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Influence of PVA on wettability alteration of carbonate rocks: the role of different ions

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Abstract

The petroleum industry faces a significant problem in enhanced oil recovery (EOR) from carbonate reservoirs because of their mostly oil-wet character, which restricts hydrocarbon extraction. To better understand how surfactant-polymer techniques can enhance oil recovery, this study investigates the effectiveness of polyvinyl alcohol (PVA) as a stand-alone agent and the function of ionically modified smart water on carbonate properties. The study focuses on its effect on critical parameters essential for effective oil displacement and recovery, such as wettability alteration, interfacial tension, and surface tension (ST) using different brines (smart water). The ability of the polymer PVA to lower interfacial and surface tension was investigated. PVA decreased surface tension in brine to 44 mN/m, although smart water solutions showed more noteworthy outcomes. When PVA was added to smart water with a 1:1 Mg^{2+}/SO_4^{2-} ratio, the lowest ST (29 mN/m) and IFT (11.8 mN/m) values were observed. The hydrophilic properties of PVA and its ionic interaction with Mg^{2+} were key factors in encouraging water adsorption on the surface of the carbonate rock. The contact angle was getting closer to zero, indicating that this interaction resulted in a full transition to a water-wet condition. Enhancing the oil displacement process in carbonate reservoirs—generally refractory to recovery—requires this complete transition to water-wet conditions. This full shift to water-wet conditions is vital for enhancing the oil displacement process in carbonate reservoirs, which are typically resistant to recovery due to their oil-wet surfaces.

Keywords: Polyvinyl Alcohol (PVA); Wettability Alteration; Brine; Contact Angle; Surface Tension; Interfacial Tension.

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1- Introduction

Wettability is defined by the tendency of a liquid to spread on a solid surface, which is quantitatively measured by the contact angle (CA). A lower contact angle indicates a more water-wet condition, which is desirable for EOR as it promotes oil displacement. The alteration of wettability can occur through various mechanisms, including ion exchange, adsorption of surfactants, and the formation of complexes at the oil-water-rock interface [1, 2] .Oil reservoirs can be classified as water-wet, oil-wet, mixed-wet, or intermediate-wet based on their wettability characteristics. Numerous studies contend that the reservoirs were salty before oil migration [2] .

For over four decades, the polymer has successfully recovered up to 30% of the initial oil retained in the reservoir. The polymer flooding method is generally less costly than water flooding since it produces more oil and less water. From 0.7 to 1.75 pounds of polymer are inundated for every barrel of excess oil produced. Mixing long-chain polymer molecules with the injected water leads to an increase in the water's viscosity which reducing mobility. Thus, by increasing the water/oil mobility ratio, this method increases the area and vertical sweep efficiency [3].

Because polymers are viscoelastic, they can undergo deformation when under stress and then regain their original shape when the stress is released. Because of this characteristic, they can adjust to different shear rates in porous media, which increases sweep and displacement efficiency and boosts oil displacement efficiency. Polymer solutions expand and contract when injected into reservoirs, releasing trapped oil by actions including tugging and stripping [4].

Large polymer molecules increase water viscosity, enhancing oil mobility by reducing the water-to-oil mobility ratio and improving oil recovery. Oil-water emulsions can also be stabilized, promoting consistent oil displacement by regulating water flow in the reservoir. The primary types of polymers used in EOR are[5, 6]:

- Hydrophilic Polymers: Water-soluble, increased water viscosity (e.g., Xanthan gum, PAM, PVA)
- Hydrophobic Polymers: Stabilize emulsions by interacting with oil (e.g., PEO)
- Biopolymers: Biodegradable and eco-friendly (e.g., cellulose derivatives, guar gum)

PVA is a synthetic polymer known for its excellent film-forming, emulsifying, and adhesive properties. In EOR applications, PVA can be used to create stable

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emulsions that help in mobilizing trapped oil within the reservoir. The polymer's ability to modify fluid properties can enhance sweep efficiency during water flooding processes by reducing the interfacial tension between water and oil, thereby improving the displacement of oil[5].the application of PVA solutions can result in large increases in recovery rates. For instance, as compared to traditional water flooding procedures, the use of a 0.5% PVA solution boosted oil recovery by 10–35% in core flooding testing. PVA's effectiveness can be increased and recovery operations in a variety of reservoir conditions can be further optimized by combining it with additional surfactants or alkaline agents [7].

Furthermore, the injection of modified low-salinity water disrupts the equilibrium between crude oil, rock, and brine systems, promoting a shift in wettability toward a more water-wet state. The complexity of the carbonate reservoir makes this process challenging to model.

In the context of brine chemistry, the presence of divalent ions such as calcium (Ca^{2+}) and magnesium (Mg^{2+}) is known to significantly reduce the contact angle, enhancing water-wet conditions. This is attributed to the ability of these ions to interact with the rock surface and displace hydrophobic oil components, thereby altering the surface properties. Conversely, monovalent ions like sodium (Na^{+}) have a lesser effect on wettability due to their lower activity in modifying the electric double layer around the rock surface[8].

Surface and interfacial tension are critical parameters in determining the behavior of fluids in porous media. The introduction of PVA and varying ion concentrations can lead to a reduction in both surface and interfacial tension. Lower interfacial tension facilitates the movement of oil droplets through the water phase, enhancing recovery rates. Studies have shown that as the concentration of divalent ions increases in the presence of PVA, there is a corresponding decrease in both contact angle and interfacial tension, which is beneficial for EOR applications.

In summary, the interaction of PVA in brine with varying ion ratios significantly alters carbonate reservoirs' wettability. By effectively reducing contact angles and interfacial tensions, PVA enhances the potential for oil recovery, making it a valuable component in EOR strategies.

Arjmand et al. studied that the polymer flooding approach is only appropriate in reservoirs with medium temperature and pressure due to hydrolyzed polyacrylamide (HPAM) decomposition at harsh conditions. HPAM is ineffective for reservoirs with high salinity and temperature because, on the other hand, the viscosity of the polymer solution increases with reservoir salinity. Polyvinyl Alcohol (PVA) is a viscous surfactant and IFT reducer that, in our opinion, can be utilized in reservoirs with high salinity and temperature since it is stable under harsh conditions, including high salinity and high temperature (over 480º F) [9].

Jalil and Hussein investigate the effect of polyethene glycol (PEG) polymer and sodium dodecyl sulfate (SDS) surfactant in comparison with nano silica in enhancing oil recovery through wettability alteration of carbonate rocks. contact angle was measured before and after treatment to recognize the extent of affinity change (wettability) of the carbonate. The polymer was observed to have an acceptable wettability alteration [10].

Zuoli et. al. characterized the microscale effects of polymer on wettability alteration and crude oil liberation in carbonates. The favorable effect of polymer on wettability alteration in carbonates revealed. They demonstrated that sulfonated polyacrylamide polymer (SPAM) enhances oil recovery from carbonates by reducing contact angles and adhesion forces, especially at elevated temperatures (70°C). Improved oil liberation efficiency, driven by wettability alteration and increased viscosity, highlights SPAM's potential in optimizing polymer flooding for enhanced oil recovery in carbonate reservoirs [11]

Al-Busaidi et.al. investigated the dependency of polymer effectiveness on carbonate reservoir characteristics, particularly initial wettability. While HPAM polymers are more effective in shifting wettability towards water-wet conditions, ATBs-based polymers offer cost advantages and remain a viable alternative. These findings are critical for developing tailored Engineered water polymer flooding strategies to optimize oil recovery in diverse carbonate formations [12]

Dehaghani et al. explored enhanced oil recovery (EOR) in carbonate reservoirs, representing over 60% of global oil discoveries. While smart water injection is effective, offshore applications face challenges due to large injection volumes. A novel continuous injection approach was tested using the Sivand offshore reservoir in Iran. Thirty-six contact angle tests and twelve flooding experiments highlighted potassium and sulfate ions as key factors. Optimal wettability alteration occurred with 4 times potassium and 0–2 times sulfate concentrations. A significant 132.8° wettability decrease was achieved with seawater containing 2 times potassium and 5 times sulfate concentrations.

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

2- Experimental work

2.1. Materials and methods

All chemicals were supplied from Alpha Chemika company. Polyvinyl Alcohol (PVA) was supplied with a molecular weight of 115000 g/mol and purity of 99.9%, Sodium chloride (NaCl) With a molecular weight of 58.44g/mol. Magnesium sulfate heptahydrate ($MgSO_4.7H_2O$) it's a molecular weight of about 246.48g/mol. Sodium sulfate (Na_2SO_4) with molecular weight 142.04. and Magnesium chloride hexahydrate ($MgCl_2.6H_2O$) with a molecular weight of 203.3g/mol.

2.2. Methodology

The experiment included three main steps: brine synthesis, rock sample preparation, and surface, interfacial, and wettability alteration.

2.2.1. Brine synthesis

Formation water was prepared by dissolving NaCl and MgSO4.7H2O in deionized water with a total dissolved salt of 20.6%. brines with different ratios of ions (Smart water) were prepared as shown in Table 1. Dissolving different salts (NaCl, Na2SO4, MgCl2.6H2O, MgSO4.7H2O) in deionized water with a constant total dissolved salt 6% for each. All ion concentrations calculated by Eq. 1:

$$\mathrm{Wt(g)} = \mathrm{Cons\ (ppm)} \times \left(\frac{1}{1000}\right)\left(\frac{\mathrm{g}}{\mathrm{mg}}\,\mathrm{ion}\right) \times \mathrm{MW\ (salt)} \times \frac{1}{\mathrm{MW(ion)}} \times \mathrm{V\ (water)} \times \frac{1}{\mathrm{purity\ of\ (salt)}} \quad (1)$$

2.2.2. Rock sample preparation

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

2.2.3. Surface tension and interfacial tension

tensiometer was used for surface tension and interfacial tension measurements, where a drop of each prepared solution was released until it reached to 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 solutions was investigated according to Eq. 2 [13].

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

Where: γ_{qw} interfacial tension of oil/water, γ_o surface tension of oil/air, γ_w surface tension of water/air
Air/water surface tension was measured for different solutions according to the experiments matrix in Table 2.

2.2.4. Contact angle measurement

A tensiometer was used for contact angle measurement, where a drop of distilled water is released Over a cut (slices) sample. After stabilizing the drop over the cut sample, the instrument software processed the image and gave the average for right and left contact angles .
Cut samples were immersed in brine (6000 ppm) NaCl with different concentrations of PVA for a different period, then dried at 80C for 30 minutes. The contact angle was then measured. the methodology was explained in Fig. 1. The same procedure was repeated for the best concentration of surfactant and polymer solutions by using different brines with different ions according to the experiment matrix, as shown in Table 3.

Table 1. brines' properties

Ions	B1	B2	B3	B4	B5	B6
Na^+	2380	1000	1000	1000	1000	1000
Mg^{+2}	0	345	340	340	325.5	0
$SO4^{-1}$	0	0	340	169	651.1	2086
Cl^{-1}	3673	3406	1288	1418	2216	0
Mg/SO_4	-	1:0	1:1	2:1	1:2	0:1
TDS	6000	6000	6000	6000	6000	3086

Table 2. Surface tension measurements matrix

constants	variables				
B1	$PVA_{1000ppm}$	$PVA_{2000ppm}$	$PVA_{3000ppm}$	$PVA_{4000ppm}$	$PVA_{5000ppm}$
Best concentration of PVA (2000ppm)	B1	B2	B3	B5	B6

Table 3. Contact angle measurements matrix

Constants	Variables				
B1 Base fluid	$PVA_{1000\ ppm}$	$PVA_{2000ppm}$			
Best concentration of PVA (2000 ppm)	B1	B2	B3	B5	B6

3- Discussion

3.1. Surface tension and interfacial tension

3.1.1. Effect of sodium chloride with PVA

Several concentrations of polymer 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 increasing the concentration of polyvinyl alcohol (PVA) generally leads to a decrease in ST and IFT. The surface tension decreased from 74 mN/m to 49 mN/m (33% reduction) at 500 ppm and 1000 ppm PVA concentrations. In comparison, surface tension of 41 mN/m was achieved at 5000 ppm PVA (44% reduction). Similarly, the interfacial tension dropped from 28 mN/m to 17 mN/m (a 39% decrease) at 500 ppm and 1000 ppm PVA, reaching its lowest value of 15 mN/m at 5000 ppm PVA (46% reduction)., due to the hydration of PVA molecules was impacted by the presence of NaCl in the solution. As a screen against electrostatic repulsions, sodium ions (Na+) enable PVA chains to pack closer together at the air-water interface.

A high concentration of NaCl can also affect on solution's viscosity, which may have an impact on the surface dynamics of PVA adsorption. However, Higher concentrations may cause a viscosity drop, which affects the PVA's total surface activity. ST and IFT have decreased slightly with increasing PVA concentration. PVA molecules may form aggregates or complex structures at higher concentrations, such as micelles or network formations. These structures can lead to less efficient reduction of ST and IFT compared to lower concentrations where individual molecules are more freely interacting at the interfaces [14]. At low concentrations of PVA (>500ppm), the attraction among the water molecules results in a water-pulling effect. Here, the inward attraction of water molecules from the surface to the bulk is greater than that in a PVA solution of higher concentration. Hence, an appreciable enhancement to the surface excess value results. But, the chaotropic ions (chloride) have hindered the interchain H-bonding and resulted in salting out decreasing the reduction rate [15].

Both 2000 ppm and 5000 ppm concentrations showed significant reductions in surface tension, about 40% and 44%, respectively, compared to the other concentrations. Therefore, 2000 ppm was chosen as the best concentration because it was more cost-effective and easier to manage while still achieving a significant reduction in surface tension.

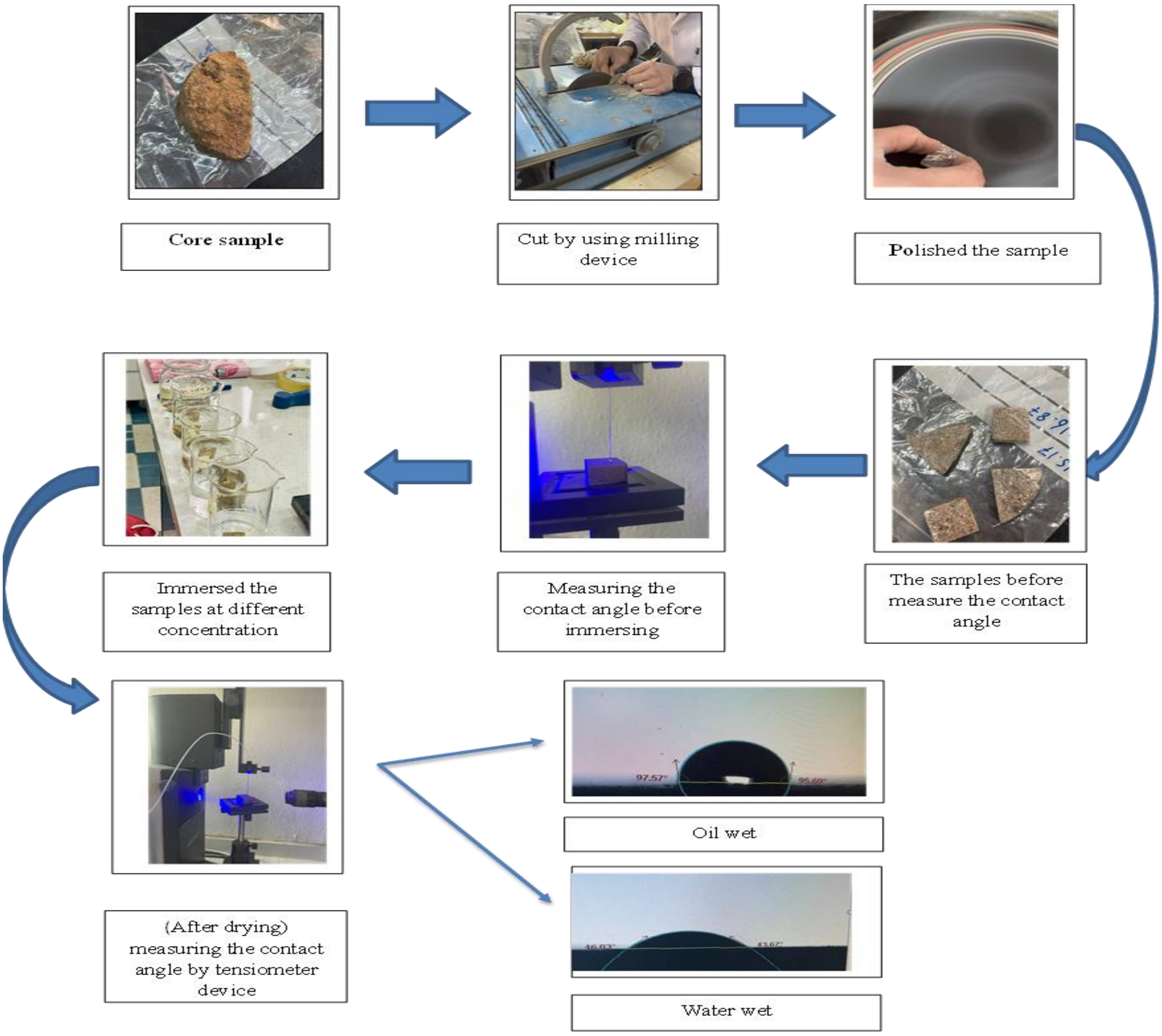

Fig. 1. Contact angle measurement methodology

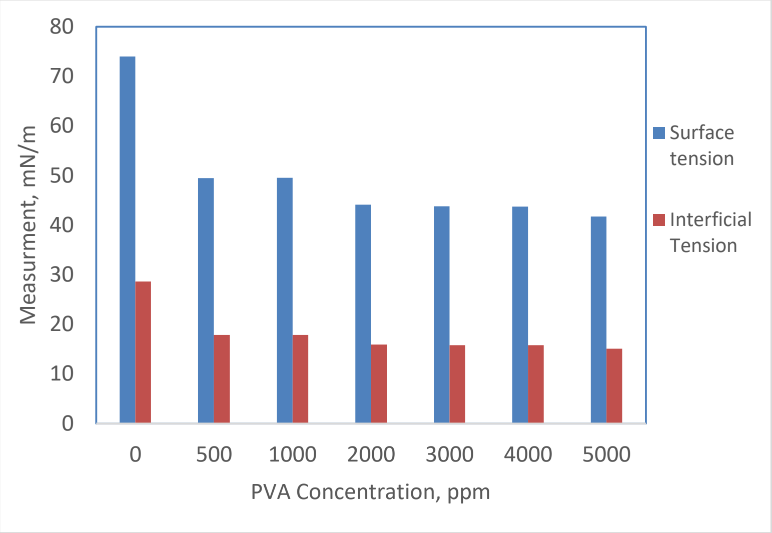

Fig. 2. Interfacial tension and surface tension for different concentrations PVA in B1

3.1.2. Effect of different ions with PVA

Different smart water was used at 2000ppm PVA to investigate the effects of various ion ratios (Na+1, Mg+2, Cl-1, and SO4-2) on surface tension and interfacial tension as shown in Fig. 3. It was found that the minimum values of ST and IFT were observed in sample B2 (Mg/SO4) The surface tension reached 29 mN/m (34% reduction) and the interfacial tension reached 11.8 mN/m (25% reduction This reduction is attributed to increased ion-ion interactions and changes in the solvation of PVA chains due to the presence of multiple salts, which collectively decrease surface activity.

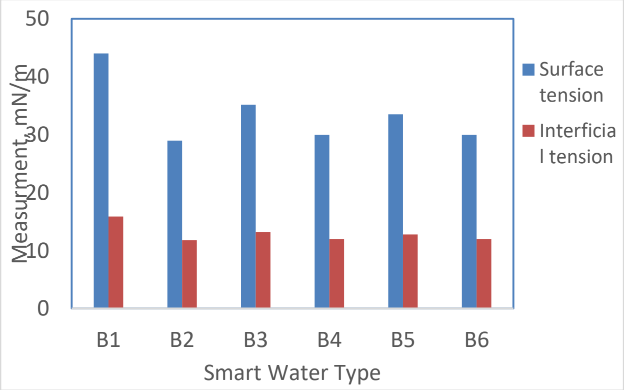

Fig. 3. Interfacial tension and surface tension for different brines at 2000 ppm PVA

B1 gives the highest values in surface tension reaching 44 mN/m and the interfacial tension at 15.8 mN/m. adding NaCl raises the friction coefficient between molecules of solution and works as charge carriers that increase the conductivity of PVA solutions. The hydrophobically modified PVA displays high air-liquid surface and liquid-liquid interfacial activity, and these activities rise with increasing NaCl content [16].

In comparison, the addition of salts such as SO_4-2 and $MgCl_2·7H_2O$ likely causes greater structural disruption in PVA solutions, leading to a reduction in surface tension (ST) and interfacial tension (IFT) .

According to the Hofmeister series, specific ions have varying abilities to 'salt-out' or 'salt-in' polymers from solution, which can alter the orientation and arrangement of PVA molecules at interfaces, such as between oil and water or air and water. The degree of reduction in surface and interfacial tensions is influenced by the type of ions present in the solution, their concentrations, and the type of PVA used .

Furthermore, high concentrations of magnesium ions can significantly enhance water absorption, modify diffusion characteristics, and improve the mechanical and thermal properties of the resulting file [17].

3.2. Contact angle

3.2.1. Effect of NaCl brine with PVA

The effect of PVA on the wettability of carbonate rock was investigated through contact angle measurement as shown in Fig. 4, where it was observed that for a concentration of 2000 ppm and 1000ppm of PVA in brine (B1), the contact angle decreased from 91.8 to 26.3 (71% reduction) after 10min from 91° to 49.36° (45% reduction) after 20 min respectively, due to PVA hydrophilicity by its numerous hydroxyl (-OH) groups along its polymer backbone [18]. that interact with carbonate surfaces forming hydrogen bonds with carbonate minerals [19] alternating to water-wet.

For a concentration of 2000 ppm of PVA, the contact angle decreases from 91.8^{o} to 26.3^{o} after 10min then increases to 76^{o} after 30^{o} min and at 1000ppm PVA the contact angle gradually decreases, from 91° to 49.36° after 20 min. then increased to 78.3 after 30 min. The surface-active characteristics of PVA and the disrupting effects of NaCl on the water structure are the main causes of the large initial drop in contact angle. In higher concentrations of NaCl, the conformation (shape) of PVA molecules can be altered, leading to the aggregation of polymer chains. This structural change can decrease the available surface area for water interaction, further reducing the hydrophilic character of PVA [20-22].

This phenomenon is explained by increased wettability and decreased surface tension. However, at sufficiently high salt concentrations, changes in surface morphology and roughness can cause the contact angle to increase again, making the surface more hydrophobic. The complex interaction of these factors highlights the diverse behavior of polymer solutions in the presence of salts.

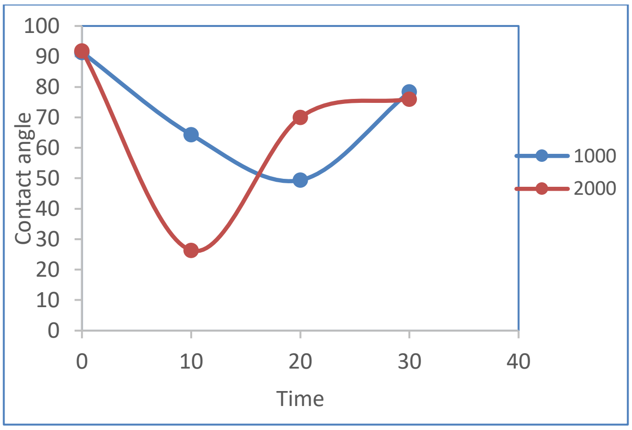

Fig. 4. The contact angle versus time for different concentrations of PVA

3.2.2. Effect of different ions with PVA

At the best PVA concentration (2000 ppm) Different types of smart water (B2, B3, B4, B5 and B6) were examined and compared with PVA in B1 to mitigate the detrimental effect of NaCl on wettability alteration as shown in Fig. 5. B3, B4, and B5, consistent behavior with a decreasing contact angle were observed over time. The addition of ions Mg^{+2} improves the PVA film's hydrophilicity and decreases the contact angle. Mg ions can interact with the hydroxyl groups (-OH) of PVA through hydrogen bonding. This increases the number of available hydrogen bonding sites, and it disrupts the crystalline structure of PVA. This increases the amorphous regions, which are more readily accessible to water molecules, further enhancing hydrophilicity. The concentration of Mg^{+2} ions can influence the degree of hydrophilicity. Higher concentrations generally lead to increased hydrophilicity up to a certain point, beyond which the material may become less hydrophilic due to excessive crosslinking or phase separation [23]. So, PVA with smart water (B2, B3 and B5) reduced the contact angle by only 6, 22 and 30% respectively.

B6 displayed an approximate behavior as well, the contact angle decreased from 116º to 67º (42% reduction) after 20 min then increased to 98º after 30º min. When Na, Cl and SO_4 are added to PVA solutions, the contact angle initially tends to decrease. The main source of this decrease is the salts' salting-out effect salts' presence promotes the development of micelles and aggregates, increasing the surface area on which water can interact with the polymer and reducing the contact angle [24].

The multivalent character of the sulfate ion (SO_4^{2-}) from Na_2SO_4 might cause a more noticeable decrease in contact angle than the chloride ion (Cl^-) from NaCl. Also, the contact angle may rise in tandem with an increase in salt concentration or the solution's saturation point. The polymer chains might entangle more during this phase, making the surface less accessible to water molecules and raising the contact angle [25].

The solution's ionic strength can also affect the hydration shell surrounding the polymer chains. Higher quantities have the potential to interfere with the polymer's hydration, making the surface more hydrophobic as the polymer becomes less soluble and more aggregated [26].

For smart water (B4) Mg/SO4 ratio (2/1), it was found that the contact angle decreased to approximately zero (fully water wet) This effect is driven by the hydrophilic nature of PVA, which promotes water adsorption, and the ionic interactions of Mg^{2+} with the carbonate rock. The electrolytes (such as sulfate ions) further enhance these interactions, collectively making the surface more favourable for water, resulting in significant wettability alteration crucial for enhancing oil recovery. This transition is often observed in carbonate reservoirs undergoing enhanced oil recovery processes using similar brines and chemical combinations [27].

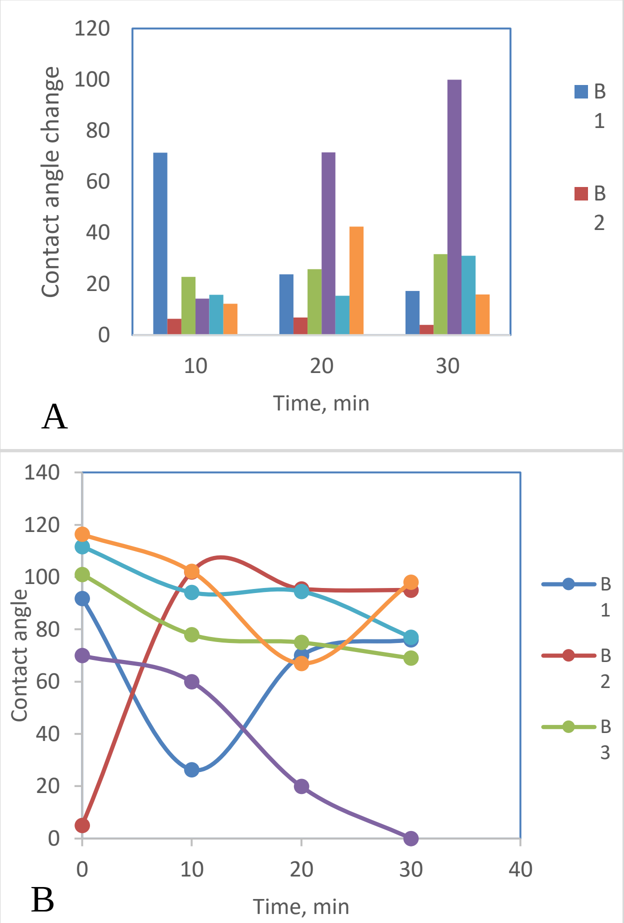

Fig. 5. Effect of different ions at 2000 PVA solution on (A) contact angle change vs time (B) contact angle vs time

4- Conclusions

Increasing Polyvinyl Alcohol (PVA) concentrations effectively reduced surface tension (ST) and interfacial tension (IFT), with a maximum reduction observed at 41 mN/m at a concentration of 2000 ppm. However, excessive PVA usage led to polymer aggregation, diminishing its effectiveness. Furthermore, variations in viscosity caused by the presence of NaCl influenced the extent of ST reduction. PVA also demonstrated a significant impact on carbonate rock wettability by reducing the contact angle. At 2000 ppm, the contact angle decreased by 71%, and at 1000 ppm, it decreased by 45%. This effect can be attributed to the hydrophilic hydroxyl groups of PVA, which form hydrogen bonds with the carbonate surface, making it more water-wet and thus improving wettability. The presence of salts in the brine further enhanced water-PVA interactions, primarily through micelle formation. Among the salts tested, sulfate ions provided a greater impact on these interactions than chloride ions. However, higher ionic strength in the solution could lead to increased hydrophobicity due to polymer aggregation. Notably, a Mg/SO_4 ratio of 2:1 was found to achieve near-complete wettability of the carbonate surface. This emphasizes the importance of optimizing the chemical composition of brine for enhanced oil recovery (EOR) applications in carbonate reservoirs.

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تأثير PVA على تغيير قابلية البلل لصخور الكربونات: دور الأيونات المختلفة

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الخلاصة

تواجه صناعة النفط تحديًا كبيرًا في تحسين استخلاص النفط (EOR) من المكامن الكربونية بسبب طبيعتها الزيتية في الغالب، مما يحد من استخراج الهيدروكربونات. لفهم كيفية تحسين تقنيات البوليمر–السطحي لاستخلاص النفط، تبحث هذه الدراسة في فعالية بولي فينيل الكحول (PVA) كعامل مستقل ودور الماء الذكي المعدل أيونيًا على خصائص الصخور الكربونية. تركز الدراسة على تأثيره على المعايير الحيوية اللازمة لإزاحة النفط واستخلاصه بفعالية، مثل تعديل قابلية البلل، التوتر السطحي البيني، والتوتر السطحي باستخدام مياه ملحية مختلفة (الماء الذكي).

تمت دراسة قدرة البوليمر PVA على تقليل التوتر السطحي والبيني. حيث قلل PVA التوتر السطحي في المياه الملحية إلى ٤٤ ملي نيوتن/متر، بينما أظهرت محاليل الماء الذكي نتائج أكثر تميزًا. عند إضافة PVA إلى ماء ذكي بنسبة ١:١ من Mg^{2+}/SO_4^{2-}، تم تسجيل أقل قيم للتوتر السطحي (٢٩ ملي نيوتن/متر) والتوتر البيني (١١,٨ ملي نيوتن/متر). لعبت الخصائص المحبة للماء لـ PVA وتفاعله الأيوني مع Mg^{2+} دورًا رئيسيًا في تعزيز امتصاص الماء على سطح الصخور الكربونية. اقتربت زاوية التماس من الصفر، مما يشير إلى أن هذا التفاعل أدى إلى انتقال كامل إلى حالة محبة للماء. يُعد هذا الانتقال الكامل إلى حالة محبة للماء أمرًا ضروريًا لتحسين عملية إزاحة النفط في المكامن الكربونية، التي تكون عادة مقاومة للاستخلاص بسبب أسطحها الزيتية.

الكلمات الدالة: بولي فينيل الكحول(PVA)، تغيير البلل، مياه مالحة، زاوية الاتصال، التوتر السطحي، التوتر البيني.