

Effects of Rock Components on Enhanced Oil Recovery by Low-Salinity Waterflooding in a Limestone Formation: A Case Study of an Iranian Oil Field

Armin Hosseini^{1,2*} and Mahdi NazariSarem¹

¹ Department of Petroleum, Mining, and Materials Engineering, CT.C., Islamic Azad University, Tehran, Iran

² Postdoctoral Fellowship, Department of Earth Science and Ocean, University of British Columbia, Vancouver, Canada

Highlights

- The mechanisms governing low-salinity water (LSW) injection in carbonate reservoirs are complex.
- Key parameters, including contact angle, zeta potential, and pH, should be investigated to better understand the underlying mechanisms.
- LSW injection demonstrated enhanced potential for improved oil recovery (EOR) in both natural and synthetic limestone rocks.
- The crude oil–brine and rock–brine interfaces exhibited different wettability responses during tertiary LSW injection.

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Abstract

The mechanisms underlying low-salinity water (LSW) flooding in carbonate reservoirs remain less well understood than those in sandstone reservoirs. This study investigates the role of rock composition through a comparative experimental analysis of natural limestone from the Shadegan oil field and pure synthetic calcite. Core-flooding experiments, contact angle measurements, zeta potential analysis, and pH monitoring were conducted under reservoir conditions of 70 °C and 100 bar. The results showed that tertiary LSW injection recovered 14.5% of the initial oil in place from the limestone, compared with only 3.4% from pure calcite. This substantial difference was attributed to a more pronounced wettability shift toward water-wet conditions in the limestone, as evidenced by a 42° reduction in contact angle compared with a 16° reduction in calcite. This wettability alteration was associated with a stronger negative shift in zeta potential in the natural limestone (−7.7 mV) than in the pure calcite (−4.1 mV). X-ray fluorescence (XRF) analysis revealed the presence of silica, sulfate, and phosphate impurities in the limestone, which enhanced the alteration of surface charge. These findings indicate that trace non-clay minerals can significantly enhance the effectiveness of LSW flooding in carbonate reservoirs, providing new insights into the optimization of enhanced oil recovery (EOR) in complex carbonate formations.

Keywords: Enhanced oil recovery, Low salinity water, Wettability variation, Surface electric charge, Zeta potential

* Corresponding author:

Email: arminh@iau.ir

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1. Introduction

Low-salinity waterflooding, also referred to as engineered waterflooding, has been reported as a promising enhanced oil recovery (EOR) method in laboratory studies (Rahevar et al., 2023). In addition, multi-salinity experiments can be used to conduct appropriate fluid-chemistry analyses of flowing brines (Navarro-Perez et al., 2024). Low-salinity water (LSW) flooding, which involves regulating the salinity of water injected into carbonate formations, has attracted increasing attention over the past decade. LSW injection can also be economically viable and environmentally favorable (Mazher et al., 1971; Evina et al., 2003). Wettability alteration is generally considered one of the primary mechanisms responsible for incremental oil production during LSW injection (Al-Shalabi and Sepehrnoori, 2016).

In sandstone reservoirs, wettability alteration has been attributed to particle migration, pH increase, multiple ion exchange, and electrical double-layer expansion (Tang et al., 1999; Ligthelm et al., 2009). The presence and type of clay minerals play important roles in these mechanisms. Because carbonate reservoirs generally contain little or no clay, LSW injection was initially assumed to provide limited improvement in oil recovery from these formations. Consequently, the mechanisms governing oil recovery during LSW injection in carbonate reservoirs remain less well understood than those in sandstone reservoirs. An additional challenge is determining whether incremental oil production results directly from LSW injection or from differences in heterogeneity between the core samples used in comparative experiments.

Numerous laboratory studies have demonstrated that reducing the salinity of injected water can improve oil recovery (Webb et al., 2008; Romanuka et al., 2012; Nasralla et al., 2014). However, other studies have reported that reducing injected-water salinity does not necessarily increase oil production (Fathi et al., 2012; Austad et al., 2012). In these studies, the presence of anhydrite was considered important for the effectiveness of LSW injection, and no improvement in oil recovery was observed in anhydrite-free limestone. Unlike sandstone reservoirs, in which injected-water salinity is often a dominant factor, other parameters, particularly brine composition, can play important roles in carbonate reservoirs (Mahani et al., 2017; Sharma et al., 2018). Therefore, a comprehensive understanding of both rock–fluid and fluid–fluid interactions is required to clarify the mechanisms underlying LSW flooding.

Yousef et al. (2011) observed substantial incremental oil recovery as seawater salinity decreased and attributed this behavior to increased rock water-wetness. Using nuclear magnetic resonance (NMR) measurements before and after LSW injection, they further associated wettability alteration with microscopic rock dissolution in lower-salinity water, as connectivity between small and large pores increased after LSW injection. Hiorth et al. (2011) developed a chemical model to investigate the effects of surface charge and dissolution of pure calcite, representing carbonate reservoir rock, during water injection. Their results indicated that oil recovery was closely related to chemical interactions involving calcite dissolution. Austad et al. (2010, 2011) reported that the effect of LSW was not observed in the absence of anhydrite in the rock or sulfate ions in the injected water. They therefore suggested that the effectiveness of LSW may depend strongly on rock composition and water chemistry (Fathi et al., 2010; Austad et al., 2012).

Mahani et al. (2015) investigated crude oil/brine/limestone systems using contact angle and zeta potential measurements while excluding the effect of calcite dissolution. They showed that decreasing salinity reduced the magnitude of the rock/brine interfacial charge, thereby promoting more water-wet

conditions. Compared with rock–fluid interactions, fluid–fluid interactions have received less attention. Sohrabi et al. (2017) conducted a series of core-flooding experiments focusing on the formation of oil-in-water microdispersions and suggested that fluid–fluid interactions may be more important than rock–fluid interactions under certain conditions (Alhammadi et al., 2017). Other studies have also emphasized the importance of fluid–fluid interactions in oil recovery by examining crude oil/brine interfacial tension and changes in oil interfacial elasticity upon contact with LSW (Wei et al., 2017; Mokhtari et al., 2019). In addition, the effects of injecting low-salinity carbonated water into oil-bearing reservoirs have been investigated analytically (Alvarez et al., 2024).

Several mechanisms have been proposed to explain LSW performance in carbonate systems, including multiple ion exchange (MIE), electrical double-layer expansion (DLE) in clay-free systems, and interactions involving potential-determining ions (PDIs), such as SO_4^{2-} , Mg^{2+} , and Ca^{2+} (Austad et al., 2010; Mahani et al., 2017). Nevertheless, an important research gap remains. Although previous studies have emphasized the importance of anhydrite, sulfate ions, or clay minerals in determining LSW effectiveness, the influence of trace non-clay impurities, such as silica and phosphate, in carbonate rocks remains poorly understood (Fathi et al., 2012). The present study addresses this gap by systematically comparing the electrokinetic and wettability responses of natural limestone and pure calcite to LSW.

In this study, the LSW flooding process was investigated using reservoir rock and fluids from the Shadegan oil field in Khuzestan Province, Iran. The objective was to experimentally evaluate the potential effects of rock composition, rock–fluid interactions, and fluid–fluid interactions on oil recovery at the core scale. The performance of tertiary LSW injection in the limestone formation of the Shadegan oil field was compared with that in synthetic pure calcite rock. X-ray powder diffraction (XRD) analysis showed that calcite was the principal mineral phase in both rock samples. Therefore, X-ray fluorescence (XRF) spectroscopy was subsequently used to identify and quantify impurities in the natural limestone.

To identify the mechanisms responsible for increased oil recovery during LSW injection, crude oil/brine interfacial tension and crude oil/brine/carbonate contact angle were measured for both natural limestone and synthetic calcite samples. Additional analyses were conducted to evaluate the effects of calcite dissolution and trace mineral impurities under low-salinity conditions. Changes in pH and variations in the surface charge at the crude oil/brine and rock/brine interfaces were also monitored as salinity decreased. These measurements enabled a more detailed assessment of the relationships among rock composition, surface charge, wettability alteration, fluid–fluid interactions, and incremental oil recovery. Accordingly, this study provides a systematic evaluation of the role of trace non-clay mineral components in controlling the effectiveness of LSW flooding in carbonate reservoirs.

2. Methodology and experimental analysis

Two types of rock were selected for analysis: natural limestone from the Shadegan oil field and synthetic pure calcite. Core-flooding experiments were conducted on the limestone using sequential injections of high-salinity water and low-salinity water. To investigate the mechanisms by which LSW improves oil recovery in the Shadegan oil field, the behavior of the natural limestone was compared with that of pure calcite under similar experimental conditions. Although calcite is the dominant mineral phase in both rocks, the incremental oil recovery obtained by LSW flooding in the Shadegan limestone was more than 10% of the initial oil in place higher than that obtained from pure calcite.

Tenfold diluted seawater used as LSW recovered an additional 14.5% of the initial oil in place following secondary seawater injection. In contrast, tertiary injection of the same LSW into synthetic pure calcite with petrophysical properties similar to those of the natural rock recovered only 3.4% of the initial oil

in place. Contact angle measurements for the crude oil/brine/carbonate system revealed that reducing the salinity of the injected water from seawater to tenfold diluted seawater shifted the wettability of both natural and synthetic rocks from mixed-wet toward water-wet conditions. However, the reduction in contact angle was substantially greater for the Shadegan limestone (42°) than for the pure calcite (16°).

This difference may explain the higher incremental oil recovery observed in the Shadegan limestone. The underlying mechanism was investigated further by measuring the zeta potentials at the crude oil/brine and carbonate rock/brine interfaces for both natural and synthetic rocks. The results indicated that decreasing the salinity of the injected water caused the zeta potentials of both interfaces to become more negative. This increased electrostatic repulsion between the interfaces, thereby increasing the thickness and stability of the aqueous film and promoting more water-wet conditions in the carbonate rock. In pure calcite, the increase in pH associated with decreasing injected-water salinity increased the concentration and adsorption of CO_3^{2-} ions, which contributed to the increasingly negative charge of the rock surface. In the Shadegan limestone, the magnitude of the negative shift in zeta potential (7.7 mV) was substantially greater than that observed for pure calcite (4.1 mV).

In other words, electrostatic repulsion between the rock/brine and brine/oil interfaces was stronger in the Shadegan limestone than in the pure calcite, consistent with the contact angle results. This interpretation was further examined using X-ray fluorescence spectroscopy to compare the compositions of the natural and synthetic rocks. The XRF results revealed the presence of silica, sulfate, and phosphate in the Shadegan limestone, whereas these components were negligible in the pure calcite. The presence of these components may therefore contribute to the more negative surface charge observed in the natural limestone.

The materials used in the experimental procedures and analyses are described in the following sections.

2.1. Materials

The materials used in the experiments included brines of different salinities, rock samples, and crude oil, as described below.

2.1.1. Saline water

Brines with different salinities, including formation water, Persian Gulf seawater, and different dilutions of seawater, were used in this study. The total dissolved solids (TDS) concentrations of Persian Gulf seawater and formation water were 41,571 and 215,591 ppm, respectively. Table 1 summarizes the compositions of the formation water and Persian Gulf seawater. Formation brine and seawater were prepared by dissolving the specified amounts of salts listed in Table 1 in deionized water.

As shown in Table 1, the TDS concentration of the formation water (215,591 ppm) was substantially higher than that of Persian Gulf seawater (41,571 ppm). NaCl was the predominant salt in both brines. The substantial difference in salinity between the formation water and Persian Gulf seawater provided an appropriate basis for evaluating the effects of water salinity under the experimental conditions. After preparation, the brines were allowed to equilibrate for 24 h and were subsequently passed through a 24- μm filter to remove any precipitated material.

Diluted seawater was prepared by mixing Persian Gulf seawater with deionized water at the required dilution ratio. For example, tenfold diluted seawater was prepared by adding 10 mL of seawater to a 100-mL volumetric flask and filling the flask to the calibration mark with deionized water. As an illustration, the Stiff diagrams for seawater and 40-fold diluted seawater are presented in Figure 1. The concentrations of the major ions, including Cl^- , Na^+ , Ca^{2+} , SO_4^{2-} , Mg^{2+} , and HCO_3^- , were substantially

higher in seawater than in 40-fold diluted seawater. The Stiff diagrams therefore facilitate comparison of the ionic compositions and salinity levels of the two brines. Because the low-salinity brines were prepared by dilution, the absolute concentrations of the constituent ions decreased while their relative proportions remained unchanged.

A schematic representation of the core-flooding apparatus, including the core holder, pumps, pressure transducers, and fluid collection system, is also presented in Figure 1.

Table 1

Ionic composition of reservoir formation water versus Persian Gulf seawater

Salt type	Persian Gulf water [mg/liter]	Formation water [mg/liter]
NaCl	152.0	28.4
CaCl ₂ .2H ₂ O	71.0	1.8
MgCl ₂ .6H ₂ O	19.2	13.7
KCl	0.0	0.8
Na ₂ SO ₄	0.5	4.5
NaHCO ₃	0.5	0.1
TDS [ppm]	215591	41571

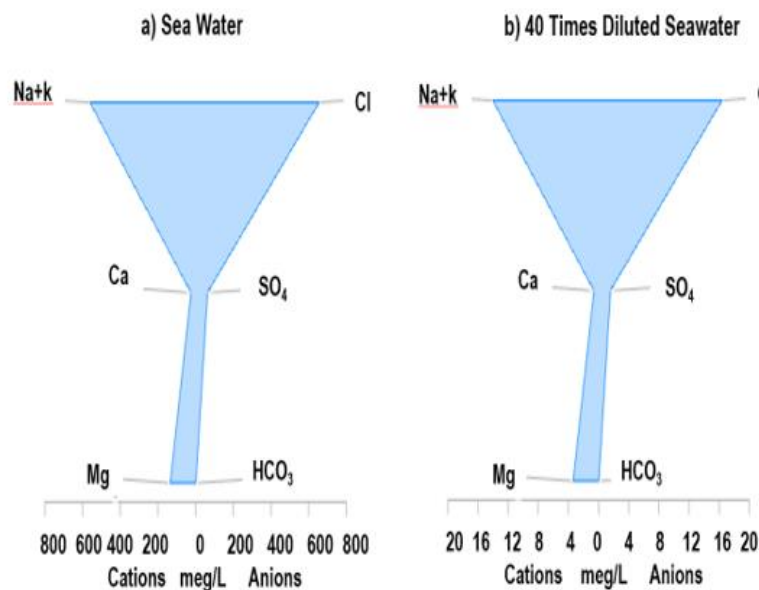


Figure 1

Stiff diagrams: (a) seawater and (b) seawater diluted 40-fold

2.1.2. Genuine rock

The limestone used in this study was obtained from the Shadegan oil field. Based on X-ray diffraction analysis, the synthetic rock was composed predominantly of calcite, as shown in Figure 2. The prominent peak in Figure 2 indicates the dominant calcite phase in the synthetic rock. X-ray fluorescence spectroscopy was also used to characterize the rock composition, and the corresponding

results are presented in the Results section. The petrophysical properties of the rock samples are summarized in Table 2.

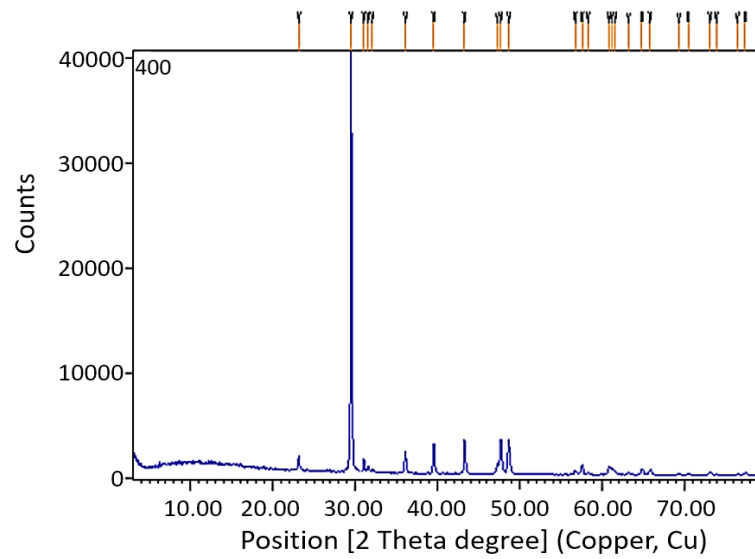


Figure 2

XRD pattern of the limestone formation in the Shadegan oil field

Table 2

Summary of experimental conditions for core-flooding tests

Material for third injection	Seawater diluted 10-fold
Material for second injection	Seawater
Dual water saturation (%)	15.26
Volume of initial oil [cm ³]	9.55
Permeability [mD]	2.49
Porosity (%)	13.87
Diameter [cm]	3.84
Length [cm]	7.02

2.1.3. Generated synthetic rock

Pure calcite powder was used to prepare a synthetic rock with petrophysical properties similar to those of the natural limestone from the Shadegan oil field. Calcite was selected because it is the dominant mineral constituent of limestone. X-ray diffraction analysis was performed to confirm the mineralogical composition of the calcite powder, and the results are presented below. The synthetic rock was prepared according to the following procedure:

- i) A calcite core was prepared using calcite powder with particle sizes ranging from 37 to 75 μm . The powder was transferred into a beaker in equal proportions on a small scale.
- ii) An appropriate amount of water was added to the beaker, and the mixture was stirred using a mixer. Because of the small particle size, sedimentation occurred slowly. Therefore, immediately after mixing,

the suspension was poured into a vertically positioned metal cylinder used as the core holder and subjected to downward water flow.

iii) During this process, the outer wall of the metal cylinder was gently tapped to promote redistribution and uniform packing of the particles. The downward water flow exerted force on the particles and facilitated their consolidation within the cylinder.

iv) After a specified period, another batch of the calcite–water mixture was prepared and added to the cylinder. This procedure was repeated until the metal cylinder was completely filled.

XRD analysis of the calcite powder confirmed that it consisted predominantly of calcite, as shown in Figure 3. After several trial-and-error iterations, the synthetic rock was successfully prepared with petrophysical properties comparable to those of the Shadegan limestone, as summarized in Table 3. To further verify the purity of the calcite powder and compare its elemental composition with that of the Shadegan limestone, X-ray fluorescence spectroscopy was performed. The corresponding results are presented in the Results section.

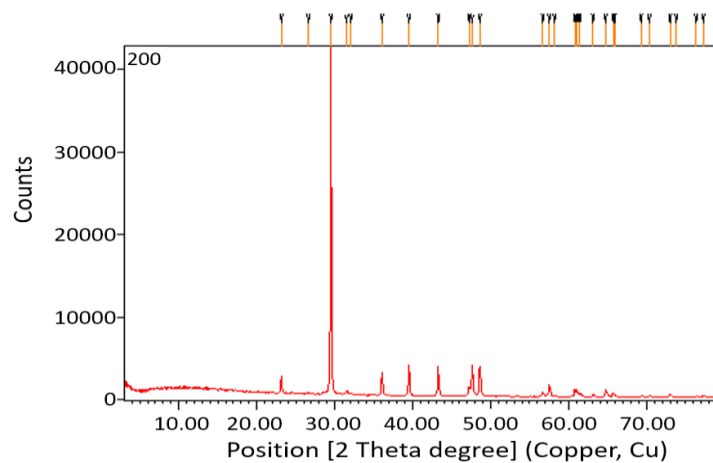


Figure 3

XRD patterns of pure calcite rock and synthetic rock

Table 3

Petrophysical properties of synthetic rock

Material for third injection	Seawater diluted 10-fold
Material for second injection	Seawater
Dual water saturation (%)	16.6
Volume of initial oil [cm ³]	10.25
Permeability [mD]	5.64
Porosity (%)	15.12
Diameter [cm]	3.84
Length [cm]	7.00

2.1.4. Crude oil

The crude oil used in this study was obtained from the Bangestan Formation of the Shadegan oil field.

Before use, the crude oil was filtered through a 24- μm filter. At ambient temperature and atmospheric pressure, the crude oil had a density of 0.86 g/cm³ and a viscosity of 8 cP. The sampled crude oil had an API gravity of 33° and was therefore classified as a light crude oil.

2.2. Experimental procedure

After preparation of the natural and synthetic rock samples, a series of core-flooding experiments was conducted, followed by measurements of interfacial tension, contact angle, pH, and zeta potential for both rock types. All core-flooding and aging experiments were performed at 70 °C to simulate reservoir conditions. The confining pressure was maintained at 100 bar, and the injection pressure was continuously monitored throughout the experiments. The experimental procedures are described in detail in the following sections.

The experimental setup consisted of a core-flooding system designed to simulate reservoir conditions at 70 °C and 100 bar. A schematic representation of the core-flooding apparatus, including the core holder, injection pumps, differential pressure transducers, back-pressure regulator, and fluid collection system, is presented in Figure 4.

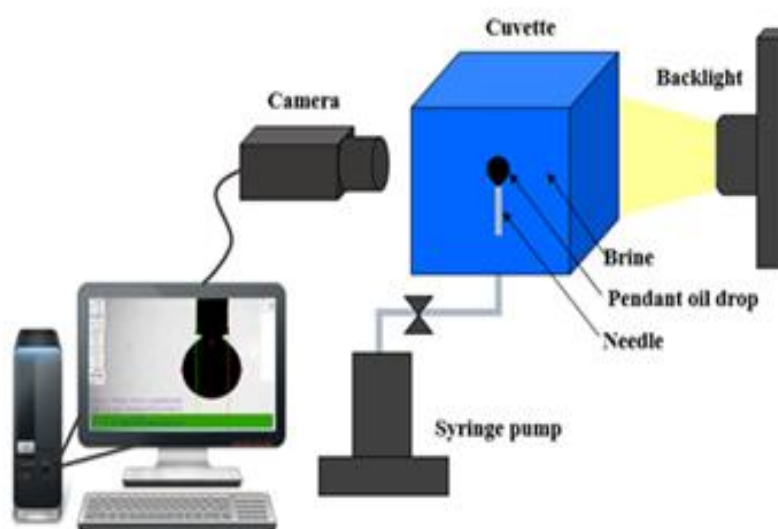


Figure 4

Schematic of an interfacial tension measurement device using the pendant drop method

2.2.1. The core flooding experiment/genuine rock

Initially, the Shadegan carbonate core was placed in a Soxhlet extractor to remove organic contaminants and was subsequently dried in an oven. The dried core was then vacuum-saturated with formation brine in a desiccator for two days. The pore volume and, consequently, the porosity were determined from the difference between the dry and saturated weights of the core. The core was then placed in the core holder, and its permeability to formation water was measured.

Crude oil was subsequently injected into the horizontally positioned, formation-water-saturated core at a flow rate of 0.2 mL/min to establish connate water saturation. To minimize capillary end effects, the oil injection rate was increased stepwise to 0.4 and 0.6 mL/min. At this stage, the initial oil in place was determined by volumetric balance. The outlet pressure was maintained at 100 bar using a pressure-control system. After establishing connate water saturation, the core was aged in the core holder at 70 °C for 31 days. The aged oil was then displaced with fresh crude oil.

Seawater was subsequently injected into the aged core at a flow rate of 0.2 mL/min. To minimize capillary end effects, the injection rate was increased sequentially from 0.2 to 0.4 and then to 0.6 mL/min. During core flooding, the produced oil volume and pressure drop across the core were continuously monitored. This stage was continued until no additional oil was produced and the pressure drop across the core reached a stable value.

2.2.2. Core-flooding experiment with synthetic rock

In the final stage, tenfold diluted seawater, designated as LSW, was injected into the synthetic rock sample to evaluate the effect of low-salinity water injection under the same experimental conditions.

2.2.3. Measurement of interfacial tension

The pendant drop method (PDM) was used to measure the interfacial tension between brine and crude oil. Figure 4 presents a schematic of the interfacial tension measurement apparatus used in this study. To initiate the experiment, an oil droplet was injected from the bottom of a transparent container filled with the desired brine through an L-shaped needle. The container had internal dimensions of $4 \times 4 \times 4$ cm³ and transparent walls. Oil injection was performed using a precision syringe pump (JMS SP-500).

The injection rate was sufficiently low to minimize the influence of hydrodynamic forces on the interfacial tension measurement. Accordingly, the principal forces acting on the droplet were capillary and buoyancy forces. A Dino-Lite digital microscope was used to record and monitor images of the suspended oil droplets. The images were subsequently analyzed using drop shape analysis (DSA) software. A uniform backlight was used to improve image contrast and measurement accuracy.

Once the suspended oil droplet reached an appropriate volume, as determined by the DSA software, the injection valve was closed. The software then continuously measured and recorded the interfacial tension as a function of time. Measurements were continued until the interfacial tension showed no significant variation with time and the system reached a quasi-equilibrium state.

2.2.4. Measurement of contact angle

The contact angle is the angle formed at the three-phase contact line between two immiscible fluids and the rock surface, measured through the denser phase. It is commonly used as an indicator of rock wettability. In an oil/water/rock system, a contact angle of less than 90° indicates water-wet conditions, whereas a contact angle greater than 90° indicates oil-wet conditions. A contact angle of approximately 90° represents an intermediate-wet or neutrally wet condition.

In this study, the sessile drop technique was used to measure the contact angle of the carbonate/crude oil/brine system. The purpose of this experimental procedure was to evaluate wettability alteration in carbonate rock samples subjected to an aging process similar to that used in the core-flooding experiments.

To measure the contact angle, the same apparatus used for interfacial tension measurements (Figure 4) was employed, except that the oil droplet was allowed to contact and adhere to the lower surface of the rock sample. The contact angle was then monitored as a function of time until no significant variation was observed and the system reached a quasi-equilibrium state. For the natural rock, a thin section of limestone from the Shadegan oil field was placed in formation brine inside a desiccator to achieve brine saturation.

To simulate the aging process, the rock section was subsequently immersed in crude oil at 70 °C for 21 days. The aged section was then removed from the oil and gently dried with an oil-absorbing cloth. To

remove the residual oil layer from the calcite surface, the rock section was immersed in a solution of cyclohexane (C_6H_{12}) and acetone (C_3H_6O). When the solution darkened because of oil dissolution, it was replaced with a fresh, clear solution. This procedure was repeated two to three times until no further oil dissolution was observed and the solution remained clear. The rock section was then placed in the desired brine in the contact angle measurement cell. After 30 min, corresponding to the approximate equilibration time of the system, the contact angle was recorded.

To age the oil-wet rock section in the presence of a selected brine, the section was first removed from the contact angle measurement cell. After removal of the attached oil droplet, the section was placed in a beaker containing the desired brine for the specified period. A thin, transparent section of pure calcite was prepared for comparison. Before further treatment, the calcite section was immersed in brine for 30 min to allow formation of an aqueous film on the rock surface. All subsequent procedures, including aging, washing, and contact angle measurement, were conducted using the same protocol as that applied to the Shadegan limestone.

2.2.5. Measurement of zeta potential

The zeta potential was measured using a Zetasizer Nano ZS (Malvern Instruments). Measurements were performed for both the crude oil/brine and calcite powder/brine systems. The experimental procedure reported by Mahani et al. (2017) was followed for preparation of the samples and measurement of the zeta potential.

2.2.6. Measurement of pH

To measure the pH of the rock/brine systems, 1 g of either pure calcite powder or limestone powder from the Shadegan oil field was added to a beaker containing the desired brine. The rock powder–brine mixture was then stirred for 5 min using a magnetic stirrer. The mixture was subsequently allowed to equilibrate for 24 h, after which the equilibrium pH was measured and recorded.

3. Experimental results and discussion

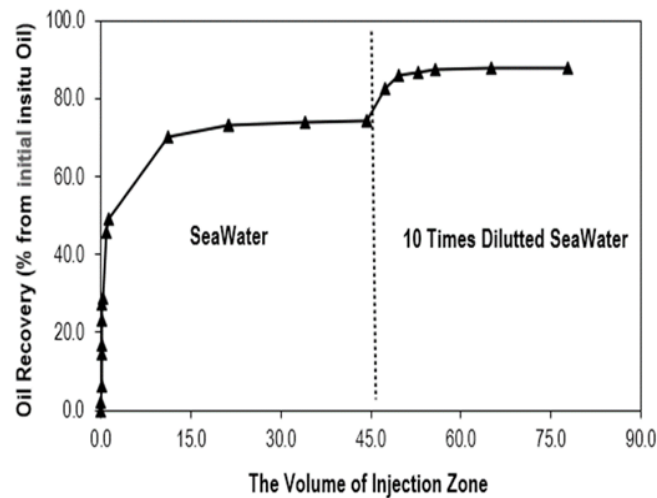
After carefully designing the experiments and implementing the laboratory procedures, the obtained results are discussed in detail in this section.

3.1. Core flooding of natural rock

To investigate the effect of salinity reduction on oil recovery from the natural core with mixed wettability, seawater and tenfold diluted seawater were injected as the high-salinity and low-salinity brines, respectively. As shown in Figure 5, high-salinity water injection resulted in a final recovery of 74.3% of the initial oil in place. The initial recovery trend was consistent with previous studies of carbonate reservoirs. For example, Saeedi Dehghani and Daneshar (2024) reported a substantial increase in oil recovery following smart water injection in carbonate formations, which is consistent with the response to LSW observed in the present study.

Following LSW injection, an additional 14.5% of the initial oil in place was recovered, increasing the ultimate oil recovery to approximately 88% of the initial oil in place. This value lies within the upper range of incremental recoveries commonly reported for tertiary LSW flooding in carbonate reservoirs, which are generally between 5% and 20% of the initial oil in place (Awolayo et al., 2018; Tetteh et al., 2021). These results highlight the substantial potential of LSW flooding in limestone formations, particularly in rocks containing specific mineral impurities such as those present in the Shadegan oil field.

A primary objective of this study was to develop a deeper understanding of fluid–fluid and rock–fluid interactions during LSW flooding. Accordingly, the performance of LSW injection in the Shadegan limestone was also compared with that in synthetic pure calcite with mixed wettability.



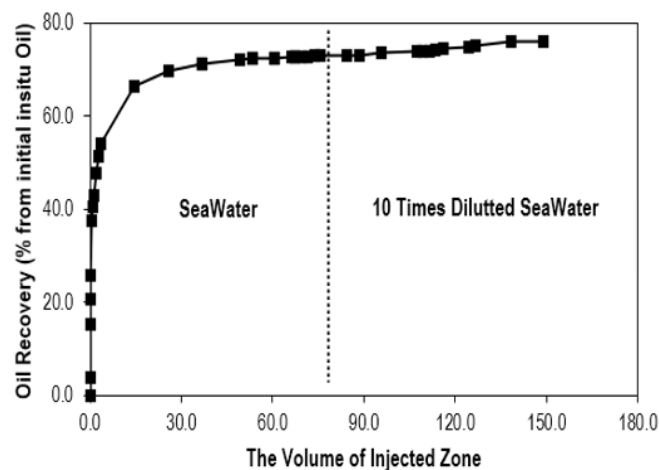
The dot line: border of secondary (left) and tertiary (right) injection for the Shadegan Oil field

Figure 5

Oil recovery during seawater and 10-fold-diluted seawater injection into mixed-wet limestone as a function of injected pore volume

3.2. Core flooding for synthetic rock

To compare the performance of low-salinity water flooding in the Shadegan limestone with that in synthetic pure calcite with mixed wettability, seawater and tenfold diluted seawater were injected as the high- and low-salinity brines, respectively, following the same procedure used for the natural rock. As shown in Figure 6, high-salinity water injection resulted in a final recovery of 72.6% of the initial oil in place.



The dot line: border of secondary (left) and tertiary (right) injection for the for Synthetic Rock

Figure 6

Oil recovery during seawater and 10-fold-diluted seawater injection into mixed-wet pure clastic rock as a function of injected pore volume

Nevertheless, following low-salinity water injection, the final recovery increased by only 3.4% of the initial oil in place, which was substantially lower than the incremental recovery observed for the natural limestone from the Shadegan oil field. To clarify the origin of this difference, the fluid–fluid and rock–fluid interactions were further investigated. These interactions are therefore important for understanding the contrasting recovery behavior of the natural and synthetic rocks and are discussed in the following sections.

3.3. Effect of interfacial tension

Figure 7 illustrates the increase in crude oil/brine interfacial tension with decreasing salinity. However, the oil recovery behavior of the natural and synthetic rocks with decreasing salinity did not follow the same trend as the interfacial tension. Therefore, changes in interfacial tension were not considered a dominant mechanism responsible for the increased oil recovery observed during LSW injection.

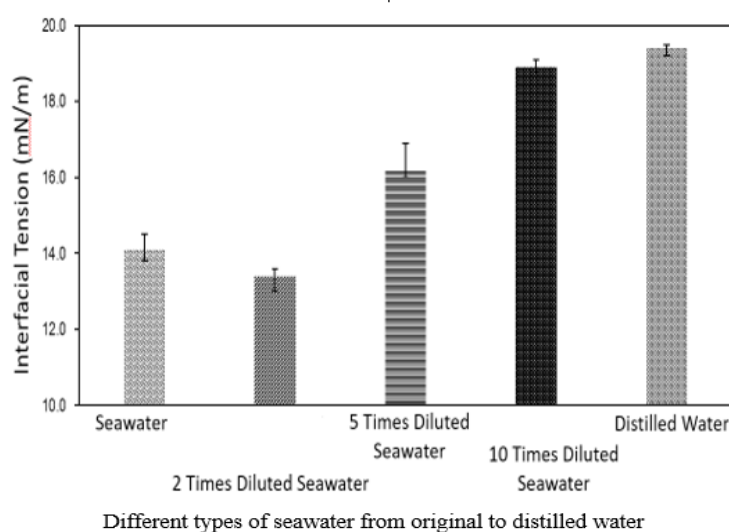


Figure 7

Dynamic interfacial tension at different seawater salinities

3.4. The effect of wettability

The contact angle of the crude oil/brine/carbonate rock system for both natural and synthetic rocks was evaluated under conditions designed to simulate the tertiary injection of tenfold diluted seawater. The results were analyzed separately for seawater, representing high-salinity water, and tenfold diluted seawater, representing low-salinity water.

3.4.1. Seawater

Initially, a thin section of oil-wet carbonate rock was placed in a contact angle measurement cell filled with seawater. The sample was exposed to seawater for 30 min to allow formation of an aqueous film on the rock surface. An oil droplet was then released and allowed to adhere to the lower surface of the oil-wet carbonate section. The contact angle gradually increased until equilibrium was reached after several minutes.

The thin sections of both natural and synthetic rocks were subsequently removed from the measurement cell and immersed in seawater for aging. The contact angle was then remeasured after each aging period. This procedure was repeated until no significant change in the contact angle was observed. This stage represented wettability alteration during the secondary injection of high-salinity seawater.

3.4.2. Tenfold diluted seawater

The same procedure described in Section 3.4.1 was followed, except that tenfold diluted seawater was used instead of seawater. The experiment was continued until no significant change in the contact angle was observed. The resulting measurements represented wettability alteration during tertiary low-salinity water injection.

Figure 8 compares the variation in contact angle associated with the reduction in salinity from seawater to tenfold diluted seawater for both natural and synthetic rocks. As shown in Figure 8, LSW shifted the wettability of both rock types toward more water-wet conditions. However, the reduction in contact angle was substantially greater for the Shadegan limestone (42°) than for the synthetic calcite (16°), indicating a stronger wettability alteration in the natural rock. This behavior is consistent with the more negative zeta potential observed for the Shadegan limestone, which enhances electrostatic repulsion between the interfaces and promotes the formation of a thicker and more stable aqueous film.

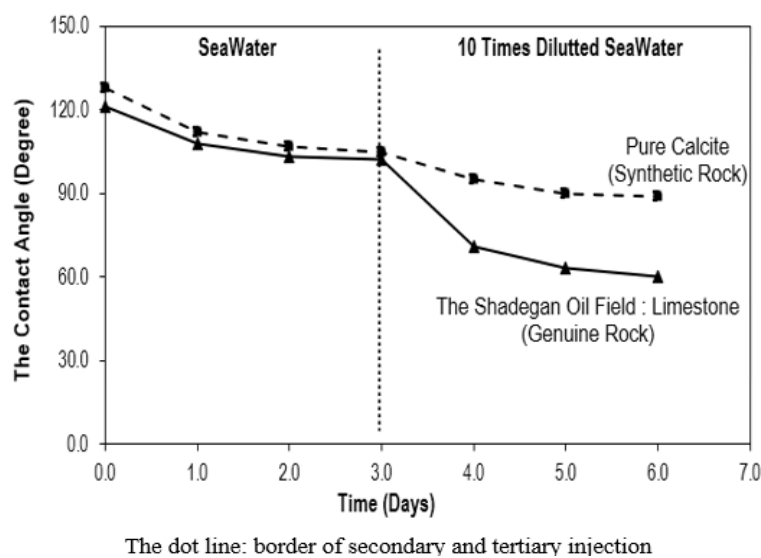


Figure 8

Variations in contact angle during secondary seawater injection and tertiary injection of 10-fold-diluted seawater into synthetic and natural rocks

3.5. The effect of rock/water and water/oil surface on wettability conversion

To understand the effect of changes in the surface charge at the crude oil/brine interface on carbonate rock wettability, the magnitude and variation of the surface charge of carbonate rock in contact with brines of different salinities must also be considered. Zeta potential measurements provide a useful means of characterizing the interfacial charge of materials in contact with a liquid medium, and the zeta potential can vary substantially with brine salinity.

As shown in Figure 9, the zeta potential at the crude oil/brine interface became more negative as salinity decreased. Similarly, Figure 10 presents the zeta potentials of the Shadegan limestone and pure calcite powder used to prepare the synthetic rock at different salinities. The results indicate that the surface charge of both rocks became more negative with decreasing salinity.

Comparison of the zeta potentials at the crude oil/brine and carbonate rock/brine interfaces for both natural and synthetic samples provides insight into the mechanism of wettability alteration. Rock wettability is strongly influenced by the thickness and stability of the aqueous film separating the oil from the rock surface, which are, in turn, affected by the interfacial electrical charges at the crude

oil/brine and carbonate rock/brine interfaces. As salinity decreased, the zeta potentials of both interfaces became more negative, thereby increasing the electrostatic repulsion between them under low-salinity conditions. Consequently, the aqueous film between the oil and rock surface became thicker and more stable, shifting the rock wettability toward more water-wet conditions.

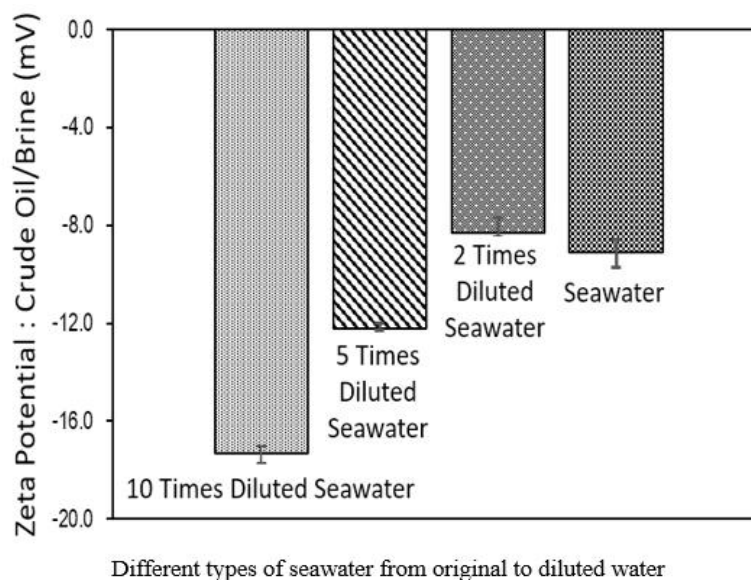


Figure 9

Zeta potential of crude oil at different seawater salinities

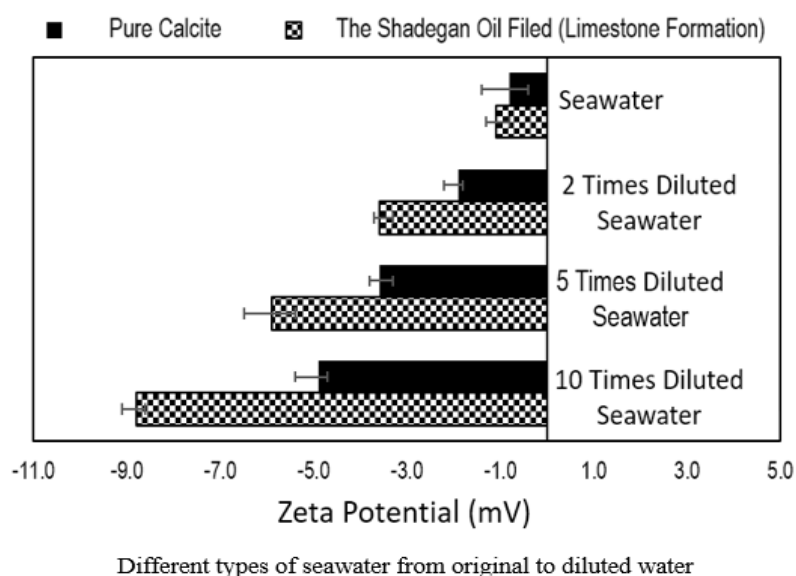
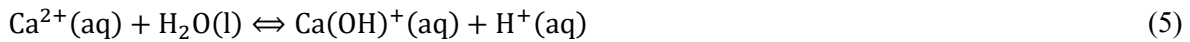
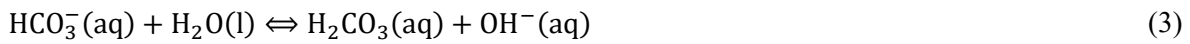


Figure 10

Zeta potential of crude oil at different seawater salinities

More specifically, when low-salinity water comes into contact with the calcite surface, Ca^{2+} and CO_3^{2-} ions are released into the aqueous phase through calcite dissolution. The dissolution behavior and concentrations of these ions are governed by the solution pH according to Equations 1–6 (Thompson and Pownall, 1989; Cicerone et al., 1992). As indicated by Equations 1–6, an increase in solution pH generally decreases the concentration of Ca^{2+} in the aqueous phase. Consequently, reduced Ca^{2+} adsorption on the calcite surface results in a more negatively charged surface (Al Mahrouqi et al., 2017).



Consistent with this interpretation, Figure 11 shows that as the salinity decreased from seawater to tenfold diluted seawater, the pH increased from 7.4 to 8.6. Therefore, in the pure calcite system, the increase in CO_3^{2-} concentration associated with increasing pH was considered the primary factor responsible for the increasingly negative surface charge of the rock. As shown in Figure 10, however, the magnitude of the shift toward a more negative surface charge was substantially greater for the Shadegan limestone than for the pure calcite rock.

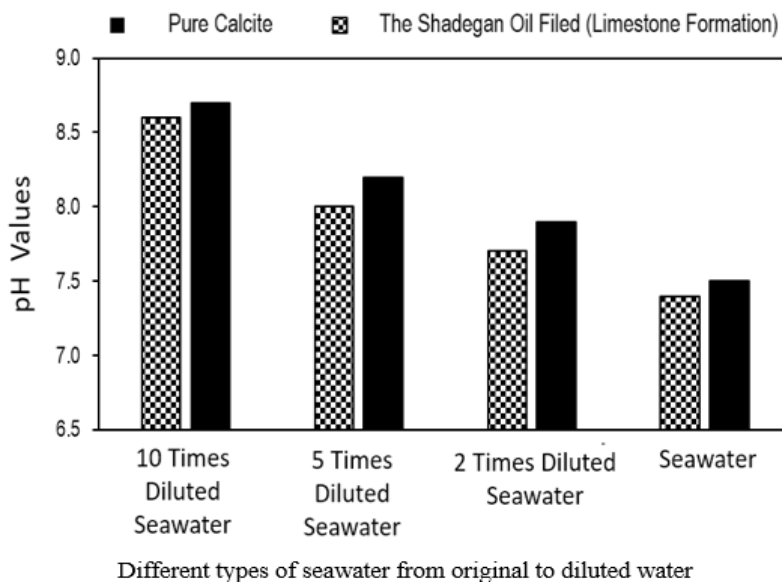


Figure 11

The pH measured in mixtures of rock powder and seawater at different salinity levels.

This behavior was associated with the reduction in salinity from seawater to tenfold diluted seawater, which produced a 7.7 mV decrease in the zeta potential of the Shadegan limestone. Under the same reduction in salinity, the zeta potential of the pure calcite rock decreased by only 4.1 mV. This difference suggests that variations in rock composition may influence the surface charge response to salinity reduction. Therefore, the elemental compositions of the natural and synthetic rocks were compared. For further analysis, the composition of the Shadegan limestone was compared with that of the synthetic pure calcite rock, as presented in Table 4.

As shown in Table 4, the Shadegan limestone contains silica, sulfate, phosphate, and aluminate, whereas these components are present only in trace amounts in the pure calcite rock. Previous studies have shown that decreasing salinity and increasing pH can make the surface charge of silica substantially more negative (Romero et al., 2018). Several studies have also suggested that the surface charge of carbonate rocks is influenced by sulfate ions, which may act as potential-determining ions (Smallwood,

1977; Zhang and Austad, 2005). Other investigations have examined the role of phosphate ions in modifying surface charge (Meng et al., 2015).

Table 4

The XRF results for genuine and synthetic rocks

Rock elements	Limestone formation	Pure calcite
CaO	53.04	54.96
Si ₂ O	0.6481	0.01≤
Na ₂ O	0.5658	0.7821
MgO	0.4069	0.1842
SO ₃	0.1225	0.01≤
Al ₂ O ₃	0.0469	0.0563
K ₂ O	0.0392	0.0665
P ₂ O ₅	0.0355	0.01≤
Cl	0.0300	0.0753
TiO ₂	0.0271	0.01≤
SrO	0.0213	0.01≤
Other elements	0.01≤	0.01≤
Loss on ignition (LOI)	45	43.9

The presence of these impurities in the Shadegan limestone likely contributes to a more negative rock surface charge and the formation of a thicker aqueous film. Consequently, these compositional differences can promote greater incremental oil recovery during low-salinity water injection in the natural limestone than in synthetic pure calcite. In the absence of clay minerals, the enhanced LSW response of the Shadegan limestone appears to be governed primarily by the surface chemistry of silica, sulfate, and phosphate impurities.

Silica surfaces become increasingly negatively charged at elevated pH because of deprotonation of silanol groups ($\text{Si-OH} + \text{OH}^- \rightarrow \text{Si-O}^- + \text{H}_2\text{O}$) (Hiorth et al., 2010). Sulfate and phosphate ions can also act as PDIs by competitively adsorbing onto calcite surface sites and increasing the negative surface charge density (Zhang and Austad, 2006). This interpretation is consistent with the findings of Mehraban et al. (2021), who reported that even trace amounts of non-carbonate minerals can strongly influence the electrokinetic behavior of carbonate rocks during LSW flooding.

Overall, the greater negative shift in zeta potential observed for the Shadegan limestone (-7.7 mV) than for pure calcite (-4.1 mV) can be attributed to the more heterogeneous mineralogical composition of the natural rock. Silica (SiO_2) develops an increasingly negative surface charge at elevated pH, particularly above approximately pH 8 (Hiorth et al., 2010; Romero et al., 2018). Similarly, sulfate (SO_4^{2-}) and phosphate (PO_4^{3-}) can act as PDIs for carbonate surfaces and contribute to a more negative interfacial charge through adsorption onto calcite sites (Zhang and Austad, 2006; Mehraban et al., 2021). The combined effects of these impurities therefore provide a plausible explanation for the stronger electrokinetic response, greater wettability alteration, and higher incremental oil recovery observed in the Shadegan limestone during LSW injection.

4. Discussion

4.1. Mineralogical control on wettability alteration and oil recovery

The substantial contrast in tertiary oil recovery between the natural Shadegan limestone (14.5% of the original oil in place [OOIP]) and synthetic pure calcite (3.4% OOIP) cannot be attributed primarily to petrophysical heterogeneity, because the two cores exhibited comparable porosity and permeability. Instead, this discrepancy highlights the dominant role of inherent mineralogical impurities in controlling the effectiveness of low-salinity water flooding. The contact angle measurements quantitatively support this interpretation. LSW induced a 42° shift toward more water-wet conditions in the natural rock, compared with only 16° in the pure calcite. This pronounced wettability alteration is consistent with the greater incremental oil mobilization observed in the Shadegan limestone, indicating that rock–fluid interactions, rather than bulk flow properties, govern the additional recovery in this carbonate system.

4.2. Electrokinetic mechanisms and the synergistic role of trace impurities

The observed wettability alteration is primarily driven by electrokinetic phenomena at the rock/brine and oil/brine interfaces. As brine salinity decreased, the system pH increased from 7.4 to 8.6, promoting partial calcite dissolution and altering the speciation and adsorption behavior of potential-determining ions. In pure calcite, this increase in pH primarily enhanced the contribution of CO_3^{2-} species to the surface charge, resulting in a moderate negative zeta potential shift of -4.1 mV.

In contrast, X-ray fluorescence analysis of the natural limestone revealed the presence of silica, sulfate, and phosphate impurities, which collectively intensified the electrokinetic response. At elevated pH, silica surfaces undergo deprotonation ($\text{Si-OH} + \text{OH}^- \rightarrow \text{Si-O}^- + \text{H}_2\text{O}$), thereby contributing additional negative surface charge. At the same time, SO_4^{2-} and PO_4^{3-} ions can adsorb onto calcite surface sites and further increase the negative charge density. The combined effects of these processes resulted in a substantially larger negative zeta potential shift of -7.7 mV in the Shadegan limestone. This stronger negative surface charge enhances electrical double-layer (EDL) repulsion between the oil and rock interfaces, stabilizes a thicker and more continuous aqueous film, facilitates detachment of adhered oil droplets, and promotes a more strongly water-wet state.

4.3. Decoupling rock–fluid and fluid–fluid interactions

In contrast to previous studies that have emphasized fluid–fluid interactions as the principal mechanism governing LSW performance, such as microdispersion formation or reduction in interfacial tension, the present results indicate that these effects are not dominant in the investigated system. The interfacial tension between crude oil and brine increased as salinity decreased, whereas oil recovery increased substantially. This opposite trend indicates that interfacial tension reduction was not the primary mechanism responsible for the observed incremental recovery.

Instead, the experimental results are more consistent with a mechanism dominated by rock surface charge modification and wettability alteration. These findings suggest that, in clay-free carbonate reservoirs, LSW performance can be strongly controlled by the mineralogical composition of the rock, particularly the presence of trace non-carbonate impurities, rather than by bulk fluid chemistry alone.

4.4. Implications for low-salinity water design in heterogeneous carbonate reservoirs

The experimental results have important implications for enhanced oil recovery screening and engineered-brine design in carbonate reservoirs. Conventional approaches often approximate carbonate formations as homogeneous calcite systems, which can lead to underestimation of the potential

effectiveness of LSW flooding in naturally impure carbonate rocks. The present results demonstrate that trace non-clay minerals, including silica, sulfate-bearing phases, and phosphate-containing components, can significantly influence the electrokinetic response of the rock surface.

Accordingly, carbonate reservoirs that may initially appear unresponsive to LSW because of the absence of clay minerals could still exhibit substantial incremental recovery if favorable trace mineral components are present. Therefore, routine XRF analysis should be considered alongside conventional X-ray diffraction and core analysis during reservoir characterization. In addition, engineered brines for carbonate LSW applications should be designed not only on the basis of ionic strength, but also by considering the affinity of PDIs for the specific mineralogical and impurity composition of the reservoir rock. Such an approach may improve the interaction between injected ions and reactive mineral surfaces and thereby enhance wettability alteration and oil recovery.

4.5. Limitations and future research directions

Although this study isolates the influence of rock impurities under controlled core-scale conditions, several limitations should be considered. First, the experiments were conducted at a single reservoir temperature of 70 °C and pressure of 100 bar. Dissolution kinetics, ion adsorption behavior, and surface charge development may vary under different temperature and pressure conditions. Second, the use of a single crude oil composition limits the extent to which the observed fluid–fluid interactions can be generalized to oils with different asphaltene contents, acid numbers, and compositional characteristics. Third, prolonged injection of diluted brines may promote mineral precipitation, including CaSO_4 or $\text{Ca}_3(\text{PO}_4)_2$, which could alter permeability, surface reactivity, and EDL behavior during extended flooding periods.

Future studies should incorporate these electrokinetic findings into pore-network and reservoir-scale simulations, evaluate the economic feasibility of field-scale brine dilution, and investigate the long-term stability of wettability alteration under continuous LSW injection. Additional experiments involving different temperatures, pressures, crude oil compositions, and mineralogical conditions would also help establish the broader applicability of the proposed mechanism.

5. Conclusions

This study experimentally demonstrates that inherent mineralogical impurities can significantly enhance the effectiveness of low-salinity water flooding in carbonate reservoirs. Core-flooding experiments conducted under reservoir conditions showed that tertiary LSW injection recovered an additional 14.5% of the original oil in place (OOIP) from the natural Shadegan limestone, compared with only 3.4% OOIP from synthetic pure calcite with comparable petrophysical properties. This substantial difference in performance was attributed to a more pronounced wettability alteration toward water-wet conditions in the natural rock, as evidenced by a 42° reduction in contact angle compared with a 16° reduction in pure calcite.

Mechanistically, decreasing brine salinity produced a greater negative shift in the zeta potential of the Shadegan limestone (−7.7 mV) than in that of pure calcite (−4.1 mV). The resulting increase in negative surface charge strengthened electrostatic repulsion between the oil/brine and rock/brine interfaces, expanded the electrical double layer, and stabilized a thicker aqueous film on the rock surface. In pure calcite, the wettability alteration was primarily associated with the pH-induced changes in CO_3^{2-} concentration and adsorption resulting from calcite dissolution. In contrast, X-ray fluorescence analysis indicated that the stronger electrokinetic response of the Shadegan limestone was associated with its heterogeneous mineralogical composition. Trace impurities, particularly silica, sulfate, and phosphate,

acted synergistically to increase the negative surface charge during LSW injection. These findings indicate that non-clay mineral impurities can strongly influence rock–fluid interactions and demonstrate that inherent mineralogical heterogeneity should be considered when designing and optimizing LSW flooding and enhanced oil recovery strategies for complex carbonate formations.

Nomenclature

DLE	Double-layer expansion
DSA	Drop shape analysis
EDL	Electrical double-layer
EOR	Enhanced oil recovery
LSW	Low-salinity water
MIE	Multiple ion exchange
NMR	Nuclear magnetic resonance
OOIP	Original oil in place
PDM	Pendant drop method
TDS	Total dissolved solids
XRF	X-ray fluorescence

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