Baghdad Science Journal

Volume 23 | Issue 2 Article 1

2-23-2026

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How to Cite this Article

Abdullah, Rafal S.; Hussein, Hussein Q.; and Jali, Rana R. (2026) "Synergistic Effect of Different Ions and Hexadecyltrimethylammonium Bromide on Wettability Alteration for Carbonate Rock," *Baghdad Science Journal*: Vol. 23: Iss. 2, Article 1.
DOI: https://doi.org/10.21123/2411-7986.5193

BAGHDAD SCIENCE JOURNAL 2026;23(2):418–428

RESEARCH ARTICLE

Synergistic Effect of Different Ions and Hexadecyltrimethylammonium Bromide on Wettability Alteration for Carbonate Rock

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ABSTRACT

Naturally carbonate reservoirs have very low oil recovery efficiency owing to their wettability and tightness of matrix. However, adding surfactant to brine solution can enhance oil recovery by changing the wettability of the carbonate rock surface from oil-wet to water-wet. In the present study, the effects of different brines in the presence of surfactant hexadecyltrimethylammonium bromide (CTAB), on the wettability of carbonate rock were investigated through different characteristics such as surface tension interfacial tension and contact angle. The best concentration of CTAB was investigated at 500 ppm in brine (NaCl) reduced surface tension and interfacial tensions to 42 mN/m and 15 mN/m respectively and prevented reservoir from damage. The effect of different type ions was studied at best CTAB concentration to prevent the negative effect of NaCl on wettability alteration. The results showed ions are only "proximally adsorbed" on the calcite surface. The contact angle changed to 39° after 20 min for 500 ppm CTAB in B1, Na + and Cl- ions reduce oil recovery. In a solution with Mg^{2+}, SO_4^{2-}, Na, Cl, and CTAB at approximately 0.1 M ionic strength, the effect of the counter-ions Mg^{2+}, SO_4^{-2} in combination with CTAB adds complexity where it can promote water-wetness by altering the surface interaction with water at a certain concentration and the contact angle reduction was about 63% after 30 min that reached to 33.9°. Furthermore, reduced surface tension and interfacial tension to 30 mN/m and 15 mN/m respectively that lead to significantly enhances oil recovery by decreasing fingering phenomena and increasing sweep efficiency.

Keywords: Brine, Contact angle, Surfactant, Ions, Wettability

Introduction

Oil has been the principal source of energy for years. Energy requests have increased throughout time as technology has developed. Based on the current energy consumption growth rate, energy demands are expected to reach over 260 peta watthours by 2050, nearly 50% more than present usage.[1] Since only 10% of the total oils in the deposits can be recovered from the reservoir using standard extraction techniques, the recovery rate of conventional oils must be enhanced to satisfy the growing need for energy.[2,3] As a result, many improved oil recovery (EOR) strategies, including wettability change, have been created and applied in the oil recovery process. It is ideally carried out to convert the oil-wet (hydrophobic) rock reservoir surface to a water-wet (hydrophilic) state, facilitating simple oil extraction.[4,5] Chemical flooding—includes surfactant flooding, polymer solution injection, alkaline flooding, and nanoparticles floating in fluids—is the most common method used in chemical EOR to modify wettability.[6] The equilibrium of the cohesive and adhesive forces between the molecules of oil

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Received 31 May 2024; revised 29 September 2024; accepted 1 October 2024.
Available online 23 February 2026

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https://doi.org/10.21123/2411-7986.5193

and the water-rock phase determines the wettability of reservoir rock. One of the most important variables affecting fluid flow in porous media and, consequently, the total hydrocarbon recovery efficiency is the wettability state of reservoir rocks.[7–9] Oil reservoirs can be classified as water-wet, oil-wet, mixed-wet, or intermediate-wet based on their wettability characteristics. Numerous studies contend that prior to oil migration, the reservoirs were salty[8] A small layer of water is preserved between the oil and the rock surface in certain reservoirs when the oil penetrates the porous medium that traps water during the reservoir's development. These reservoirs are found to be water-wet. Other reservoirs developed oil-wet conditions as a result of the water films collapsing during the multiphase flow's development.[2]

The mineral content of rocks is frequently diverse, resulting in chemical heterogeneity on the surfaces that come into contact with water and oil. Wettability for homogeneous systems can vary from very water-wet to strongly oil-wet based on the interactions between the solid surface, water, and oil.[10,11] The wetting state of rock surfaces can change by different modifiers that have different qualities, causing the formation to react differently. The nature of the formation rock,[9] oil characteristics, formation pH, temperature, and salinity should all be considered when selecting modifiers for a certain formation.[10,11]

In general, the Wettability Alteration Mechanism by Surfactant depends on the type of surfactant. Cationic surfactants are accountable for the cleaning mechanism's modification in wettability in oil-wet carbonate rock,[12] the formation of ion pairs between the acidic components of crude oil adsorbed on the surface of carbonate rock and the cationic heads of surfactant molecules.[13,14] It could reveal the once water-wet carbonate rock by removing the adsorbed coating of crude oil components from the rock's surface, retaining rock to the originally water-wet calcite rock.[15] Conversely, the van der Waals contact between the tails of the surfactant molecules and the adsorbed crude oil components (carboxylate molecules) on the surface of the carbonate rock results in the formation of a monolayer of anionic surfactant molecules on the carbonate rock.[13,16] The initially oil-wet rock surface may become increasingly water-wet due to a layer of adsorbed surfactants with hydrophilic head groups covering it; this process is known as the coating or adsorbed mechanism. Given that the ion-pair interactions are substantially stronger than the hydrophobic contacts. Because of the higher ionic interaction, cationic surfactants are thought to be superior than anionic surfactants.[17,18]

Even at high temperatures, naphthenic acids adsorb onto the calcite surface and change the surface wettability towards the oil-wet state. The electrostatic forces generated by the electrical charges at the calcium carbonate surface submerged in aqueous solutions control the amount of adsorption. Similar to this, the reduction in surface charge caused by treating the carbonate surface with low-salinity/engineered/smart water affects the degree of carboxylate desorption from the carbonate surface and the wettability change towards the water-wet state. The main mechanism for enhanced oil recovery in carbonate reservoirs has been shown to be wettability change; nevertheless, the contributing factors and prerequisites for wettability alteration are not well known By separating the impacts of salinity, ionic composition, and oil composition on the wettability change of solid surface, more information about the underlying mechanism for the incremental oil recovery caused by low-salinity/smart water could be collected.[19] Some studies have shown that CTAB alters wettability. In this regard, Saeedi Noorizadeh and Saeedi[20] used CTAB in smart fluid with various $SO4^{-2}$ concentrations[20] showed that CTAB was more effective in lowering the IFT and contact angle in brine when the concentration of SO_4^{2-} was increased 4 times Zargham Salari[21] conducted contact angle studies on carbonate rock and discovered that the in comparison to SDS and TX-100, the combination of smart water and surfactant has a more significant effect in improving oil recovery. Additionally, 1% of CTAB in brine may further reduce the contact angle.

Also, Moradi et al[22] used a plant-based natural surfactant and potential-determining ions (Mg^{2+}, Ca^{2+}, and SO_4^{2-}). Their findings showed that in the presence of four times the concentration of SO_4^{2-}, a 72% increase in oil recovery can be achieved by increasing the concentration of the plant-based surfactant to 3 weight percent (its critical micelle concentration, or CMC) value. Moreover, Mofrad and Dehaghani[23] examined the impact of both SDS and CTAB on wettability alteration in using smart water, it was shown that CTAB significantly outperforms SDS in terms of reducing the hydrophobicity of the carbonate surface. Furthermore, using DTAB, as a cationic surfactant, in conjunction with the Mg^{2+}, Karimi et al[24] showed that Mg^{2+} and DTAB in smart water will change rock to a more water-wet.

The present study aimed to investigate the wettability alteration by CTAB through measuring different characteristics (contact angle, surface tension and interfacial tension) in the presence of NaCl. Furthermore, the study aimed to investigate the effect of different ions on wettability alteration of carbonate rocks.

Materials and methods

CTAB is a cationic surfactant that is also known as hexadecyltrimethylammonium bromide. It has the molecular formula of $C_{19}H_{42}BrN$ and a purity greater than 98%. It's molecular weight about 364.45 g/mol. Sodium chloride (NaCl) with a molecular weight of 58.44 g/mol. Magnesium sulfate hepta hydrate ($MgSO_4.7H_2O$) it's a molecular weight of about 246.48 g/mol. Sodium sulfate (Na_2SO_4) with molecular weight 142.04 g/mol. Magnesium chloride hexahydrate ($MgC_2.6H_2O$) with molecular weight 203.3 g/mol.

Methodology

The experiment part included a main three steps, brine synthesis, rock sample preparation, and wettability alteration as shown in Fig. 1.

Brine synthesis

Different types of brines were prepared, brines with different ratio of ions shown in Table 1.

Rock sample preparation

The samples of the rock were supplied from Mishrif Formation of the Nasiriyah Oil Field. Plug core samples were cut into several pieces with a thickness of 2 cm and then polished. The cut sample dried to 80 °C for 30 min.

Contact angle measurement

A Tensiometer was used for contact angle measurement, where a drop of distilled water is released over cut sample. The instrument software processed the image and gave the average for right and left contact angles after stabilizing the drop over the cut sample. The effect of salinity water (NaCl) was studied with different concentrations of CTAB on the rock sample then the contact angle was measured. The optimum concentration of CTAB was chosen, then the same procedure was repeated for optimum concentration of surfactant solution for different ion solutions.

Surface tension and interfacial tension

A Tensiometer was used for surface tension measurement, where a drop of each prepared solution in step (1) points 2 and brine (NaCl solution) was released until reached maximum drop volume. The instrument software processes the image and gives the surface tension between drop and air. Furthermore, the interfacial tension between the crude oil and CTAB solutions were investigated according to Eq. (1) where, CTAB molecules can aggregate to form micelles in the brine solution.[25]

$$\gamma_{qw} = \gamma_o + \gamma_w - 2\emptyset(\gamma_o\gamma_w)^{0.5} \quad (1)$$

Where:
γ_{qw} interfacial tension of oil/water
γ_o surface tension of oil/air
γ_w surface tension of water/air

Results and discussion

Surface tension and interfacial tension

Effect of sodium chloride with CTAB

Several concentrations of surfactants were added to the brine (B1), and the surface tension (solution/air) and interfacial tension (solution/ crude oil) were measured, the results illustrated in Fig. 2 showed that a sufficient reduction in surface tension and interfacial tension with increasing CTAB concentration. The highest surface and interfacial tension from 74 and 28 mN/m to 51 mN/m and 18 mN/m respectively at concentrations 250 and 100 ppm CTAB compared to the lowest value at 1000 CTAB, which reached 32 mN/m and 12 mN/m respectively. This behavior can be interpreted in terms of the critical micelle concentration (CMC) of CTAB. Initially, individual CTAB molecules are dispersed in the solution. The presence of electrolytes causes the CMC to drop because the salt anions lessen the electrostatic repulsion between the CTAB molecules' positively charged head groups. The CTAB molecules are more likely to assemble into micelles as a result of this decrease in repulsion.[26] Additionally, if CTAB concentration is raised higher, molecules begin to migrate toward bulk rather than adsorb at the surface.[25] Those reasons lead to less effective on reducing surface tension and constant surface tension of 51 mN/m. However, micelles formation was increased at 500 ppm and 1000 pm CTAB concentrations. This could lead to a slightly lower surface tension of 42 mN/m and 32 mN/m respectively. Also, the interactions between CTAB, NaCl, and water molecules could influence surface tension, Sodium ions may compete with CTAB molecules for adsorption sites at the liquid-air interface. Higher concentrations of sodium ions can displace CTAB molecules from the interface,

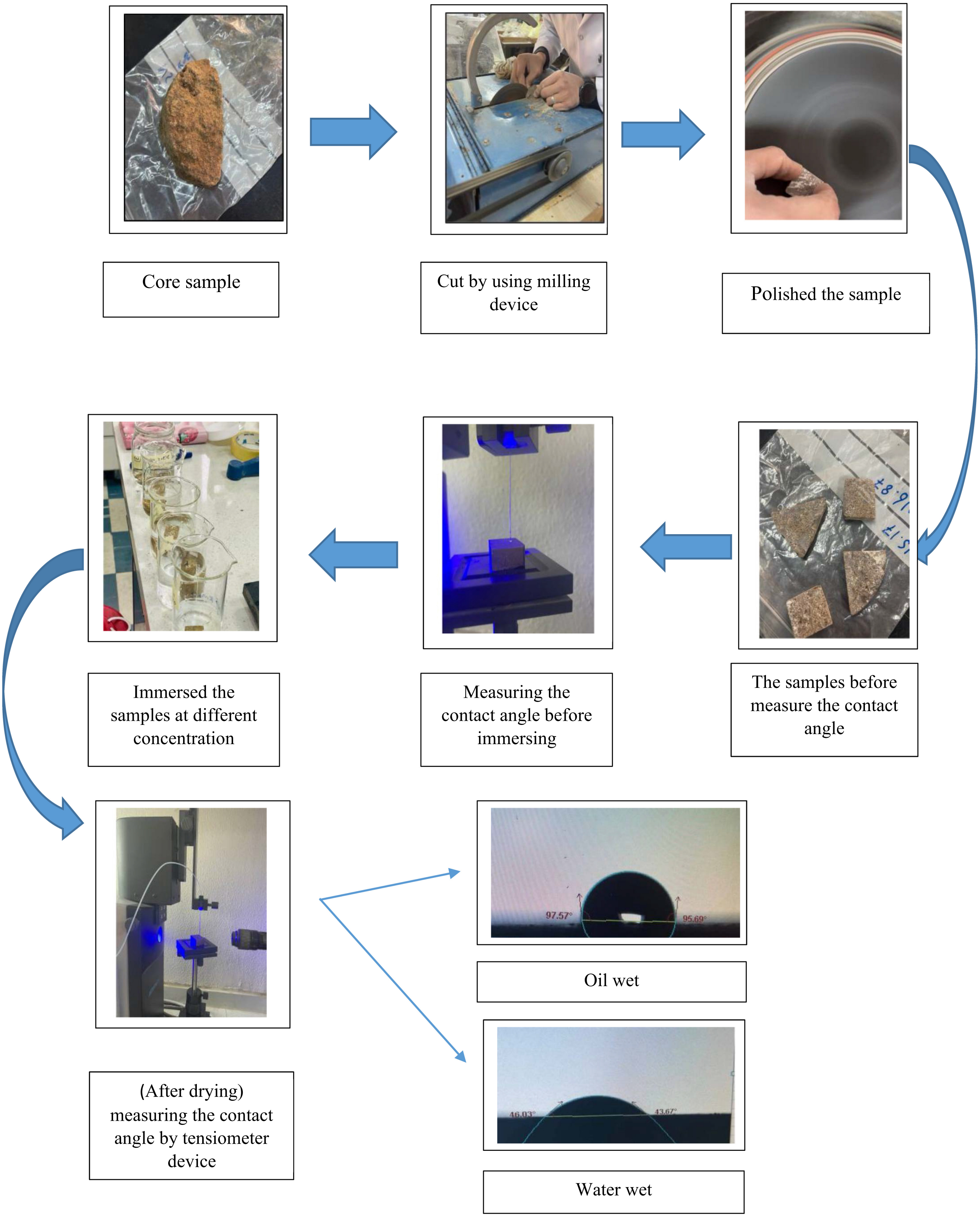

Fig. 1. Experiments methodology.

Table 1. Brines' properties.

	B1	B2	B3	B4	B5	B6
Na^{+}	2380	1000	1000	1000	1000	1000
Mg^{+2}	0	345	340	340	325.5	0
$SO4^{-2}$	0	0	340	169	651.1	2086
Cl^{-1}	3673	3406	1288	1418	2216	0
Mg/SO4	–	1:0	1:1	2:1	1:2	0:1
TDS	6000	6000	6000	6000	6000	3086
CTAB CMC	232.08	61.74	209.53	210.47	209.39	249.91

leading to reduced coverage of CTAB and potentially slight reduction in surface tension.[27]

Effect of different ions with CTAB

High surfactant concentrations and salinity can increase the risk of surfactant adsorption onto the reservoir rock, which might lead to reduced permeability and potential formation damage. The micelles can also precipitate in the presence of divalent cations, which are often present in reservoir brines.[28] So, the impact of Different ratios of ions (Na^{1+}, Mg^{2+}, Cl^{1-} and SO_4^{2-}) were investigated in the presence of 500 mg CTAB on surface tension and interfacial tension. Fig. 3 shows the minimum surface and interfacial tension at the ratio 1:1 of Mg:SO_4 were 30.15 and 12 mN/m respectively, where Magnesium ions and sulfate ions can interact with CTAB molecules at the liquid-air interface, these interactions may affect the adsorption of CTAB molecules at the interface, altering their ability to reduce surface tension. The nature of these interactions (e.g., electrostatic, hydrophobic) depends on factors such as ion charge, size, and hydration properties. Also, Magnesium ions and sulfate ions can form complexes with CTAB molecules in solution. These complexes may have different surface-active properties compared to free

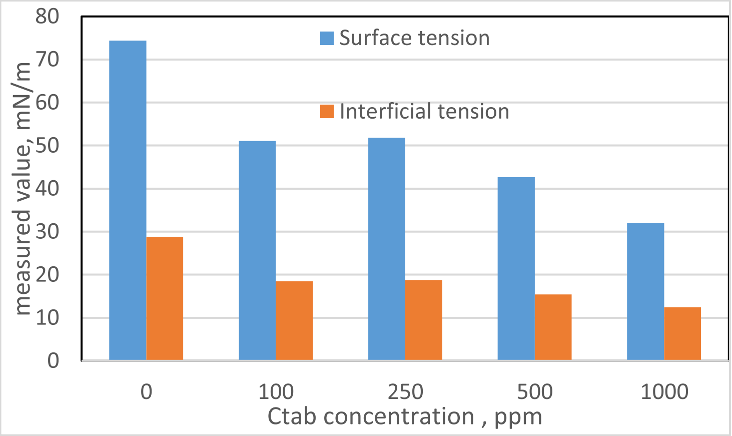

Fig. 2. Interfacial tension and surface tension for different concentrations CTAB in B1.

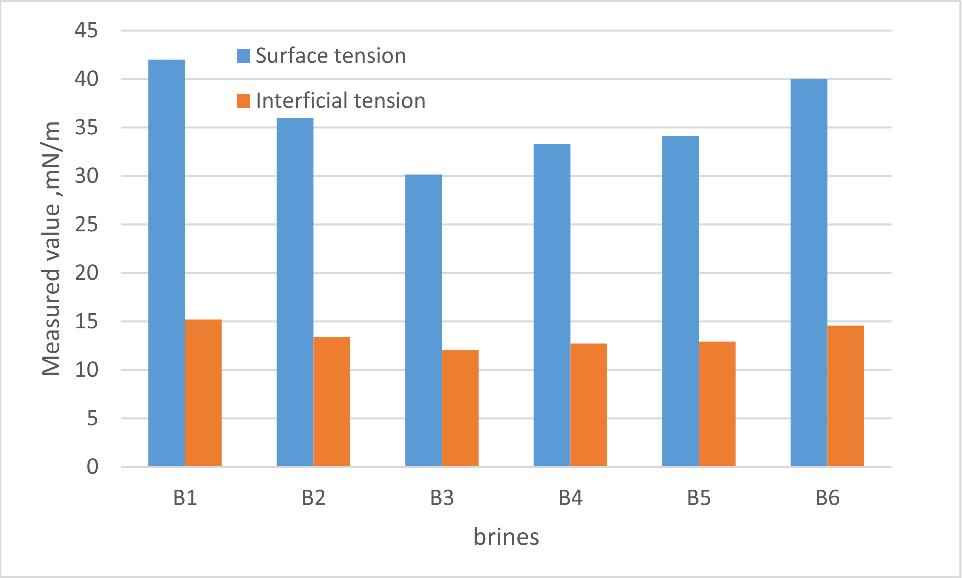

Fig. 3. Interfacial tension and surface tension for different brines at 500 ppm CTAB.

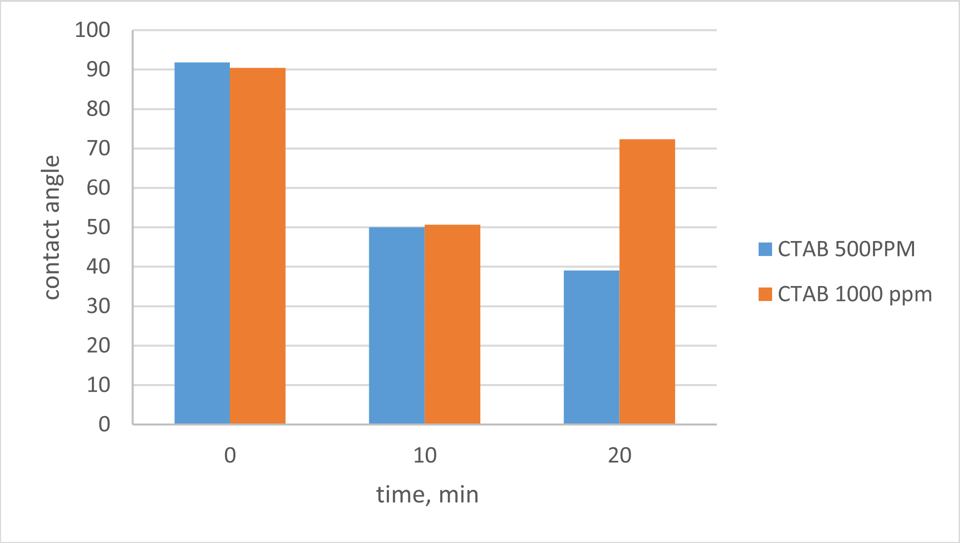

Fig. 4. The contact angle versus time for different concentrations of CTAB.

CTAB molecules, leading to changes in surface tension.[28] On the other hand, high concentration of Magnesium ions or sulfate ions can displace CTAB molecules from the interface, reducing their surface activity and resulting in higher surface tension as shown in Fig. 3 where Mg^{2+} alone and SO_4^{2-} alone had higher surface tension reaching 36 and 40 mN/m respectively.

Contact angle

Effect of NaCl brine with CTAB

Wettability alteration was investigated by measuring the contact angle after aging rock sample in brine solutions (B1) with two CTAB concentrations 500 and 1000 ppm for different periods, as shown in Fig. 4. For concentration (500), the contact angle decreased from 90° to 40° after 20 min for brine (B1). In this case, the positive charge of CTAB creates a complicated structure on the rock that is negatively charged by carboxylic acid. As a result, the wettability can be adjusted to a more water-wet condition, because the CTAB and carboxylic may interact electrostatically to generate ion pairs. Furthermore, there are positively and negatively charged organisms on the carbonate surface.[20]

On the other hand, Fig. 4 shows ununiform behavior for contact angle by increasing concentration of CTAB to 1000 ppm in B1, where contact angle decreased from 90° to 50° after 10 min then increased to 72° after 20 min and retained to 50° after 30 min. Additionally, the carbonate surface has both negatively and positively charged species. The increase in contact angle for concentration 1000 ppm was attributed to the CMC value of CTAB as shown in Table 1, where above this value, CTAB monomers have a tendency to form micelles, and micelle formation reduces the single free ability to create cationic-anionic ion pairs with carboxylic acid.[9]

Moreover, it could be explained by the existence of NaCl salt in the calcite surface region, which forms a double layer; this layer does not require a charged surface to exist[29]. As was previously demonstrated, the calcite surface is highly hydrophilic; water molecules from the first two wetting layers are heavily adsorbed to it. But the solvation shells of Na^{1+} and Cl^{1-} ions are established, and they interact with the calcite surface via the hydrogen-bonding network of the first two wetting layers. As a result of the Na^{1+} and Cl^{1-} ions' "proximal adsorption," the initial wetting layer somewhat depletes from the calcite surface. As a result, the interfacial water structure is drastically altered, especially the orientation angle and O-H bond angle distributions, whereby the adhesion between the calcite surface and the NaCl brine weakens and the state reduced water-wettability (more oil-wet).[29]

Effect of different ions with CTAB

At the best concentration of CTAB (500 ppm), different brine ions (B2, B3, B4, B5 and B6) were studied and compared with CTAB in B1 to prevent the negative effect of NaCl on wettability alteration. So, Fig. 5a and 5b show that the combination of two influencing ions Mg^{2+} and SO_4^{2-} can significantly decrease the contact angle with uniform reduction reached to 62% and 30% after 30 min. for B3 and B5 respectively compared to the case where Na^{1+}, Mg^{2+}

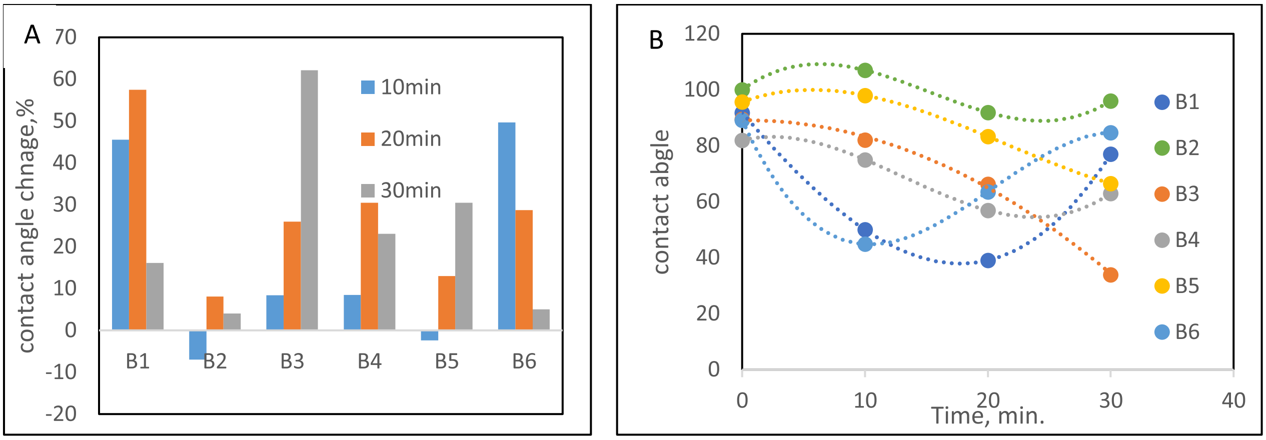

Fig. 5. Effect of different ions at 500 ppm CTAB solution on contact angle. (a) contact angle change, (b) contact angle vs time.

or SO_4^{2-} ions were used alone where contact angle decreased to 39°, and 91° for B1 and B2 respectively after 20min then increased to 77° and 96° for each respectively after 30 minutes,the same behavior has appeared in B4 where the contact angle decreases to 56° after 20 minuted then, increased to 63° after 30 minutes also, B6 suffered a uniform behavior where the contact angle decreased from 89° to 44° after 10 minutes then retained increasing sharply to 84° after 30 minutes. It has been observed that the addition of Mg^{+2} alone can raise the zeta potential of the rock surface to a more positive value, replace Mg^{2+} on the surface, desorb attached carboxylate groups, and form complexes with Mg^{+2}. All of these processes lead to the irreversible desorption of lipophilic groups on the rock surface.[30]

According to the data, the optimum way to alter wettability was to use CTAB dissolved in low salinity water containing Mg/SO_4 at a ratio of = 1 (B3). After 30 minutes, the contact angle decreased from 89° to 33°. Mg^{+2} and $SO4^{-2}$ are the primary wettability modifiers in carbonate rocks, according to several studies. But each ion has a distinct effect on how wettable carbonate rocks[26]. It is generally accepted that Mg^{2+} ions by themselves can lessen the carbonate surface's oil-wetness. In this way, adding sulfate ions to a solution reduced the contact angle due to three factors. First of all, positive charges can be reduced on the surface of carbonate rock via SO_4 adsorption. Consequently, Mg^{2+} can readily reach the rock surface and change the wetting conditions. Second, divalent cations and $SO4^{2-}$ may combine to create ion pairs. Consequently, they will be accessed easily to the surface, as SO_4^{2-} is adsorbed on the surface.[31]

Ionic strength

Ionic strength for all brines was calculated as shown in Fig. 6, it seems that all solutions approximately

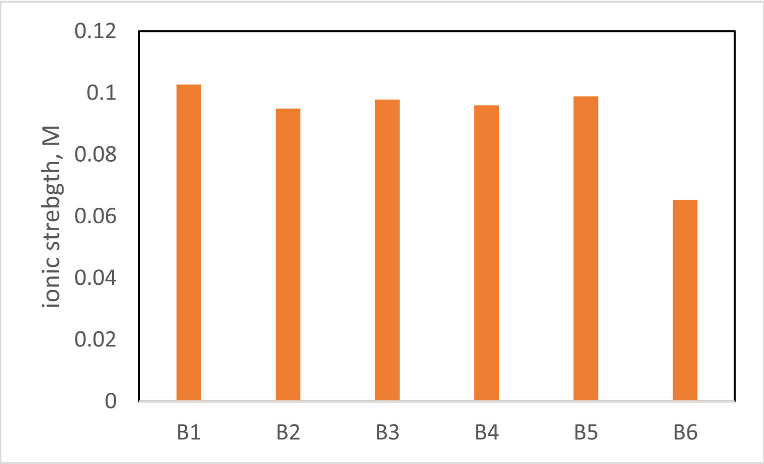

Fig. 6. Ionic strength for prepared brines.

have the same values between 0.094 to 1M except B6 where ionic strength 0.065 M. However, they have different effects on wettability alteration as described previously.

Increasing the solution's ionic strength (e.g., to 0.1 M) increased the electrical double layer around the rock surface. This increase can reduce the electrostatic repulsion between the rock surface and the oil, making it easier for the oil to adhere to the rock surface. At the calcite surface, Na^{1+} ions can also create Na-naphthenates, which can adhere the oil molecule more firmly to the mineral surface. However, the negatively charged carboxylate groups in the oil and the negatively charged sites on the rock surface are connected by the presence of divalent cations, such as Mg^{2+}. At the calcite surface, Na^{1+} ions can also create Na-naphthenates, which can adhere the oil molecule more firmly to the mineral surface. However, the negatively charged carboxylate groups in the oil and the negatively charged sites on the rock surface are connected by the presence of divalent cations, such as Mg^{2+}.[32]

In a solution with Mg^{2+}, $SO_4{}^{2-}$, Na, Cl and CTAB at approximately 0.1 M ionic strength, the wettability of carbonate rock can be altered to a more water-wet condition under certain conditions. The combination of Mg^{2+} and $SO_4{}^{2-}$ ions generally promotes water-wetness due to charge neutralization and surface complexation. The presence of CTAB adds complexity; while it may initially promote oil-wet conditions due to its hydrophobic tail, at an optimal concentration, it can promote water-wetness by altering the surface interaction with water.[33]

Conclusion

The impact of different salts including NaCl, $MgSO_4$, $NaSO_4$ and $MgCl_2$ with different concentrations on the carbonate rock sample is investigated. In this way, the main concluding results are summarized as follows:

The presence of CTAB and salt ions decreases the interfacial tension between solution and crude oil due to different factors formation of complexes between ions and CTAB and interaction force that leads to enhanced oil recovery by decreasing effect of fingering and increasing mobility of oil. The combined presence of Mg^{2+}, $SO_4{}^{2-}$, Na^{+1} and CTAB at an optimal concentration can alter the wettability of carbonate rock to a more water-wet condition. Mg^{2+} and $SO_4{}^{2-}$ promote hydrophilicity, while properly managed CTAB concentrations, so that more oil will be extracted from rock.

The optimum concentration of different ions was studied to prevent the negative effect of NaCl on wettability alteration. Complete wettability alteration toward water wet is demonstrated for carbonate rock with strongly hydrophilic wearability modifier Na^{1+}, Cl^{1-}, Mg^{2+},$SO_4{}^{2-}$ from reaching the surface, which was hindered by the first two wetting layers. There are two ways that this proximal adsorption modifies wettability: Na^{+} and Cl^{-1} ions settle closer to the surface, distributing the interfacial water structure and decreasing the amount of moisture on the calcite surface (which affected oil recovery) Nevertheless, $SO_4{}^{2-}$ and Mg^{2+} ions continued to adsorbed on the surface, altering the surface charge and raising the calcite surface's water content (which enhanced oil recovery). The recent work opens the door to understanding the wettability change process at the micro-scale and offers physical insights into the essential role of wettability modifiers. Also, the compression of the double layer due to increased ionic strength typically leads to a more water-wet condition. Due to the effect the counter-ions Mg^{2+} and $SO_4{}^{2-}$. The presence of CTAB adds complexity where it can promote water-wetness by altering the surface interaction with water at a certain concentration.

Acknowledgment

Sincere thanks and appreciation to the staff of the College of Engineering, Chemical Engineering Department for their cooperation throughout the research period.

Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all figures and tables in the manuscript are ours. Furthermore, figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Baghdad.

Authors' contribution statement

R. S. A, H. Q. H, and R. R. J. contributed to the design and implementation of the research, the analysis of the results, and the writing of the manuscript.

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التأثير التآزري للأيونات المختلفة وبروميد هيكساديسيلتريميثيل أمونيوم على تغيير قابلية البلل لصخور الكربونات

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الخلاصة

تتمتع المكامن الكربونية بشكل طبيعي بكفاءة منخفضة للغاية في استخلاص النفط بسبب قابليتها للبلل بالنفط وانخفاض نفاذيتها ومساميتها. ومع ذلك، فإن إضافة المادة الخافضة للتوتر السطحي إلى المحلول الملحي يمكن أن تعزز عملية استخلاص النفط عن طريق تغيير قابلية بلل سطح الصخور الكربونية من محبة للنفط إلى محبة للمياه. في هذه الدراسة تم دراسة تأثير المحاليل الملحية المختلفة بوجود المادة الخافضة للتوتر السطحي سيتيل ثلاثي ميثيل بروميد الأمونيوم (CTAB) على قابلية بلل الصخور الكربونية من خلال خصائص مختلفة مثل الشد السطحي زاوية التلامس.، تمت دراسة التركيز الأفضل لـ Ctab عند 500 جزء في المليون في محلول ملحي (كلوريد الصوديوم) مما أدى إلى تقليل التوتر السطحي والتوترات السطحية إلى 42 ملي نيوتن / م و15 ملي نيوتن / م على التوالي ومنع الخزان من التلف وأظهرت النتائج أن الأيونات "ممتصة بشكل تقريبي" فقط على سطح الكالسيت ان زاوية التلامس تغيرت إلى 39^{o} بعد 20 دقيقة لـ 500 جزء في المليون من CTAB في B1 وتقلل أيونات الصوديوم والكلوريد من استخلاص الزيت. في محلول يحتوي على Mg^{2+} و SO_4^{2-} وNa^{1+}، Cl^{-1} وCTAB بقوة أيونية تبلغ حوالي 0.1 مول، فإن تأثير الأيونات المضادة Mg^{2+} SO_4^{2-} مع CTAB يضيف تعقيدًا حيث يمكن أن يعزز رطوبة الماء عن طريق تغيير تفاعل السطح مع الماء عند تركيز معين وكان انخفاض زاوية التلامس حوالي 63٪ بعد 30 دقيقة ووصل إلى 33.9^{o}. علاوة على ذلك، تم تقليل التوتر السطحي والتوتر البيني إلى30 ملي نيوتن / م و15 ملي نيوتن/م على التوالي مما يؤدي إلى تعزيز استخلاص الزيت بشكل كبيرعن طريق تقليل ظاهرة الأصابع وزيادة الكفاءة.

الكلمات المفتاحية: محلول ملحي، زاوية الاتصال، خافض التوتر السطحي، ايون، قابلية الابتلال.