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Experimental Investigation of Using Tetramethylammonium Chloride on Chemical Enhanced Oil Recovery in Carbonate Rocks

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The chemical enhanced oil recovery (CEOR) is the process of using surfactants, alkalis, polymers for increasing oil production in the tertiary production stage. Cationic surfactants were used to minimize the IFT and altering the wettability of carbonate rocks which is essential for facilitating the mobilization of the trapped oil from porous media. The capability of cationic surfactants will depend on many factors including the surfactant concentration, salinity, temperature, and surfactant chain length. Tetramethylammonium chloride (The alkyltrimethylammonium, R-N$(CH_3)_3$) is one of quaternary ammonium compounds which has a short chain length. TMAC can tolerate the harsh reservoir conditions (high temperature and high salinity). Using the high concentration of TMAC can ensure sufficient monomer availability to induce the IFT reduction and wettability alteration and can enhance the oil recovery by ensuring sufficient monomer availability. In this experimental work, we used TMAC as an EOR fluid. Using high concentration of TMAC (5000 ppm) which prepared in high salinity water (seawater) for chemical EOR and compare the results with low concentration to figure out the role of increasing concentration of short chain surfactant in EOR. Two brines (formation water & seawater) and two Indiana limestone core samples were used for Spontaneous Imbibition and surfactant flooding experiments under reservoir conditions which mimic the Middle East reservoir conditions. FTIR, IFT, and ICP were performed to characterize the surfactant. Spontaneous Imbibition was also used to confirm the ability of TMAC to change the wettability at high concentration. The results of spontaneous imbibition showed that using high concentration of TMAC (5000 ppm) can alter the wettability more than the low concentration and gave about 16% of accumulative oil recovery. While the core flooding outcomes revealed that the TMAC at concentration of (5000 ppm) increased oil recovery about 24% when using it at tertiary stage. The total oil recovery of core flooding test was about 74% of OOIP. The FTIR test was carried out for TMAC powder and TMAC/SW solution with rock. FTIR results showed a slight change in TMAC bonding when added to the rock. Furthermore, the ICP analysis of effluent showed a slight increase in Ca^{+2} concentration alongside with a slight decrease in Mg^{+2} concentration.

Keywords: Cationic surfactants, wettability, spontaneous imbibition, core-flooding, EOR

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The global demand of oil is assumed to raise by 35% through the next two decades due to the fast industrial development and diminishing resources of supplies [1]. Approximately about 30–40% of the oil initially in place (OIIP) can be retrieve by the conventional primary and secondary stages of oil production processes and that mean more than 60% of the oil is trapped in the hydrocarbon reservoirs [2]. Around 60% of the world's oil reserves and 70% of the conventional oil reserves in the Middle East are trapped in the carbonate reservoirs [3, 4]. The oil production from oil-wet or mixed-wet carbonate reservoir rock is a challenge because these reservoirs have complex and heterogeneous properties [5]. They reveal variable porosity and permeability which affect the fluid storage and flow through the reservoir leading to high rate of water production [6]. To fulfil higher rates of oil production, tertiary recovery or the enhanced oil recovery (EOR) is applied to improve the recovery of the trapped oil from these reservoirs [7]. EOR methods fall into broad categories, such as chemical flooding, gas injection, microbiological

recovery, and thermal recovery [8]. Chemical enhanced oil recovery is one of the EOR techniques that involves using chemical like surfactants, polymers, alkalis, and others as an EOR fluid to reduce the interfacial tension (IFT) and change the wetting properties [9]. The surface-active agents (i.e. surfactants) can improve the oil recovery from oil or mixed wet reservoirs by reducing the IFT to ultra-low levels and modify the initial wetting properties to water wet. The fluid-fluid interactions (IFT) and rock-fluid interactions (wettability alteration) are very important factors for oil recovery. But the performance of them are affected by many factors like surfactant type, rock lithology, temperature, salinity, and pH. Therefore, examining the surfactants is very essential factor for prosperity the operations of EOR [10]. The surfactants are identified by the hydrophilic head and hydrophobic tail. Depending on the charges of their head, they can be categorized as cationic (positive), anionic (negative), non-ionic (no charge), and zwitterionic (positive and negative) [11]. EOR operations in oil wet carbonate reservoirs have significant problems like thermal stability, high salinity, high rate of adsorption and then lack of their ability to reach deeply into the reservoirs [12]. Cationic surfactants are known as the surfactants that have one or more positive charge. They are quaternary ammonium salts (QAS), that commonly used in EOR operations and have the capability to lower the IFT and shift the oil wet carbonate to more water wet in the harsh conditions (high temperature and high salinity) by forming ion pairing [13, 14]. Different types of cationic surfactants are utilized in carbonates reservoirs for EOR such as CTAB, DTAB, and others. CTAB with formula $C_{19}H_{42}BrN$ which it referred as "cetrimonium bromide" [15, 16]. It is used to reduce IFT, foam generation, modifying the rock wettability, and improve the oil recovery when it is used with DI and high salinity water [17-19]. Other type of the cationic surfactant with a molecular formula $C_{15}H_{34}BrN$ is known as Dodecyltrimethylammonium bromide or (DTAB) [20]. DTAB raised the oil recovery in limestone rock at high temperature when prepared with brines of different salinities [21, 22].

This study evaluates the performance of using a quaternary ammonium salt as a cationic surfactant; Tetramethylammonium chloride, which is soluble and compatible in order to improve the oil recovery in carbonate reservoir under the harsh reservoir conditions. QAS are used as a pharmacological research agent that blocks selective potassium channels, as a basic laboratory chemical reagent, and as a low-residue bactericide in hydrofracking. They are not toxic at concentrations bellow 2630 mg/kg (2,630 ppm). TMAC used as chemical reagent, phase-transfer catalyst, surfactant in cleaning products, and in biochemical applications. In oil industry, TMAC also used hydraulic fracturing because it acts as a low-residue bactericide which allow to be used in oil and water- based fracturing fluids.

EXPERIMENTAL SECTION

Rock Samples

The experimental work was acquired on two core samples/Indiana limestone with different dimensions to perform the spontaneous imbibition and core flooding tests. Before each test the samples are cleaned by toluene and methanol at room temperature to remove any salts or contaminations and then dried at temperature 100℃. The mineral composition that obtained by X-ray diffraction analysis for the powder core sample shows the main mineral is calcite.

Oil

A crude oil with an API gravity 28.7, density 0.982 gm/cc, viscosity 12.18 cp at ambient temperature is obtained from an oil field in the Middle East. It is filtered to remove undissolved particles using 5μm in-line filter. The total base number (TBN=0.76 mg of KOH/g) and total acid number (TAN= 0.04 mg of KOH/g) are determined by 916 Ti-Touch Metrohm apparatus. Then the SARA analysis are performed using IATROSCAN MK-7s FID device as (Saturates 23.05%, Aromatic 44.22%, Resin 22.97%, and Asphaltene 9.76%).

Surfactant

The cationic surfactant Tetramethylammonium chloride [$(CH_3)_4N(Cl)$], termed TMAC, is used as an EOR fluid in this study. TMAC was provided through Sigma Aldrich with purity ≥ 98% and molecular weight 109.6 gm/mol.

Brines and Solution

Two high salinity brines are used; formation water (FW) with a salinity 241,000 ppm to mimic the Middle East reservoir conditions and seawater (SW) with a salinity 67,745 ppm in order to mimic the Arabian Gulf water [23]. Both brines are synthesized by adding the salts ($NaHCO_3$, Na_2SO_4, NaCl, $CaCl_2.2H_2O$, and $MgCl_2.6H_2O$) to the deionized water (DI). The surfactant solution (TMAC/SW) was prepared in SW with a concentration (5000 ppm) to use it for the experimental work later. The brines and solutions were stirred and filtered by 0.22 μm Millipore filter to ensure that all the salts particles have been dissolved.

Fourier Transform Infrared Light Spectroscopy (FTIR)

A powder sample from Indiana limestone were prepared to check the ion interaction and chemical bonding of the surfactant with SW and the rock sample by the analytical technique (FTIR) using TENSOR 27 apparatus. The procedure involves passing the infrared light across the powder sample causing the atomic bond within the sample to absorb the infrared light at

a different energy or the light of the spectrum transmitted at a specific wavelength with transmission region (500-4000 cm^{-1}). At each region, the peaks that represent the chemical bond and functional group are identified and compared with the reference database.

Interfacial Tension (IFT)

The interfacial tension of oil/surfactant solution was performed using the Drop Shape Analyzer device/Pendant Drop method which provided by Kruss. IFT measurements were employed the at ambient temperature (25°C) and reservoir temperature (90°C). The method depends on analyzing the shape of oil drop that is hung in the surfactant solution. Then the camera captures the shadow of the drop, and the image is processed by the software to fit the drop to the Young-Laplace equation. IFT measured depending on the radius of curvature of the oil drop, density difference between the two fluids, and the gravitational acceleration.

Spontaneous Imbibition (SI)

The spontaneous imbibition test was conducted by Amott cell (Figure 1). SI was performed on the core sample S. The porosity and permeability were measured using He Porosimeter and Gas Permeameter respectively. Then the sample was immersed in the formation water (FW) for one week to ensure that the FW saturate the sample.

The initial oil saturation Soi and irreducible water saturation Swi measured through displacing FW by oil. Subsequently, S1placed inside the Amott cell and TMAC/SW solution surrounded it. The oil volume is monitored and recorded daily for 30 days until there is no oil production.

Core Flooding (CF)

The other sample was used to find out the oil recovery using the TMAC/SW solution. After cleaning, drying, measuring porosity and permeability, and aging the sample in the FW for a week, F sample loaded in the core holder inside the core flooding system (Figure 2). To establish Soi and Swi, the oil flooded through the F sample with several pore volumes. Then the reservoir conditions are applied (confining pressure 4000 psi, back pressure 3000 psi, and temperature 90℃) and SW are flooded as a secondary recovery fluid until there is no oil production. Following that, the surfactant flooding with a flow rate 0.1 cc/min. The oil and water produced were collected in 15 ml cylindrical glass each 30 min.

Induced Couple Plasma (ICP)

The effluent of SI and CF tests after injection TMAC/SW solution was analyzed by Shimaduz ICPE-9820 plasma atomic emission spectrometer. It involves ionizing the solution sample, detecting and quantifying the concentration of elements that present in the effluent sample.

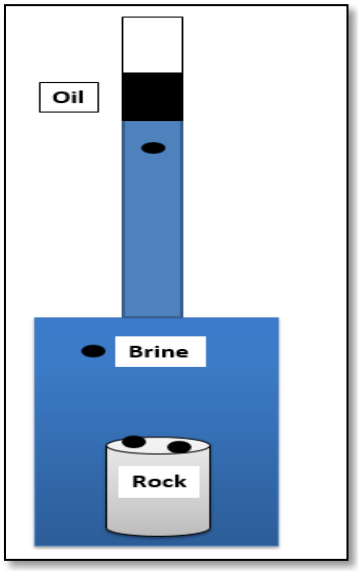

Figure 1. Schematic illustration of Amott Cell.

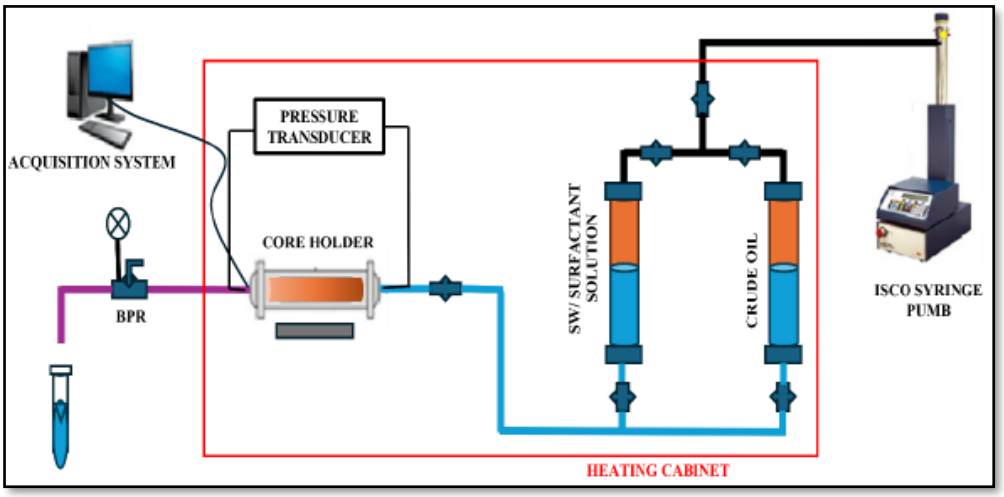

Figure 2. Schematic illustration of core flooding system.

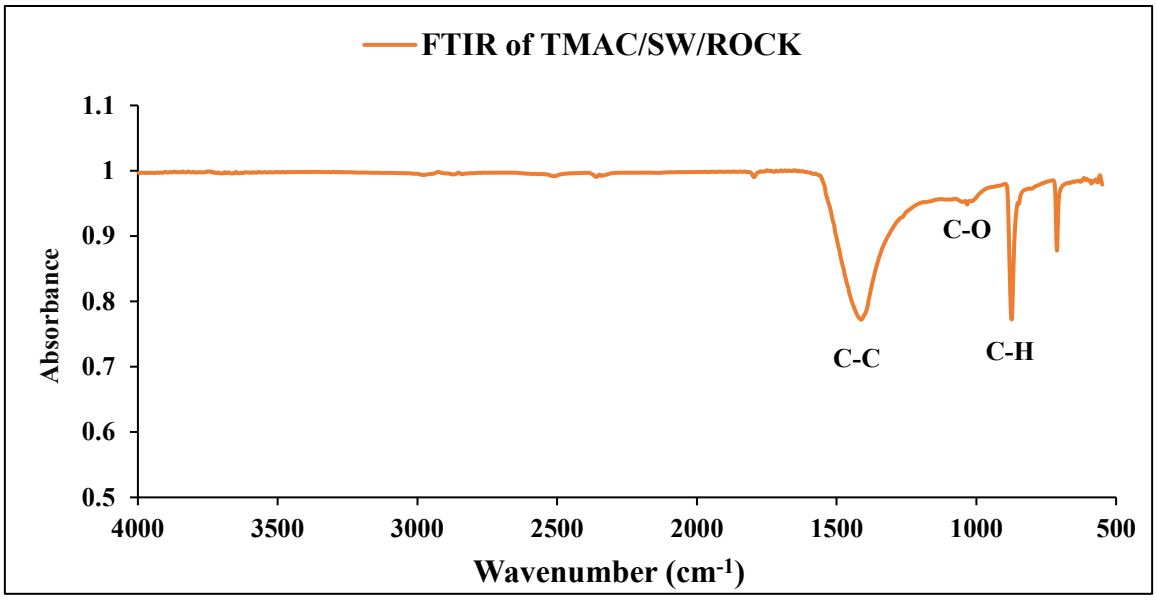

Figure 3. FTIR of 5000 ppm TMAC/SW/rock.

RESULTS AND DISCUSSION

Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) was performed using a Bruker (TENSOR 27) infrared spectrometer to determine the chemical bonds and functional groups present in the rock/SW/surfactant system. Figure (3) showed the IR spectra of the aforementioned system. The spectrum shows three prominent peaks. The first one is at wavenumbers of 1407.9 cm^{-1} which represent the stretching of carbon-carbon (C-C) bond. While another peak appears at 1033.7 cm^{-1} which obviously represent (C-O) bond. The corresponding peak of stretching of carbon-hydrogen (C-H) bond in an out-of-plane ("oop") appears at 873.7 cm^{-1} [24-26].

Interfacial Tension (IFT)

The Drop Shape Analyzer device/Pendant Drop method which is provided by Kruss was utilized to evaluate the interfacial tension between crude oil and varying concentrations of surfactants solution using. The solutions were prepared by mixing the TMAC with SW at temperatures 25 °C. the concentration range was (100-5000) ppm. The IFT value of SW/oil was 22.3 mN/m. Figure (4) depicts the IFT values of TMAC/SW versus concentration. The TMAC reduced the IFT values from 22.3 mN/m to moderate range. The IFT values declined with increasing concentration

from 12.3 mN/m for 100 ppm to 10.9 mN/m for 5000 ppm. The CMC could not be exactly determined because of the continual decline in IFT values within the selected range. All the IFT values are in moderate range, and this can be attributed to the short chain of TMAC which affect its ability to gather tightly at the oil-water interface, leading to less effect than long chain surfactants [27, 28]. Additional cause may decrease TMAC ability to achieve ultra-low IFT which is the all-selected concentration may below the CMC while the effective IFT reduction requires surfactant concentration near or above CMC [29, 30]. Numerous studies also reported the effect of salinity on IFT values specially the existence of divalent ions Ca^{2+} and Mg^{2+} [20, 31-33].

Spontaneous Imbibition Test

In this study, the spontaneous imbibition test was used as a toll to monitor the wettability alteration in Indiana limestone core sample. The core samples were subjected to centrifugation to measure the initial oil and water saturations and conduct the spontaneous imbibition experiments. The surfactants mixture with SW were used as the surrounding phases in the Amott cells. The properties of the core samples that used in this study are listed in Table 1. The oil recovery mechanisms are reflected in the spontaneous imbibition recovery curves and oil production data from the core surfaces. The imbibition is driven by gravity and capillary forces. The oil production from the upper part of the core samples reflect the gravity effect which is led to shape the recovery slope to be almost straight because oil recovery progress at a steady and slow rate. While the oil recovery through imbibition, driven by capillary force, flows from the side and upper surfaces because of the counter-current flow of water and oil [34-36]. Further the capillary force effect reflect the initial stage of oil production in SI [37].

The initial oil saturation (irreducible water saturation) was 66.93 % for the Indiana limestone core sample (S) used in this test. The Figure (5) represents the oil production curve with time (30 days of production). The production starts from the first day with about 4 % of production and continued till day 12 where reach to its plateau with about 16% of production.

The initial production during first day proved that the wettability alteration and capillary force effect improved more than the low concentration (100) ppm where they were negligible (as published in our previous study) [38]. Also, the production rate is higher when the concentration increased to 5000 ppm with 16% while it was about 9% for concentration 100 ppm. Therefore, the spontaneous imbibition test revealed that a partial improvement occurs in wettability and capillary force activation for TMAC/SW with the higher concentration 5000 ppm more than the lower concentration 100 ppm.

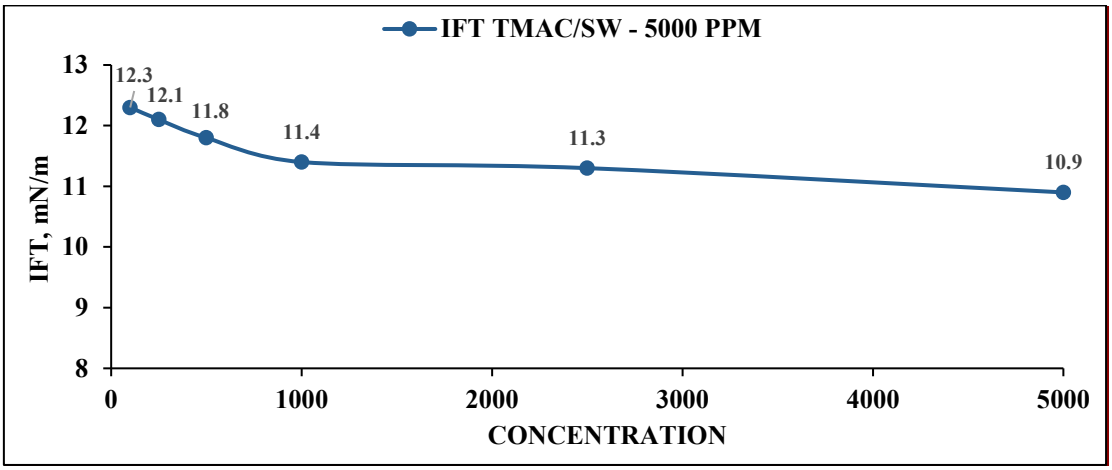

Figure 4. IFT measurements of TMAC/SW solutions vs. concentration at 25˚C.

Table 1. Rock Sample Properties of Spontaneous Imbibition Test.

Sample ID	Length, inch	Diameter, inch	Porosity, %	Permeability, md	S_{oi} %	S_{wi} %	Recovery, %
S	2.5	1.5	13.13	48	66.93	33.07	16

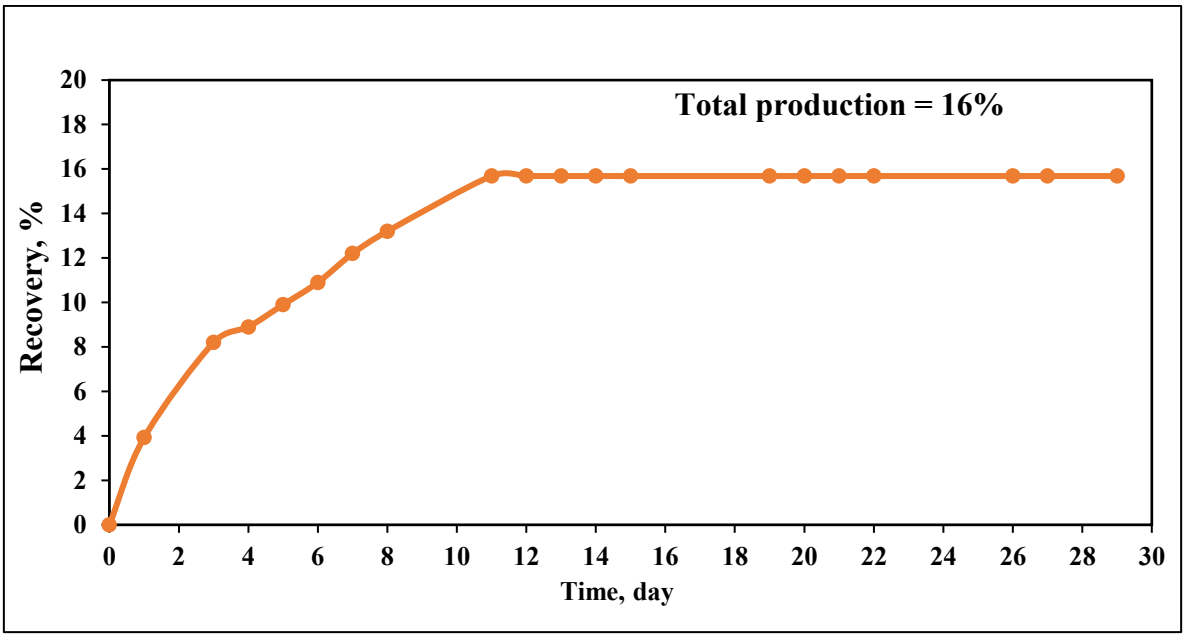

Figure 5. Spontaneous imbibition recovery curves for TMAC/SW solution at 70˚C.

Table 2. Rock Sample Properties of core-flooding Test.

Sample ID	Length, inch	Diameter, inch	Porosity, %	Permeability, md	S_{oi} %	S_{wi} %	Recovery, %
F	3.5	1.5	14.16	34.5	75.76	24.2	23.76

Core-Flooding Experiment

The core-flooding test was performed on Indiana limestone core sample as representative of carbonate rock. The core properties, porosity, and permeability is presented in Table 2. The porosity and permeability of core (F) were 14% and 34.5 md respectively.

The core sample saturated with formation water (241,000 ppm) and then the crude oil injected into the core samples using four flow rates (0.1,0.25, 0.5, and 0.75 cc/min) until no water production. The amount of original oil in place (OOIP) is equal to the amount of produced water while the irreducible water saturation is the amount of water left in core sample. The irreducible water saturation (S_{wi}) and initial oil saturation (S_{oi}) were calculated using equations (1) and (2).

$$Soi = OOIP / PV * 100\% \quad (1)$$

$$Swi = (PV - OOIP) / PV * 100\% \quad (2)$$

Subsequently, the reservoir conditions were applied in the flooding system (temperature 90°C, back pressure 3000 psi, and confining pressure 4000 psi). The high salinity seawater (67,745 ppm) was flooded into carbonate core sample seawater as a secondary oil recovery and surfactant solution (5000 ppm) utilized as a tertiary oil recovery. In secondary mode, about 8 PVs of high salinity sea water were injected into the core samples. The water and oil produced were collected every 30 minutes. The calculation of oil recovery factor was done by using the separated volume of oil. The residual oil saturation (S_{or}) was calculated using the following equation:

$$Sor = (OOIP - \text{volume of oil produced}) / PV * 100\% \quad (3)$$

After seawater flooding, the surfactant flooding (tertiary recovery) had been performed. TMAC/SW solution (5000 ppm) was used as EOR fluid. The oil recovery was calculated using equation (4):

$$\text{Oil recovery efficiency} = \text{volume of oil produced} / OOIP * 100\% \quad (4)$$

Figure (6) represents the oil recovery curve of secondary (seawater) and tertiary (surfactant) recovery using 0.1 cc/min injection rate. In the secondary stage, the oil recovery increased rapidly in the first 1.8 PV. After that the graduated increase continued till about 5 PV where the recovery reached its flat plateau. The cumulative oil recovery of the secondary stage was 50% of OOIP. After secondary stage the flooding scheme switched to tertiary stage using TMAC/SW solution with concentration 5000 ppm. Additional oil production of 24% of OOIP in the tertiary stage obtained as a result of the injection of TMAC/SW (5000 ppm). The total oil recovery was about 74% of OOIP in both secondary and tertiary modes.

The incremental oil recovery (24%) in core flooding test, the production profile (16%) of spontaneous imbibition test, and also the IFT results proved that the TMAC/SW at concentration 5000 ppm resulted in better outcomes from the low concentration 100 ppm, as mentioned in our previous studies [38, 39]. and this can be attributed to the same reasons that are aforementioned in section 3.2. Table (3) shows the comparison between TMAC with commercial surfactants used in different studies.

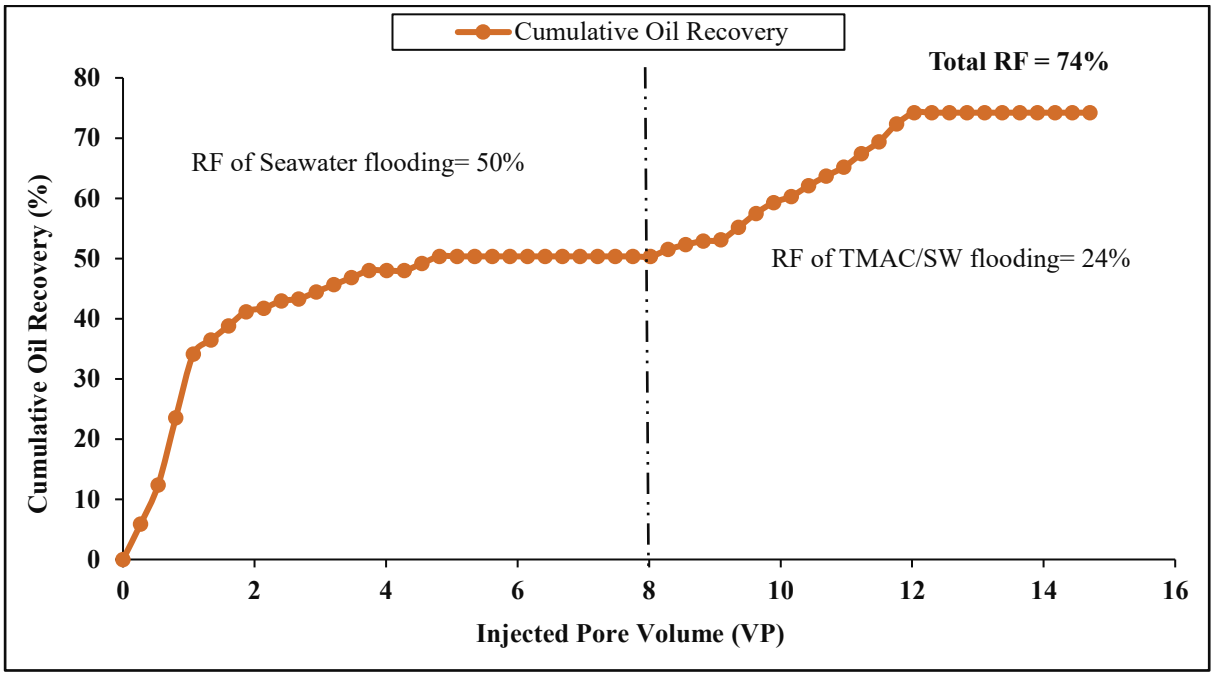

Figure 6. Oil recovery results of core (F) as 5000 ppm TMAC/SW injected at 0.1 cc/min, back pressure 3000 psi and confining pressure 4000 psi.

Table 3. Comparison between TMAC and HTMAC with Commercial Surfactants.

Surfactant	Concen.	Condition	IFT	Wettability alteration	SI (Total)	EOR incremental
TMAC	5000 ppm	HTHP High salinity	√	√	16%	24%
CTAB [18]	range	3WT% NaCl	√	-	-	32-55%
CTAB [19]	3%	3% salinity	√	-	-	18.3%
DTAB [21]	1%	High salinity	-	√	72%	-
DTAB [22]	0.5%	High salinity	-	√	55.9%	-

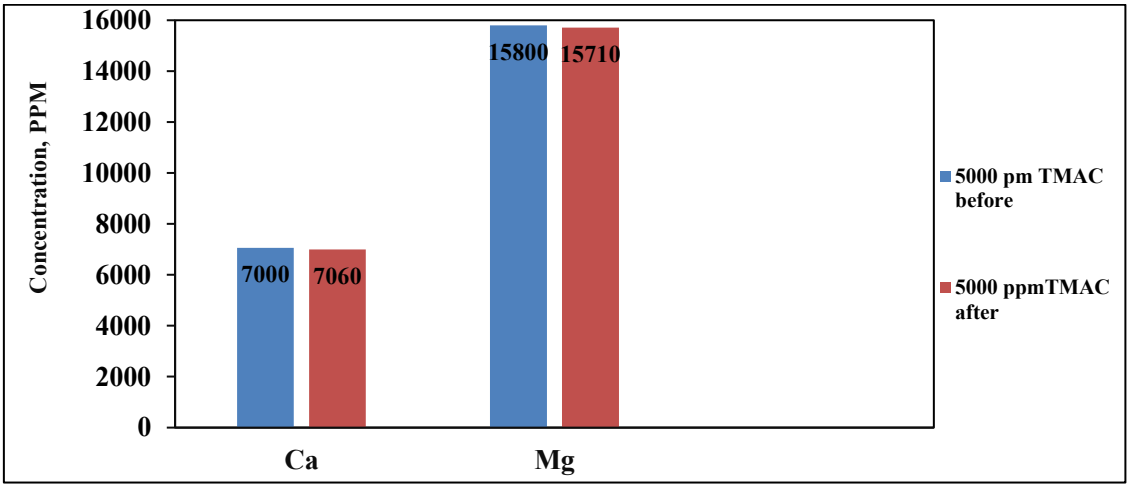

Figure 7. Effluent analysis of spontaneous imbibition test using ICP.

Induced Couple Plasma (ICP):

Induced Couple Plasma test was performed for the effluent fluids of spontaneous imbibition test across Indiana limestone sample that flooded by TMAC/SW solution (5000 ppm). Calcium $Ca^{2+}$and magnesium Mg^{+2} were the ions with the highest percentage in ICP analysis. Figure (7) shows that the Ca^{2+} cation is slightly increased to 7060 ppm while the magnesium concentration was slightly reduced.

It has been documented that Ca^{2+}, Mg^{+2}, and SO_4^{2-} are strong potential determining ions (PDI) which are naturally present in seawater and formation water. These ions can greatly influence the surface charge of carbonate [40-43]. Aging the sample in formation water (241000 ppm) can significantly impact the rock surface chemistry and develop a positive surface charge due to the dominant divalent ions (Ca^{2+}, Mg^{+2}). Consequently, the sample flooded by TMAC that was prepared in high salinity seawater (SW). A partial dissolution or ion exchange can lead to this slight change in Ca^{2+} and Mg^{+2} concentration. This agree with the previous observations that the initial brine composition play a crucial role in determining the wettability and surface charge [44-46].

CONCLUSION

In this work, the application of TMAC surfactant which prepared in high salinity seawater has been applied for chemical enhanced oil recovery. The efficiency of oil recovery in low-permeability Indiana limestone has been investigated using spontaneous imbibition and core-flooding experiments. The IFT results confirmed that the high concentration (5000 ppm) of TMAC/SW reduced the IFT more than the lower concentration. This impact on IFT reflects on SI and core-flooding outcomes. The total oil production was 16% in SI test while the cumulative oil recovery was 24% of OOIP in the core-flooding test.

NOMENCLATURES

TMAC	Tetramethylammonium chloride
CTAB	Cetrimonium bromide
DTAB	Dodecyltrimethylammonium bromide
EOR	Enhanced oil recovery
CEOR	Chemical enhanced oil recovery
IFT	Interfacial tension
WA	Wettability alteration
SW	Sea water
FW	Formation water
DI	Deionized water
QAS	Quaternary Ammonium Salts
SI	Spontaneous Imbibition
CF	Core flooding
CA	Contact Angle
XRD	X-Ray diffraction
FTIR	Fourier-transform infrared spectroscopy
ICP	Induced Couple Plasma
Soi	Initial oil saturation
Swi	Irreducible water saturation
Sor	Residual oil saturation
OOIP	Oil originally in place
PV	Pore volume

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