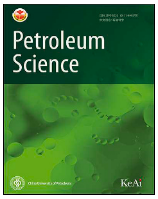
Petroleum Science 22 (2025) 4753–4765

Contents lists available at ScienceDirect

Petroleum Science

journal homepage: www.keaipublishing.com/en/journals/petroleum-science



Original Paper

Synergistic effects of chelating agents and surfactants for chemical EOR in carbonates

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ARTICLE INFO

Article history:
Received 26 February 2025
Received in revised form
14 July 2025
Accepted 18 July 2025
Available online 25 July 2025

Edited by Yan-Hua Sun

Keywords:
Chelating agent
Surfactant
Enhanced oil recovery
Carbonate reservoir

ABSTRACT

Surfactants play a critical role in enhanced oil recovery (EOR) applications; however, their performance is often compromised in harsh reservoir conditions, such as high temperature and high salinity, due to precipitation caused by interactions with multivalent metal ions. Chelating agents were introduced into oilfields for various purposes due to their ability to sequester metal ions. In this work, we conducted a comprehensive investigation about chelating agent-surfactant (CS) flooding for carbonate reservoirs, as an alternative to the well-established alkaline surfactant (AS) flooding used in sandstone. The tested surfactants include sodium dodecyl sulfate (anionic) (SDS), dodecyltrimethylammonium bromide (cationic) (DTAB), Triton X100 (nonionic), and a locally synthesized zwitterionic surfactant. The tested chelating agents include diethylenetriaminepentaacetic acid (DTPA), ethylenediaminetetraacetic acid, and glutamic acid *N*,*N*-diacetic acid. pH and temperature, as dominant factors in chelating agent solubility and brine stability, were modified to test chelating agent solutions of different concentrations and their mixtures with surfactants. Interfacial tension reduction by chelating agents alone, surfactants alone, and their mixtures were measured. Wettability alteration brought by chelating agents and surfactants on carbonate rock surfaces was evaluated using the static contact angle method. Based on the obtained results, chelating agents can be applied as low-cost additives for surfactant stabilization in high salinity conditions. The addition of chelating agents significantly improved the stability of SDS and DTAB in salt solutions and seawater. At a relatively low concentration (0.25 wt%), DTPA was able to stabilize DTAB of 1.00 wt% in seawater at high temperature (90 °C). DTPA, among the tested three chelating agents, exhibited a stronger stabilization effect on surfactants of different ion types. When chelating agents are to be applied in brine, an optimal applicable pH range of 5–9 is recommended so not to induce solubility issue of chelating agents or stability issues of metal ions. In this range, IFT reduction is more significant at high pH, while wettability alteration is more significant at low pH. The combination of a cationic surfactant with a chelating agent forms a low adsorption wettability modifier which can change strongly oil-wet rock to water-wet conditions, thus significantly increasing the residual oil recovery from oil-wet carbonate formations. Zwitterionic and nonionic surfactants are also applicable to combine with a chelating agent for EOR purposes. Anionic surfactant SDS, however, showed a growing inhibition on the wettability alteration effect induced by EDTA as the concentration of SDS increased.

1. Introduction

A chelating agent can form coordinate bonds with a central metal atom (Almubarak et al., 2021). By this process known as chelation, the metal atom is captured. This process can trigger two impacts: 1) enhanced dissolution due to the extraction of metal

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Peer review under the responsibility of China University of Petroleum (Beijing).

https://doi.org/10.1016/j.petsci.2025.07.009

atoms from the solid; 2) improved salinity-sensitive chemical stability in the solution due to the sequestration of metal ions. Chelating agents were introduced into oilfields for various purposes including iron control, carbonate stimulation, sandstone stimulation, scale removal, scale inhibition, filter cake removal, hydraulic fracturing, and enhanced oil recovery (EOR) (Hassan et al., 2020).

Chemical EOR faces challenges in carbonate rock, especially in harsh conditions. Popular chemicals such as surfactants and polymers can have stability issues when they are injected into formations with high salinity brine or at high temperatures. Besides, many carbonate reservoirs are naturally fractured, posing a challenge for water flooding and chemical flooding in the rock matrix due to the low pressure gradient caused by high permeability fractures. As a result, wettability alteration plays an even more important role in naturally fractured carbonate formations.

Recently, the EOR potential of chelating agents became the other focus of interest (Mahmoud et al., 2017). Chelating agents alone can contribute to oil recovery by interfacial tension (IFT) reduction and wettability alteration. Studies observed additional oil recoveries up to 20% (Mahmoud and Abdelgawad, 2015) and 32% (Hassan et al., 2021) from carbonate, and 19% from sandstone (Alarifi et al., 2018). For instance, chelating agents are found to be effective in altering rock wettability (Hassan et al., 2020). For carbonate rock, the reaction between the chelating agent and calcite results in the leaching of calcium ions from the rock surface. Removal of positively charged calcium ions from the rock surface leads to less adsorption of negatively charged organic materials such as carboxylic acids (Hou et al., 2015), resins, and asphaltenes (Tajikmansori et al., 2023). Mineral dissolution is also recognized as an important mechanism (Daneshfar et al., 2023), though there are debates about the relationship between mineral dissolution and wettability alteration. Some researchers observed a relationship between calcite dissolution and wettability alteration (AlMahri et al., 2024; Loreto et al., 2024; Noruzi et al., 2024), though some studies claim that mineral dissolution is unrelated to wettability change (Opoku Boampong et al., 2023). Apart from mineral dissolution, the adsorption of chelating agent on carbonate rock surfaces is claimed to be the dominant factor for wettability alteration (Hassan et al., 2021). For sandstone rock, factors including the chlorite dissolution (El-Din Mahmoud, 2018), the chelating of iron from clay minerals (Mahmoud and Al-Hashim, 2019), and wettability alteration (Alarifi et al., 2018; Mohammadzadeh et al., 2023) are found to be dominant mechanisms in EOR by DTPA. On the other hand, chelating agents can also reduce the oil/water IFT, alone or with other EOR materials. Chelating agents can reduce the oil/water IFT, especially at lifted pH (Mohammadi Khanghah and Parhizgar Keradeh, 2024; Mohammadzadeh et al., 2024). For example, Mohammadi Khanghah and Parhizgar Keradeh (2024) observed an IFT reduction by DTPA from 29.52 to 3.37 mN/m.

Combining a chelating agent with a surfactant is a promising idea since a chelating agent has the potential to sequester multivalent metal ions, thus enhancing the stability of surfactants in high salinity brines. Commonly used EOR chemicals such as polymers (Algharaib et al., 2014) and surfactants (Hou et al., 2024; Mohamed et al., 2017) face performance degradation and stability challenges in harsh conditions (high temperature, high salinity). Counterions in water shield the charge on surfactant head groups, thus reducing the repulsion among surfactant molecules, resulting in aggregation and precipitation. To address these issues, high-cost additives or novel developed surfactants are usually applied, which can drastically increase the cost of the project. Chelating agents can sequester the metal ions and possibly reduce the impact of salts on surfactant stability, thus helping stabilize low-cost surfactants in high salinity conditions and enhance their effectiveness (Velásquez et al., 2024; Yunusov et al., 2023b; Zheng et al., 2025).

Apart from potential stability enhancement, the addition of a chelating agent in surfactant can reduce the interfacial tension as well (Yunusov et al., 2023b). There are tests showing that the IFT reduction was strongly dependent on the interactions between surfactants and chelating agents (Mohammadi Khanghah and Parhizgar Keradeh, 2024). There are also reports about the enhanced wettability alteration by mixing chelating agents with surfactants (Deng et al., 2022a, 2022b; Yunusov et al., 2023a). Besides, adding chelating agents can have an impact on surfactant behavior in other aspects such as viscosity (Ahmed et al., 2023). Significant incremental oil recovery was observed by the chelating agent alone, and the chelating agent + surfactant mixture. Janjua et al. (2021) flooded oil-wet carbonate rock with DTPA and VES, separately and combined, and obtained additional oil recovery of 5.82%–9.64%. Mahmoud and Abdelgawad (2015) flooded sandstone and carbonate cores with 5 wt% HEDTA and EDTA in seawater. More than 20% OOIP was recovered. Hassan et al. (2021) obtained 19%–32% OOIP incremental oil recovery by flooding cores with GLDA. Al-Azani et al. (2024) combined DTPA with an in-house synthesized cationic Gemini surfactant to form an effective chemical flooding composition the displaced an incremental 37% OOIP. Deng et al. (2022a) combined DTPA with two surfactants and obtained an imbibition oil recovery of 10%–13%. These studies demonstrate the promising potential of the chelating agent alone as well as chelating agent + surfactant mixtures in carbonate EOR.

To date, there is limited study of the EOR potential of the combinations of chelating agents and surfactants. It is necessary to ensure the stability and compatibility of the EOR fluid before application. Solubility is the first factor to consider. The solubility of chelating agents in the aqueous phase largely depends on their protonation stage (Kragten and Decnop-Weever, 1983). The more chelating agents are protonated, the less soluble they are in water (Hassan et al., 2020). The protonation stage depends on the pH condition. The lower the pH, the more chelating agents are protonated (Chen and Reid, 2011; Fredd and Fogler, 1998). As a result, chelating agents are more soluble at lifted pH conditions (Almubarak et al., 2021). The suggested concentration is usually no lower than 5 wt% for advantages including a much lower IFT value (Mohammadi Khanghah and Parhizgar Keradeh, 2024), a strong chelation process on the rock surface (Daneshfar et al., 2023), less clay swelling, reduced cost in dilution (Parhizgar Keradeh and Tabatabaei-Nezhad, 2023), and so on. A higher concentration of chelating agent usually requires a higher pH for dissolving. However, the application of high-pH liquids into the reservoir is not always practical. For example, the commonly adopted alkaline-surfactant-polymer (ASP) flooding technique is not applied to high salinity formations due to precipitation issue (Almubarak et al., 2021). It is important to explore the efficiency of chelating agents in neutral or acidic conditions.

In this study, three types of commonly used chelating agents, DTPA/EDTA/GLDA, were tested regarding their solubility, enhancement in surfactant compatibility with brines, IFT reduction, and wettability alteration potential. Multiple types of surfactants were tested and combined with chelating agents to better understand the interactions between the chelating agent and the surfactant and find potential solutions for carbonate EOR projects.

2. Experimental

2.1. Crude oil

Crude oil (0.914 g/cm^3 at 23.0 °C) collected from an oilfield in the Kingdom of Saudi Arabia was used in this study. It has a viscosity of 91.216 mPa·s at 22 °C. Its SARA composition is shown in Table 1.

2.2. Indiana limestone

Low permeability (< 10 mD) Indiana limestone outcrops were cored and sectioned into substrates (25.4 mm in diameter, 3–4 mm in thickness). XRD study showed that carbonate mineral constitutes 100% of the outcrop, making it an ideal representative for carbonate rocks. Substrates were cleaned with toluene and methanol in a Soxhlet extractor to remove organic and inorganic contaminants. They were then dried and soaked in crude oil in the oven at 90 °C for at least 2 weeks to render the initial oil-wetness. After soaking in the tested solutions, the wettability changes were measured, recorded, and compared.

2.3. High salinity water and brine

Brines of different compositions were prepared by dissolving salts (NaCl, $MgCl_2$, $CaCl_2$, and Na_2SO_4, purchased from Sigma-Aldrich) into deionized water. Concentrations and salinities are given in Table 2. The prepared "2 × HSW" has a salinity close to twice that of the high salinity water.

2.4. Surfactants and chelating agents

Table 3 lists the chemicals of interest in this study, including three chelating agents and four surfactants covering all ion types. DTPA/EDTA/GLDA are the three most used chelating agents. Sodium dodecyl sulfate (SDS), dodecyltrimethylammonium bromide (DTAB), a locally synthesized zwitterionic surfactant (ZW), and Triton X100 are representatives of surfactants from different ion types.

The following experiments were conducted to assess the applicability and efficiency of applying chelating agents and surfactants together for EOR purposes. Solubility tests show the applicable pH range and the impact of the concentration of chelating agents in brines. Compatibility tests show the impact of different salts, brines, and chelating agents, on the stability of different types of surfactants. IFT measurements were conducted to evaluate the effectiveness of different surfactants, different chelating agents, and their mixtures in reducing the IFT between oil and water. Contact angle measurements show the effectiveness of these materials and their mixtures in altering strongly oil-wet carbonate rock towards water-wet conditions.

2.5. Solubility and compatibility tests

Stock solutions of 20.00 wt% of chelating agents (DTPA/EDTA/GLDA) were prepared. DTPA and EDTA were dissolved in deionized water by gradually adding NaOH. 20.00 wt% GLDA was prepared by diluting from 40.00 wt% GLDA solution. 20.00 wt% solutions were then diluted to different concentrations. Since the received stock solutions were of high pH, HCl was added to the dilutions to modify the pH when needed. A SevenExcellence pH meter manufactured by METTLER TOLEDO was used for real-time pH monitoring. Three readings were taken and averaged for each liquid.

Table 1
SARA composition of the crude oil.

Composite	Saturates	Aromatics	Resins	Asphaltenes
Content, %	23.39	46.63	25.69	4.29

Table 2
Salt compositions of the diluted high salinity water, high salinity water, and double high salinity water.

Water	Concentration, g/L					TDS, g/L
	$NaHCO_3$	Na_2SO_4	NaCl	$CaCl_2$	$MgCl_2$	
0.5 × HSW	0.09	3.17	20.59	0.90	4.14	28.87
1 × HSW	0.17	6.34	41.17	1.80	8.27	57.74
2 × HSW	0.26	10.07	68.02	3.13	13.07	94.55

A high salinity environment poses a challenge to the stability of surfactants. Different surfactant solutions were tested in their compatibility with different brines prepared by dissolving a single type of salt, or by combining different kinds of salts. The stability of the mixture between the surfactant and chelating agent in brines was also tested to show the possible improvement of compatibility brought by the chelating agent.

2.6. IFT measurement

Interfacial tension measurements were conducted using the drop shape analyzer (DSA 25B) (adopting the pendant drop method (Herd et al., 1993)) and the spinning drop tensiometer (adopting the spinning drop method (Manning and Scriven, 1977)) manufactured by KRUSS. All measurements were done at 22 °C and 1 atm.

2.7. Contact angle measurement

Contact angle measurements (Manning and Scriven, 1977) were conducted using the drop shape analyzer (DSA 25B) (adopting the sessile drop method, accuracy 0.1°). At least four readings were taken from each sample and then averaged to represent the overall wettability condition of the sample. Contact angle changes before and after treatment were adopted to evaluate the wettability alteration performance of the tested EOR fluids.

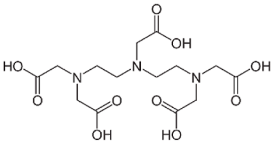
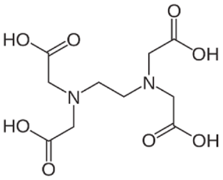
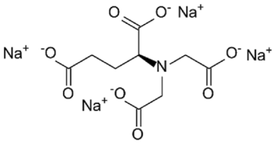
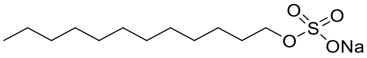
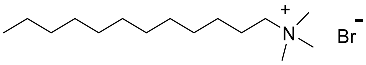
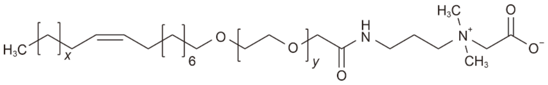
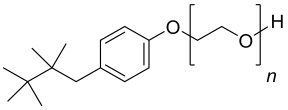
3. Results and discussion

3.1. Solubility of chelating agent in deionized water

DTPA/EDTA/GLDA was dissolved in water with modified pH (by adding HCl or NaOH) values at various concentrations. DTPA and GLDA showed better solubility than EDTA in water. Under the tested conditions (0.10–1.00 wt%, pH = 2–13), both DTPA and GLDA dissolved completely to form clear solutions. EDTA, however, is less soluble at low pH values of 2–4, as shown in Fig. 1.

In most field applications, chelating agents are used at a higher concentration of up to 20.00 wt% (Hassan et al., 2020). Higher concentrations of the most commonly used chelating agent, EDTA, were tested. Results showed that even at weak acidic conditions (pH = 5), EDTA did not fully dissolve when its concentration was lifted to 4.00 and 5.00 wt%. DTPA has a better solubility in water than EDTA (Barri et al., 2022) because DTPA has one more carboxylic group that helps form hydrogen bonds with surrounding water molecules (Rämö et al., 2000). GLDA has a better solubility than EDTA possibly (Mahmoud et al., 2011) because it has a better molecular flexibility to adapt better to the hydration shell (Hazrina et al., 2018). It is recommended to use DTPA or GLDA instead of EDTA in acidic conditions.

Table 3
Chelating agents and surfactants used in this study.

Name	Ion type	Formula
DTPA		
EDTA		
GLDA		
SDS	Anionic	
DTAB	Cationic	
ZW	Zwitterionic	
Triton X100	Nonionic	

3.2. Solubility of chelating agent in brine

When chelating agents were dissolved in high salinity water (1 × HSW), precipitation was observed at high pH conditions. At a neutral condition (pH = 6–7), DTPA/EDTA/GLDA (0.10–1.00 wt%) in high salinity water are stable. At high pH, though the solubility of chelating agents is enhanced, excess hydroxyl ions can cause precipitation issues. Metal ions in the brines form precipitates with OH^- ions. Fig. 2 shows the precipitations in two vials containing EDTA solutions (1.00 wt%) in diluted high salinity water (0.5 × HSW) and high salinity water (1 × HSW) at lifted pH of 10.3.

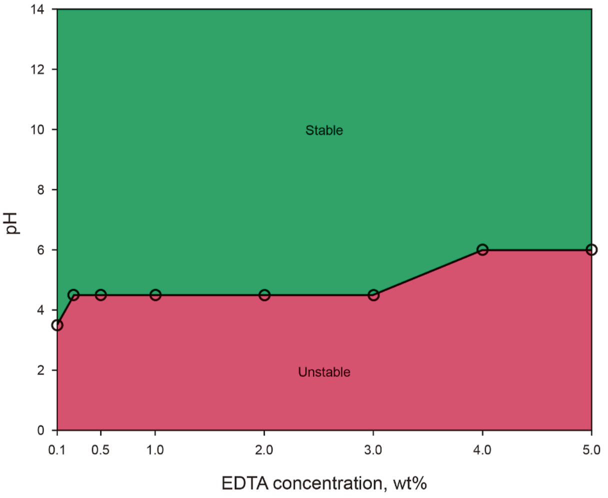


Fig. 1. Solubility of EDTA in water at modified pH and various concentrations.

Fig. 2. Precipitation when 1.00 wt% EDTA at high pH value (pH = 10.3) was added to different brines.

High salinity water (1 × HSW) tested at different pH levels shows no precipitation at temperatures between 22 and 90 °C when the pH is 9 or lower. At a pH of 9.9, precipitation was observed. Temperature change (22–90 °C) seemed to have no impact on the amount of precipitation (as shown in Fig. 3).

In summary, apart from considerations of the low-pH-caused crystallization of chelating agents in water, the high-pH-caused metal ion precipitation is also an important factor to consider before the application. An applicable pH range of 5–9 is recommended.

3.3. Solubility of surfactants in brine

SDS, being one of the most recognized surfactants (Massarweh and Abushaikha, 2020), is not stable at lifted salinity. Compatibility tests were conducted in brines of different salts and under varying temperatures. Concentrations were selected to cover the salts' concentrations in the high salinity water. The concentrations of $CaCl_2$, $MgCl_2$, and NaCl in high salinity water are 0.180, 0.827, and 4.117 g/L, covered in the tested ranges. In this way, the obtained results can be utilized in understanding the solubility of SDS in high salinity water.

Results shown in Table 4 indicate that multivalent metal ions, Ca^{2+} and Mg^{2+}, have a much stronger impact on SDS than monovalent metal ion Na^+. At 3.54 wt%, NaCl failed to precipitate SDS, while $MgCl_2$ did. $CaCl_2$ precipitated SDS at much lower concentrations. Multivalent metal ions have a stronger impact on SDS stability since they form insoluble salts, calcium dodecyl sulfate, and magnesium dodecyl sulfate, with SDS.

DTAB and Triton X100 were found to have far better compatibility than SDS in different brines. DTAB remained stable in high salinity water and 2 × HSW, at concentrations of 1.0–2.0 wt% in room conditions for 1 day.

After 1 week, precipitations were observed in DTAB solutions. The sample at lifted temperature (90 °C) showed quicker and more precipitation than the sample in room conditions (22 °C), as shown in Fig. 4.

Triton X100 was found to have the best compatibility with different brines. Triton X100 remained stable in all tested conditions (22–90 °C, 1 × HSW, 2 × HSW, 1.00–2.00 wt%) for 2 weeks.

3.4. Solubility of chelating agent-surfactant (CS) solutions in brine

3.4.1. Solubility of SDS in different chelating agents

A series of tests about the impact of high salinity water on the compatibility of surfactant + chelating agent mixtures were conducted.

Table 5 lists the results from all tested SDS + EDTA mixtures at various brine compositions, concentrations, and temperatures.

Table 4
Impact of different salts and temperature on the stability of SDS in brines (0.50 wt% SDS).

Salt	Salt concentration, wt%	Stability of SDS in brine at different temperatures			
		22 °C	50 °C	70 °C	90 °C
$CaCl_2$	0.09	F	F	T	T
	0.18	F	F	T	T
	0.36	F	F	T	T
	0.71	F	F	T	T
	1.43	F	F	T	T
$MgCl_2$	0.44	F	T	T	T
	0.89	F	T	T	T
	1.77	F	T	T	T
	3.54	F	T	T	T
	7.09	F	F	T	T
NaCl	3.54	T	T	T	T
	7.09	F	T	T	T
	14.17	F	T	T	T

Note: T stands for stable, F for unstable.

Fig. 4. DTAB solutions (1.00 wt%) in 1 × HSW at 22 and 90 °C after 1 week.

Multivalent metal ions (Ca^{2+}, Mg^{2+}, Al^{3+}) are believed to be the prime factor in the precipitation of SDS. In the tests, an increased concentration of $CaCl_2$ produced more precipitation, as shown in Fig. 5. In such cases, adding EDTA can largely mitigate the

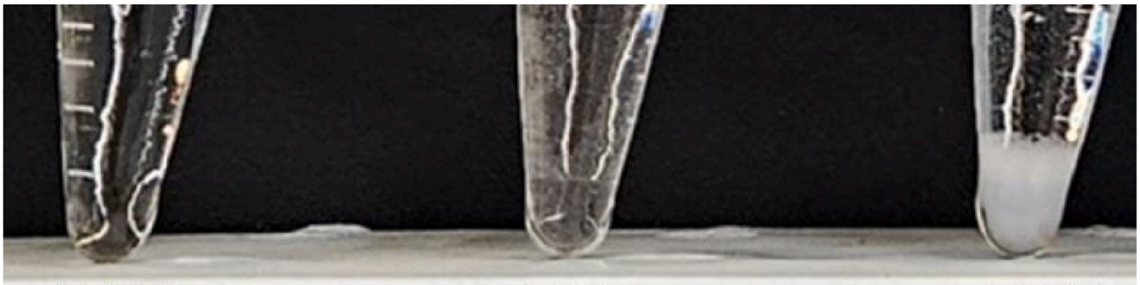


Fig. 3. Stability of high salinity water (1 × HSW) at different pH values.

Table 5
Solubility of SDS-EDTA in $CaCl_2$ solution.

SDS, wt%	EDTA, wt%	$CaCl_2$, wt%	Stability of SDS-EDTA at different temperatures			
			22 °C	50 °C	70 °C	90 °C
1.0	3.00	0.05	T	T	T	T
	3.00	0.10	T	T	T	T
	3.00	0.20	F	T	T	T
	3.00	0.40	F	T	T	T
	3.00	0.80	F	T	T	T
1.0	1.00	0.20	F	T	T	T
	2.00	0.20	F	T	T	T
	3.00	0.20	F	T	T	T
	1.00	0.40	F	T	T	T
	2.00	0.40	F	T	T	T
	3.00	0.40	F	T	T	T
0.5	3.00	0.20	F	T	T	T
1.0	3.00	0.20	F	T	T	T
2.0	3.00	0.20	F	T	T	T
0.5	3.00	0.40	F	T	T	T
1.0	3.00	0.40	F	T	T	T
2.0	3.00	0.40	F	T	T	T

negative impact of $CaCl_2$. When the EDTA concentration increased, the amount of precipitated solids was reduced.

A higher concentration of SDS led to more precipitation, as shown in Fig. 6. The higher the EDTA concentration, the smaller the amount of precipitation, as shown in Fig. 7. This observation indicates that EDTA contributed to stabilizing SDS. Besides, a lifted temperature also largely improves the compatibility of SDS in $CaCl_2$ solutions with a concentration of 1/4 to 4 times that of $CaCl_2$ in high salinity water. At a temperature no lower than 50 °C, no solid was observed. This behaviour may be attributed to the chelating ability of EDTA. Before the addition of EDTA, the positive part of $CaCl_2$ (Ca^{2+}) was freely binding with the negative part of SDS (SO_4^-). More addition of $CaCl_2$ means more attraction of positive and negative parts of $CaCl_2$ and SDS, respectively, which results in more precipitation. The addition of EDTA minimizes the binding of $CaCl_2$ and SDS through interaction with $CaCl_2$ which ultimately reduced the amount of precipitation.

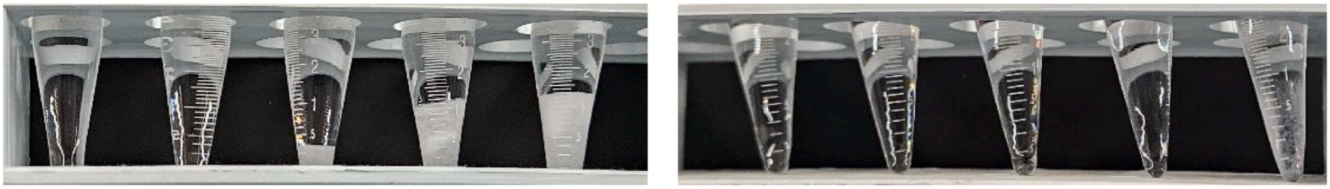
DTPA and EDTA were compared in the aspect of SDS compatibility enhancement in high salinity water, with solution composition listed in Table 6.

Results are shown in Fig. 8. DTPA (0.50 and 1.00 wt%) stabilized the 1.00 wt% SDS in high salinity water. EDTA, however, failed to stabilize SDS in this concentration range (0.10–1.00 wt%). In stabilizing SDS in brines, DTPA is stronger than EDTA. This can be due to the fact that DTPA has a stronger chelating efficiency than EDTA, thus further eliminating the negative impact of metal ions on the stability of SDS (Norvell, 1984).

3.4.2. *Solubility of DTAB in different chelating agents*

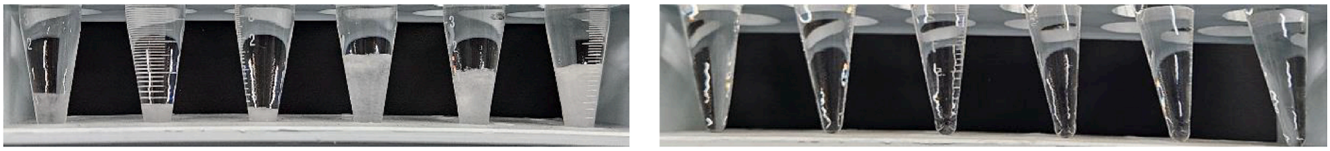
A comparison of GLDA and DTPA in enhancing DTAB compatibility with brines is summarized in Table 7.

Results are shown in Fig. 9. After 1 week, the solution with DTPA remained clear. The two solutions with GLDA were not as



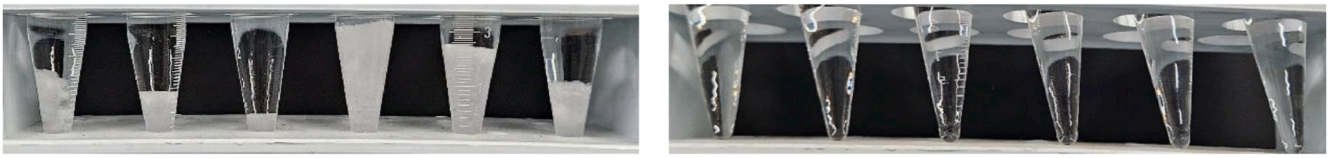
22 °C 50 °C

Fig. 5. Compatibility results of SDS (1 wt%)-EDTA (1 wt%) solutions at different $CaCl_2$ concentrations (left to right: 0.05/0.10/0.20/0.40/0.80 wt%).



22 °C 50 °C

Fig. 6. Compatibility results of 0.20 wt% $CaCl_2$ + 2.00/1.00/0.50 wt% SDS + 3 wt% EDTA (three in the left) and 0.40 wt% $CaCl_2$ + 2.00/1.00/0.50 wt% SDS + 3.00 wt% EDTA (three in the right).

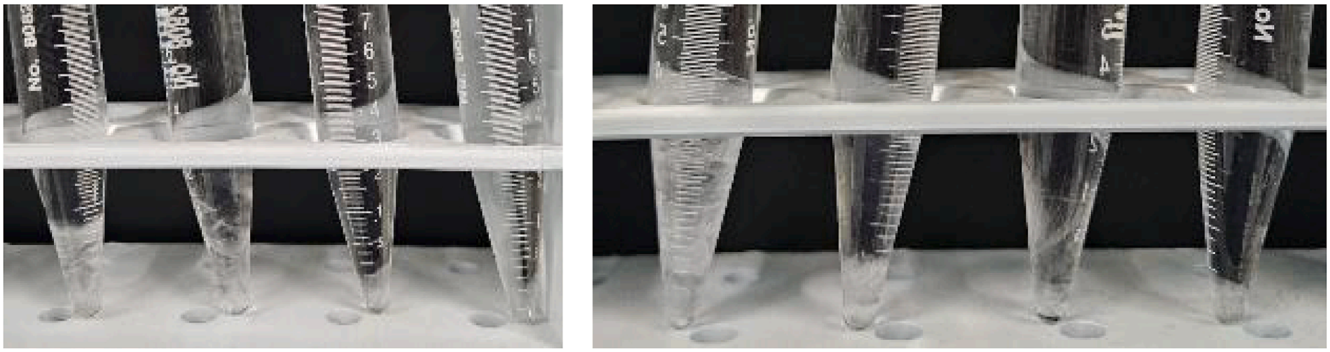


22 °C 50 °C

Fig. 7. Compatibility results of 0.20 wt% $CaCl_2$ + 1.00 wt% SDS +1.00/2.00/3.00 wt% EDTA (three in the left) and 0.40 wt% $CaCl_2$ + 1.00 wt% SDS + 1.00/2.00/3.00 wt% EDTA (three in the right).

Table 6
Composition of the solutions in the test of SDS compatibility enhancement by DTPA and EDTA.

No.	SDS concentration, wt%	DTPA concentration, wt%	EDTA concentration, wt%
1	1.00	0.10	0
2	1.00	0.25	0
3	1.00	0.50	0
4	1.00	1.00	0
5	1.00	0	0.10
6	1.00	0	0.25
7	1.00	0	0.50
8	1.00	0	1.00


1.00 wt% SDS + 0.10/0.25/0.50/1.00 wt% DTPA

1.00 wt% SDS + 0.10/0.25/0.50/1.00 wt% EDTA

Fig. 8. Compatibility results of the SDS + chelating agent mixtures in high salinity water.

Table 7
Composition of the solutions in the test of DTAB compatibility enhancement by DTPA and GLDA.

No.	DTAB concentration, wt%	DTPA concentration, wt%	GLDA concentration, wt%	Temperature, °C
1	1.00	0	0	90
2	1.00	0.25	0	90
3	1.00	0	0.25	90
4	1.00	0	0.50	90


No chelating agent

With 0.50 wt% GLDA

With 0.25 wt% GLDA

With 0.25 wt% DTPA

Fig. 9. Compatibility results of the DTAB + chelating agent mixtures in high salinity water.

Table 8
Composition of the solutions in the test of DTAB compatibility enhancement by DTPA.

No.	DTAB concentration, wt%	DTPA concentration, wt%	Temperature, °C
1	1.00	0	90
2	1.00	0.25	90
3	2.00	0	90
4	2.00	0.25	90

clear as the DTPA case but can still be considered stable. The solution with no chelating agent showed significant precipitation. Results indicate that DTPA is stronger than GLDA in enhancing the compatibility of DTAB with high salinity water. The high salinity water contains different ions which hinder the solubility of DTAB. The addition of GLDA minimizes the precipitation by interacting with various ions in high salinity water. The reason behind the clear solution using DTPA compared to GLDA could be the stronger

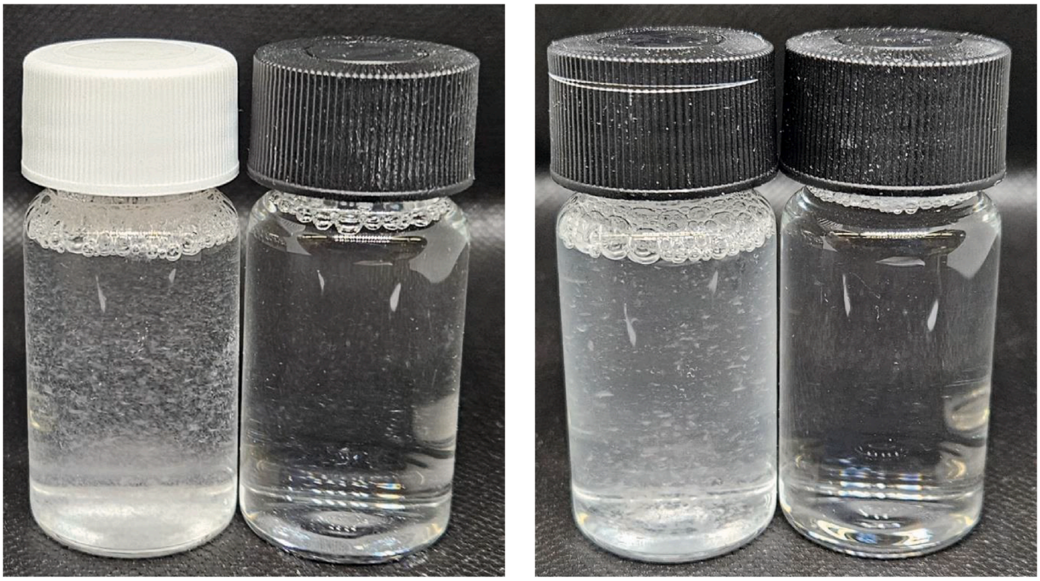

1.00 wt% DTAB 1.00 wt% DTAB + 0.25 wt% DTPA 2.00 wt% DTAB 2.00 wt% DTAB + 0.25 wt% DTPA

Fig. 10. Impact of DTAB concentration on the long-term (39 days) stability of the DTAB + DTPA mixtures in high salinity water (1 × HSW).

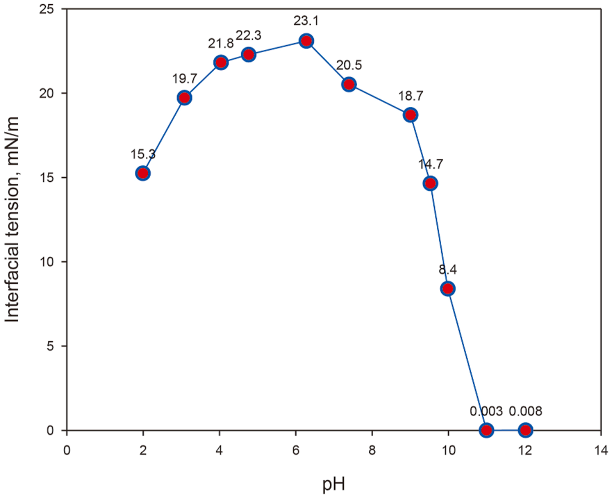


Fig. 11. IFT values between crude oil and water at different pH values.

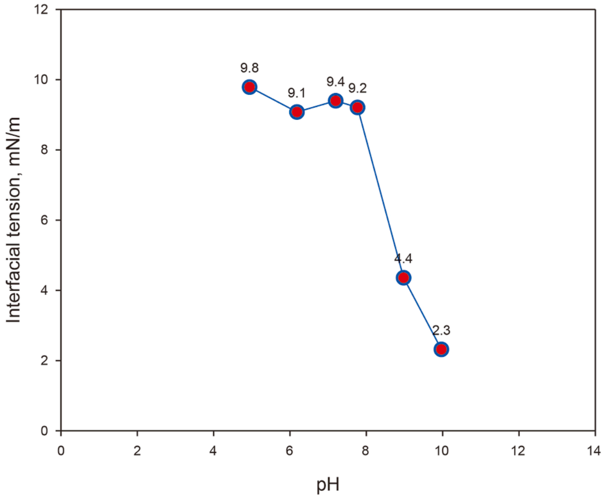


Fig. 12. IFT values between 1 wt% EDTA solution and crude oil.

Table 9
IFT values between crude oil and different EOR fluids.

Chelating agent	Chelating agent concentration, wt%	Surfactant	Surfactant concentration, wt%	pH	Stable IFT, mN/m
		SDS	1.00	6.4	1.74
		SDS	1.00	11.2	0.44
		DTAB	1.00	5.5	0.38
		DTAB	1.00	11.0	0.12
		Triton X100	1.00	4.2	0.56
		Triton X100	1.00	11.0	0.53
		ZW	1.00	3.8	2.72
		ZW	1.00	10.9	0.39
		GS	1.00	3.2	2.28
EDTA	1.00			6.2	9.08
EDTA	1.00			10.2	4.36
EDTA	1.00	SDS	1.00	10.9	0.46
EDTA	1.00	SDS	0.10	10.9	1.38
EDTA	1.00	SDS	0.01	11.2	15.88
EDTA	1.00	SDS	0.001	11.1	8.75
EDTA	1.00	DTAB	1.00	10.8	0.31
EDTA	1.00	Triton X100	1.00	10.8	0.28
EDTA	1.00	Triton X100	0.10	11.2	0.31
EDTA	1.00	Triton X100	0.01	11.1	0.23
EDTA	1.00	Triton X100	0.001	10.4	7.00
EDTA	1.00	ZW	1.00	11.0	0.35

Table 10
IFT recovery under high pH conditions.

EDTA (chelating agent) concentration, wt%	Surfactant	Surfactant concentration, wt%	pH	Immediate IFT, mN/m	Stable IFT, mN/m
1.00	SDS	0.10	10.9	0.57	1.38
		0.01	11.2	0.013	15.88
		0.001	11.1	0.014	8.75
	Triton X100	0.10	11.2	0.32	0.31
		0.01	11.1	0.24	0.23
		0.001	10.4	0.019	7.00

Table 11
Surfactant solution compositions for the treatment of strongly oil-wet Indiana limestone substrates.

Surfacant solution	Concentration, wt%	Original pH	Low pH	Medium pH	High pH
SDS	1.00	7.3	3.3	7.3	10.7
DTAB	1.00	5.7	2.8	7.3	10.6
Triton X100	1.00	4.4	3.3	6.0	10.8
ZW	1.00	3.9	3.1	7.4	10.5

chelation power. The DTPA contains 8 coordination sites while GLDA has 5 coordination sites. Due to extra coordination sites, DTPA chelates strongly with the ions of high salinity water which results in enhancing the compatibility of DTAB with high salinity water.

A series of tests were conducted to study the impact of DTAB. Details of the four tested solutions are given in Table 8.

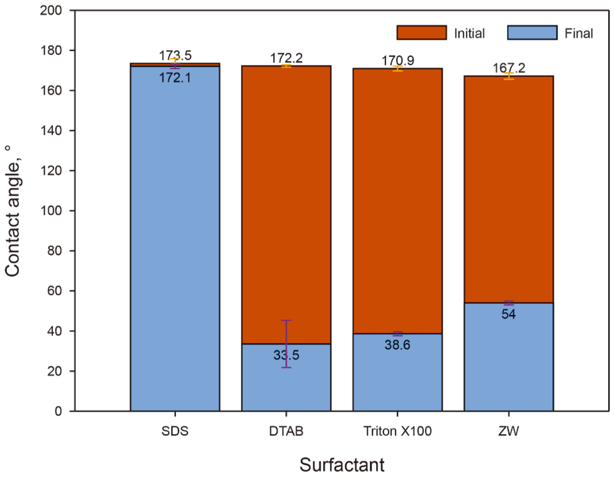
Results are shown in Fig. 10. After 39 days, both DTAB + DTPA mixtures remained stable, confirming the strong enhancement brought by DTPA at a low concentration of 0.25 wt%.

3.5. IFT reduction

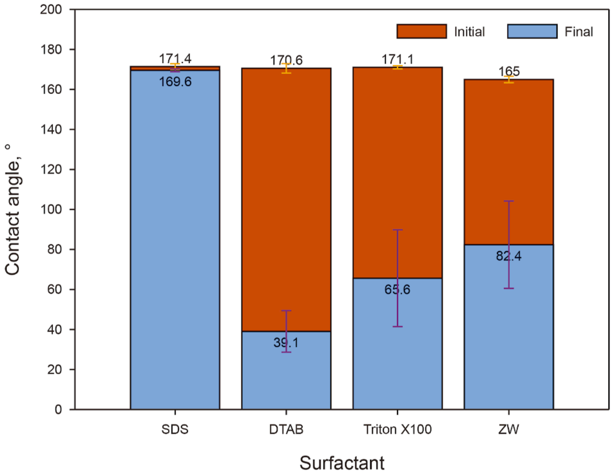
IFT between crude oil and DI water was measured at different pH conditions to see the impact of pH on IFT value. As shown in Fig. 11, results indicate that both acidic and basic conditions facilitate the reduction of IFT, likely due to the generation of in-situ surfactant. Comparably speaking, for the tested crude oil, alkali reduced IFT more significantly than acid.

1.0 wt% EDTA solution was modified by adding HCl to reduce the pH value. The IFT values between 1.0 wt% EDTA solution and crude oil at different pH values were obtained, as shown in Fig. 12.

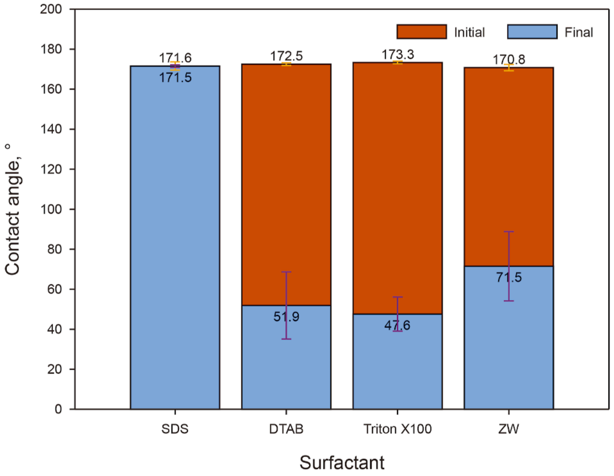
When only surfactant was applied, the resulting IFT values were generally higher than the values obtained by the mixture of EDTA and surfactants at high pH values. Table 9 summarizes the IFT values obtained between crude oil and different EOR fluids. Comparing surfactants and their mixtures with EDTA at high pH values, the surfactant + EDTA combination achieved lower IFT. Chelating agents are found in several previous studies to enhance surfactants' interfacial behavior (Zheng et al., 2025) to form stronger foams (Al-Darweesh et al., 2023, 2024), and further reduce the IFT (Velásquez et al., 2024; Yunusov et al., 2023b).



(a) Low pH



(b) Medium pH



(c) High pH

Fig. 13. Contact angle values before and after the soakage treatment in four surfactant solutions at different pH levels.

Possible explanations include sequestration of the metal ions to mitigate the salting-out impact on surfactants, and the interactions between surfactant and chelating agent molecules.

The main mechanism of the significant IFT reduction in the EDTA + surfactant at high pH cases was in-situ surfactant generation caused by the reaction between excess OH^- ions and acidic components in the crude oil. However, an IFT recovery was frequently observed in high pH conditions. The low IFT obtained in high pH cases did not last long. The IFT underwent a sharp decrease and then a gradual increase process. A low IFT was achieved in the beginning (referred to as “immediate IFT” in Table 10) and then stabilized at a higher value (referred to as “stable IFT” in Table 10). This phenomenon could be a result of the partitioning of in-situ generated surfactants into the aqueous phase.

3.6. Wettability alteration

Strongly oil-wet Indiana limestone substrates were soaked in surfactant concentrations of 1.00 wt% at three pH levels: low, medium, and high, as detailed in Table 11. The treatment lasted for 20 days. After that, contact angles on the substrates’ surfaces were measured and compared with the initial angles, as plotted in Fig. 13.

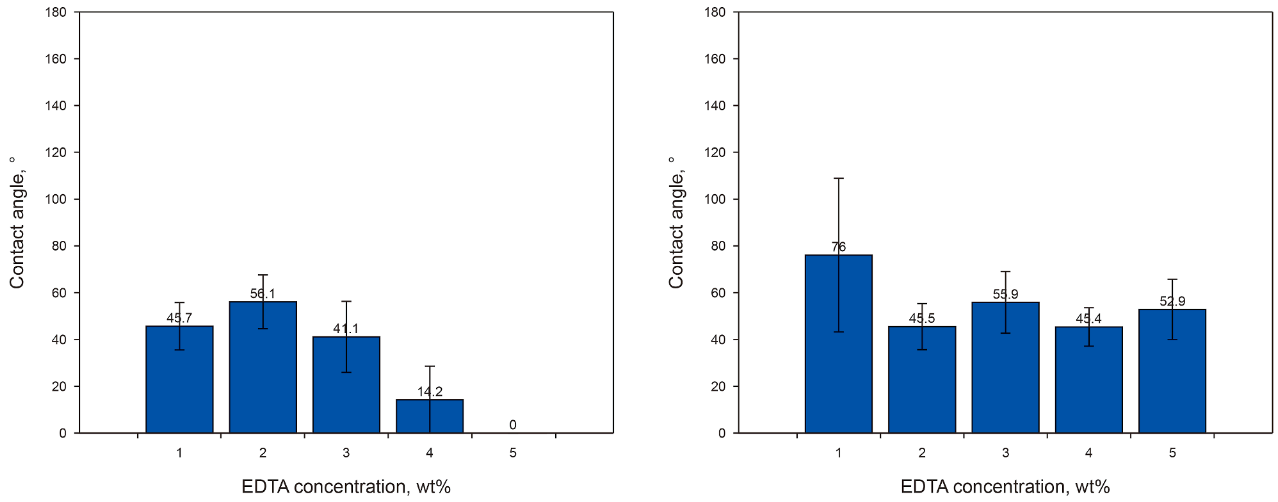
Among the tested four surfactants, DTAB appeared to have the strongest wettability alteration performance, while SDS showed nearly no impact. An increased pH hurts the wettability alteration induced by DTAB. However, this effect was not pronounced in Triton X100 and ZW cases. A possible explanation is that excess OH^- ions can bind with the cationic surfactant molecules, thus limiting the interaction between the cationic surfactant and negatively charged organic molecules adsorbed on the rock surface.

Chelating agents can modify carbonate rock wettability. pH is found to be an important factor. A series of tests were conducted to measure the contact angle values after treating oil-aged rock in EDTA solutions at different concentrations (1.00–5.00 wt%) and pH values. Data was plotted as Fig. 14. Generally, lower pH and higher concentration lead to stronger wettability alteration. The impact of a lower pH can be explained by enhanced mineral dissolution, which can contribute to wettability alteration. The impact of a higher concentration can be explained by enhanced surface chelation of calcium ions from the rock.

The wettability alteration performance of the mixture between EDTA and different types of surfactants was checked, as plotted in Fig. 15.

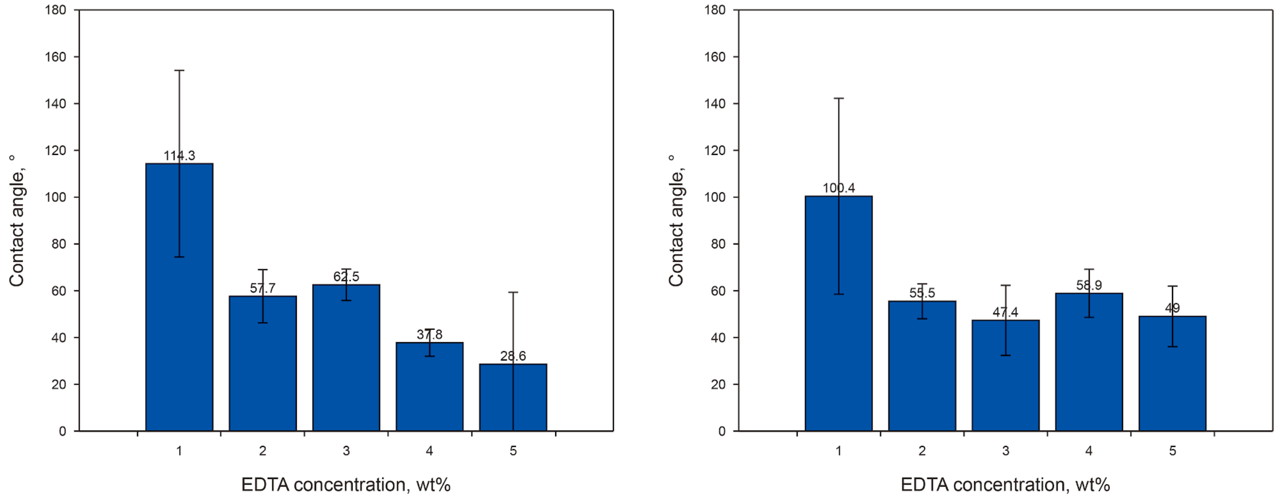
Without surfactant, 1.00 wt% EDTA at pH around 11 achieved neutral-wet on the rock surface. Among all tested surfactants, the locally synthesized zwitterionic surfactant has the best synergic effect with EDTA on wettability alteration. Cationic and nonionic surfactants DTAB and Triton X100 also changed strongly oil-wet surface to water-wet conditions. Mixing EDTA and SDS hurts the wettability alteration. A more detailed study comparing the impact of SDS and Triton X100 was conducted, as plotted in Fig. 16.

The negative/positive impact of surfactant on the wettability alteration potential of the mixture between 1.00 wt% EDTA and the surfactant is enhanced by the increased concentration. A possible explanation for the negative impact of anionic surfactant is that chelating agents alter rock wettability by adsorption,



(a) pH = 3

(b) pH = 7



(c) pH = 9

(d) pH = 13

Fig. 14. Wettability alteration of carbonate rock by EDTA at different concentrations (1.00–5.00 wt%) and different pH (3–13) conditions.

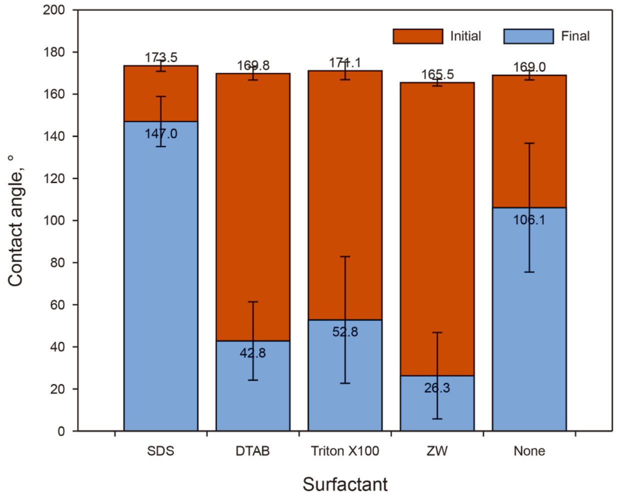
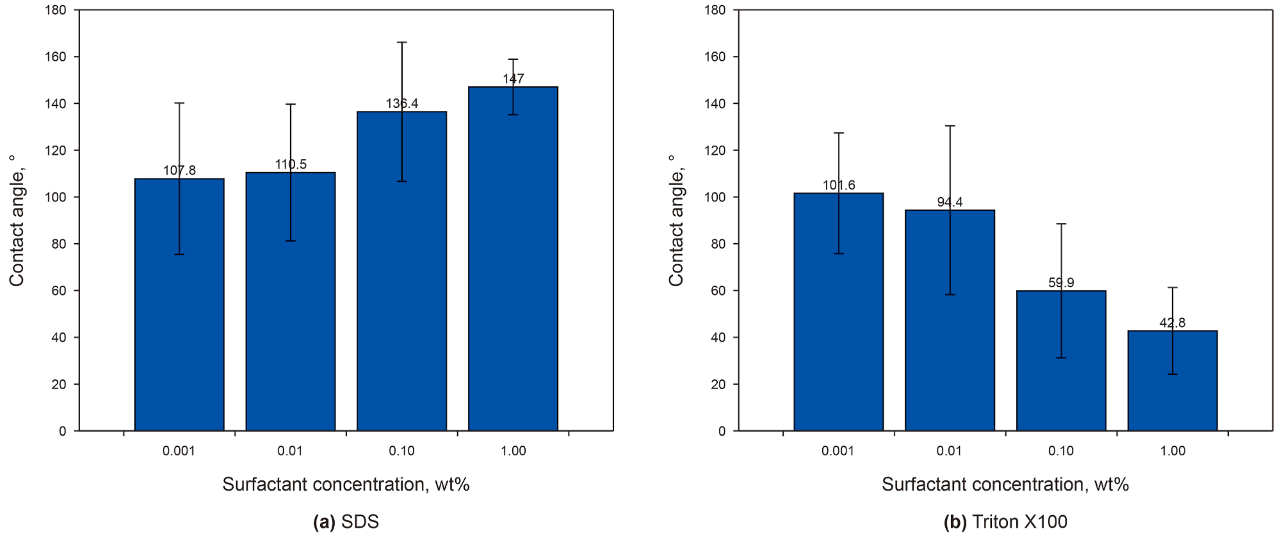


Fig. 15. Contact angle changes of strongly oil-wet Indiana limestone substrates soaked in 1.00 wt% EDTA with/without 1.00 wt% surfactant.

while anionic surfactant molecules are also prone to the positively charged carbonate surface. As a result, there is a competitive adsorption between the anionic surfactant and EDTA, limiting the fulfillment of EDTA's wettability alteration potential.

Demands for carbonate EOR come largely from the residual oil trapped in highly heterogeneous carbonate formations, where low-permeability regions remain unflooded (Dong et al., 2002; Lu et al., 2014). To recover residual oil, it is recommended that wettability alteration be strongly enhanced so that a positive capillary pressure can be the contributing force that induces spontaneous imbibition in low-permeability regions (Deng et al., 2021; Zhang et al., 2021). In this condition, a very low IFT is not favorable since it will limit the magnitude of the capillary force, thus weakening the main oil recovery driving force. In a word, in carbonate reservoir EOR, significant IFT reduction may not be necessary. Chelating agents alone, or the combinations of chelating agents and surfactants that can modify oil-wet rock back to water-wet, are potential EOR materials for carbonate reservoirs.



(a) SDS **(b)** Triton X100

Fig. 16. Resulting contact angle of strongly oil-wet Indiana limestone substrates soaked in 1.00 wt% EDTA with SDS or Triton X100.

4. Conclusions

In this paper, a comprehensive study from the preparation of chelating agent solutions to the EOR contribution was conducted and discussed. The covered information includes the applicability of chelating agents in different pH conditions, concentrations, and salinities, enhancement of surfactant solution compatibility with brines by using chelating agents, and the IFT reduction and wettability alteration by chelating agent alone, surfactant alone, or their combinations. Based on the obtained results, chelating agents, especially DTPA, are effective in enhancing the compatibility of surfactants (SDS and DTAB) with brines, and in wettability alteration. The surfactant + chelating agent mixture holds promising potential as a chemical EOR material for oil-wet carbonate reservoirs.

The following conclusions are drawn in detail.

(1) Chelating agents, including DTPA, EDTA, and GLDA, are low-cost additives for surfactant stabilization in high salinity conditions. DTPA stabilized DTAB of 1.00 wt% in seawater at high temperature (90 °C).

(2) The surfactant + chelating agent mixture can significantly alter rock wettability, except for cases where anionic surfactants were used. 1 wt% EDTA reduced contact angle by 62.9°. The EDTA + SDS, EDTA + DTAB, EDTA + Triton X100, EDTA + ZW mixtures achieved contact angle reductions of 26.5°, 127.0°, 118.3°, 139.2°, respectively. The combination of a cationic surfactant with a chelating agent forms a low adsorption wettability modifier which can significantly increase the residual oil recovery from fractured oil-wet carbonate formations.

(3) The application of chelating agents into the reservoirs requires consideration of pH conditions prior. A low pH can induce chelating agent precipitation. EDTA precipitated at 0.25–5.00 wt% when pH was modified to around 4. Seawater is not stable at pH higher than 9. pH can induce precipitation in seawater. A pH range of 5–9 is recommended.

(4) Chelating agents can reduce the oil/water IFT (from 20.5 to 9.4 mN/m at pH = 7), though not as effectively as surfactants (1 mN/m level). When applied with surfactants, a synergic effect was observed. Though a higher pH can promote the generation of in-situ surfactants, achieving even lower IFT values (0.01 mN/m level), the significant IFT reduction effect gradually vanishes with time (probably due to the partitioning of in-situ generated surfactants into the aqueous phase), and precipitation issues in brine.

CRediT authorship contribution statement

Xiao Deng: Writing – original draft, Investigation, Formal analysis. **Mohammad Otaibi:** Supervision. **Mohanad Fahmi:** Validation, Funding acquisition. **Mobeen Murtaza:** Investigation. **Muhammad Shahzad Kamal:** Writing – review & editing, Supervision, Conceptualization. **Shirish Patil:** Writing – review & editing. **Syed Muhammad Shakil Hussain:** Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgment

The research was supported by the College of Petroleum Engineering & Geoscience (CPG) at King Fahd University of Petroleum and Minerals (KFUPM).

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