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Experimental Study of the Chemical Stimulation of Iranian Fractured Carbonate Reservoir Rocks as an EOR Potential, the Impact on Spontaneous Imbibition and Capillary Pressure

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Abstract. *Beside their worldwide abundance, oil recovery from fractured carbonate reservoirs is commonly low. Such reservoirs are usually oil-wet, thus, waterflooding leads into early breakthrough and low recovery due to the high conductivity of the fracture network, negative capillary pressure of the matrix and, consequently, the poor spontaneous imbibitions of water from fractures into the matrix during the course of waterflooding. In such problematic reservoirs, changing the wettability of the matrix toward water-wetness can improve spontaneous imbibition by changing the sign and, thus, the direction of capillary forces, resulting in an improvement of waterflood efficiency and, consequently, oil recovery. A study of this technique on the most significant Iranian oil-producing reservoir, Asmari, seems necessary. Some surfactants of different ionic charges have been examined in this study. Asmari reservoir rock samples were used and the petrophysical and mineralogical properties of the rock samples were determined by both thin section analysis and core flooding techniques. Interfacial tension measurements have been done to decide surfactant solution concentrations. Capillary pressure measurements were conducted both before and after wettability alteration. Amott-Harvey and USBM wettability indices were determined. Among the surfactants, a cationic one could best raise the level of spontaneous imbibition. Favourable changes in the wettability indices were observed.*

Keywords: *Wettability alteration; Surfactants; Carbonate reservoirs; Spontaneous imbibition; Capillary pressure.*

INTRODUCTION

Wettability is considered to be one of the influential parameters in multiphase flow and affects porous medium parameters such as capillary pressure, relative permeability and the efficiency of waterflooding. The oil recovery from naturally fractured carbonate reservoirs is usually low; about 20% of the Original Oil In Place (OOIP) is recovered by pure pressure depletion. Treiber et al. [1] evaluated the wettability of 50 reservoirs. They showed 64% of the carbonate reservoir rocks they studied were intermediate-wet, 28% were oil-wet and 8% were water-wet. Nevertheless, waterflooding in such reservoirs leads to poor results due to the oil-wet nature of the matrix, negative capillary pressure, and the highly permeable fracture network. In such conditions, the spontaneous imbibition of the water phase is very poor and the water bypasses the matrix blocks without adequate displacement of the oil from the matrix. Oil-wetness in carbonates is brought about by the adsorption of organic acids and carboxylates onto the rock surface during aging [2]. Altering the wettability of the matrix from oil-wet to water-wet states can solve such a problem. Some surfactants have the capability of modifying the wettability of the rock matrix into water-wet through adsorbing onto the surface. In this way, the injected water will imbibe spontaneously from fractures into the matrix blocks and the oil is displaced. Hamon [3] measured drainage and imbibition capillary pressure curves using reservoir fluids and both oil-wet and water-wet semi-permeable membranes. He reported trends between the amount of

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spontaneous imbibition, the wettability index to water and the residual oil saturation and both structural position and permeability of the samples. Standnes and Austad [4] proposed the mechanism for wettability alteration by surfactant solutions to be the formation of ion-pairs between surfactant monomers and the adsorbed carboxylates. Their laboratory tests with chalk cores showed that oil recovery can reach 70%. Xie et al. [5] applied a cationic surfactant and a non-ionic surfactant to over 50 cores from three dolomite reservoirs. The incremental oil recovery ranged from 5% to 10% OOIP; wettability alteration was identified as the main factor. Ayirala et al. [6] studied the beneficial effects of wettability altering surfactants in oil-wet fractured reservoirs by conducting contact angle measurements using a DDDC technique under ambient conditions. They proposed a sequential process of diffusion and imbibitions, in which the surfactant first diffuses into the rock matrix and alters its wettability enabling the imbibition of even more surfactant solution for significant improvement in oil recovery.

Qualitative and quantitative methods of characterizing wettability have been proposed in the literature [7]. Qualitative measurements include spontaneous imbibition, microscopic visualization of fluid distribution and wettability evaluation using relative permeability curves [8]. Quantitative methods include contact angle measurements, the Amott method [9] and the USBM method [10].

The relative influence of capillary and gravity forces acting on the fluids inside the core in the displacement process is characterized by the inverse bond number [11], N_B^{-1}:

$$N_B^{-1} = C\sigma(\phi/k)^{1/2}/\Delta\rho g h, \tag{1}$$

where $C = 0.4$ for the capillary tube model [11], $\Delta\rho$ is the density difference between the water phase and the oil phase (kg/m^3), σ is the IFT (N/m), ϕ is the porosity (fraction), k is the permeability (m^2), h is the height of the core (m), and g is the gravitational acceleration (m/s^2). In general, spontaneous imbibition is dominated by capillary forces, if $N_B^{-1} > 5$; for $5 > N_B^{-1} > 0.2$ both gravity and capillary forces are active and, if $N_B^{-1} < 0.2$, imbibition is dominated by gravity forces.

In the Amott method, three different wettability indices were defined:

1. Water wettability index (I_w); the ratio of V_{osp} to $(V_{osp} + V_{ofc})$.
2. Oil wettability index (I_o); the ratio of V_{wsp} to $(V_{wsp} + V_{wfc})$.
3. Amott-Harvey wettability index (I_{AH}); the difference between I_w and I_o.

The USBM index was defined as the logarithm of the ratio of the areas under the forced imbibition curve and that of those under the forced drainage curve [12]. The objective of this work is to determine the wettability of Iranian carbonate reservoir rocks and investigate the effect of surfactants on the wettability of the samples to improve spontaneous imbibition. We applied both quantitative and qualitative methods to evaluate wettability and wettability alteration.

EXPERIMENTS AND RESULTS

Materials

Porous Medium

Asmari reservoir formation samples were used as the rock sample whose petrophysical properties will be discussed in the proceeding section.

Brine

Asmari reservoir brine with a high salinity (180*10^3 ppm) and a density of 1.17 g/cc was used as the brine phase. We used reservoir brine to see if it was possible to re-inject it into the reservoir, as it is best at ionic equilibrium with the reservoir rock.

Oil

Asmari stock tank oil was used as the oil phase in all of the experiments. The oil had a viscosity of 0.0197 Pa.s and an API gravity of about 25.7 at 25°C.

Surfactants

Surfactants termed SDS, C12DTAB and Triton X100, which are anionic, cationic and non-ionic, respectively, were used as the surface active agent to be studied.

Sample Preparation

Six carbonate sample rocks were selected from Iranian oil producing carbonate formations, as listed in Table 1. First, thin sections were prepared and analyzed using a polarizan microscope to study the lithology and mineralogy of samples. The first four samples were calcite; the porosity was mainly secondary porosity comprised of micro-fractures with a small percent of dissolution porosity. Samples 5 and 6 were also calcite; some micro-fossils and macro-fossils were evident in the samples as well. The porosity was comprised of inter-granular and dissolution porosity. The first four samples were more homogeneous than the last two, from the mineralogical point of view. Then, the core plugs were prepared with a 1.5 inch core cutter; the dimensions of the plugs are listed in Table 1. The samples were washed with a Dean Stark and Soxhlet Extractor, according to Anderson [13]. Toluene was used as the cleaning solvent. All samples were placed

Table 1. Core properties, measured with core flooding apparatus.

No.	L (mm)	D (mm)	ϕ	K (md)	Lithology
1	57.39	37.39	8.95%	18	Calcite
2	53.47	37.63	10.38%	21	Calcite
3	57.20	36.97	9.18%	21	Calcite
4	54.38	37.12	9.39%	20	Calcite
5	58.86	37.71	15.54%	58	Calcite
6	56.61	37.43	14.88%	51	Calcite

in a furnace at a temperature of 200°C for 48 hours. A nitrogen porosimeter and an air permeameter were used for porosity and absolute permeability measurements, respectively.

The measured porosity and absolute permeability are listed in Table 1. There is good agreement between the measured values and those evaluated from thin section analysis. After the initial core preparation and evaluation of the petrophysical properties, the following steps were taken to restore the samples to a reservoir wettability state for spontaneous imbibition experiments:

1. Plugs were placed into a saturator apparatus and a vacuum was applied for 1 hour.
2. The plugs were saturated by reservoir oil.
3. The samples were submerged in oil and allowed to age for 30 days at 100°C in order to restore reservoir wettability.

Experiments were started after preparation of restored samples.

IFT Measurement

The measured IFT data are listed in Table 2. The IFT measurements between the oil and the brine phase containing surfactants were taken under ambient conditions by means of a pendant drop instrument termed IFT700 with a high sensitivity as well as a spinning drop instrument.

Spontaneous Imbibition Experiments

Four of the plugs were subject to spontaneous imbibition experiments, set into oil-brine-rock systems, shown in Table 2 and accompanied by the inverse bond number for each system. The core samples were 100% saturated with oil and submerged, respectively, in formation brine, SDS, Triton X100 and C12DTAB solutions (the water phase in all the systems was the formation brine) in an Amott cell under ambient conditions, (25°C), and the water phase was allowed to imbibe into the matrix and displace the oil. The recovery was recorded versus time for each system.

System 1; Formation Brine-Oil-Rock

The spontaneous imbibition of reservoir brine containing no surfactant was very slow and stopped very soon (Figure 1). This demonstrates the initial oil-wet nature of the rock sample. The initial recovery of oil is due to the heterogeneities brought about during core preparation and refers to pores in the immediate vicinity of the core surface, which are open to the surface. The oil in such pores might have been washed by the brine contact and expelled by buoyancy. The inverse bond number calculated for this system turns out to be 71.834; emphasising that it would be controlled by capillary forces, should a displacement occur in this system at all. Oil production in this system terminates at a recovery of about 1.5%. This confirms the oil-wetness of the reservoir rock, in other

Table 2. Systems used in spontaneous imbibition experiments and their specifications.

Sys. No.	Water Phase	Surfactant wt %	Oil Phase	IFT (mN/m)	Core	N_B^{-1}
1	Pure brine	-	Asm. Oil	11.475	1	71.834
2	SDS	0.2	Asm. Oil	0.183	2	0.999
3	C12DTAB	1.0	Asm. Oil	1.469	3	9.295
4	Triton X100	0.05	Asm. Oil	0.007	4	0.0424

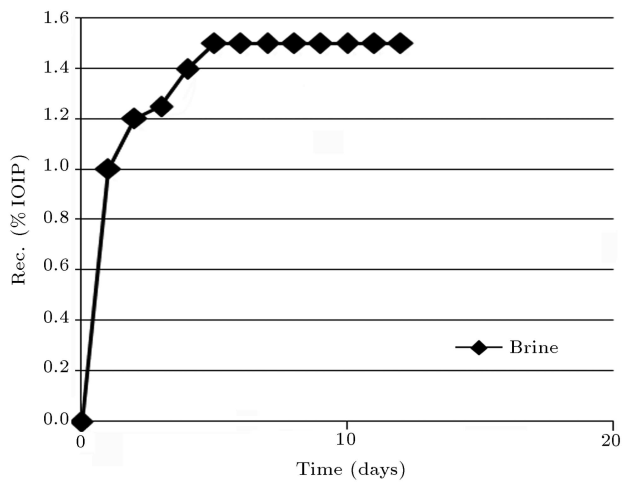

Figure 1. Spontaneous imbibition result, recovery versus time for system 1.

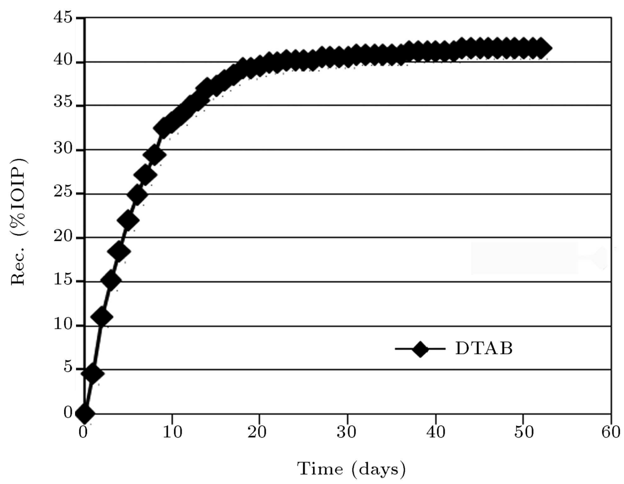

Figure 3. Spontaneous imbibition result, recovery versus time for system 3.

words, negative capillary pressure resists water invasion into the pore space.

System 2; SDS Solution-Oil-Rock

The imbibition of SDS solution ceased within 15 days. The imbibition rate was low and oil production appeared to be a nearly linear function of time, which is typical of imbibition processes where both capillary and gravity forces are active [14] (Figure 2). The imbibition experiment was stopped after 15 days at an oil recovery of about 12%. The inverse bond number for this system was calculated to be 0.999 indicating a process controlled by gravity forces in line with the shape of the imbibition curve.

System 3; C12DTAB Solution (1wt% in Reservoir Brine)-Oil-Rock

The rate of imbibition in this case was very fast, so that about 30% of the OOIP was recovered within only 8 days, then, it slowed down and about 42% of the OOIP was recovered over 51 days. After that, plateau production was reached. The curved shape of the imbibition curve in Figure 3 shows a counter-current imbibition displacement controlled by capillary forces, which is indicative of a change in the wettability of the porous medium [13]. The inverse bond number for this system, 9.295, shows the predominance of capillary forces over gravity forces for this system. The high rate of imbibition in this case is due to the adsorption of the surfactant onto the rock surface from the very beginning of the process, which results in a change in the wettability of the rock and, consequently, a change in the direction of capillary forces.

System 4; Triton X100 Solution-Oil-Rock

The recovery versus time for the spontaneous imbibition in this system is shown in Figure 4. The rate of spontaneous imbibition in this system was lower than that in the C12DTAB system; plateau production

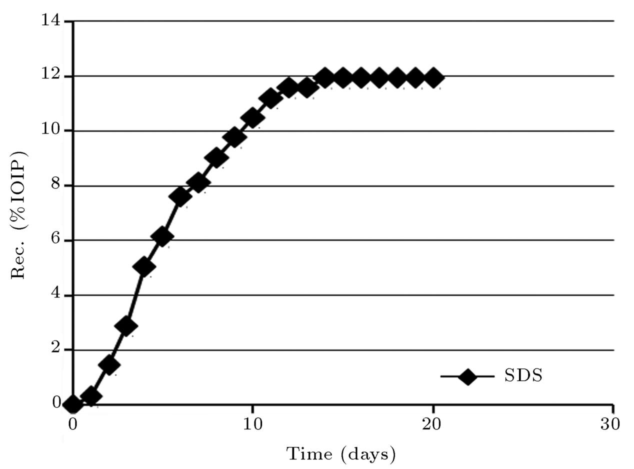

Figure 2. Spontaneous imbibition result, recovery versus time for system 2.

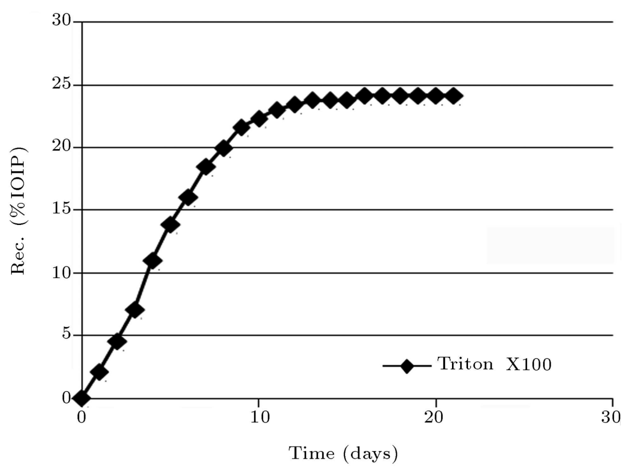

Figure 4. Spontaneous imbibition result, recovery versus time for system 4.

was reached after 20 days at a recovery of about 24% OOIP. The linear trend of the imbibition curve is in line with the inverse bond number (0.044) for this system, which conveys that spontaneous imbibition is strongly controlled by gravity forces in this case. The ultra-low IFT value caused by the concentration of Triton monomers in the oil-water interface has increased the effect of gravity forces in this system, in contrast to system 3, where a counter-current imbibition controlled by capillary forces resulted in a curvature in the imbibition profile.

For further analysis of the difference in trends, the normalized recovery for each system, which is the recovery divided by the final recovery, was plotted versus time. As shown in Figure 5, the recoveries in systems 2 and 3 have similar trends, indicating the predominance of the same driving forces in both cases. The trend in spontaneous imbibition in system 4 deviates from a straight line with a curvature. Here, the imbibition profile carries a different trend, emphasizing the difference in driving forces controlling the displacement, which are capillary forces in this case. This is also in agreement with the inverse bond number and the IFT for this system (Table 2). According to

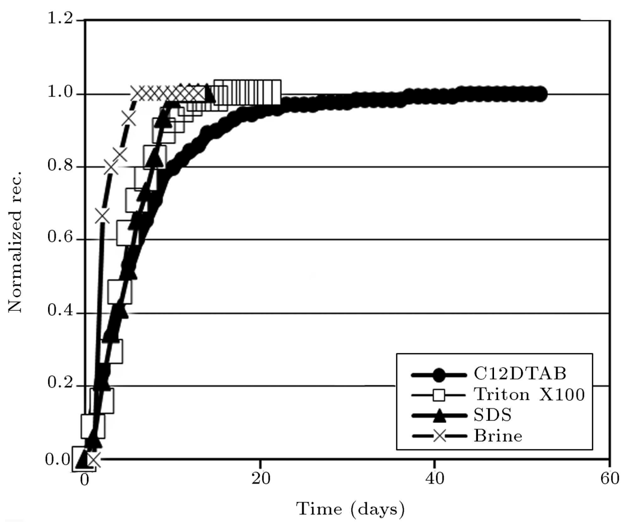

Figure 5. Normalized recovery versus time, spontaneous imbibition experiments.

spontaneous imbibition experiments, C12DTAB was selected for further studies, and capillary pressure experiments were conducted using this surface active agent. To investigate the changes in the wettability of the rock, powerful quantitative methods of Amott-Harvey and USBM were used.

Capillary Pressure Experiments

Capillary pressure experiments were conducted using a core flooding system under reservoir conditions by means of CAPRI. The oil-water-rock systems used in these experiments are shown in Table 3. For quantitative experiments, a combined Amott/USBM test was conducted to measure Amott, Amott-Harvey and USBM indices in a sequence of tests; complete capillary pressure tests were conducted for the purpose. The volume of the displaced phase at each step was accurately measured by means of downstream and upstream metering pumps that accumulate the displaced phases at constant pressure.

In these tests, the following steps were taken to displace water by oil and, subsequently, displace oil by water. The capillary pressure at each saturation point was recorded after saturation stabilization; each point took from several hours to several days to stabilize. Semi-permeable ceramics were used at downstream and upstream of the floods:

1. Primary displacement of water by oil down to irreducible water saturation.
2. Spontaneous displacement of oil by water; the displaced oil was recorded as V_{osp}.
3. Forced displacement of oil by water until no more oil was displaced; recording the volume of oil displaced as V_{ofc}.
4. Spontaneous displacement of water by oil; the displaced water was recorded as V_{wsp}.
5. Forced displacement of water until no more water was displaced; the volume of displaced water was measured as V_{wfc}.

Table 3. Oil-brine-rock systems used in capillary pressure experiments.

Sys. No.	Oil	Brine	Surfactant (wt%)	Core
5	Asm. Oil	Res. Brine	-	5
6	Asm. Oil	Res. Brine	C12DTAB, 1	5
7	Asm. Oil	Res. Brine	-	6
8	Asm. Oil	Res. Brine	Triton X100, 0.05	6

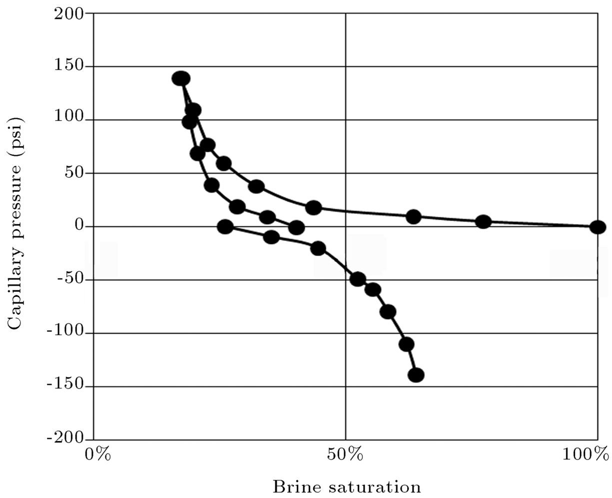

Figure 6. Capillary pressure-saturation curve for system 5 (using reservoir brine as the water phase).

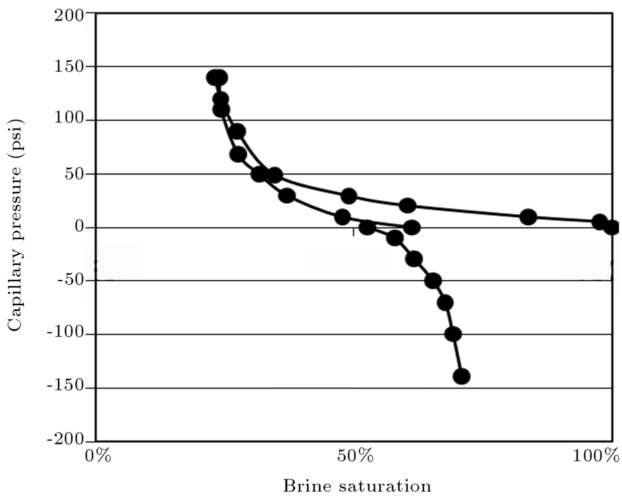

Figure 7. Capillary pressure versus water saturation, system 6 (1wt% C12DTAB in reservoir brine).

System 5

The full-set capillary pressure curve for system 5 is shown in Figure 6. Table 3 includes the specifications of the systems in capillary pressure experiments.

The water, oil, Amott-Harvey and USBM indices for this system are listed in Table 4. The value of the Amott-Harvey Index, ($I_{AH} = -0.314$), and the USBM index ($I_{UABM} = -0.288$) show the oil-wet nature of the rock sample. This explains the resistance of capillary forces against the spontaneous invasion of water into the plug in naturally fractured carbonate reservoirs. Furthermore, it is in good agreement with the lack of spontaneous imbibition in the experiment with reservoir brine, in system 1. The residual oil saturation and irreducible water saturation for this system are listed in Table 4.

System 6

Figure 7 shows the capillary pressure curve for system 6, whose specifications are listed in Table 3. The oil, water, Amott-Harvey and USBM indices associated with this system are listed in Table 4.

In this system, where the reservoir brine has been replaced with 1wt% C12DTAB solution in the reservoir brine, the Amott-Harvey system was calculated to be 0.415, which shows that the rock sample became water-wet due to the adsorption of the surfactant onto the rock surface rendering the surface in the pore scale water-wet. The USBM index for this case was calculated to be 0.194. The change in the wettability of the rock has brought about a rise in irreducible water saturation conveying a reorder in saturation distribution inside the matrix, in comparison with the previous case where the water phase was pure reservoir brine. A greater portion of the pore space is occupied by water, which is retained by narrower pores.

The change in the sign of Amott-Harvey and USBM indices shows the change in the direction of capillary forces, also, that the spontaneous tendency of the matrix to water outweighs that for oil, as can be concluded from Figure 7. This explains the relatively high rate of spontaneous imbibition in the qualitative analysis in system 3. In this case, a rise in the primary drainage curve was observed (Figure 7). Compared with system 5, the irreducible water saturation shows a rise of about 6%.

Systems 7 and 8

The specification and components for system 7 are listed in Table 4. The trend of spontaneous imbibition for system 4, Table 3, showed that Triton X100 was not as efficient as C12DTAB in modifying the wettability of the rock sample and, thus, improving spontaneous imbibition by wettability alteration. However, Triton X100 could lower the IFT between the oil and the brine to ultra-low values (Table 2) leading to the conclusion that the recovery in system 4 was dominated by gravity forces; further confirmed by the value of the inverse bond number (0.0424). Regarding the properties of

Table 4. Results from capillary pressure experiments for systems 5 and 6.

Sys. No.	I_w	I_o	I_{AH}	I_{USBM}	S_{wir}	S_{or}
5	0.193	0.507	-0.314	-0.288	17.12%	36.02%
6	0.618	0.203	0.415	0.194	23.14	29.06

the Triton X100 observed in the experiments, this surfactant could be a good candidate for processes where IFT reduction to ultra-low values is favourable, such as alkali-surfactant-polymer flooding. Therefore, we conducted single drainage (wetting phase displaced by non-wetting phase) both with and without Triton X100, to see the extent to which this surfactant could change the capillary pressure. Lowering the capillary pressure, in this case, can favor oil displacement in waterflooding, especially in conventional reservoirs. In these tests, fully oil-saturated rock plugs were flooded with the brine phase, displacing the oil down to residual oil saturation. Semi-permeable membranes were applied at the upstream and downstream of the plugs. Figure 8 shows the capillary pressure-water saturation curve for both systems 7 and 8. Curve 1 in Figure 8 is the one associated with system 7, and curve 2 is that of system 8. As shown in the figure, after addition of the surfactant to the brine phase i.e. the system whose brine phase contains Triton X100 (Figure 8, curve 2), a considerable reduction in capillary forces occurred, which contributed to the high IFT reduction capability of Triton X100. The residual oil saturation decreased for an amount of about 25% in system 8, compared with system 7, this implies that the surfactant can be a good candidate for ASP flooding yielding a considerable change in displacement efficiency. Here, a greater portion of the pore space could be invaded by the brine phase after surfactant addition due to the reduction of IFT and, consequently, reduction in the resistance of capillary forces. It is worth mentioning that these experiments do not imply a change in the direction of the capillary forces, which is assumed to be the consequence of wettability alteration.

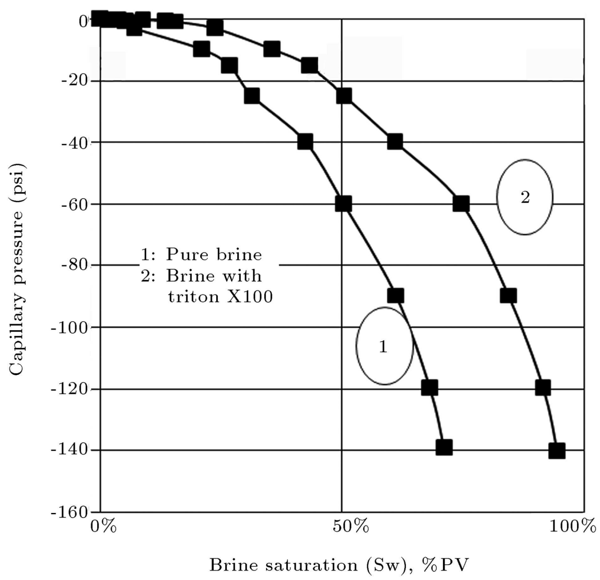

Figure 8. Drainage capillary pressure versus saturation, systems 7 and 8.

DISCUSSION

Imbibition experiments show that the initial wettability of the reservoir rock restored by aging the samples is oil-wet. This is verified by the rock not imbibing the pure reservoir brine spontaneously. Nevertheless, the capillary pressure experiments for system 5 (which is comprised of reservoir brine, oil and rock) from which the Amott-Harvey and USBM indices were calculated, demonstrate the initial oil-wet nature of the rock sample after aging. In such conditions, the negative matrix capillary forces resist the entrance of water as they are directed outwards, i.e. in the direction opposite to the favourable water movement. This shows that a favourable water exchange between fracture and matrix through spontaneous imbibition of reservoir brine might not occur in naturally fractured reservoirs. According to spontaneous imbibition experiments, C12DTAB could best improve spontaneous imbibition through changing the wettability of the matrix, thus, changing the capillary forces' direction assisting water to displace oil out of the matrix. This surfactant could favorably change the wettability of the rock. The USBM and Amott-Harvey indices in this case turned out to be positive. The rise at the beginning of the primary drainage curve in system 6, in comparison with system 5 (oil-wet state), conveys that an entry pressure was created as the rock became water-wet. The C12DTAB solution could shift the imbibition capillary pressure curve to the right causing a 26% rise in the spontaneous imbibition saturation end-point; the beginning point of forced imbibition. This also verifies the improvement in spontaneous imbibition that can, thereby, be brought about in fractured reservoirs. Triton X100 is highly capable of reducing the IFT between the oil and the brine used in the experiments, but it still has a lower rate of imbibition compared with that of C12DTAB. Triton X100 seems not to change the wettability of the rock sample. Thus, the recovery is explained to be governed by gravity forces, which overcome the capillary forces after Triton X100 weakens the capillary forces by IFT reduction.

CONCLUSIONS

- The cationic surfactant, C12DTAB, is the most efficient among the surfactants studied. It can improve spontaneous imbibition in the reservoir rock samples studied through wettability alteration.
- C12DTAB can favorably alter the wettability of the carbonate rock samples used in this study toward more water-wetness, according to Amott-Harvey and USBM test results. This is also consistent with spontaneous imbibition experiment results.
- There is fairly good agreement between Amott-Harvey and USBM indices in the oil-wet state for

the rocks studied. In the water-wet state, USBM turns out to show intermediate-wet.

- SDS did not make, relatively, a considerable improvement in spontaneous imbibitions, and turned out to be incapable of altering the wettability of the rock samples studied.
- Although Triton X100 seems not to change the wettability of the rocks studied in these experiments, it has the capability of decreasing the IFT down to ultra-low values. This surfactant recovers the oil under gravity forces by reduction of capillary forces rather than changing their direction. Its recovery might increase with increasing block height. Triton X100 can be a suitable surfactant for ASP flooding; regarding its IFT reduction ability and the shift it could make in capillary pressure curves.

NOMENCLATURE

ASP	Alkali-Surfactant-Polymer
C12DTAB	C12dodecyl trimetyl ammonium brimide
DDDC	dual drop dual crystal
g	gravitational acceleration
h	height
I_{AH}	Amott-Harvey wettability index
IFT	interfacial tension
I_o	oil wettability index
I_{UABM}	USBM index
I_w	water wettability index
k	permeability
N_B^{-1}	inverse bond number
OOIP	original oil in place
P_c	capillary pressure
PV	pore volume
SDS	sodium dodecyl sulfonate
S_{wir}	irreducible wetting phase saturation
USBM	U.S. Bureau of Mines
V_{ofc}	volume of oil displaced by forced imbibition
V_{osp}	volume of oil displaced by spontaneous imbibition
V_{wfc}	volume of oil displaced by forced drainage
V_{wsp}	volume of water displaced by spontaneous drainage
σ	interfacial tension
ϕ	porosity
$\Delta\rho$	density difference

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BIOGRAPHIS

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