Evaluation of a New Agent for Wettability Alteration During Enhanced Oil Recovery

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ABSTRACT: *Wettability alteration is an important mechanism when applying surfactants during enhanced oil recovery (EOR). Moreover, in carbonate reservoirs, the rock has more tendencies to be oil-wet than those of sandstone reservoirs. This mainly arises from the interaction amongst the carbonate surface chemistry exposed to the asphaltenic/aromatic components of crude oil. Therefore, it is of great significance to develop surfactants with high efficiency in wettability alteration of carbonate rocks. In this work, a cationic surfactant n-dodecyltriethylammonium bromide (DTEAB), was prepared and applied in a wettability alteration experiment for the first time. Then, the critical micelle concentration and wettability properties were studied by means of electrical conductivity and sessile drop techniques, respectively. Consequently, it was observed that this surfactant can alter the wettability of Bangestan rock pellets from oil-wet to water-wet state, and the results are better than that of wettability alteration observed for Khark rock pellets. Based on this mechanism, nearby 22% Original Oil in Place (OOIP) was obtained by tertiary surfactant flood during core displacement in excess of ultimate recovery factor of secondary water injection. As a consequence, the suggested cationic surfactant in this study can be a good agent for increasing oil recovery factor in the petroleum industry.*

KEYWORDS: *n-dodecyltriethylammonium bromide (DTEAB); Cationic surfactant; Critical micelle concentration (CMC); Wettability alteration; Core displacement.*

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INTRODUCTION

Enhanced oil recovery is one of the most promising methods for increasing oil recovery from oil reservoirs [1, 2]. For the past 50 years, improving the oil recovery *via* wettability alteration has been grasped attentions especially for carbonate reservoir which covers nearby 50% of the world's known reservoirs [3-5]. Based on the preceding investigations, wettability is recognized as the leading parameter influencing the distribution, flow and location of fluid in the porous media; thereby, there will be a satisfactory wettability condition in which the highest amount of oil production from the petroleum reservoirs could be produced [6-10]. In accordance to the proposed definition by *Anderson* [11], wettability is defined as the propensity of a fluid for adhering to or spreading on the surface of a solid when other immiscible fluids are existing. For example, in an oil-wet system, the oil has the preference of coating or wetting the majority of the surface of rock occupying the small pores [12].

Several quantitative methods including contact angle method, *Amott* [13] and U.S. Bureau of Mines (USBM) [11, 14] have been extended to measure the degree of rock wettability. In contact angle method, the angle of a liquid droplet in the presence of the other immiscible fluids is measured through the denser phase as a representative of wettability disregarding the effect of surface roughness and heterogeneity of rock [15]. When we are dealing with reservoir properties, the USBM and Amott tests are utilized in which an average value is assigned for the wettability by such techniques [15]. In addition to the quantitative methods, for qualitative analysis of reservoir wettability, various approaches including imbibition rates, reservoir logs, and capillary pressure curves have been broadly applied in the literature [16].

Using the technique of water advancing contact angle, wettability state of 55 oil reservoirs were evaluated by *Treiber* and *Owens* [17]. In their study, variation of contact angle from 0 to 75°, 75° to 105° and 105° to 180° are termed as water-wet, intermediate-wet and oil-wet, respectively. As an important finding of their study, they observed that in about 84% of the studies, carbonate reservoirs were oil-wet. In 1993, a comprehensive investigation was conducted on wettability of 161 dolomite, calcareous dolomite, dolomitic limestone, and limestone cores by *Chilingar* and *Yen* [18]. The authors understood that about 80% of the studied carbonate core samples which include most of the world's oil reservoirs, were either oil-wet (i.e., 100° to 120°) or strongly-oil wet (i.e., 120° to 180°).

Initial wettability of petroleum reservoir can be influenced by a number of parameters including rock mineralogy, temperature, oil composition, pressure and chemistry of brine, including pH and ionic composition [11]. In this regard, it has been thought that the deposition of organic compounds or/and the adsorption of polar constituents of crude oil have a crucial impact on wettability alteration of reservoir rock [3]. For adsorption of polar constituents of crude oil rock surface, four mechanisms were proposed by *Buckley et al.* [19] and *Buckley* and *Liu* [20] including: polar interactions, which happens when the water film between rock and oil is not existing; acid/base interaction, wherein it happens between the liquid/solid and liquid/liquid interfaces; ion binding, where the brine/oil interface can be connected to the rock surface by the existing divalent or multivalent ions in the brine; surface precipitation of asphaltenes, when the crude oil component has a very low solubility in the reservoir oil.

In the center of the all EOR technologies, surfactant flooding [21-26] and steam injection [27, 28] have been proposed so as to alter the wettability of reservoir rock. There is a bulk of researches focusing on wettability alteration as a key mechanism for EOR processes [29-31].

In present study, a cationic surfactant was prepared in order to evaluate its potential application in wettability alteration of Bangestan and Khark carbonate rocks for the first time. At first step, n-dodecyltriethylammonium bromide (DTEAB) as cationic surfactant was synthesized. Then, by means of electrical conductivity approach the critical micelle concentration (CMC) of the surfactant was measured. Applying the sessile drop technique, alteration in rock wettability was monitored as surfactant concentration changes. Finally, core displacement experiments were executed in order to have an estimation of the oil recovery potential of DTEAB surfactant.

EXPERIMENTAL SECTION

Materials

Preliminary materials for surfactant synthesis

The following materials were used in the preparation of DTEAB surfactant: Triethylamine, dodecyl bromide and acetonitrile. All chemicals were purchased from Merck Chemical Company.

Oil phase

A crude oil sample was obtained from Bangestan reservoir. Bangestan's oil density and viscosity are 0.88 g/cm^3 and 26 cp, respectively, at ambient temperature.

Aqueous phase

Solutions with different concentration of surfactant were made up using distilled water. These solutions were applied in order to carry out the electrical conductivity, contact angle and core displacement measurements.

Rock sample

For implementing wettability assessment, two types of rock pieces (pellets) were used. These pellets were prepared from Bangestan group located in southwest of Iran, Khouzestan province, and coral-reef formation of Khark group located in south of Iran, Persian Gulf. Moreover, in core displacement test, the carbonate core sample of Bangestan group was utilized only in this study. Core sampling and core cutting systems are used to make core plug in an appropriate size.

Methods

Synthesis of dodecyl triethyl ammonium bromide

To prepare the cationic surfactant, about 10 mL of dodecyl bromide was poured into a round-bottom flask, and then approximate volumes of 5 ml tri ethylamine and 20 ml acetonitrile as solvent were added to the container. The round-bottom flask was connected to the reflux system and heated for 24 hours. The flask content was poured into a beaker and cooled in an ice water bath. The white solid surfactant was filtered and washed with small amount of ethanol, and then it was dried at room temperature. The molecular structure of dodecyltrimethylammonium bromide is indicated in Fig. 1 [32]. Some chemical and physical properties of DTEAB is reported in Table 1.

CMC Measurement

CMC is defined as the point in which a sudden alteration in physical properties of the utilized surfactant will be observed. This is due to the agglomeration of surfactant monomers leading to the formation of micelles. For CMC determination, several techniques have been developed including refractive index, pH, and Interfacial Tension (IFT), electrical conductivity and osmotic pressure. Among these methods, electrical conductivity is one of the most widely used methods in literature for CMC determination.

Table 1: Some chemical and physical properties of DTEAB [32].

Molecular formula	$C_{18}H_{40}BrN$
Molecular weight (g/mol)	350.429
Exact Mass (g/mol)	349.234
IUPAC name	n-dodecyl(triethyl) ammonium;bromide
Covalently-Bonded Unit Count	2
Defined Bond Stereocenter Count	0
Heavy Atom Count	20
Compound Is Canonicalized	Yes

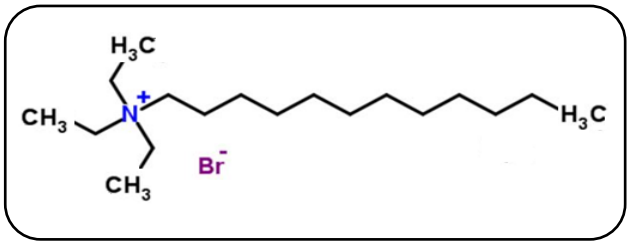

Fig. 1: Molecular structure of synthesized surfactant used for wettability alteration [32].

In this study, JENWAY-4510 apparatus was applied. At first, by using an appropriate solution of KCl the conductivity-meter was calibrated. Then, the most concentrated solution of the surfactant was prepared and by dilution method the other concentrations were made ready for the experiment. About 19 surfactant solutions with concentrations ranging from 0.01 to 1.00 were prepared in this study. For each conductivity measurement, the probe was fully immersed in the beaker containing the surfactant solution. After each measurement, the probe was washed carefully by distilled water.

Wettability measurement

For wettability measurement, the rock pellets of both Bangestan and Khark groups were aged into the filtered Bangestan crude oil for about 14 days in the oven at the temperature of 50˚ C. Afterwards, the pellets were washed up with the kerosene and made it ready for contact angle measurement. VIT-6000 apparatus (Fars EOR Group Co.) was employed in order to implement the sessile drop approach for measuring the contact angle of the drop through the denser fluid (water) which is sitting on the pellet. The schematic illustration of the VIT-6000 apparatus is shown in Fig. 2. As can be seen, this apparatus has four parts of video camera system, illuminating system,

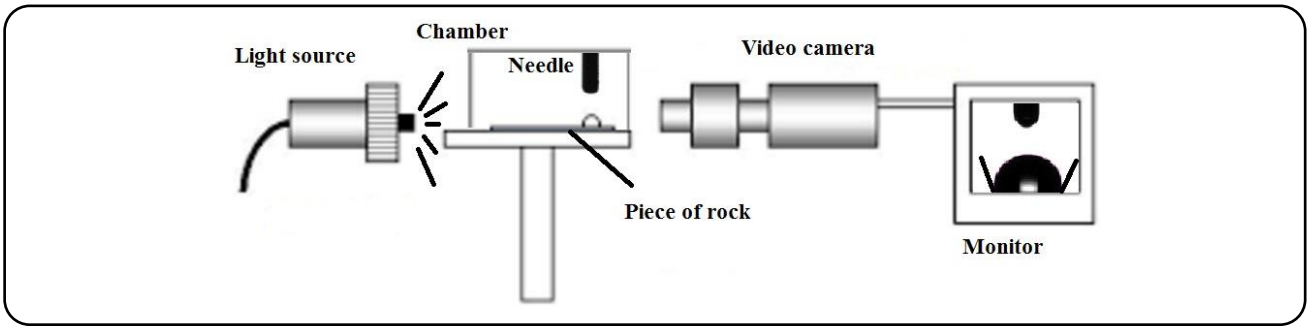

Fig. 2: Contact angle measurement by sessile drop method.

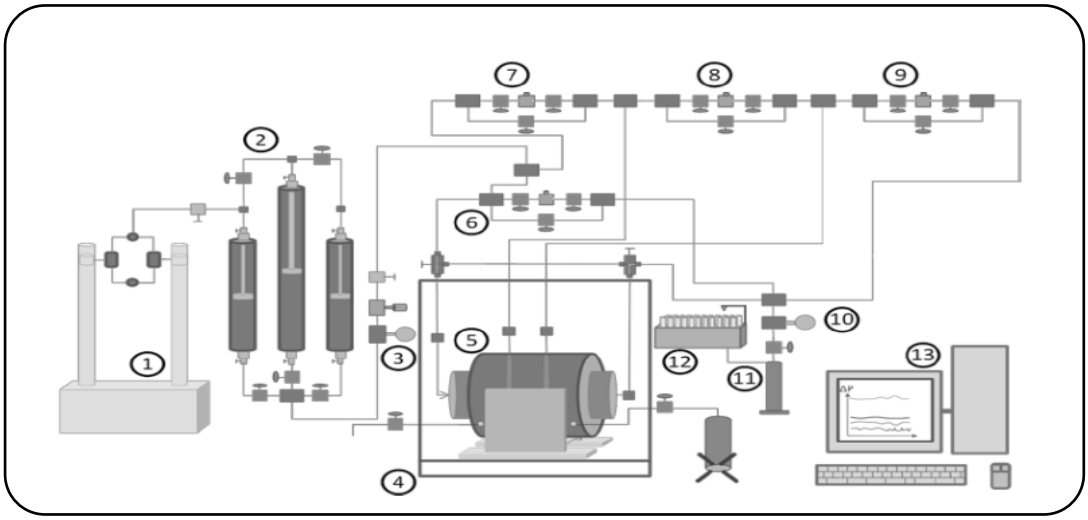

Fig. 3: Schematic of built-in-house coreflood system. Pulse-less syringe pumps (1); High-pressure accumulators for reservoir fluids (2); Upstream pressure gauge (3); Oven (4); Hassler core holder with internal pressure taps (5); Overall differential pressure transducer (6); Differential pressure transducer for first third of core (7); Differential pressure transducer for the middle third of the core (8); Differential pressure transducer for the final third of the core (9); Downstream differential pressure transducer (10); Backpressure regulator (11); Fraction Collector (12); PC and control software (13).

experimental chamber and data acquisition or monitoring system. In detail, the pellet was put into the experimental chamber in an appropriate place. Then, the chamber was filled with kerosene as the representative of the oil phase. By injecting the surfactant solution with a syringe pump at a very low rate, a droplet of surfactant solution would be dropped on the pellet through the needle locating in the top of the chamber. The shape of the liquid droplet on the pellet would be illustrated by the computer connected to VIT-6000 in which the contact angle could be easily calculated by the Digitizer software.

Core displacement experiment

Before executing the flooding experiments, the core plug was washed up by methanol for about 48 hours in the soxhlet extractor apparatus. Then, the core was put in the oven for 24 hours at the temperature of 100° C. In the next step, the plug was placed in core holder. One side of the core holder was sealed and the other side was connected to the vacuum pump. The core plug was then evacuated completely for about 5 hours. When the vacuum process was completed, the vacuum pump was disconnected and the core holder was connected to the transducer. In this manner, the core flood system would be set up in order to start the experiment. Fig. 3 represents the schematic illustration of the core flood set up used in this study [33]. By means of the manual pump connected to the top of the core holder, the core sample would be under the constant overburden pressure of 2500 psi. It is mentionable that both sides of core holder were sealed at this stage. Transducer was filled with distilled water and the line connected to the core holder would be under different

constant pressures up to 1500 psi. At this time, the valve connected to the core holder was opened, and the other side was opened as well. For permeability measurement, under different constant rates, the pressure drop was recorded by the operator. Using Darcy equation, the absolute permeability could be found. Moreover, the difference between the initial volume of the syringe pump at the time of valve opening and final volume of pump at which the first drop was produced at the end of core holder, shows the Pore Volume (PV) of core plug.

For measuring irreducible water saturation (S_{wi}), transducer was filled with crude oil and the core plug was flooded with oil for at least 3 PV. The produced water was collected and subtracted from the pore volume of the core plug showing the volume of the irreducible water trapped in the porous media. As a result, the S_{wi} was also calculated. Now the core plug was ready for implementing the displacement process. At first, the core plug was swept by the distilled water at secondary stage, and then it was flooded by surfactant solution at tertiary stage. For both secondary water injection and tertiary surfactant flood, the process was continued until no more volume of crude oil would be produced at the end of the system at the injection rate of 0.5 cc/min. Core specifications data are inserted in Table 2.

RESULTS AND DISCUSSIONS

In order to apply DTEAB as cationic surfactant, it is necessary to determine the value of CMC. The CMC shows the point where the surfactant monomers are agglomerated to form micelles. Surfactant micelles are of great importance in which the rate of change of surfactant physical properties experiences a crucial variation. Moreover, surfactants are applied in EOR at concentrations above the CMC. Fig. 4 displays the results of the electrical conductivity measurements versus the surfactant concentration at standard condition. The CMC value is determined to be 0.29 wt. % at which the micelles of DTEAB will be established. In accordance with Fig. 4, the slope of electrical conductivity curve is decreased at 0.29 wt. %. It is notable that the uncertainty related to this measurement is about ±0.02 wt. %. So, we can report the CMC value as 0.29 ± 0.02 wt. %. Before reaching CMC, surfactant molecules are in the form of monomers which can carry electrical charges at very high speed than the micelles. For this reason, for concentrations above the CMC, an alteration in the trend of electrical conductivity curve will be observed.

Table 2: Specifications of core sample in tests No. 1 and 2.

Diameter (cm)	3.69
Length (cm)	7.13
Bulk volume (cm^3)	76.21
Pore volume (cm^3)	7.014
Porosity (%)	9.2
S_{wi} (%)	23
Absolute permeability (mD)	1.10

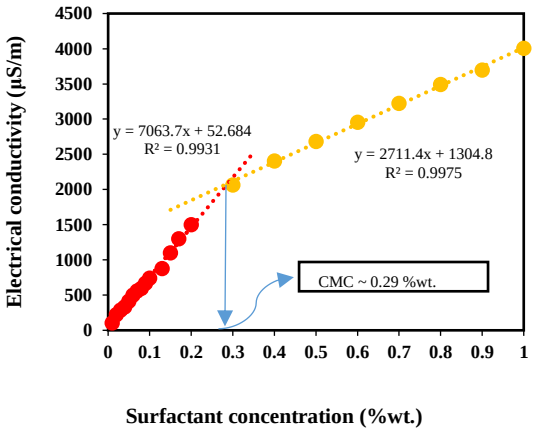

Fig. 4: CMC determination of DTEAB as surfactant by electrical conductivity method.

By knowing the CMC value, wettability alteration experiment is designed for concentrations at the vicinity of such value. The pictures of the settled drops on the rock pellets are shown in Fig. 5 for both Bangestan and Khark pellets. Another illustration type of wettability alteration was illustrated in Fig. 6. When the concentration of DTEAB increases, the contact angle of the droplet with the rock through the denser phase would be decreased. It means that the rock wettability changes from oil-wet to water-wet state for both types of carbonate rock pellets. The reason that can be put forth for such phenomenon is the adsorption of the surfactant monomers/micelles on the rock and their replacement with the aromatic and asphaltenic compounds existing on the rock surface.

Furthermore, the result of wettability alteration for Bangestan rock pellets is slightly better than those of Khark. In other words, the Bangestan rock pellets exhibits more water-wet tendencies than the Khark pellets due to the more adsorption and more replacement of the surfactant molecules with the aromatic and asphaltenic

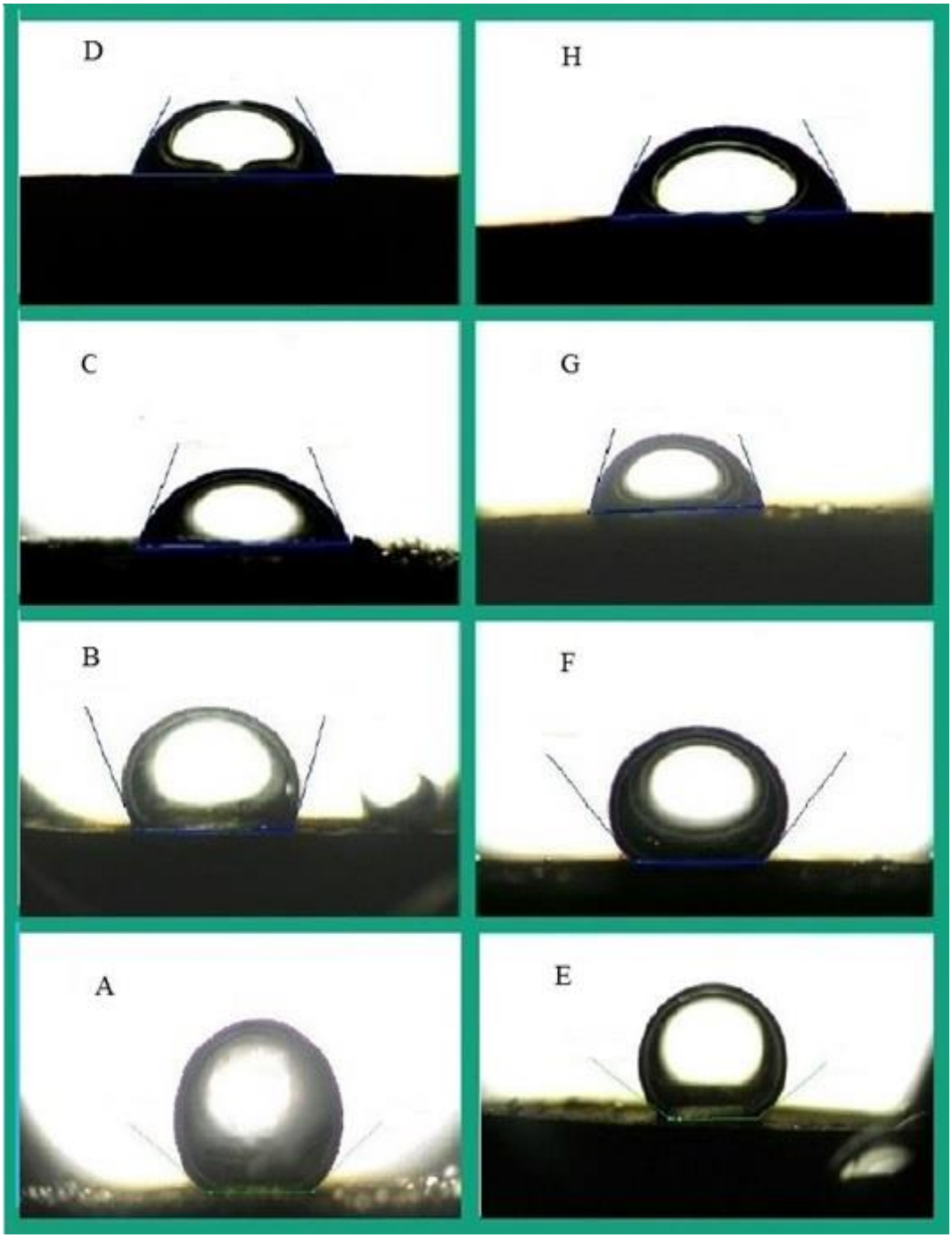

Fig. 5: Wettability alteration of Khark and Bangestan pellets by several concentrations of suggested surfactant in this study; (A) Treated Bangestan pellet in distilled water, (B) Treated Bangestan pellet in 0.08 %wt. of DTEAB, (C) Treated Bangestan pellet in 0.2 %wt. of DTEAB, (D) Treated Bangestan pellet in 0.5 %wt. of DTEAB, (E) Treated Khark pellet in distilled water, (F) Treated Khark pellet in 0.08 %wt. of DTEAB, (G) Treated Khark pellet in 0.2 %wt. of DTEAB, and (H) Treated Khark pellet in 0.5 %wt. of DTEAB.

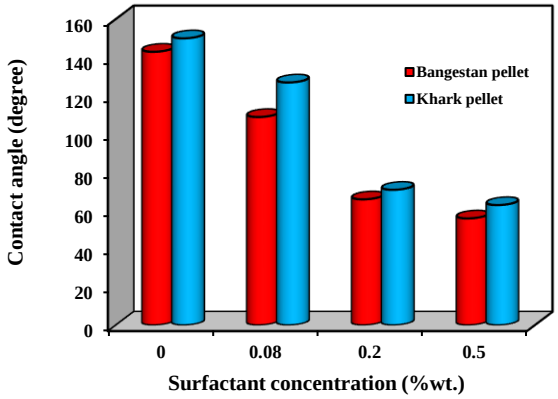

Fig. 6: The contact angle of the treated carbonate Bangestan and Khark pellets at different concentrations of DTEAB.

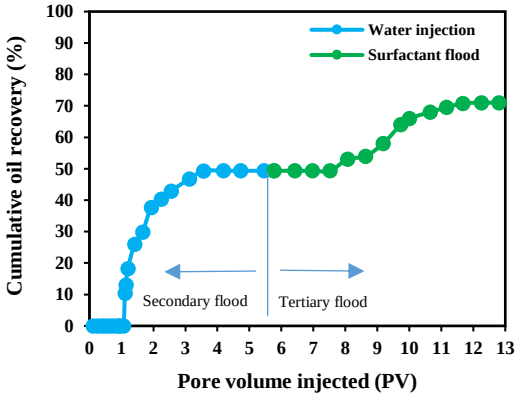

Fig. 7: Cumulative recovery curve for water injection followed by surfactant flooding during core displacement experiment.

compounds adsorbed on the rock. This is because of the surface chemistry of the Bangestan rock which adsorb lower amount of asphaltenic compounds on its surface; therefore, the replacement of surfactant with such aromatic material on Bangestan pellets can be considered easier than the adsorption on the Khark pellets.

Based on the wettability alteration results, this process can be considered as an active mechanism for EOR; therefore, a core displacement analysis is designed so as to have an estimation of recovery potential of the proposed surfactant in this study. Cumulative recovery factor of secondary water injection followed by tertiary surfactant flood is illustrated in Fig. 7. It is obvious that the ultimate recovery factor at the end of water injection and surfactant flood are approximately 49% and 71% of original oil in place (OOIP), respectively.

In other words, injection of surfactant at tertiary stage of production adds about 22% OOIP to the cumulative recovery obtained by water injection. This additional recovery is attributed to the wettability alteration obtained during core flood experiment. In other hands, when the DTEAB enters to the porous media, it will be adsorbed on the pores and pore throats and eliminates the asphaltenic compounds by replacement with such functional groups. As a result, the trapped oil in the porous media can be extracted and produced in addition to the oil recovered by secondary water injection. In conclusion, the results of this study may have significance for future application in petroleum industry.

CONCLUSIONS

In this study, dodecyltrimethylammonium bromide, as cationic surfactant, was prepared. Then, it was applied in EOR for the first time. For this, the CMC value of the proposed surfactant was measured by use of electrical conductivity method. Measurement of wettability by sessile drop demonstrated that this mechanism is an active EOR process. Based on this process, it was observed that this surfactant can alter the rock wettability from oil-wet to the water-wet. Moreover, this surfactant has a better effect on wettability alteration of Bangestan rock than that of Khark one. Conducting recovery potential analysis by core flood experiment demonstrated that the proposed surfactant can achieve nearly 22% OOIP in addition to the ultimate recovery obtained from water injection. Finally, the suggested cationic surfactant could be a potential nominee for future EOR studies.

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