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WETTABILITY EVALUATION IN PRE-SALT CARBONATES USING TUNED WATER

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Abstract. *Tuned water injection (TWIN) method has been proved to increase the oil recovery in reservoirs. Considering, that the pre-salt carbonates present high heterogeneity, it was believed that the technique would not work in this type of rock, the predominant oil to mixed wettability, associated with low permeability matrix and high fracture density, being major challenges to the application of the method. The wettability alteration is one mechanism that could increase the oil recovery, where the rock wettability alters to a more water-wet condition due to TWIN, with the presence of the potential determining ions (PDI). This work evaluates the wettability alteration through oil recovery, with the qualitative technique of forced displacement. The tests were performed with carbonate reservoir samples at pre-salt conditions of pressure and temperature. Three tests were conducted to analyze the wettability behavior in terms of secondary, tertiary, and ultimate recovery. This work presented prominent results regarding the effects of tuned water inducing the wettability alteration in carbonate reservoirs. Important factors as brine composition, chemical alterations, and petrophysical parameters can have influenced the results in terms of oil recovery and led to divergent values of recovery during the wettability evaluation.*

Keywords: *Enhanced Oil Recovery, Wettability Alteration, Low Salinity Water Injection, Carbonates, Forced Displacement.*

1. INTRODUCTION

The tuned water injection (TWIN) method mainly consists of the addition or removal of specific ions from the injection water. Due to the low operating cost, low capital investment, environmentally friendliness compared to other chemical flooding methods, the technique is attractive in the oil industry (BARADARI *et al.*, 2020). Considering that more than 60% of the world's oil reserves are from carbonate reservoirs, researchers have conducted a number of studies regarding the effect of the TWIN in carbonates (DERKANI *et al.*, 2018).

It was first believed that the method would not work in carbonates given the absence of certain minerals and clays. The carbonates have a complex nature and present high heterogeneity and multimodal porous systems; these are not the only challenges for the application of the technique since the wettability is predominantly oil and mixed-wet (AL SHALABI *et al.*, 2013). The wettability alteration is one mechanism that could increase the oil recovery, since a previously oil-wet rock should change to a more water-wet condition. The potential determining ions (PDI) mechanism, in which the concentration of specific ions is altered, is the most common technique to explain the wettability alteration phenomenon (AUSTAD *et al.* 2015; 2013; 2012; 2005). Qualitative and quantitative methods are used to measure the wettability condition and alteration. The most common quantitative techniques are the contact angle (AHR, 2008), Amott (AMOTT, 1959), and the United States Bureau of Mines (USBM) (TIAB & DONALSON, 2004). In terms of qualitative methods, the most used are spontaneous imbibition (MORROW & MASON, 2001) and forced displacement (LIMA, 2016). In this work, the wettability alteration analysis occurs through the evaluation of oil recovery graphs obtained from forced displacement experiments considering the rock/fluid system, which is similar to the work conducted by Agbalaka *et al.* (2009).

The present study was performed using pre-salt carbonate reservoir samples from the Santos Basin in Brazil, and evaluated the wettability behavior and alteration as a result of the oil recovery due to the TWIN.

2. EXPERIMENTAL METHODOLOGY

2.1 Sample Characterization

The samples used during the experiments were from the Brazilian pre-salt, specifically the Barra Velha formation from the Santos Basin. The carbonates in this location present high heterogeneity, which can affect the oil recovery, considering it is difficult to predict the behavior of the fluid in the sample (BASSO *et al.*, 2020). The rock samples are from three different facies and were chosen for their representativeness in the field under study. Table 1 shows the rock properties, such as length (L), diameter (D), dry mass (m), porosity (Φ), pore volume (PV), and permeability (k).

Table 1. Core sample parameters.

Sample ID	**L (cm)**	**D (cm)**	**m (g)**	**Φ (%)**	**PV (cm^3)**	**k (mD)**
BM – 1	7.5	3.8	177.0	25.0	21.3	145
BM – 2	7.5	3.8	208.8	10.3	8.9	30.3
BM – 3	7.5	3.8	202.7	13.1	11.2	44.1

2.2 Fluid Preparation

The fluids used during the experiment consist of formation water (FW), seawater (SW), tuned water (TW), crude oil, and recombined oil. Table 2 shows the chemical composition of the brines, in which the tuned waters are $SW_{(x10)}$, $SW_{(x10)+SO_4^{2-}}$, SW_{Ds}, $SW_{(x2)Ds}$, and $SW_{(x10)Ds}$, respectively, seawater ten times diluted, seawater ten times diluted with the addition of sulfate, desulfated seawater, and desulfated seawater two and ten times diluted.

Table 2. Chemical Composition of Brines in Salts Concentration (g/L).

Brine Composition	**FW**	**SW**	**$SW_{(x10)}$**	**$SW_{(x10)+SO_4^{2-}}$**	**SW_{Ds}**	**$SW_{(x2)Ds}$**	**$SW_{(x10)Ds}$**
HCl (mL)	0.87	-	-	-	-	-	-
NaCl	217.64	23.47	2.35	2.35	23.47	11.74	2.35
$CaCl_2.2H_2O$	13.43	1.47	0.15	0.15	1.47	0.73	0.15
$MgCl_2.6H_2O$	4.95	10.55	1.05	1.05	10.55	5.28	1.05
$SrCl_2.6H_2O$	9.49	0.04	0.01	0.01	0.04	0.02	0.01
$Na_2B_4O_7.10H_2O$	1.40	-	-	-	-	-	-
KCl	5.34	0.72	0.07	0.07	0.72	0.36	0.07
LiCl	0.56	-	-	-	-	-	-
$BaCl_2.2H_2O$	0.05	-	-	-	-	-	-
KBr	1.41	-	-	-	-	-	-
Na_2SO_4	0.09	3.90	0.39	4.29	0.10	0.05	0.01
$NaHCO_3$	1.29	0.19	0.02	0.02	0.19	0.09	0.02
TDS	**245.22**	**34.18**	**3.44**	**7.34**	**30.55**	**15.28**	**3.06**

The crude oil used in the experiments is from a pre-salt field located in the Santos Basin. Table 3 shows the crude oil parameters measured at reservoir conditions.

Table 3. Crude Oil Parameters.

Parameter	**Measurement**
Density [g/L]	865.80
Viscosity [cp]	21.12
API Gravity [°API]	23.94

Table 4 shows the results of the SARA (saturated, aromatic, resins, and asphaltenes) analysis. The high percentage of saturates indicates that the oil is categorized with high paraffinic content.

Table 4. SARA Analysis (%, m/m).

Hydrocarbon Fraction	Results
% Saturates	90.06
% Aromatics	2.69
% Resins	3.19
% Asphaltenes	4.76

To mimic the reservoir conditions at a laboratory scale, the recombined oil is crucial. It consists of combining crude oil with a simplified gas composition based on the PVT (pressure, volume, and temperature) data of the gas found in the field; the simplified commercial gas used is shown in Table 5.

Table 5. Commercial Gas Composition.

Component	Gas Concentration (mol%)
CO_2	28.09
N_2	0.48
C1	60.28
C2	4.57
C3	3.74
iC4	2.84

The preparation of the recombined oil is based on the pressure (above to 8000 psi), temperature (69 ºC), and gas-oil ratio (187.7 std m^3/std m^3) in-situ. To prepare one liter of recombined oil under these conditions, a total of 500 mL of crude oil and 185.4 mL of the simplified gas composition was needed. The preparation begins with the transfer of the gas into the container. A high-pressure displacement pump was used to transfer the gas to the crude oil container. The oil container was covered with a heating blanket to provide reservoir temperature conditions. During this part of the experiment, it is critical to maintain the temperature constant because, if a drop-in temperature occurs, the saturation pressure will also drop and the gas will disassociate from the oil. After the gas transfer, the container is removed, and the oil container is allocated in a mechanical agitator with constant pressure for 24 hours.

At the start of the coreflooding experiments, it is crucial to build the system's pressure to achieve reservoir conditions. So, crude oil was first injected into the rock until the system conditions were stable. Recombined oil was then injected into the system (already pressurized at 8270 psi). Roughly 5 pore volumes were flowed through the rock sample to obtain the analogous condition of the reservoir.

2.3 Sample Preparation

The first stage of preparation was core cleaning. It is crucial to remove any contaminant in the porous medium, such as hydrocarbon, drilling fluids, and inorganic substances. According to the API RP 40 (2010), the selection of which solvent use is important since it should not alter and/or destroy the structure of the sample. The technique applied was the flow-through cleaning system, where the sample is assembled in a core-holder and the solvents are injected under pressure into the core. It is also possible to inject the solvent continuously or periodically and under ambient or elevated temperatures. During the performance of the process, the solvents used were low aromatic kerosene, heptane, and distilled water. These solvents are considered mild-cleaning because they reduce the solubility of larger oil components due to their low aromaticity. Their injection purpose is to displace the residual oil in the sample without promoting the substantial removal of the polar organic compounds adsorbed to the rock surface, preserving the rock's original wettability (PIÑEREZ *et al.*, 2020). Finally, the core was dried at 90ºC until reached a constant weight.

The second stage of preparation was obtaining the initial water saturation (Swi) of the samples. All core samples were placed in an accumulator and vacuumed for at least 8h. Then fully saturated with FW. The FW saturation (S_{FW}) occurs in two stages, the first is through gravitational drive (the water will penetrate the cores until the samples are saturated). To assure the S_{FW} condition, the second stage is a pressurized saturation in which FW is continuously injected under constant pressure for 24h. With the samples saturated with FW, the Swi process initiates. Due to the crude oil, which presents high paraffinic content, it was necessary to heat the system with heating blankets to maintain the temperature at 69 ºC. The process started with the injection of 2 PVs of FW to confirm the saturation condition of the samples. Subsequently, 3 PVs of crude oil were injected until the water production was stable. The displaced volumes were quantified in glass graduated cylinders. The Swi was calculated using Eq. (1) for each sample, where V_{WP} is the volume of produced water (achieved with Eq. (2)), and PV_{total} is the sample's total pore volume.

$$S_{Wi} = \frac{V_{WP}}{PV_{total}} - 1 \quad (1)$$

$$V_{WP} = V_{GC} - V_D \quad (2)$$

Where, V_{GC} is the volume of water displaced by the oil accumulated in the graduated cylinder, and V_D is the dead volume of water contained in the diffuser, valves, and flow lines.

The last stage is the wettability restoration. Steel cells were used to restore the wettability condition of the samples. The cells containing the samples in the Swi condition were filled with crude oil and submitted to 90ºC for at least 14 days (GRAUE *et al.*, 1999).

3. COREFLOODING EXPERIMENTS

The qualitative method of coreflooding was used during the performance of the experiments, and the objective of the technique is to evaluate the recovered volume after each stage of the brine's injection.

Figure 1 illustrates the apparatus constructed to evaluate the wettability behavior through the oil recovery at reservoir conditions of pressure (above to 8000 psi) and temperature (69 °C). During the tests, the overburden pressure (P_{ovb}) settled at 9200 psi. Approximately 1000 psi pressure differential between the injection pressure and overburden pressure is important to maintain a safety margin to avoid the communication within the core-holder. If the P_{ovb} is lower than the P_{inj} the overburden water will saturate the sample. The fluid flow rate was 0.1 cm³/min. To obtain a more precise reading of the produced oil in the separator, a digital camera was installed in front of the separator and took pictures in a pre-defined time interval to follow and record the production over time. Measurements of the differential pressure in the sample were also obtained during the test.

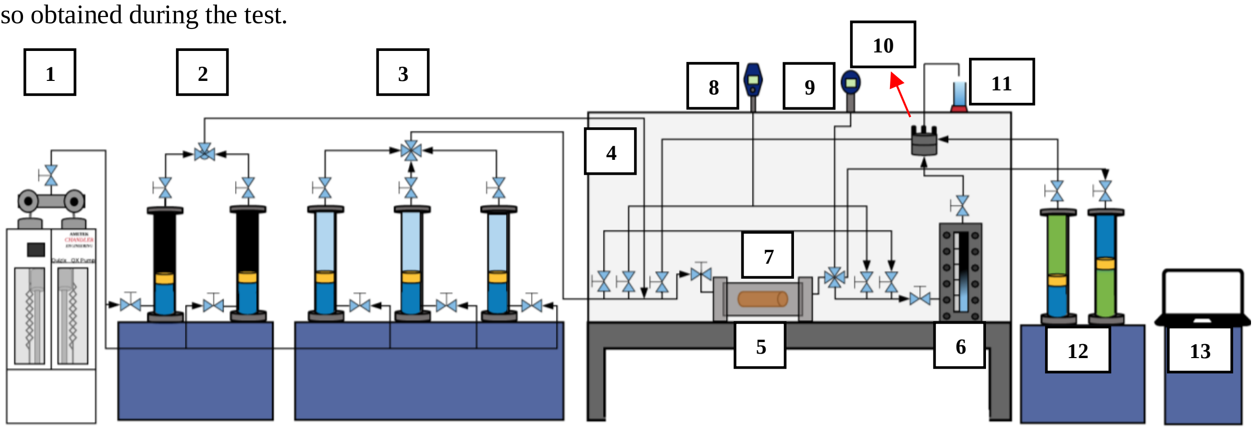

Figure 1. Experimental apparatus schematics.

The enumeration presented in Figure 4 represents, respectively:

1. Quizix QX pump
2. Oil injection system (CO and RO)
3. Water injection system
4. Oven
5. Core-holder
6. Biphasic Separator
7. By-pass
8. Differential pressure transducer
9. Manometer
10. Backpressure valve
11. Production system
12. Nitrogen injection system
13. Data acquisition system

4. RESULTS AND DISCUSSION

The tests provide the results as a function of oil recovery as original oil in place (% OOIP) and the differential pressure (psi) measured in the samples for the different brines, versus the pore volume injected (PVi).

Reactive and non-reactive fluids are used to evaluate the condition and the wettability alteration during the performance of the experiments. FW was used as a non-reactive fluid because it will not disturb the system`s equilibrium since, in the reservoir, the rocks contain FW and oil. The FW injection aims to produce the mobile oil until the balance of capillary forces in the water/oil/rock system is broken, simulating a primary recovery. Therefore, the FW injection will provide the first reading of the original wettability in the rock previous to the injection of the reactive fluids. The purpose of the reactive fluids (brines with different ionic composition than FW) is to break the initial chemical equilibrium of the water/oil/rock system, then additional oil will be produced through the wettability alteration.

Distinct scales of production can be reached in the experiments due to the different rock's mineralogy, facies, and type of carbonate reservoirs since the samples are extremely heterogeneous, considering that the composition of the carbonate rocks is strongly linked to wettability.

Figure 2 illustrates the set of experiments performed and the brines utilized during each test. SW is used as a secondary method, in Test 3, to evaluate its potential as a wettability modifier at the start of the injection process.

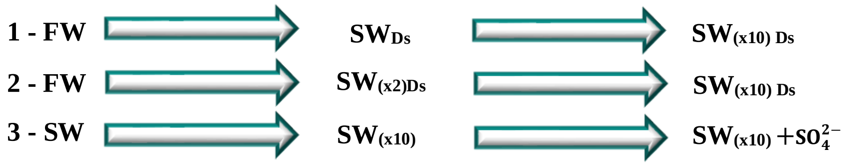

Figure 2. Set of experiments.

Figure 3 illustrates the results obtained in Test 1, which was performed with the sample BM-3. The test yielded a recovery factor (RF) of 17.5% (OOIP) with the injection of 22 PVs. Table 6 shows the rock sample properties.

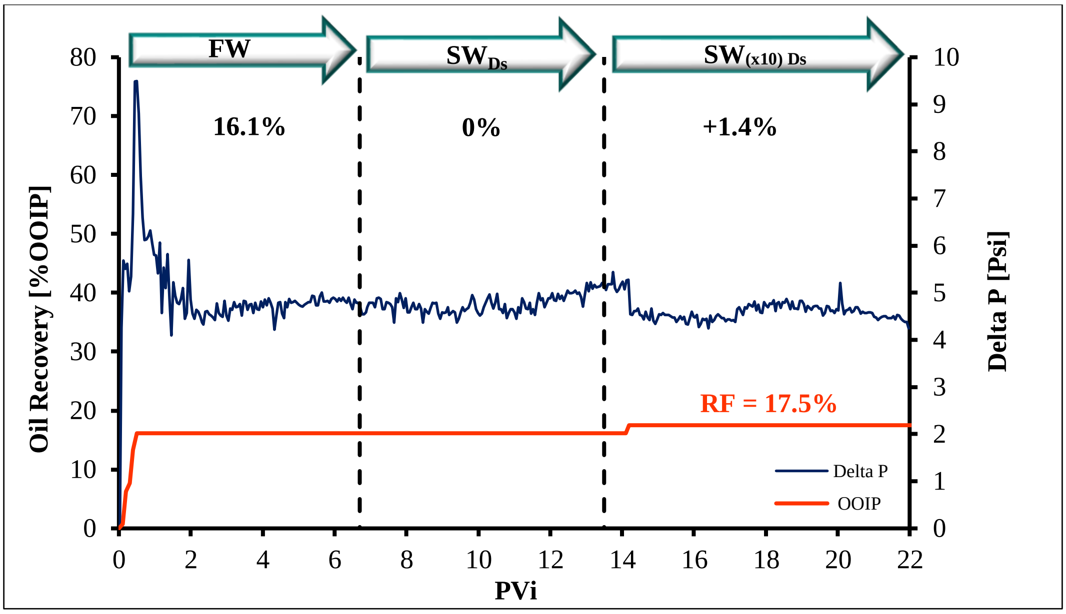

Figure 3. Test 1: Oil recovery and pressure differential x pore volume injected.

Table 6. Sample BM – 3.

Sample ID	Φ (%)	PV (cm³)	k (mD)	Swi (%)
BM – 3	13.1	11.2	44.1	37.62

The first recovery can be noted around 0.6 PVi with FW, which achieved the production plateau at 16.1% (OOIP), and the injection continued until 6.6 PVs to assure that the residual oil saturation was reached. The oil's polar compounds may have contributed to the low value of recovery, in which the sample wettability presented preference to oil. The injection of SW_{Ds} as a tertiary method did not present any increment in production, as an addition was observed in the ultimate recovery using $SW_{(x10)Ds}$.

The absence of increment in production during the SW_{Ds} injection can be correlated with the small quantities of sulfate in the solution and, along with the brine composition, was not enough to break the system equilibrium and alter the rock's wettability. As shown in the mechanism proposed by Austad *et al.* (2012) the sulfate acts as a catalyst to the process of ionic exchange, which promotes wettability alteration.

In altering the brine composition, $SW_{(x10)Ds}$ promoted an additional recovery of 1.4% (OOIP). Though according to Zhang & Sarna (2012), recoveries until 1.8% can be associated with operational errors. Furthermore, no increments in recovery were observed when utilizing $SW_{(x10)Ds}$ as the ultimate recovery method.

The pressure drop curve presents a spike at the beginning of the injection process and, according to Seyyedi *et al.* (2018), this increase followed by a decrease in pressure can indicate the moment that the FW started to flow. Also, the almost constant behavior during the performance of the test can be correlated to the constant flow rate; the behavior can still be associated with the rock's low permeability and may indicate that the injection sequence does not cause any alteration in the fluid/rock system.

Figure 4 provides the results of Test 2 using the sample BM-2. During the test, 18.6 PVs were injected to observe the system behavior with the alteration of brines. The first water (FW) was the only one to produce oil, achieving a recovery of 11.5% (OOIP) at the beginning of the test at 0.2 PV. Table 7 shows the properties of the rock sample.

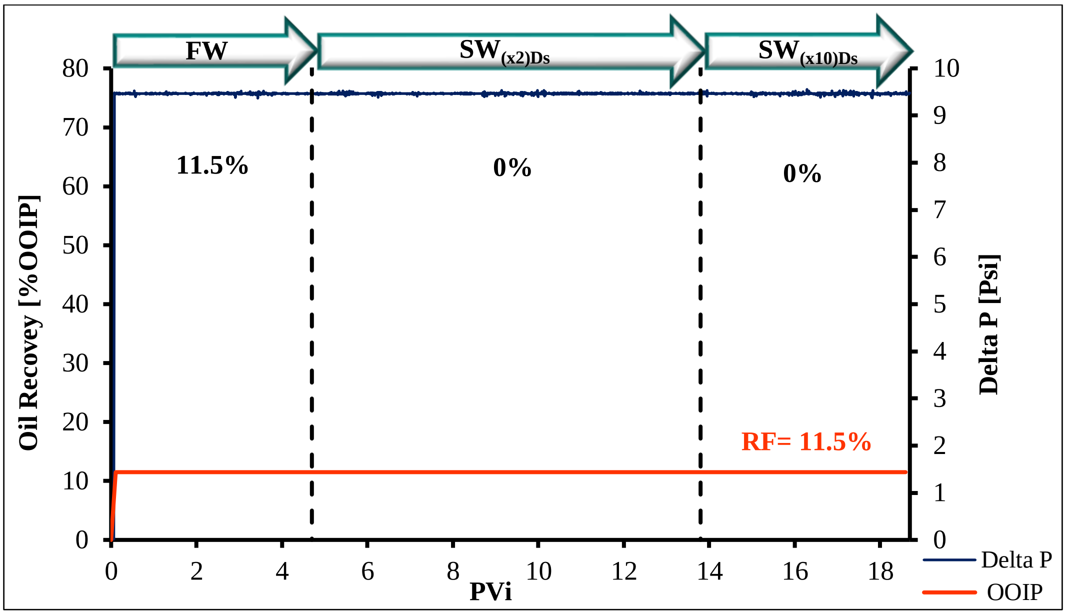

Figure 4. Test 2: Oil recovery and pressure differential x pore volume injected.

Table 7. Sample BM – 2.

Sample ID	Φ (%)	PV (cm³)	k (mD)	Swi (%)
BM – 2	10.3	8.85	30.3	46.88

The initial production could be associated with the injection of FW, which simulates the primary recovery of a reservoir. With the injection of the second and third brine, the production was kept constant. In addition, the oil's polar compounds may have contributed to the low oil recovery that can be an indicator of the rock wettability's strong preference for oil.

When altering the injection brines to the ones with lower salinities, no additional oil was achieved. Is possible to correlate this behavior to the lack of sulfate (usually observed as anhydrite in carbonate reservoirs) originally presented in the formation that tends to present a more strongly preference to water (ARGYRI, 2017). Also, the small quantity of sulfate present in the $SW_{(x2)Ds}$ and $SW_{(x10)Ds}$ may have contributed to the non-break of the system equilibrium that could induce the wettability alteration to a more water-wet state. As mentioned, the sulfate acts as a catalyst for the process that promotes the ionic exchange and induces wettability alteration.

In the pressure drop curve, is possible to observe a relatively constant behavior kept around 9.5 psi during the test development. This occurred due to the low permeability of the sample that extrapolates the reading capability of the transducer.

Figure 5 shows the results of Test 3 with the BM-1 sample (Table 8). During the test, 18.6 PVs were injected with the hopes to confirm the production stabilization and that the residual oil saturation would be achieved. The SW injection, as a secondary method, obtained a recovery of 54.7% (OOIP) and, in the second fluid injection, $SW_{(x10)}$, was utilized and an increment of 5.6% (OOIP) was observed in the production. As the ultimate recovery, a combination of diluted seawater with the addition of sulfate ion was tested, although no additional oil was recovered, thus achieving a final recovery factor of 60.3% (OOIP).

Divergent values of recovery were obtained with the injection of SW as the first fluid, the recovery of 54.6% (OOIP) is much higher than that achieved with FW in Tests 1 and 2. The higher recovered value can be a result of the water composition, considering that FW is in equilibrium with the system. The higher concentration of sulfate and the lower concentration of NaCl in the SW could be an important factor when considering the electrical double layer effect. Fathi *et al. (*2011) and Liu &Wang (2020) affirm that the decrease in the non-active ion concentration enables the more prominent action of the active ions towards the rock surface. Also, Mamonov *et al.* (2019) state that the higher production can be a result of the injection of a reactive fluid (SW) as a secondary method, which may have induced the wettability alteration (1.7 to 2.5 PVi) to a more water-wet state due to the break in the system equilibrium. Another factor to highlight is the rock mineralogy; in the case of the sample BM-1, the rock matrix presents a more homogeneous aspect with higher values of permeability and porosity, which may have contributed to the higher value of recovery.

The increment achieved with $SW_{(x10)}$, between 8 and 10 PVi, could have occurred because, when injecting a brine with lower salinity, a desaturation of the non-active ions of the rock surface occurs, which favors the wettability alteration given the presence of active ions. Authors as Austad *et al.* (2013; 2012; 2005) and Zhang *et al.* (2006a) conducted studies that have proved the influence of the sulfate ion acting as a catalyst in the process of wettability alteration. However, the results obtained in Test 3 with a higher concentration of sulfate in the $SW_{(x10)+}SO_4^{2-}$ conflict with the previous studies since this addition did not present any increase in recovery. This raises the idea that perhaps the sulfate concentration was not increased enough to induce an increment in production, or the critical value was achieved (AL-ATTAR *et al*., 2013).

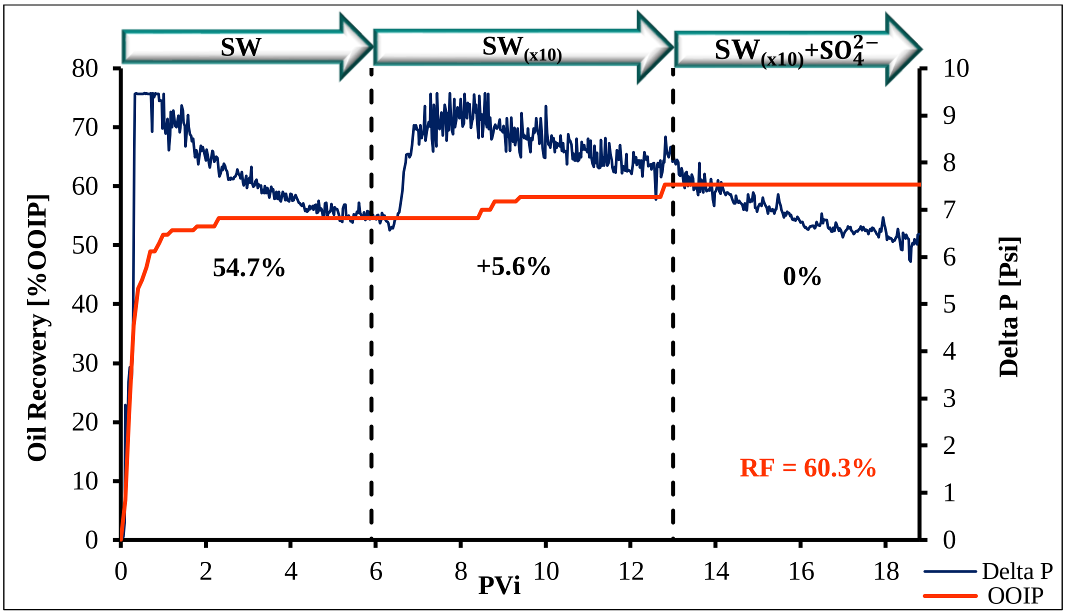

Figure 5. Test 3: Oil recovery and pressure differential x pore volume injected.

Table 8. Sample BM – 1.

Sample ID	Φ (%)	PV (cm³)	K (mD)	Swi (%)
BM – 1	25.0	21.3	145	34.59

The spike followed by the decrease in pressure at the beginning of the test indicates the moment that the FW started to flow in the rock sample. Another increase is observed after the brine change, which may indicate that the chemical interaction between the rock and the new brine elevated the pressure due to the wettability alteration of the rock to a more water-wet condition.

5. CONCLUSION

- In secondary mode, SW was more efficient than FW presented a higher value of recovery. This could be associated with the increase in the sulfate concentration and reduction of NaCl in the SW, which induced a wettability alteration at the start of the test.
- In terms of tertiary recovery, $SW_{(x10)}$ proved to be the more effective solution, inducing the wettability alteration to a more water-wet state and promoting an increment of 5.6% (OOIP) in recovery for Test 3, while SW_{Ds} and $SW_{(x2)Ds}$ did not present any additional oil on Tests 1 and 2, respectively.
- As an ultimate recovery, the injection water that presented the potential to induce a break on system equilibrium and promote the wettability alteration was the $SW_{(x10)Ds}$, though is not possible to conclude that the 1.4% obtained was due to the water composition or because of operational errors.

6. ACKNOWLEDGMENTS

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