1. Introduction

Surfactant flooding has been regarded as a potential tertiary oil recovery technology in depleted reservoirs after water flooding. This method is being more and more interested as the oil price is getting higher. Extensive laboratory studies concerned with surfactant systems have been done during past decades. Many efforts of enhanced oil recovery tend to displace the remaining oil in sandstones and chalks by the use of surfactants. Pilot and commercial scale projects have employed surfactant flooding as a promising oil recovery method.

The success of this enhanced oil recovery method is crucially dependant on surfactant selection. Extensive experimental research is necessary prior to implementation of the process to assure that the surfactant chosen is right for the reservoir of interest. Surfactant adsorption is also an important aspect of surfactant flooding which has striking effect on the process. Surfactant loss due to its adsorption on the rock surface has been studied by many researchers. While surfactant is adsorbed, surfactant molecules transfer from bulk solution to the surface. It can happen if the surface is energetically more favored by the surfactant than the bulk solution. Effectiveness of surfactant solution to reduce oil-water interfacial tension is impaired by the adsorption of surfactant in porous media and renders the process economically unfeasible. If the adsorption is too high, the large amount of surfactants would be required to produce small amount of extra oil. Although there are some theories to predict surfactant adsorption, experimental methods are the most appropriate way to determine the adsorption isotherm.

In the present study, the feasibility of surfactant flooding in sandstone of one of south Iranian reservoirs was investigated using core flooding experiments. Three different kinds of surfactants containing an anionic, a cationic and a new nonionic biosurfactant, *Zizyphus Spina-Christi* extract were used for the purpose of oil recovery in a core flood system. Core flooding tests were conducted on sandstone to test the effectiveness of surfactants in tertiary oil recovery. Extensive research including adsorption behavior of surfactants on the rock surface and interfacial tension (IFT) measurements were used to analyze the surfactant flooding performance. An additional recovery varying between 6.15 % and 12.17 % of original oil in place (OOIP) was obtained by surfactant solution injection. The results indicated that the new biosurfactant can be used as an enhanced oil recovery (EOR) agent either for IFT reduction or wettability alteration purpose. Furthermore, availability and the low cost of the *Zizyphus Spina-Christi* extract in comparison to common chemical surfactants in petroleum industry make it economically viable for surfactant flooding.
2. Materials and Experiments

2.1. Materials

2.1.1. Surfactants

The following types of surfactants were selected for the experiments:

(a) Anionic surfactant

The anionic surfactant used in this study was Sodium Lauryl Sulfate (SLS) with molecular weight of 288.4 Daltons, supplied from IndiaMART.

(b) Cationic surfactant

The cationic surfactant used in this work was Cetyl Trimethyl Ammonium Bromide (CTAB) supplied from Merck Co. The molecular weight is 364.45 Daltons.

(c) Nonionic surfactant

Nonionic saponin is a natural biosurfactant extracted from leaves of Zizyphus Spina-Christi tree (collected from south of Iran) by spray dryer method. The total extracted powder contains saponin and flavonoids. Powder has light brown color and soluble in water and alcohol. The density of the powder is 0.103 g/cm³ and 1% of this powder in water has the pH of 4.6-5. The chemical composition of the oil of wild Zizyphus Spina-Christi leaves obtained by hydrodistillation was examined by capillary GC and GC/MS. Thirty-four components were characterized, representing 92.2% of the oil. The main components in the oil were geranyl acetone (14.0%), methyl hexadecanoate (10.0%), methyl octadecanoate (9.9%), farnesyl acetone C (9.9%), hexadecanol (9.7%) and ethyl octadecanoate (8.0%). Pordel et al. used saponin surfactant for the purpose of wettability alteration in carbonate rocks. This surfactant has been limitedly used as an enhanced oil recovery agent in core flooding systems.

2.1.2. Rock Samples

Rock samples were taken from Asmari sandstone. Based on the petrographic observations, the rock composition of the Asmari reservoir varies between silica clastic and carbonates lithologies. In this reservoir, quartz and dolomite are dominant minerals where calcite, anhydrite, clay minerals, potassium feldspar and iron oxides are among other abundant minerals. The properties of the samples used in this study are similar to each other in order to obtain comparable results. Porosity and permeability of the cores were measured as part of the characterization work.

2.2. Experimental Procedure

2.2.1. Interfacial Tension Measurement

Low residual oil concentration is achieved only when oil-water interfacial tension is reduced to a small value by the use of surface active agents or surfactants. The interfacial tension between kerosene and aqueous surfactant solutions was determined from axisymmetric drop shape analysis of a captive drive instrument (pendant drop method) shown in Fig. 1(a) to see if acceptable IFT reduction occurs. A typical pendant drop apparatus consists of three parts: an experimental cell, an illuminating and a viewing system to visualize the drop and a data acquisition system to infer interfacial tension from the pendant drop profile. The IFT values were obtained using following empirical relationship:

$$\gamma = \frac{D_s \Delta \rho g}{H} \quad (1)$$

Where $\gamma$ is the interfacial tension, $\Delta \rho$ is the density difference, $g$ is gravitational constant, $D_s$ is the equatorial diameter of the drop and $H$ is a correction factor which is related to the shape factor of the pendant drop, $S$, defined as:

$$S = \frac{D_s}{D_e} \quad (2)$$

Where $D_e$ is the drop diameter measured horizontally at a distance $D_s$ away from the apex of the drop. The values of $H$ were obtained by solving the Bashforth and Adams equation. The droplet profile was recorded in PC with a CCD scope with an appropriate time interval.

Figure 1(b) indicates the final state of surfactant solution drops in kerosene at specified concentration of 6000 ppm just before drop falling which is used for $D_s$ and $D_e$ determination.

2.2.2. Solution Preparation and Determining Calibration Curves

Surfactant solution was prepared by adding accurate amount of surfactant powder to a specific volume of water (measured by a disspensette), in a 1000 mL vessel. Dissolving process was carried out by using a magnetic stirrer. Continuous heating was applied to overcome Kraft temperature for 4 h. Each solution was prepared separately to eliminate the error caused by stock solution preparation and dilution. The conductivity and density were measured at a constant temperature (25 ± 1 °C) to determine calibration curves.

2.2.3. Adsorption Measurement

During a surfactant flood, surfactant is usually lost by adsorption onto the porous medium. This phenomenon was experimentally studied to assess the performance of the surfactants. Adsorption isotherms were determined using batch equilibrium techniques. Sand rocks were initially crushed into small rock fractions into single grains using a mortar and pestle. The resulting sand was sieved using meshed sieves under the agitation of a Ro-Tap Testing Sieve Shaker. Different grain sizes were obtained and the grain ranges with maximum frequency were used to measure the adsorption. A mass ratio of 1 : 5 of rock to surfactant solution was applied. The equilibrium time needed for complete surfactant adsorption during agitation is less than 24 h. As an example, adsorption amount of SLS surfactant was measured at different contact durations.
to ensure the equilibrium occurrence after 24 h. The calibration curves of density and conductivity of the solution built by the use of Anton Paar model DMA 5000 density-meter and Sartorius professional PP-20 conductivity-meter were used for concentration measurement. The surfactant solution and sand particles were equilibrated in flask containers in gentle agitation for 24 h which is more than the time required to reach equilibrium as mentioned before. The surfactant solution and sand sediments were then separated by centrifugation. Different centrifuging speeds and durations were tested to find optically the optimum conditions in which all rocks deposit in the solution. The optimum condition was the speed of 7000 rpm for 15 min. Finally, the surfactant concentration was determined using surfactant properties like density, conductivity and interfacial tension.

It is worth mentioning that salinity partly affects the adsorption process, however, this influence depends on the surfactant type, brine composition and rock type. Since this paper is aimed at comparing the adsorption behavior of the surfactants, deionized (DI) water is used for all adsorption experiments.

2.2.4. Core Flood Tests

Flood experiments were performed in sandstone cylindrical cores. The cores were first carefully washed to remove any liquid and then oven dried for 24 h. Each core was placed inside a rubber sleeve and the sleeve with the core was inserted into the core holder. The sleeve would grip and squeeze around the core when a confining pressure of around 3000 psi was applied. Figure 2 shows a schematic of the core flood set up. It simply consists of an ISCO pump (syringe pump) which is very sensitive to flowrate (low flowrate of 10-5 mL/min up to at most 25 mL/min) with maximum pressure of 7000 psi, a core holder which can tolerate the pressure of 5500 psi (3.74 × 10^7 Pa), a 10,000 psi (6.80 × 10^7 Pa) pressure gauge and two distributors.

The porosity of each core was determined by the volume of injected water after putting it in evacuation for one hour. The absolute permeability of any core was obtained by injecting distilled water through the core at five different rates under steady state conditions. The flowrates were selected low enough to achieve laminar flow conditions. Assuming linear, incompressible, and one-dimensional flow, Darcy’s equation was used to calculate the absolute permeability of each core. The saturated cores were then brought to connate water conditions in a primary drainage oil flood. The oil used
was from one of Iranian reservoir with density of 849.6 kg/m³ and viscosity of 6.2 mPa s measured at 25 °C. Core properties are given in Table 1.

After establishing the initial conditions, the cores were ready for water flooding as a secondary oil recovery process. Water was injected with a constant rate of 1 mL/min. Water injection was continued to about one pore volume when the oil production became negligible.

The residual oil saturations can be seen in Table 1. The effluent was collected in 1 mL tubes with 0.2 mL accuracy to obtain the incremental oil recovery. Then, a surfactant slug with specified size of one pore volume was injected with the same rate and additional recovery was obtained. Finally, another pore volume of water was injected as a post flush until the oil cut at the production outlet became nearly 0%.

The common methods of distinguishing between water and oil in surfactant flooding are resting the solution, centrifuging and adding some demulsifiers. The first two methods are applied for all core flooding tests to ensure that the recovery is exactly measured.

3. Results and Discussion

3.1. Interfacial Tension

Prior to core flood tests, surfactant properties of interfacial tension reduction and adsorption behavior were studied. The IFT between the oil and water is reduced due to the adsorption of surfactant on interface. Low IFT is a result of strong interactions between surfactant and oil molecules. Change in IFT was investigated as a function of surfactant concentration. The results showed that the IFT decreased as surfactant concentration increased until its changes became negligible at some concentration called critical micelle concentration. The minimum IFT maintains 0.003, 0.0015 and almost 0.013 N m⁻¹ for SLS, CTAB and saponin, respectively as shown in Fig. 3. The IFT values for saponin/water/oil system are in good agreement with the measured values for other surfactants which are used in chemical flooding and indicate good surfactant characteristics of saponin for chemical flooding. The saponin IFT can also be decreased to ultra low IFT by
the addition of salt and alcohol but in this study since the purpose was to compare surfactant solutions solely, it was not done.

3.2. Surfactant Adsorption

The variations of conductivity and density of SLS surfactant solution with respect to concentration are shown in Fig. 4. The calibration curves were validated with predetermined points of two specific concentrations and the maximum relative error was 5% and 0.0028% for conductivity and density curves, respectively. This showed the reliability of the methods of concentration measurement. The critical micelle concentration (CMC) of the surfactants can be determined by conductivity curves. It was done for three surfactants and the CMC was 2203.13, 716.387 and 48925.802 ppm for SLS, CTAB and saponin, respectively. Figure 4(b) shows the CMC determination of SLS surfactant using conductivity curve.

The loss of surfactant during chemical flooding is a major determinant in this process performance and economic feasibility. Surfactant adsorption is mainly due to the electrostatic interactions between charged sites on the rock surface and those of a surfactant. The non-equilibrium adsorption was determined for SLS concentration of 1000 ppm by the methods of conductivity and density which are in very good agreement (Fig. 5). As it can be seen in Fig. 5, adsorption amount remains almost constant after passing 5 h. Also, 90% of the adsorption occurs in the first 2 h, regarding to non-equilibrium adsorption. This obviously reveals that the 24 h contact time is more than enough to reach equilibrium conditions in adsorption process.

Equilibrium adsorption was measured in a large concentration range. In the case of ionic surfactants three different regions can be seen in adsorption isotherm as shown in Figs. 6(a) and 6(b). Region 1 shows a gradual increase of adsorption with increasing concentration in which surfactant molecules adsorbs on the rock surface and do not interact with each other. In Region 2, adsorption isotherm shows an increase in the slope in which surfactant molecules move toward the solid surface and form hemi-micelle which was originally postulated by Gaudin and Fuerestenau56). In other words, CMC of hemi-micelle formation also can be called critical aggregation concentration (CAC) that is lower than CMC, in which a two dimensional aggregation is formed on the rock surface. In 3rd region, adsorption is hindered by electrostatic repulsion between hemi-micelles and the slope of the adsorption isotherm is reduced. Measuring saponin adsorption, three regions appeared (Fig. 6(c)). In Region 1 a monolayer of surfactants is adsorbed on the surface and interaction between surfactants causes a graduate decrease in the slope. As the surfactant concentration reaches the CMC, the surfactants tend to aggregate and orient in vertical direction which causes more cross section area and results in increasing the slope. In Region 3, the adsorption slope decreases because the interaction between surfactants hemi-micelle on the surface is the same as the bulk solution43). High adsorption of saponin nonionic surfactant at high concentrations is useful in the cases in which it is favorable to alter the wettability57). It must be mentioned that the adsorption values obtained were consistent with previous works58).

It is a proven fact that the amount of surfactant adsorption is a function of surface area. As such, since the surface exposed to surfactant solution in core flooding is smaller than static test, dynamic adsorption is slightly lower than static one. The previous experiments conducted on SLS revealed that the adsorption amount of dynamic test is approximately 70% of static one.

3.3. Flood Experiment

Considering interfacial tension reduction, adsorption behavior and economic issues, concentration of 6000 ppm was selected for all surfactants in flooding experiments. For the concentration of interest
the adsorption amount was the least for new nonionic (1.66 mg/g rock) and the most (2.42 mg/g rock) for cationic surfactant.

Three core flooding experiments were performed to evaluate the displacing efficiency of various surfactant systems. The rock properties shown in Table 1 were determined by core flooding tests described in previous section. The porosity and permeability of all cores were almost the same and made it possible to correlate the flooding performance to the nature of surfactant and not of the rock. The results were evaluated in terms of incremental oil recovery as presented in Fig. 7. The average water flooding recovery was found to be about 55 % of the OOIP for all experiments. Surfactant systems were all successful in producing additional oil. Tertiary oil recoveries were 12.17, 9.25 and 6.15 % of original oil in place for SLS, saponin and CTAB, respectively. The difference between recoveries obtained by each surfactant may be due to several reasons. Overall, it can be explained by the difference between IFT and adsorption values for the concentration used in core floods. In other words, a combination of IFT and adsorption amounts determines the ultimate effectiveness of surfactants in recovery improvement. In the case of anionic surfactant (SLS), the adsorption amount is near to saponin while the minimum IFT value is near to CTAB. The low IFT value and small surfactant loss result in maximum additional recovery. Although the cationic surfactant (CTAB) has the lowest IFT value, the large amount of its adsorption results in poor oil recovery respect to nonionic saponin with higher IFT value. The new biosurfactant, saponin, also has considerable recovery enhancement due to low adsorption value at that specific concentration and acceptable IFT reduction.

In addition to technical reports, economic aspect is one of the most important factors to screen a surfactant. The economic viability is essentially dependent on parameters such as the cost of surface-active substance and the oil price. *Zizyphus Spina-Christi* is a natural surface active substance found in N. Africa east to Afghanistan and NE India. It is easily available. The *Zizyphus Spina-Christi* extract surfactant is quite inexpensive and can be obtained around 2.5-3.0 US$/kg in Middle Eastern countries such as Iran. From the other point, the proposed surfactant is natural and biodegradable. Thus, causes minimal or no harm to the environment compared to common chemical surfactants in petroleum industry.

**4. Conclusions**

The probability of surfactant injection in Asmary sandstone was investigated using core flooding experiments. Three core flood experiments were carried out using three kinds of surfactants. The results showed that the additional recovery was a strong function of surfactant type. Each surfactant had its own specific IFT reduction and adsorption behavior which were both crucial parameters in surfactant flood performance. However, the results showed that the adsorption is more effective respect to IFT reduction.

The amount of tertiary oil recovery observed for anionic surfactant or SLS was 12.17 % of original oil in place after primary water flood in which the average recovery was 55 % but high adsorption of CTAB surfactant caused minimum amount of recovery. For new nonionic saponin, injection of surfactant solution into a fully water flooded core resulted in almost considerable

Fig. 6 Equilibrium Adsorption Isotherm

Fig. 7 Tertiary Oil Recovery Obtained by Surfactants Injection
additional recovery in which final residual oil saturation was equal to the case of anionic surfactant. Also, availability and the low cost of the Zizyphus Spina-Christi make it economically viable for surfactant flooding. Moreover, the newly introduced biosurfactant poses minimal or no harm to the environment.

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