Experimental Study on Enhanced Oil Recovery by Low Salinity Water Flooding on the Fractured Dolomite Reservoir

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ABSTRACT: *Enhanced Oil Recovery from carbonate reservoirs is a major challenge especially in naturally fractured formations where spontaneous imbibition is a main driving force. The Low Salinity Water Injection (LSWI) method has been suggested as one of the promising methods for enhanced oil recovery. However, the literature suggests that LSWI method, due to high dependence on rock mineralogy, injected and formation water salt concentration, and complexity of reactions is not a well-established technology in oil recovery from carbonate reservoirs. The underlying mechanism of LSWI is still not fully understood. Due to lack of LSWI study in free clay dolomite fractured reservoir, and to investigate of anhydrate composition effect on oil recovery in this type of reservoir, the main purpose is the experimental evaluation of oil recovery from one of the Iranian naturally fractured carbonated (dolomite containing anhydrate and free clay) reservoirs using LSWI. For this purpose, a set of experiments including spontaneous and forced imbibition is conducted. To obtain the optimum salt concentration for oil recovery, the secondary mode of the spontaneous imbibition tests is performed by seawater in various salt concentrations at the reservoir temperature (75°C). Also, the tertiary recovery mode is subsequently applied with optimum brine salinity. The lab results reveal that by decreasing the injected water salt concentration, oil production increases. Furthermore, in order to upscale the experimental results to the field scale, a more precise dimensionless-time correlation is used. Due to some inconsistencies over the influence of mechanisms on LSWI oil recovery, the mineral dissolution, pH-increase mechanisms, and wettability alteration are also studied. The results indicate that wettability alteration is the main mechanism and mineral dissolution may be the predominant mechanism of the improved oil recovery in the studied reservoir. It is noticed, the elevation of pH led to enhanced oil recovery when high dilution of low salinity water is implemented.*

KEYWORDS: *Low salinity; Naturally fractured dolomite reservoir; Wettability alteration; Spontaneous imbibition; Mineral dissolution; pH increase.*

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INTRODUCTION

More than half of the world's oil reserves are stored in carbonate reservoirs [1]. Due to heterogeneity, the complexity of minerals (limestone, chalk, and dolomite), low permeability, and wettability condition, Enhanced Oil Recovery (EOR) methods for such reservoirs are challenging. Because of the inherent oil-wet nature of the carbonate rock, a large volume of oil remains in the matrix with injected water failing to flush most of the oil from the pore spaces [2]. Hence, it is necessary to increase the potential of oil production through wettability alteration by EOR methods such as additive chemicals, nanomaterials, and low-salinity water.

The low salinity water injection method has been suggested as one of the promising methods for enhanced oil recovery. However, the literature suggests that LSWI method, due to high dependence on rock mineralogy, injected and formation water salt concentration, and complexity of reactions, is not a well-established technology in oil recovery from carbonate reservoirs, and the underlying mechanism of LSWI is still not fully understood. Due to this result in this work, LSWI in a fracture carbonated reservoir is studied.

Austad et al. (2011) attributed easy accessibility of active ions to the rock surface due to salt concentration reduction causes increase of oil production in LSWI in the carbonated reservoir [3].

Mahani et al. (2015) found that the low salinity EOR effect was influenced by the mineralogy of the rock where the effect was mostly caused by rock-brine and rock-oil interaction rather than brine-oil interaction [4]. Based on this result, our study is focused on the evaluation of the low salinity water effect on rock (such as wettability change, dissolution, and precipitation process). The spontaneous imbibition (Sp. imbibition) process is one of the major recovery mechanisms in where most of the oil is stored in a low permeability matrix block in the fractured reservoir [5]. Some Sp. imbibition researcher results on carbonated samples are reviewed in this part of the study. *Shariatpanahi et al*. (2016) performed some Sp. imbibition tests on dolomite plug sample with diluted seawater (10 and 100 times) [6]. They observed that 10-time dilution succeeded to increase the recovery factor by 15%, while 100-time dilution did not change the oil recovery significantly. *Zaeri et al.* (2018) performed a set of Sp. imbibition tests on limestone plug samples and observed that the highest oil recovery occurred in the 20-time dilution of seawater while the lowest was found in the 40-time dilution [7]. Hence, improved oil recovery by LSWI is not a direct function of the degree of salt concentration reduction but *Austad et al*. (2011) indicated oil recovery in carbonate reservoirs increases when water salinity concentration decreases.

The results obtained from some studies suggested that at 70°C oil recovery in Sp. imbibition by LSWI in chalk /limestone ($CaCO_3$) plugs is more effective than in dolomite plugs ($CaMg(CO_3)_2$) [3,4]. In this study comparison of low salinity water, Sp.imbibition test results on limestone and dolomite are in line with this.

Table 1 summarizes the results of low salinity Sp. imbibition on carbonated reservoirs found in the literature. To the best of our knowledge, most LSW Sp. imbibition experiments have been performed on limestone/chalk core samples.

Several mechanisms have been suggested to explain the performance of LSWI in all types of reservoirs [8]. However, there is still no general agreement on the underlying mechanisms [9]. Mechanisms such as wettability alteration (the main underlying mechanism) and other proposed mechanisms can be categorized into the solid-liquid interface (mineral dissolution, pH effect, fines migration, multiple ion exchange, double layer expansion, and salt-out) or liquid-liquid interface (osmosis effect, IFT, micro-dispersion) have been presented to describe the incremental oil recovery in LSWI. Table 2 reports some literature about the LSWI oil recovery mechanisms. In this work, some mechanisms such as wettability alteration (as the main mechanism in LSWI), mineral dissolution, and pH effect are reviewed and experimental studied.

Zhang and *Morrow* (2006) showed that the key reactions in LSWI on the carbonated rock are calcite dissolution (Eq. (1)) and cationic change (Eq. (2)) by producing OH^- or increasing the pH value. Based on these reactions, the oil droplets attached to the rock surface can be desorbed from the surface [10].

$$CaCO_3 \rightarrow Ca^{2+} + CO_3^{2-} \tag{1}$$

$$CO_3^{2-} + H_2O \rightarrow HCO_3^- + OH^- \tag{2}$$

Table 1: Sp. imbibition in various dilution Salinity IW of seawater (SW) or formation water (FW) for LSWI in carbonates.

Author	Rock type	T (°c)	Imbibition type	Salinity IW	Salinity FW	μ_{oil}(cp)	AN* (mg KOH/ gr oil)	Injection model	RF%
Zhang and Austad 2005[11]	Chalk	40-120	Sp. imbibition	33390 ppm	68000	2.5-3	0.17-2.07	Secondary	1.1-65.3
Yi& Sarma, 2012[12]	Limestone fracture	70	Sp. imbibition	40 ×SW (1090 ppm)	201022	1.674 @ 70°c	-	Tertiary	21
Al-Harras et al. 2012[13]	Limestone fracture	70	Sp. imbibition	100×FW	193230	10.2 @ ATM	-	Secondary	22
Karimi et al. 2016[14]	Limestone fracture	75	Sp. imbibition	FW	196000	9.3 @ ATM	0.37	Secondary	10
				100× FW					58
Rashid et al. 2015[15]	Limestone fracture	75	Sp. imbibition	FW	202150	1@ 70°C			2
				SW			0.58	Secondary	21
				2×SW				Tertiary	8
Shariatpanahi et al. 2016[6]	Dolomite fracture	70	Sp. imbibition	10×SW (33430 ppm)	222190	20.8@ ATM	0.52	Tertiary	15
Zaeri et al. 2018[7]	Limestone fracture	75	Sp. imbibition	20× SW (1463 ppm)	-	5.04@ 75 °C	0.1	Secondary	13.9

**AN is the acid number of the oil that is dependent on oil carboxylic acid ions*

Table 2: A literature review of mechanisms in LSWI on the carbonated reservoir.

Mechanisms	Researchers
Wettability alteration	*Standness &Austad* 2000; *Strand et al.* 2006; *Hognesen et al*. 2005; *Webb et al.* 2005; *Mohani et al*. 2015; *Wickramathilaka* 2011; *Emadi* and *Sohrabi*, 2013; *Mahani et al.* 2013; *Al-Shalabi et al.* 2014; *Alameri et al.* 2014; *Shariatpanahi et al.* 2016; *Zaeri et al.* 2018[16,17,18,19,4,20,21,22,1,5,6,7]
Mineral dissolution	*Hiorth et al.* 2010; *Yousef et al.* 2011*; Mahani et al*. 2015; *Pu et al*. 2010; *Qiao et al.* 2015[23,24,4,25,26]
pH effect	*Austad* 2013; *Zaeri et al*. 2018[27,7]
Fine migration	*Zahid* 2012; *Hamouda* and *Valderhaug* 2014[28,29]
Multiple ion exchange	*Safavi et al*. 2019[30]
Double layer expansion	*Lighthelm et al*. 2008[31]
Osmos effect	*Sandengen* & *Arntzen* 2013[32]
IFT reduction	*Yousef et al*. 2011[24]
Microdispersion	*Emadi* and *Sohrabi* 2013; *Bartels et al.* 2019[21,33]
Salt effect	*Rezaeidoust et al*. 2009[34]

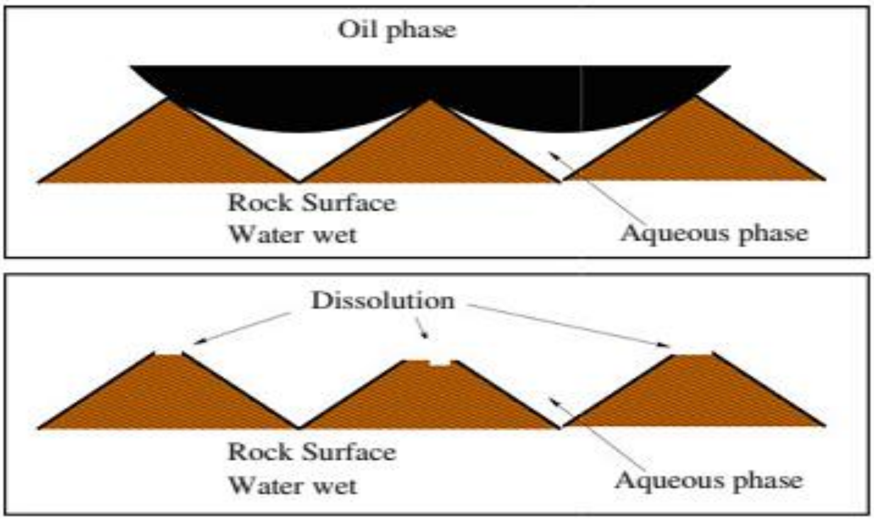


Fig. 1: Schematic of the proposed rock dissolution mechanism ([23] with permission)

Austad et al. (2011) attributed the OH^- elevation in LSWI to the replacement of H^+ with Ca^{2+} adhered to the clay of the surface of the rock (Eq. (3))[27].

$$Ca^{2+} - Clay + H_2O \rightarrow H^+ - Clay + Ca^{2+} + OH^- \quad (3)$$

Zhang et al. (2007), *Austad* (2013), and *Zaeri et al.* (2018) showed that there is no clear relationship between pH value and oil recovery [5, 27,73]. They observed that the pH-increase mechanism may not be effective for LSWI on carbonated rock as pH value depends on chemical reactions.

Aksulu et al. (2012) stated that in LSWI, pH was affected by the presence of calcite and anhydrite in the core structure where the dissolution of calcite and anhydrite from the core surface into water affected the pH value in brine [36]. *Chandrasckar* and *Mohanty* (2018) concluded that elevation of pH caused decreased adhesion forces for seawater and in turn, increased oil recovery [37].

Hiorth et al. (2010) observed that the dissolution of calcite could enhance water wetness in LSWI. The amount of calcite dissolved appears sufficient to account for the extra oil production, especially if, as expected, the calcite is preferentially dissolved exactly where the oil wets the calcite (Fig. 1) [23]. *Mahani et al.* (2015) observed that oil recovery can be enhanced without mineral dissolution mechanism in LSWI on the carbonated surface, but this mechanism occurred at up to 25-time dilution of SW[4]. On the other hand, *Sohrabi et. al.* (2015) showed that the main mechanism was due to rock/fluid interaction through mineral dissolution[38].

Austad et al. (2011) and *Yousef et al.* (2010) reported that LSWI oil recovery on limestone plugs improved when the plugs contained anhydrite ($CaSO_4$) in response to anhydrite dissolution causing in-situ generation of SO_4^{2-} ions [3,24]. *Qiao et al.* (2015) also suggested that anhydrite dissolution can be added to calcite dissolution. Also, the extent of anhydrite dissolution is determined based on the production of sulfate ions (Eqs. (4) and (5))[26].

$$CaCO_3(S) + H^+ \rightarrow Ca^{2+} + HCO^{3-} \quad (4)$$

$$CaSO_4(S) \rightarrow Ca^{2+} + SO_4^{2-} \quad (5)$$

Shariatpanahi et al. (2016) reported the catalyst role of sulfate ions in the system for the wettability alteration process (either through the injected brine or anhydrite dissolution). Sulfate ions are essential to oil recovery in calcite rocks while being very trivial in dolomite. They also found that the low brine salinity can increase the surface reactivity of dolomites to produce key ions (Ca^{2+}, Mg^{2+}, SO_4^{2-})[6].

In the present study, because of contradictions on LSWI effect and mechanisms as well as very limited studies on low salinity water Sp. Imbibition on dolomite plug sample (due to high dependence of LSWI oil recovery on mineralogy), the application of LSWI is evaluated in one of the Iranian naturally fractured carbonate reservoirs (mostly dolomite) at 75°C. In the following section, the general characteristics and properties of the studied reservoir are described. Next, methods including a summary of laboratory procedures with the corresponding challenges is presented. Due to the importance of Sp. imbibition in naturally fractured carbonate reservoirs, in the next section we deal with the secondary oil recovery through Sp. imbibition experiments[5]. For this purpose, the prepared dolomite plugs are imbibed with a sufficient volume of different diluted seawaters to ensure that no more oil is produced and the optimum diluted seawater for the reservoir is determined. Due to the complexity of reactions, the experimental conditions, and mineralogy in the system of crude oil/brine/rock, there is no general agreement on mechanisms including mineral dissolution (dolomite and anhydrate dissolution) and pH effect in LSWI. In particular, previous literature suggests that pH may not be used as a criterion for verifying the LSWI effect; therefore, it requires further investigations. Thus, the concentrations of key cations or anions (Ca^{2+}, Mg^{2+}, SO_4^{2-}) are measured and the pH-increase mechanism is assessed in the following section of the paper. Then, the tertiary recovery mode is subsequently applied to reduce residual oil saturation with optimum diluted seawater.

EXPERIMENTAL SECTION

Reservoir description

The reservoir under study is a low permeability naturally fractured oil reservoir located in the southwest of Iran with more than 50 years of production history. It is currently under crustal gas injection. Due to the long-term production history, the reservoir pressure has significantly declined and water has encroached through fractures in the oil zone. Therefore, improving the imbibition mechanism can enhance the recovery factor from the water-invaded zone.

Oil property

The general PVT properties of the reservoir oil are summarized in Table 3.

Rock properties

The selected plug samples were cut from the low permeability whole core of the reservoir that they are very similar based on routine properties. The plug samples had been recovered from a water-invaded zone. The results of plug scanning showed no signs of micro-fracture or vugs. Plugs XRD results and thin section micrograph of studied mostly dolomite reservoir show evidence of anhydrite and free clay content (one of the plug samples scans in Fig. 2). Important properties of the rock samples are reported in

Table 3: Crude oil physical property (the measured oil property in this study point with *)

property	result
API	30
Viscosity*(cp)	4.756
AN*(mg KOH/g)	0.2
Asphalten content*(wt%) [38]	3.3

Table 4. Mostly limestone C5 plug sample was selected for the comparison of LSWI effect on limestone and dolomite lithology.

Brine

The brine used in spontaneous and forced imbibition tests is diluted by seawater. Table 5 shows the physical properties of low salinity water with different concentrations (0, 10, 20,100 times dilution of seawater). The geochemical analysis of low salinity is reported in Table 6.

METHODOLOGY

Experimental methods

In this study, the experiments start from the preparation of plug samples to mimic the reservoir conditions and the measurement of fluid/rock properties. Then Sp. imbibition tests are performed to evaluate low salinity potential in the fractured reservoir. Due to low values of the plug heights, the gravity force is negligible and the capillary force would be the main driving force in the Sp. imbibition process. In addition, forced imbibition tests are performed to determine the endpoint of relative permeability (K_{rw}^0), Amott wettability index (I_w), and oil saturation (S_{or}) within a wide range of low salinity dilutions.

Plug sample preparation

After cleaning the plug samples in the Soxhlet apparatus for about 1 month, the plug samples are saturated by filtered formation water and then flooding is performed with oil to obtain irreducible water saturation (S_{wi}). for more precise measurement of S_{wi}). The water and oil produced in the previous stage are separated by a centrifuge and demulsifier. Then, the plug samples are aged in oil at reservoir temperature for up to 40 days (the aging duration is detected by contact angle test). Next, fresh crude oil is re-injected into the plugs for different reasons including

Table 4: Rock property.

Sample Name	Lithology	Length (mm)	Diameter (mm)	Porosity (%)	Permeability (MD)	S_{wi} (%)
C1	Mostly Dolomite	52.19	38.05	12.66	4.19	12
C2	Mostly Dolomite	51.60	37.60	15.47	3.19	31
C3	Mostly Dolomite	52.31	38.03	14.90	3.57	24
C4	Mostly Dolomite	52.25	38.11	14.17	2.75	20
C5	Mostly limestone	52.22	31.38	12.26	0.6	27.5

Table 5: Brine physical property.

brine	Density (gr/cc)@ATM	Viscosity (cp) @ 26.8ºC	Viscosity (cp) @ 75ºC	IFT (dyne/cm)
FW(Formation water)	1.1125	1.1258	-	-
SW(Seawater)	1.0274	0.969	0.48	34.1
10×SW*	0.9994	0.926	0.466	32.6
20×SW	0.989	0.901	0.45	32.2
100×SW	0.9867	0.895	0.413	32

**10*SW=10 times dilution of sea water*

Table 6: Geochemical analysis of low salinity.

Brine	Key ion component(ppm)			TDS (ppm)
	Ca^{2+}	Mg^{2+}	SO_4^{2-}	
SW	440	1632	3110	43120
10×SW	44	163.2	311	4312
20×SW	22	81.6	155.5	2100
100×SW	4.4	16.32	31.1	431.2
FW	8917	552	142	224000

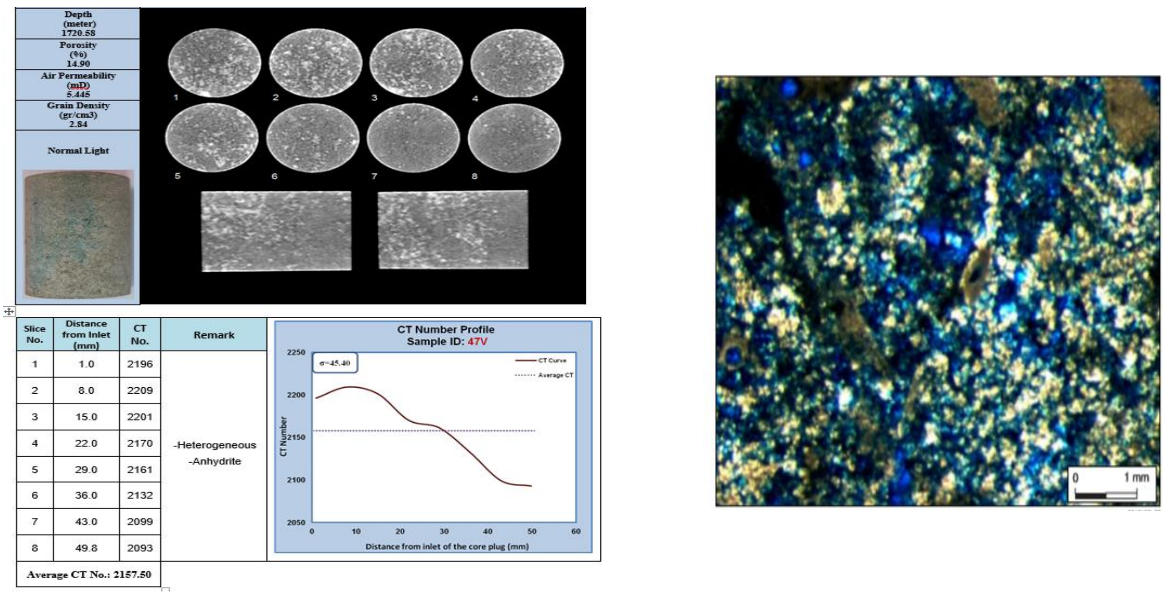


Fig. 2: The result of one of the rock samples: XRD scan (left) and thin section micrograph with mostly dolomite facies (right).

refreshing the crude oil inside the plug, discharging heavy compounds such as asphaltene, and determining K_{ro}^0 (the oil endpoint of relative permeability) at initial water saturation.

Wettability measurement

Contact angle measurement is one of the common methods to measure the wettability of a surface. There are different methods for contact angle measurement and in all methods, plate samples (thin section of the core samples) are used to measure the contact angle [15,24]. In this work, a contact angle greater than 115 degrees is observed after the completion of the oil aging process. This suggests that the plate is oil-wet before applying LSWI (Fig. 3). To investigate the influence of LSWI on the rock wettability alteration, the oil-wet plates are placed in low salinity waters under the reservoir temperature for two weeks [39].

Spontaneous imbibition test

The secondary Sp. imbibition is performed after aging the plug samples and placing them in the seawater with different dilutions (0, 10, 20,100 times dilutions) at the ambient pressure and the reservoir temperature. The volume of low salinity water imbibed to the plug sample is obtained by measuring the volume of produced oil, considering the material balance equation. In the tertiary mode, we follow the same procedure, though the Amott cell is filled with the seawater first and then filled with the optimum dilution of low salinity water (Fig. 4).

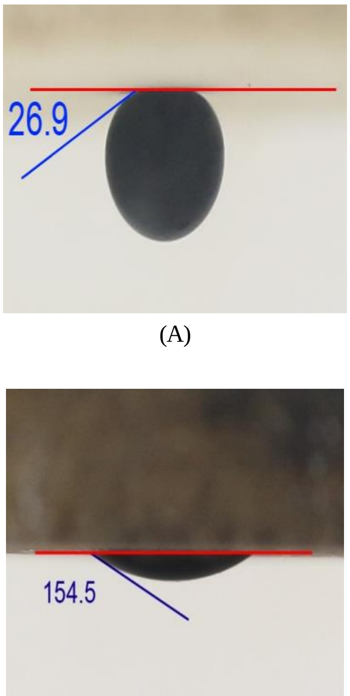
Forced imbibition

After ensuring that the oil recovery curve versus time reached a plateau or no further oil is produced in Sp. imbibition test, the next step is performing forced imbibition tests by LSWI in the reservoir temperature to determine the endpoint of relative permeability (K_{rw}^0), residual oil saturation (S_{or}), and I_w. In a wide range of dilutions, the core is flooded with low salinity water by a pump. In the forced imbibition test despite the Sp.imbibition test, low salinity is injected by the rate until the pressure difference between the inlet and the outlet of the core- the holder was stabilized (Fig. 5). The Amott wettability index to water, I_w, is determined based on *Ma et al.* (1997)[40].

RESULTS AND DISCUSSION

Precipitate formation

Due to the incompatibility between the injected water with formation water, the possibility of precipitation is investigated using a commercial simulator based on kinetics reactions. The results show that the Scale Index (SI) for all sediments such as hemihydrates (water of crystal), celestine ($SrSO_4$), halite (NaCl), calcite ($CaCO_3$), and gypsum ($CaSO_4$+H_2O) is less than zero suggesting the absence of precipitation at different mixing ratios (Fig. 6)[41]. The concentrations of ions in the seawater and the Formation Fater (FW) used in the experiments are listed in Table 7.



(A)

(B)

Fig. 3: Contact angle before aging (A) and after aging (B).

Fig. 4: Experimental Amott cells set up in the oven.

Table 1: Ions concentration in seawater (SW) and formation water (FW).

Ions	SW	FW
TDS(PPM)	43120	224000
Na^{+}	12000	12.414
K^{+}	0,00	0.00
Mg^{2+}	1632	480,00
Ca^{2+}	440,00	2.084
Sr^{2+}	33,00	0.00
Ba^{2+}	0,00	0.00
Fe^{2+}	0,00	0.00
Zn^{2+}	0,00	0.00
Cl^{-}	2141	24.5
SO_4^{2-}	3110	96.00

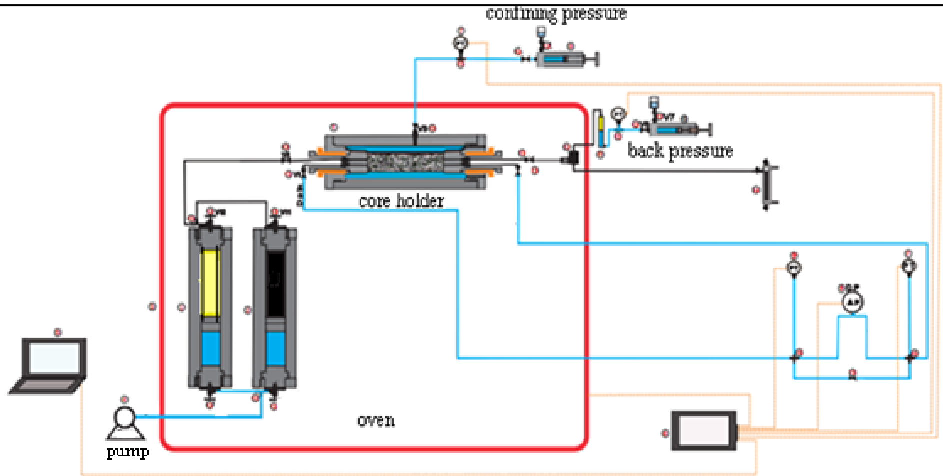


Fig. 5: Schematic forced imbibition experimental setup.

As there is no precipitation in the presence of seawater with higher salt concentrations (the worst-case scenario), it can be assured that there will be no precipitation in the other dilutions of seawater either. This inference has been similar to the deduction made by *Sohal et al.* (2016)[42]. They did not consider the chance of precipitation in the presence of reactive anions (such as SO_4^{2-}) in the Sp. imbibition test.

Spontaneous imbibition test results

Oil Recovery Result

In a Sp. imbibition test, the rate of oil recovery is partially controlled by the rock wettability [40,43]. The rock wettability depends on the aging process. In this study, Sp. imbibition experiments are performed using four plug samples with a high dolomite content (free clay) and oil-wet wettability in reservoir temperature (75°C) to investigate the effect of saltwater composition on oil production. The results are shown in Table 8 and Fig. 7.

The results indicate an increase in oil production upon salt concentration reduction. This is in line with the work of *Austad et al.* (2010), *Yousef et al.* (2011), *Hiorth et al.* (2010), while in contrast to *Zaeri et al.* (2018) and *Shariatpanahi et al.* (2016)[6,7,23,24].

Fig. 7 shows the impact of various seawater dilutions (SW, 10×SW, 20×SW, and 100×SW) on oil recovery during the imbibition process. Note that the optimum salt concentration in LSWI is 430 ppm which corresponds to 100×SW dilution.

Table 8: Sp. imbibition experimental results on the mostly dolomite sample in various dilutions of SW in 75°C.

Core name	Type	Second imbibing fluid	Recovery (%)
C1	Dolomite	seawater	4.5
C2	Dolomite	10×seawater	12.2
C3	Dolomite	20×seawater	13.5
C4	Dolomite	100×seawater	14.9

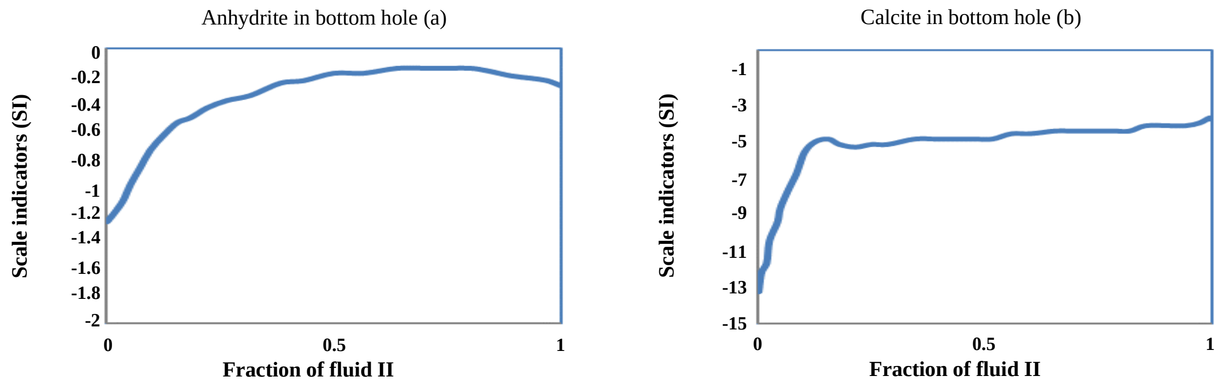


Fig. 6: Result of precipitate formation in compatibility FW and SW a) for anhydrite b) for calcite.

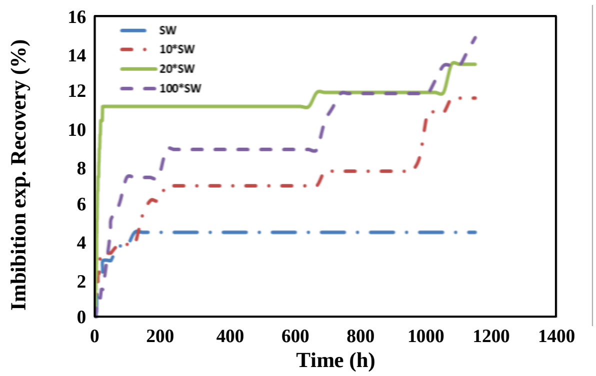


Fig. 7: Oil recovery percentage in Sp. imbibition in various seawater dilutions.

As can be seen from Fig. 7, all the recovery curves have leaped about 1 hour after the start of Sp. imbibition tests. This can be due to the capillary suction of the low salinity water into the water-wet pores of the core. Each curve has its own start of plateau due to differences in properties such as pore wettability and salt concentration of the low salinity water. As can be seen, all diluted seawaters represent a leap at the late time of the recovery process. This may be due to wettability alteration of the pores from oil-wet to water-wet. Although enough time has passed in the case of seawater, no wettability change is observed and this result is due to higher salt concentration.

The ultimate oil recovery by Sp. imbibition for 100×SW corresponded to 14.9% which is higher than that of other dilutions of SW. For a description of 20×SW behavior in Fig. 7, it can be due to routine property of plug sample (porosity and permeability) and many small pores that their wettability has not changed from water-wet to oil-wet (in oil aging process). So at the first of the Sp.imbibition test most of the oil in the water-wet parts of the plug the sample was produced by capillary pressure and at the end of this test, the oil production rate decreased.

Comparison of secondary and tertiary imbibition

Table 9 and Fig. 8 show the oil recovery in the optimum low salinity water imbibition (100×SW dilution of seawater) of a dolomite plug sample in the secondary and tertiary imbibition modes respectively in reservoir temperature. In comparison, oil recovery in secondary imbibition is greater than that in tertiary imbibition.

Table 2: Comparison of oil recovery in the secondary and tertiary mode in 75 °C.

Recovery in secondary imbibition by seawater before the tertiary mode	Recovery of tertiary imbibition by optimized low salinity	Recovery of secondary imbibition by optimized low salinity
4.5%	8.3%	14.9%

Table 10: Comparison of oil recovery in limestone and dolomite plug sample in 75 °C.

Core name	Type	Second imbibing fluid	Recovery (%)
C4	Dolomite	100×seawater	14.9
C5	Limestone	100×seawater	20.6

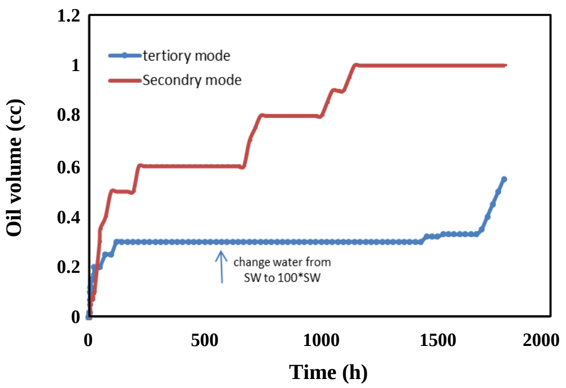


Fig. 8: Oil recovery in the secondary and tertiary modes in 75°C.

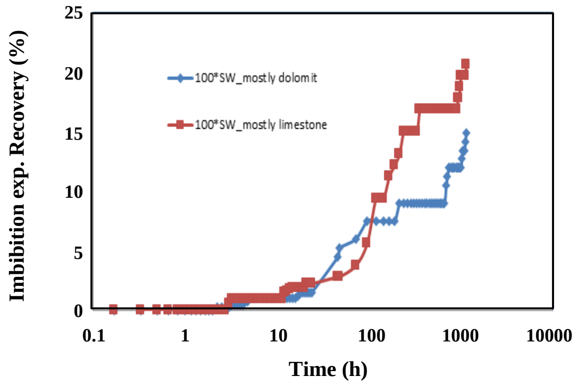


Fig. 9: Oil recovery in limestone and dolomite plug sample in 75°C.

The optimum low salinity experiments finished after 45 days in both secondary and tertiary modes.

This result can be explained that in tertiary Sp. imbibition mode, the low salinity water (100×SW) as soon as imbibes the core, mixes with the existing seawater in the pores, thereby brine salt concentration increases in the pores. Increasing brine salt concentration causes low oil recovery. Therefore, low salinity injection in the tertiary mode is not recommended for this fractured reservoir.

4.2.3. Comparison of secondary and tertiary imbibition

For comparison of lithology effect on LSWI oil recovery, Sp.imbibition test performs on a mostly limestone plug sample and the result compare with C4 core behavior (Table 10 and Fig. 9).

Based on Sp. imbibition test result, oil recovery of limestone plug sample is more than dolomite therefore LSWI in limestone ($CaCO_3$) plugs is more effective than in dolomite plugs ($CaMg(CO_3)_2$. These results are in line with *Austad et al.* (2011), *Aghaeifar et al.* (2015), *Mahani et al.* (2015)[4,44,45].

Flooding test results

Forced imbibition tests in secondary mode and reservoir temperature are performed to determine the endpoint relative permeability (K_{rw}^0, K_{ro}^0) and the Amott wettability index (I_w), which are shown in Table 11. In this work, K_{rw}^0 and K_{ro}^0 are used as key parameters in the dimensionless time function. The low value of k_{ro}^0 may be due to the presence of heavy components such as asphaltene components in crude oil.

Effect of clay content effect

Here according to thin section micrograph results (Fig. 2), the plugs are free clay but oil recovery is shown by LSWI. This can suggest that the presence of clay as one of the conditions for LSWI effect is not necessary and this result is in line with *Aksulu et. al.* (2012)[36]. He found that

Table 11. Forced imbibition experimental results (75°C).

Sample name	Low salinity imbibition	Recovery of force imbibitions (%)	K_{rw}^0	I_w (%)	K_{ro}^0
C1	SW	-	0.02	-	0.018
C2	10×SW	62	0.07	19.7	0.06
C3	20×SW	60	0.08	23	0.036
C4	100×SW	45	0.14	25	0.18

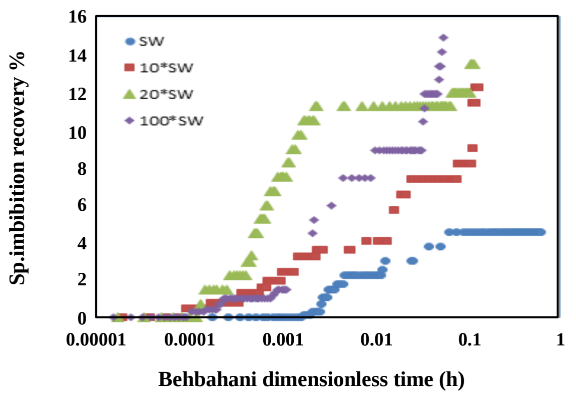


Fig. 10: Oil recovery in various dilutions of low salinity by Behbahani & Blunt equation (2005).

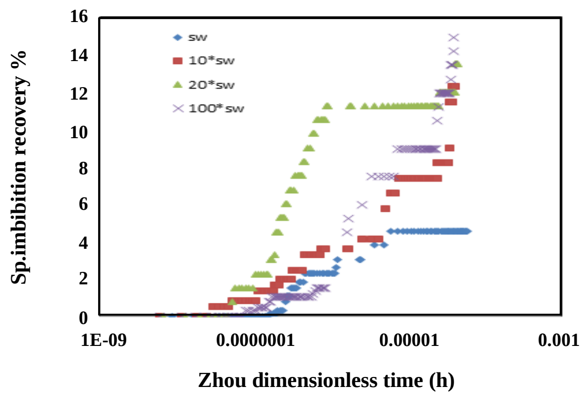


Fig. 11: Oil recovery in various dilutions of low salinity by Zhou equation (2002).

the presence of clay mineral in carbonated reservoir gives optimum performance when salinity range of water injection is 20000-33000 ppm; thus, since in this study, the salinity contents in low salinity water (10, 20, 100 times of dilution of seawater) are lower than 20000 ppm, the clay contact effect is negligible.

Laboratory scale-up

To apply Sp. imbibition test results in the field scale, a suitable dimensionless time should be introduced for the oil-wet system. *Zhou et al.* (2002) and *Behbahani* and *Blunt* (2005) suggested Eq. 6 and Eq. 7 for oil-wet systems, respectively[46,47].

$$t_D = t\sqrt{\frac{k}{\Phi}}\frac{\sigma}{L_c^2}\sqrt{\lambda_{rw}^0\lambda_{ro}^0}\frac{1}{\sqrt{M^*}+\frac{1}{\sqrt{M^*}}} \tag{6}$$

$$t_D = t\sqrt{\frac{k}{\Phi}}\frac{\sigma}{L_c^2}\lambda_{rw}^0 \tag{7}$$

Where t is time, K represents permeability, Φ denotes porosity, σ indicates interfacial tension, L_c is the characteristic length, $\lambda r^0 = kr^0/\mu$ shows mobility, and $M^0 = (\lambda rw^0/\lambda ro^0)$ is mobility ratio.

Figs. 10 and 11 display oil recovery versus (1) Behbahani & Blunt and (2) Zhou dimensionless times respectively for LSWI in various dilutions of seawater. The comparison of Figs. 10 and 11 show that Zhou's equation is more acceptable than that of *Behbahani* & *Blunt* for the reservoir because the curves in the Zhou's equation are closer together; this can be due to the effect of more factors such as oil viscosity and oil endpoint relative permeability in Zhou's dimensionless time equation.

Also for improving Zhou dimensionless time equation for LSWI and overlap of all curves, it is suggested to modify this equation by the parameters accounting for low salinity properties such as key ions concentration, TDS, Ion strength, and other measurable parameters.

Wettability alteration mechanism

The results of contact angle measurement at various dilutions of low salinity water (10×SW, 20×SW, 100×SW) indicate wettability alteration from oil-wet to water-wet (Fig. 12). A lower contact angle or more water-wet wettability can be seen in higher dilutions of seawater.

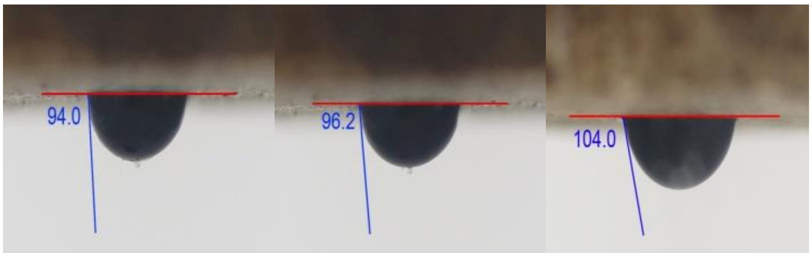


Fig. 12: The contact angle of the oil droplet in various dilutions of low salinity water (degree).

The results show that the diminishing salt concentration is directly related to changes in the wettability from oil-wet toward water-wet thus enhancing oil production.

Also, the results of I_w measurement suggest that the plug sample imbibed by 100×SW is more water-wet than other samples (5th column of Table 11). Thus, reduction of the salt concentration leads to further water wetness and more oil production.

Various mechanisms affect the behavior of wettability alteration; accordingly, in this study two mechanisms including mineral dissolution and pH effect are investigated.

Oil recovery and mineral dissolution mechanism

Ca^{2+}, Mg^{2+}, and SO_4^{2-} are the ions whose concentrations in the aqueous solution control the polarity and density of electrical charge on the mineral surface and influence interactions between oil and the rock surface [48]. In this section, the mineral dissolution mechanism has been evaluated by measuring Ca^{2+}, Mg^{2+}, SO_4^{2-} concentrations, and permeability measurement. No precipitation has been assumed during LSWI in the core composed of dolomite and anhydrite.

Dolomite dissolution

The dissolution of dolomite contributes to the presence of Ca^{2+} and Mg^{2+} in the effluent [25]. Table 12 shows Ca^{2+} and Mg^{2+} concentrations in the effluent of Sp. imbibition test in each dilution of low salinity water on dolomite core samples. All concentrations increase after the Sp. imbibition tests. A comparison of the concentrations of Ca^{2+} and Mg^{2+} produced at 10×SW and 100×SW show that the solubility of Ca^{2+} and Mg^{2+} in high dilution of the low salinity water is greater than in lower dilutions. Also, comparison of these results with LSWI oil recovery (Table 8) shows that the mineral dissolution mechanism (dolomite dissolution) can be important in LSWI on this type of core sample. It can be concluded the rate of oil, recovery depends on the rate of dolomite dissolution which is also directly influenced by the dilution of water.

Anhydrite dissolution

As mentioned earlier, the plug samples are composed of anhydrite which is more important for LSWI oil recovery by wettability alteration. Also, the high amounts of SO_4^{2-} in various dilutions (Table 12) are related to the anhydrite dissolution in low salinity water imbibition tests. One may conclude that SO_4^{2-} ions act as a catalyst to improve the LSWI oil recovery (Table 8). The mechanism behind this improvement may be related to the altered wettability of the plug sample in response to the replacement of the adsorbed carboxylic group with SO_4^{2} (mineral dissolution).

pH-increase mechanism

As mentioned previously, there is some disagreement among researchers regarding the role of pH in LSWI oil recovery of sandstone and carbonate reservoirs. In particular, only limited studies have been conducted to evaluate the role of PH in LSWI oil recovery from carbonate rock samples.

Based on experimental results (Fig. 13), pH value decrease in all dilutions except in 100×SW therefore based on this result and Sp.imbibition oil recovery results (Table 8), the pH-increase mechanism can be one of the effective mechanisms for improving oil recovery in LSWI by 100×SW (lower salt concentration) in the mostly dolomite reservoir.

Table 12: Key ions concentration before and after the Sp.imbibition test.

on Mostly dolomite core sample($MgCa(CO_3)_2$) at 75°c		
Low salinity type	Before Sp.imb test	After the Sp.imb. test
Concentration of Ca^{2+}		
10×SW	44	180
100×SW	4.4	230
Concentration of Mg^{2+}		
10×SW	163.2	166.75
100×SW	16.32	46
Concentration of SO_4^{2-}		
10×SW	311	543
100×SW	31.1	583

As a justification, by diminishing salt concentrations as much as possible in brine and in order to maintain the ionic system stability, dolomite rocks dissolve more in brine and carboxylic acids in crude oil composition dissolve in the brine too (Eqs. 8, 10). As brine-rock reactions are more effective than oil-brine reactions (Mahani, 2015) and because of lower solubility of carboxylic acids than dolomite rock (Eqs. (8, 9, and 10)), OH^- is produced more than H^+, where finally pH increases at lower salt concentrations of brine or greater dilutions of seawater. Moreover, it can be concluded the other reason for producing more OH^- or increasing pH in high dilution of low salinity water is the easy possibility of active ions of producing of rock solubility to water due to decreasing of salt ions concentration.

Dolomite dissolution:

$$CaMg(CO_3)_2 \rightarrow Ca^{2+} + Mg^{2+} + 2CO_3^{2-} \quad (8)$$

$$CO_3^{2-} + H_2O \rightarrow HCO_3^- + OH^- \quad (9)$$

And carboxylic acid dissolution:

$$HCOOH + H_2O \rightarrow H^+ + COOH^- \quad (10)$$

At last, it may be suggested that for validation of this conclusion, the carboxylic ion, and H^+, CO_3^{-2} production should be measured while considering the solubility constant of key reactions.

Generally, it can also be concluded that there is no clear relationship between the pH value and LSWI oil recovery as there are no relations between pH and salt concentration in brine. Bearing in mind that pH is dependent on the type of chemical reactions, the pH mechanism may not be effective in the LSWI.

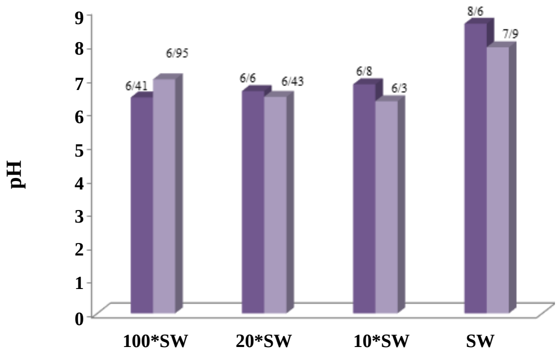


Fig. 13: pH value before and after of Sp. imbibition experiments.

CONCLUSIONS

There are a number of contradictions in the mechanisms suggested for LSWI oil recovery of carbonate rocks. In this study, LSWI (in various dilutions of SW) was evaluated in one of the Iranian naturally fractured carbonate reservoirs (mostly dolomite/free clay). In particular, the mineral dissolution (dolomite dissolution and anhydrate dissolution) and pH-increase mechanisms are validated, performing Sp. imbibition experiments by LSWI in various dilutions of seawater on a dolomite rock surface. In all experiments, it was assumed that there is no precipitation, and the plug samples were homogenous. The following conclusions can be drawn from the experimental observations:

1. Incremental oil recovery was observed in the Sp. imbibition tests of the dolomite plugs, injecting low salinity water. The optimum dilution of SW was obtained at 100×SW dilution. This value corresponds to a total salt concentration of 430 ppm is.

2. The LSWI had a significant impact on secondary imbibition oil recovery; however, its impact on tertiary mode was almost negligible.

3. For reservoir under the study, *Zhou et al*.'s (2002) equation provided more appropriate results for up-scaling laboratory results to field results.

4. The measurement of I_w before and after LSWI revealed that the wettability of the carbonate plug changed. Oil recovery improved by injecting low salinity water. The dilution of 100×SW could alter the wettability of the surface toward water-wet more than other dilutions.

5. The oil recovery obtained by LSWI in the clay-free core could suggest that the presence of clay was not necessary.

6. The mineral dissolution mechanism occurred due to the dissolution of dolomite and anhydrite in various dilutions of SW. The rate of dissolution directly depended on the dilution of water.

7. Based on pH measurement, it was observed that the pH-increase mechanism occurred at a high dilution of seawater (100×SW). To explain such a finding, it may be justified that in order to maintain ionic system stability, dolomite and carboxylic acids are dissolved in brine. Due to more solubility of dolomite compared to carboxylic acids, OH^- is produced more than H^+, thus increasing pH.

These results can help in interpreting the potential of field tests in the future.

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Nomenclature

θ	Contact angle
μ	Viscosity
ρ	Density
σ	Interfacial tension
Φ	Porosity
C	Constant
dx	Variation in the length of the core
dp	Pressure difference
K	Permeability
Swi	Initial water saturation
Sor	Oil residual saturation
t	Time
t_D	Dimensionless time
Lc	Characteristic length
K_{rw}^0	Endpoint water rel perm
K_{ro}^0	Endpoint oil rel perm
λ_{rw}^0	Water mobility
λ_{ro}^0	Oil mobility
M^0	Mobility ratio
Sp	Spontaneous

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