C with low flow rate (0.02 cc/min) by using oil pump in order to establish the irreducible water saturation experimentally. After that, brine was flooded into the core to 2 pore volumes at the same rate and temperature but using the water pump. We also sampled the effluent after an interval of breakthrough and measured the pH instantly. A closed lid was used to test the effluent to ensure that the ion concentration does not change as water vaporizes. It is important to note we stirred brine with evacuation to make sure that there was no air bubble retained in the brine. Accordingly, that was done to ensure minimization of carbon dioxide in the core before flooding the core. Postma and Appelo (2004) [7] argued that CO₂ can increase dissolution of the calcite as it can form carbonate acid through lowering its pH. Next, repeating the soltrol step with the same flow rate and pore volume injected to achieve the same initial water saturation. Then, we switched to de-ionized water flooding so the residual oil saturation was introduced at distilled water. We measured the volumes of produced oil and water at time. The oil recovery factor was measured as % OOIP. The next step of analysis of effluent water samples to quantify pH and the concentration of Ca^{2+}, Mg^{2+}, SO_{4}^{2-}, Cl^- and Na^+ ions using the ion chromatography and inductively coupled plasma optical emission spectrometry (ICP-OES). Soxhlet method was again used as cleaning method to remove the soltrol from the composite core by using 100% isopropanol for four hours only. The core plugs were then dried at 60 °C in the oven for 42 hours. The final step took images of SEM and EDS for the outlet and the inlet of the composite core. These were done after seawater injection was completed as well as injection of oil and de-ionized water. Figure-1 shows the experimental setup for the recovery.

Fig.1 Experimental setup of LSWI.
6. Results and Discussion
pH, effluents concentration, oil recovery and pressure drop results are shown in Figs. 2 – 9 for both the sea water and de-ionised water.

6.1 pH Measurements
The measurements of pH, active and non-active ions concentrations in the effluent and pressure drop curve examined the dissolution of calcite in this experiment. From figure-2 initially we have only one point due to two phase flow. In two phase flow when we did waterflooding, initially we only produce oil and to measure pH. We need to get a sample of water. During the sea water imbibition, the produced water was collected after breakthrough so in two phase flow experiment we cannot have lots of pH measurement. Moreover, the initial pH of the produced sea water recorded around 7.8 which was higher pH value than the injected SW. It can be clearly seen that CaCO₃ react with water throughout the core and kinetic dissolution of it was happening. In contrast, the pH went up to 9.2 through low salinity waterflooding but at the end of de-ionized water injection slightly decreased to around 8.8 which was not that much difference. This difference might be due to the dissolution was happening more at the beginning. According to the following equilibria

\[
\text{CaCO}_3 + \text{H}_2\text{O} \leftrightarrow \text{Ca}^{2+} + \text{HCO}_3^- + \text{OH}^-
\]

Calcite (CaCO₃) reacts with water, such reaction makes Ca²⁺ with OH⁻ and OH⁻ is the reason why pH is going up as well as the reason why we produce more cations, especially Ca²⁺ in the effluent due to calcium hydroxide is liquid. As a result, effluent pH of de-ionized water was higher than the effluent of SW due to active and non-active ions were presented in the injected SW. Sea water might illustrate increasing in the rate of kinetic dissolution.

Fig.2 Pore volume injected Vs pH of SW&DW production.

6.2 Effluent Concentration Data
The results obtained from the effluent concentrations for Ca²⁺, Mg²⁺, SO₄²⁻, Cl⁻ and Na⁺ are given in figures 3-7. Furthermore, cations especially Ca²⁺ and pH have increased in the effluent due to the dissolution. The concentration of Ca²⁺ in the effluent is recorded after breakthrough for both flooding SW and DW that is given in Fig-3. In the sea water there are some dissolution inhibitors that work to decrease the rate of dissolution in the limestone reservoirs like SO₄²⁻, Mg²⁺, Si, Sr, Ba, PO₄³⁻, metal ions and Ca²⁺ by itself (Morse and Arvidson, 2002) [8]. Preventing the interaction between the seawater and the surface
of calcite molecules could have happened when the metal ions are presented. However, the Ca\(^{2+}\) ion concentration in the effluent of sea water is recorded higher than that of de-ionized water due to the higher rate of dissolution in the sea water. Hence, when the initial Ca\(^{2+}\) ion and/or high salinity conducted, the kinetic of calcite dissolution takes place faster. It can be seen that the increase of pH in the effluent of low salinity water flooding could pertain not only to the dissolution but to other interaction took place inside the rock and/or some CO\(_2\) consumption related to the de-ionized water which was not vacuumed before flooding into the composite core. The difference between injected and effluent value of Ca\(^{2+}\) ion for SW was (150 mg/l) whereas in DW recorded less than the sea water by almost double (Fig-3). It can be concluded that dissolution in carbonate rocks occur higher during high salinity waterflooding than that in low salinity waterflooding. From Fig-4, the concentration of Mg\(^{2+}\) ion in the effluent and the injected one was identical in the sea water due to the small percentage of dolomite which represents only 3.4% in our sample. We did not have seen any change in Mg\(^{2+}\) ion concentration throughout high salinity water injection. Therefore, dolomite dissolutions definitely did not happen during high salinity injection. In contrast, in the low salinity the injected concentration of Mg\(^{2+}\) was zero but we still produced a lot of Mg\(^{2+}\) because there is dissolution of Mg\(^{2+}\). An anion might adsorb into the surface of rock at the same time reduce the positive charge of carbonate surface so it will increase the electrostatic attraction. From Figs-5 and 6 during injected sea water more SO\(_4^{2-}\) ion was reacted with the negatively charged carboxylic acid group at the carbonate surface. This reaction between an anion and carboxylic group leads to releasing several of the oil from the surface of carbonate as well as changing the wettability of rock to more water wet. For this reason, the concentration of sulfate and chloride ions in the effluent for high salinity was lower 10 times than the injected level. Therefore, the dissolution of calcite might increase during the sea water injection.

We have seen that Ca\(^{2+}\) and pH have increased in the effluent of SW due to the dissolution of calcite whereas SO\(_4^{2-}\) and Cl\(^{-}\) concentrations decreased. According to Zhang et al. (2007) [9], reducing of SO\(_4^{2-}\) and Cl\(^{-}\) concentrations into 10 times of the injected level caused by the existence of Mg\(^{2+}\) which is interacted with SO\(_4^{2-}\) at the early stage. It can be concluded that improving oil recovery was because of both the adsorption of ions especially SO\(_4^{2-}\) on the carbonate surface and dilution of the injected fluid.

![Fig.3](image-url)

**Fig.3.** Eflluent ion concentration for Ca\(^{2+}\) samples of SW & DW production
Fig. 4. Effluent ion concentration for Mg$^{2+}$ samples of SW & DW production.

Fig. 5. Effluent ion concentration for SO$_4^{2-}$ samples of SW & DW production.

Fig. 6. Effluent ion concentration for Cl$^{-}$ samples of SW & DW production.
6.3 Oil Recovery

The best results of oil recovery data for both high salinity and low salinity waterflooding are given in Fig-8. After flooding 2 PV of sea water, the recovery was recorded as 37% OOIP, while a higher cumulative oil recovery was recorded 50% OOIP after flooding about 8.26 PV of de-ionized water. There is around 13% difference corresponding to low salinity water injection. There are many reasons why recovery factor different? Initially the increase in pH was significantly higher in the de-ionized water injection than that of in sea water injection because of the dissolution of calcite. Another important reason is that due to the relative permeability is different and the residual oil saturation was less in low salinity by almost 12% than in high salinity. Therefore, we are improving water relative permeability by fines migrations which only happen in water so the oil relative permeability should be decreased in the low salinity waterflooding. As we used heterogenous composite rock in our experiment, the injected water goes to high permeable channels so the low permeable channels remain unsweep. When we reduce the salinity of the injected water, the water go to low permeable channels.

This would improve volumetric sweep efficiency. Hence, the oil recovery also would increase by decreasing the salinity of injected fluid.
6.4 Pressure Drop

The figure 9 down shows the measurements of pressure drop that were done throughout the core plugs with the pore volumes receiving an injection from sea water and de-ionized water. For sea water, the pressure drop decreased and almost stabilized at 11 psi. However, it went up to 22 psi in de-ionized waterflooding. This indicates that fines migration have taken place due to dissolution as shown by the SEM-images. Furthermore, the difference of pressure drop between high salinity and low salinity was almost double. So that’s mean relative permeability to water become half which should get better oil recovery. Eventually, despite the increase in pressure, it can be interpreted to be emanating from fines migration and dissolution. Pressure drop decrease in high salinity by dissolution can be compensated by increase in pressure drop by fines migration at low salinity.

![Fig.9, Pressure drop for Sea water and De-ionized water.](image)

6.5 SEM and EDS images

There were about 150 SEM and EDS images of both interfaces of outlet and inlet of the composite core. It is important to note that in most of these pictures, fines migration was detected. However, it was tough to identify and figure out the calcite and dolomite dissolution prior and after the flooding by EDS. As opposed to the discovery of Zahid et al. (2012) [10], dissolution mentioned early was confirmed using the experimental measurement of pH, Mg$^{2+}$ and Ca$^{2+}$ ions concentration in the samples of effluent. From these SEM and EDS images, we have managed to find out some fines migrations, which probably might have happened due to dissolution and therefore occurrence of fines dissolution from the rock. It is important to note that when fines migration occurs, the pressure drop is registered as the fines block some of the pores diverting the flow to a less permeable area. In comparison, pressure drop should be on the decline due to dissolution. Because these phenomena were evident in our experiment, we noticed a significant decrease in pressure. This is associated with enhanced pore throat connectivity. Pressure drop made significant effect and thus cannot be interpreted in an unambiguous manner, because dissolution and fines migration can compensate each other.

When it comes to the recovery of oil, both fines migration and dissolution may end up resulting in a rise in oil recovery in a significant manner. Fines migration blocks the throats of the large pores representing the primary flow of the fluid diverting it to an area with less unswept permeability.
Additionally, dissolution can restore an original water-wet carbonate system before aging of oil dissolves and removes the oil-wet layer on the surface of the carbonate rock [11].

7. Conclusion
With the above findings, the following conclusions can be drawn.

- Both sea water and de-ionized water have the capacity to dissolve carbonate rock as it was evident when the concentration of Ca$^{2+}$ and pH increased. The higher the salinity rate, the higher the rate of dissolution, although to some degree the dissolution of rocks stops after attaining the equilibrium.

- When dissolution induces pH increase, it can have positive effects on how wettability is altered to a more water-wet through conversion of the flood to alkaline, therefore, higher oil recovery was noticed.

- The rate of dissolution of carbonate is a gradual process and equilibrium may not be attained when kinetic dissolution takes place, even at a slow rate of flow (0.02 cc/min).

- Dissolution of carbonate may induce fines dissolution from the surface of the rock and on fines migration as shown in the SEM and EDS images.

- The drop increased in pressure, because of fines migration which was improving water relative permeability.

- Both the dissolution of the rock and the fines migration that emanate from dissolution induction may significantly affect the oil recovery from the carbonate rocks. Fines migration sometimes blocks the throats of the large pore and changes the flow to an area with less permeability. Moreover, the dissolution may end up rendering the surface of the rock as more water-wet. Both mechanisms, however, have the capacity to attain higher recovery of oil.

Appendix

SEM and EDS images:

The Inlet Interface
Abreviations:

- LSWI: Low Salinity Water Injection
- EOR: Enhanced Oil Recovery
- SW: Sea Water
- DW: De-ionized Water
- H.S: High Salinity
- L.S: Low Salinity
- SEM: Scanning Electron Microscope
- EDS: Energy Dispersive Spectroscopy
- EWI: Engineered Water Injection
- OOIP: Original Oil in Place
- PV: Pore Volume
- TDS: Total Dissolved Solids
- So: Oil Saturation
- CaCO₃: Calcite
- CaMg(CO₃)₂: Dolomite
- Ca²⁺: Calcium
- Na⁺: Sodium
- Mg²⁺: Magnesium
- SO₄²⁻: Sulfate
- Cl⁻: Chloride
- Sor: Residual Oil Saturation
- Swirr: Irreducible Water Saturation

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