

# Experimental Investigation on Spontaneous Imbibition of Surfactant Mixtures in Low Permeability Reservoirs

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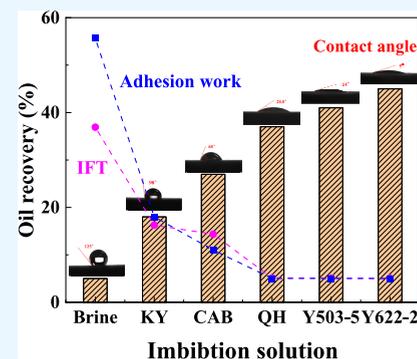
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**ABSTRACT:** Spontaneous imbibition of surfactants could efficiently enhance oil recovery in low permeability sandstone reservoirs. The majority of studies have considered the application of individual surfactants to alter wettability and reduce interfacial tension (IFT). However, a significant synergistic effect has been reported between different types of surfactants and between salts and surfactants. Therefore, this study systematically studied the capability of a binary surfactant mixture (anionic/nonionic) and a ternary surfactant mixture (anionic/nonionic/strong base–weak acid salt) in imbibition enhanced oil recovery (IEOR). The interfacial properties and the cores' wettability were explored by IFT and contact angle measurements, respectively. Subsequently, the imbibition performances of different types of surfactant solutions were discussed. The results suggested that the surfactants' potential to enhance oil recovery followed the order of ternary surfactant mixture > binary surfactant mixture > anionic > nonionic > amphoteric > polymer. The ternary surfactant mixture exhibited strong capacity to reverse the rock surface from oil-wet ( $125^\circ$ ) to strongly water-wet ( $3^\circ$ ), which was more significant than both binary surfactant mixtures and individual surfactants. In addition, the ternary surfactant mixture led to an ultralow IFT value of 0.0015 mN/m, achieving the highest imbibition efficiency (45% OOIP). This research puts forward some new ideas on the application of the synergistic effects of surfactants in IEO from low-permeability sandstone reservoirs.



## 1. INTRODUCTION

Low permeability oil reservoirs have become one of the most important energy sources. The combination of hydraulic fracturing treatments and horizontal well technology is widely used.<sup>1,2,4</sup> Due to small pore throats and cracks formed by hydraulic fracturing treatment, low permeability reservoirs show great potential for imbibition.<sup>3,5</sup> Since the 1950s, spontaneous imbibition has been studied and rapidly developed as a mechanism of oil recovery. It was an invasion process in which the wetting phase replaces the nonwetting phase through capillary force or gravity in porous media.<sup>6,7</sup>

According to the Young–Laplace equation, wettability and interfacial tension (IFT) are the primary factors of imbibition. Surfactants can both reduce the IFT and change the wettability.<sup>8,9</sup> The International Organization for Standardization divides surfactants into four types (anionic surfactants with anionic groups, cationic surfactants with cationic groups, amphoteric surfactants with both anionic and cationic groups, and nonionic surfactants with nonionic groups). It has been proposed that there are three main mechanisms for surfactants to reverse the wettability in sandstone reservoirs (ion-pair formation, surfactant adsorption, and micellar solubilization).<sup>10–12</sup> Feng and Liang found that anionic surfactants have much more potential than cationic surfactants to enhance oil recovery in sandstone reservoirs, and they confirmed that the displacement efficiency is slightly higher for anionic

surfactants compared to that for cationic surfactants.<sup>13</sup> Alvarez and Schechter believed that anionic surfactants enhanced oil recovery more efficiently and performed better in reversing wettability in sandstone reservoirs than nonionic–cationic surfactants.<sup>14</sup> Xu et al. studied the static imbibition efficiencies of different surfactants based on molecular structures. The authors reported that sulfate anionic surfactants could reverse the wettability of sandstone rocks more effectively compared to sulfonate surfactants and achieved the highest imbibition recovery. They pointed out that the addition of ethylene oxide (EO) groups into the surfactant structures could increase the hydrophilicity of surfactants and help alter wettability and improve the imbibition recovery.<sup>15</sup>

In recent years, the mixture of two surfactants could reduce IFT to a low value, which has attracted attention in the oil industry. Rosen and Hua conducted an experimental investigation, and the results demonstrated that the molecular interaction with nonionic surfactant groups in a binary

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surfactant mixture is in the order anion > cation > nonionic.<sup>16</sup> Das et al. also investigated the synergism between the two synthesized anionic surfactant and nonionic surfactant.<sup>17</sup> They pointed out that the critical micelle concentration (CMC) of the binary surfactant mixture is lower than those of individual surfactants. However, the binary surfactant mixture could reduce IFT to the same value and perform better under salinity tolerance, reducing the cost effectively.<sup>18</sup> Kesarwani et al. also indicated that a binary surfactant mixture had more outstanding performance than the individual surfactants and achieved an ultralow IFT value.<sup>19</sup>

Despite the numerous research on the wettability alteration ability of individual surfactants as well as a few reports on the capability of binary surfactant mixtures to reduce IFT, limited investigations have focused on the application of ternary surfactant mixtures. Thus, it is necessary to study the availability of the ternary surfactant mixture to achieve low IFT, wettability alteration, and increased oil recovery.

In this work, we aim to investigate the wettability reversal and IFT reduction capability of individual surfactants, binary surfactant mixtures, and ternary surfactant mixtures. In addition, the EOR abilities of different surfactant mixtures were studied by conducting spontaneous imbibition experiments, and the imbibition enhanced oil recovery (IEOR) mechanisms were further studied.

## 2. MATERIALS AND METHODS

**2.1. Materials.** Sandstone cores applied in this research were from the outcrop of the Yumen oilfield. One outcrop core sample was used, which was cut into many pieces to ensure similar lithology. The characteristics of cores are given in Table 1. Dehydrated crude oil from the Yumen oilfield was used. The

**Table 1. Characteristics of Cores Used in Tests**

parameters	value
diameter (cm)	2.5 ± 0.1
length (cm)	3.1 ± 0.1
porosity (%)	12.1 ± 0.1
permeability to air (mD)	0.8 ± 0.1

density of the crude oil is 0.83364 g/cm<sup>3</sup> and the viscosity is 43.1 mPa·s at a room temperature of 25 °C. The crude oil contained 11.7% wax, 45.9% alkanes, 8.0% asphaltene, 15.7% aromatics, and 19.9% nonhydrocarbons. Formation water was also from the Yumen oilfield. The name and nature of imbibition solution used in this study are given in Table 2. The five chemical agents used in the study include polymer KY (1 wt % polyacrylamide), amphoteric surfactant CAB (5 wt % LHSB), anionic surfactant QH (5 wt % petroleum sulfonate), binary surfactant mixture Y503–5 (2.5 wt % APG12 + 5 wt %

HABS), and ternary surfactant mixture Y622–2 (2 wt % APG-12 + 2 wt % petroleum sulfonate+0.8 wt % Na<sub>2</sub>CO<sub>3</sub>). The concentration was the optimum concentration of each chemical agents which was determined according to Sun's conclusions of the static imbibition experiment with oil sand.<sup>20</sup>

**2.2. Oil Saturation and Aging of Cores.** A vacuum pressure saturating device was used to saturate the cores with crude oil. First, the cores were placed in an oven at 100–105 °C for 8 h, and then, the cores' dry weight was weighed. Afterward, these cores and crude oil were put in different vacuum pressure tanks for different times, respectively, 12 h for cores and 30 min for oil. Furthermore, the vacuumized oil was released into the vacuum pressure tank with cores, and then, the pressure of the tank was increased to 20 MPa. After 12 h, the cores were taken out and immersed into the same oil at 60 °C for subsequent tests. The cores were aged seven days to establish an oil-wet state. Prior to imbibition, removing the cores out of the oil and wiping off superfluous oil covered the core surface. Next, the cores were weighed, the previous dry weight of these cores was compared, and the saturated oil volume was calculated.

**2.3. IFT Measurements.** The spinning drop interfacial tensiometer (TX-500C, CNG CO., USA) was used to measure the IFT between the imbibition solution and crude oil. The capillary tube was filled with the solutions of polymer and surfactants. Then, a drop of oil was injected into the center of the tube with a syringe. In all cases, the measurements of IFTs were conducted at a rotating speed of 3000 rpm. Images of the oil droplets were captured using a high-resolution video camera, and the IFTs were calculated according to the following eq 1.<sup>21</sup> During the tests, the data were collected by the device automatically once every 30 s until the values remained stable for half an hour, and the temperature was maintained at 60 °C, the same with that of the imbibition temperature.

$$\sigma = 1.2336(\rho_w - \rho_o)\omega^2\left(\frac{D}{n}\right)^3\frac{L}{fD} \cong 4$$

where  $\sigma$  is the interfacial tension between the solution and oil (mN/m),  $\rho_w$  is the density of solution (g/mL),  $\rho_o$  is the density of oil (g/mL),  $\omega$  is the rotating speed (rpm),  $L$  is the length of the oil drop (mm),  $D$  is the width of the oil drop (mm),  $n$  is the refractive index of the water phase, and  $f(L/D)$  is the correction factor.

**2.4. Contact Angle Measurements.** The JC 2000D2 contact angle measuring instrument was used (shown in Figure 1). Prior to contact angle (CA) measurement tests, the core samples were cut into small slices (2.5 × 0.3 cm) using a trimming machine, and the slices were sanded to make the surface flat and smooth. Then, the core slices were saturated and aged for seven days to establish an oil-wet state in order to

**Table 2. Imbibition Solution Used in the Study**

code	name	nature	concentration/ppm	molecular structure
KY	polyacrylamide	polymer	100	(C <sub>3</sub> H <sub>5</sub> NO) <sub>n</sub>
CAB	LHSB	amphoteric surfactant	500	C <sub>11</sub> H <sub>23</sub> CONH(CH <sub>2</sub> ) <sub>3</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> CH <sub>2</sub> CH(OH)CH <sub>2</sub> SO <sub>3</sub> <sup>-</sup>
QH	petroleum sulfonate	anionic surfactant	500	R
Y503–5	1 wt %APG12 + 5 wt %HABS	anionic + nonionic (binary surfactant mixture)	750	C <sub>12</sub> H <sub>25</sub> O(C <sub>6</sub> H <sub>11</sub> O <sub>5</sub> ) <sub>n</sub> , C <sub>12</sub> H <sub>25</sub> SO <sub>3</sub> NA
Y622–2	2 wt %APG12 + 2 wt % petroleum sulfonate + 0.8 wt % Na <sub>2</sub> CO <sub>3</sub>	anionic + nonionic + strong base-weak acid salt (ternary surfactant mixture)	600	C <sub>12</sub> H <sub>25</sub> O(C <sub>6</sub> H <sub>11</sub> O <sub>5</sub> ) <sub>n</sub> , R, Na <sub>2</sub> CO <sub>3</sub>

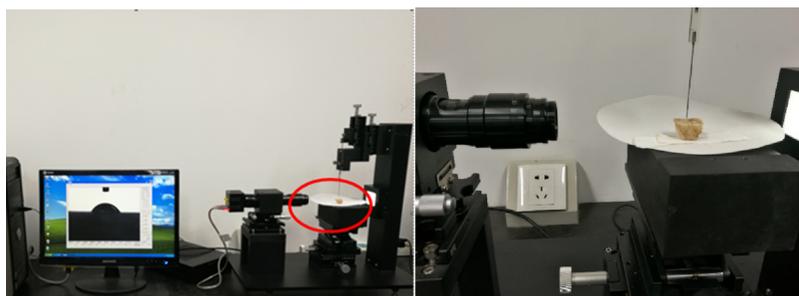


Figure 1. Attention Theta Lite static CA measuring instrument.

obtain oil-wet core slices. To begin with, the initial CA of the oil-wet core slices were measured. In addition, to measure the CA after imbibition tests, the oil-wet core slices were immersed in selected imbibition solutions at 60 °C for the same period of time. Afterward, a drop of solution was dropped onto the core sample surface using a syringe, and the final CA was measured from the profile of the droplet shape. In order to ensure the reliability of the results, each measurement was repeated at least three times.

**2.5. Spontaneous Imbibition Experiments.** Spontaneous imbibition tests were performed to study the efficiency of various chemical solutions in improving the oil recovery from porous media. To begin with, the cores were saturated with oil and aged for 7 days in order to ensure full saturation. Prior to imbibition, the chemical solutions and the saturated cores were all placed in an oven at 60 °C for over 1 h to preheat,<sup>22</sup> eliminating the thermal expansion effects caused by temperature change. The cores were then put in modified Amott cells (shown in Figure 2) filled with preheated chemical



Figure 2. Modified Amott cell used for imbibition tests.

solutions (formation water, polymer solution, and different surfactant solutions). Then, the cells were put in a water bath at 60 °C to maintain a constant temperature.<sup>15</sup> During the spontaneous imbibition process, the volume of the produced oil was recorded continuously in a graduated cylinder at the top of the modified Amott cell with an accuracy of  $\pm 0.01$  mL until the oil production ceased.

### 3. RESULTS AND DISCUSSION

**3.1. IFT between Crude Oil and Solutions (Formation Water, Polymer, and Different Surfactant Mixtures).** IFT is a crucial parameter affecting the mobilization of residual oil. According to Figure 3, it was found that surfactants could reduce the IFT effectively. Only one type of oil sample was used during all experiments. For amphoteric surfactant CAB, the IFT was 5.612 mN/m, while anionic surfactant QH

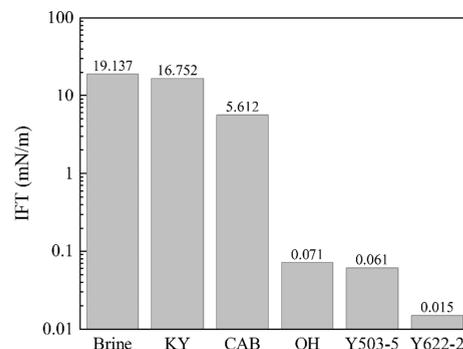


Figure 3. IFT results for different solutions at 60 °C.

lowered the IFT to 0.071 mN/m. Moreover, the addition of a nonionic surfactant into an anionic surfactant further decreased the IFT to 0.061 mN/m, thanks to the addition of the nonionic surfactant and reducing the electrostatic repulsion between the negatively charged anionic surfactant heads, resulting in a compact arrangement of surfactant mixture molecules.<sup>19,23</sup> In addition, in the presence of the strong base–weak acid salt, the ternary surfactant mixture (anionic/nonionic/strong base–weak acid salt) was found to be more effective in reducing the IFT (0.015 mN/m). The solution was alkaline due to the existence of the strong base–weak acid salt. In this system, acidic species in the crude oil diffused into the interface and reacted with the hydroxides in the chemical solution to form surface-active petroleum soaps,<sup>24–26</sup> which further decreased the IFT.

**3.2. CA in Formation Water, Polymer, and Different Surfactant Mixtures.** The wettability plays a leading role in the movement of the water–oil interface and the associated displacement of oil through porous media. To study the efficiency of surfactants to change the wettability, the CA of the initial oil-wet core slices and the CA of core slices after imbibition for different times were gauged. The initial CA of cores saturated with oil was close to 125°, indicating the strongly oil-wet condition. The results implied that the CA reduced with imbibition time to different values for different imbibition solutions (as shown in Figures 4 and 5). For example, the initial CA was 125° measured in formation water. It then decreased to 98° in the presence of KY solution, confirming that the polymer solution exhibited weak efficiency in reversing rock wettability. On the other hand, for each surfactant solution, the CA decreased to a lower value (68° in CAB solution, 30° in QH solution, 14° in Y503–5 solution, and 3°~5° in Y622–2 solution) within a short period of time. The capability of surfactants to decrease the CA followed the order ternary surfactant mixtures Y622–2 (anionic/nonionic/strong base–weak acid salt) > binary surfactant mixtures Y503–

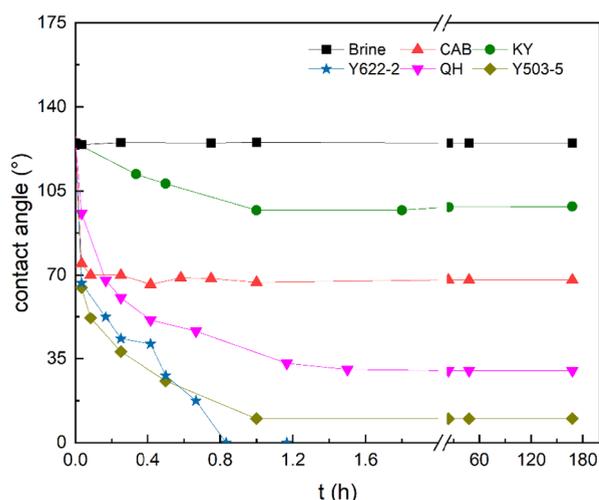


Figure 4. Dynamic oil CAs for different solutions at 60 °C.

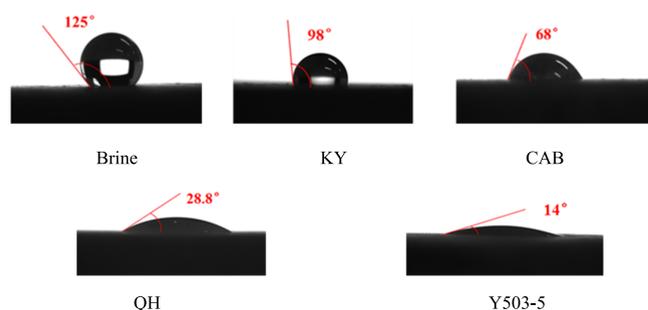


Figure 5. Equilibrium CAs for different solutions at 60 °C.

5 (anionic/nonionic) > anionic surfactant QH > amphoteric surfactant CAB.

The initial core samples in brine were negatively charged.<sup>15</sup> In anionic solution, the negatively charged heads of anionic surfactants adsorbed by hydrophobic interactions on both the oil-wetted core surface and the oil/water interface result in electrostatic repulsion that made the oil strip from the core surface easily,<sup>27,28</sup> restoring the water-wetness of the sandstone core surfaces. Therefore, the CA decreased in anionic surfactant solution QH. In addition, the CA in the binary surfactant mixture solution Y503–5 (anionic/nonionic) was found to be lower than that in the presence of QH or CAB (individual surfactant), indicating that there was synergism upon increasing water-wetness between the anionic surfactant and the nonionic surfactant. It could be due to the alternating distribution of nonionic surfactant molecules and anionic surfactant molecules, and the electrostatic repulsion between the anionic surfactant was reduced by electrostatic shielding.<sup>29–31</sup> This favored the anionic surfactant molecules by more closely adsorbing on the core surface and the interface between oil and water, enhancing the repulsion between the two interfaces. This resulted in the binary surfactant mixture effectively altering the oil-wetted rock surfaces to water-wetted surfaces. What is more, the introduction of the strong base-weak acid salt into the ternary surfactant mixture further reduced the CA. It was because the acidic components of the crude oil would interact with the hydroxide in solutions to form petroleum carboxylate or petroleum sulfonate, which is surface-active,<sup>24,25,32</sup> enhancing the wettability reversal ability of the binary surfactant mixtures.

**3.3. Imbibition Results Using Formation Water, Polymer, and Different Surfactant Mixtures.** The imbibition efficiencies of different chemical solutions (formation water, polymer KY, amphoteric CAB, anionic QH, binary surfactant mixtures Y503–5, and ternary surfactant mixtures Y622–2) were investigated at 60 °C. The results of imbibition are shown in Figure 6. All these results confirmed

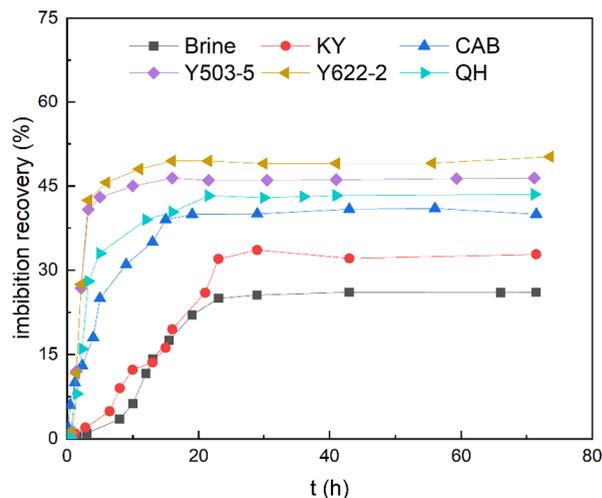


Figure 6. Imbibition results using six types of imbibition solutions at 60 °C.

that the ternary surfactant mixture (anionic/nonionic/strong base-weak acid salt) Y622–2 had the highest imbibition efficiency (45% OOIP), closely followed by binary surfactant mixtures (anionic/nonionic) Y503–5 (41% OOIP). Both the ternary and binary surfactant mixtures exhibited a higher recovery ability compared to the anionic QH surfactant (37% OOIP). However, both amphoteric CAB and polymer KY resulted in low imbibition recovery between 18 and 27% OOIP. This was much lower than that using the mixed solutions containing anionic surfactant, suggesting the poor efficiency of both amphoteric and polymer to enhance oil recovery in sandstone reservoirs.

According to research,<sup>15</sup> the lowering of IFT and the change of wettability would change the mechanisms of imbibition. On the one hand, the increase of water-wetness could lead to the increase of capillary pressure, strengthening the capillary pressure as a driving force in the process of spontaneous imbibition. On the other hand, the capillary pressure decreased with the lowering of IFT, resulting in gravity force being the main imbibition driving force.

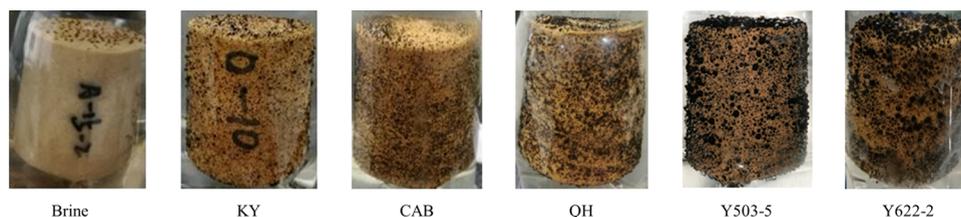
Surfactants would both change wettability and reduce IFT in the process of spontaneous imbibition. Adhesion work was found to be a critical indicator to discuss the imbibition mechanisms of various types of surfactants. Adhesion work is the work required to detach per area of an interface between the solid and liquid in the presence of a third phase.<sup>33</sup> In this study, the third phase was chemical solution, and the interface between the solid and liquid was the interface between the core and oil. The adhesion work between the core and oil could be described by eq 2:

$$W_{SL} = \sigma (1 - \cos\theta) \quad (2)$$

where  $W_{SL}$  is core-oil adhesion work;  $\sigma$  is the interfacial tension between oil and imbibition solution; and  $\theta$  is the

**Table 3. Imbibition Results Using Different Solutions and the Corresponding Adhesion Work at 60 °C**

imbibition solution	concentration (mg/L)	IFT (mN/m)	final CA (°)	$W_{SL}$	imbibition recovery (%)
formation water		19.137	125.9–127.8	30.39	5
polymer KY	0.01	6.752	98.5–99.7	7.81	18
amphoteric CAB	0.02	5.612	68.1–70.7	3.60	27
anionic QH	0.05	0.071	28.8–29.5	0.0089	37
binary surfactant mixtures Y503–5	0.08	0.061	14.2–16.8	0.0021	41
ternary surfactant mixtures Y622–2	0.06	0.015	5.6–6.1	0.000057	45

**Figure 7.** Core images of spontaneous imbibition with different solutions at 60 °C.

contact angle. It was shown that the adhesion work decreased with the decrease in  $\sigma$  or  $\theta$ . This indicated that the adhesion work is lower in more water-wet or lower IFT conditions. According to the definition of adhesion work, the reduction of adhesion work might be profitable to detach oil from the core surface more easily, bringing about higher oil displacement efficiencies.

Subsequently, the  $W_{SL}$  values were figured according to eq 2. The results are shown in Table 3. Compared with brine and polymer KY, the IFTs and CA values both decreased with the addition of surfactants, and this significantly decreased the adhesion work. The ternary surfactant mixture Y622–2 showed the strongest power to decrease both the CA and IFT, causing the greatest decrease of the adhesion work value, thus achieving the highest imbibition recovery. Furthermore, the efficiency of the binary surfactant mixture Y503–5 to decrease the adhesion work exceeded that of the anionic surfactant solution QH, which resulted in higher imbibition recovery. In comparison, the polymer and brine did not significantly change the wettability and IFT, and this resulted in high adhesion work, which led to the failure of the polymer solution and brine to achieve high recovery during the imbibition process.

The points made above are further supported by the core images at the end of imbibition (Figure 7). As shown in Figure 7, only few scattered oil was present on the top surface of the core sample used for brine imbibition. In comparison, a large amount of oil droplets emerged from all surfaces of the core sample in the presence of the Y622–2 solution, indicating that the binary surfactant solution was more easily imbibed into the core and that the oil was drained out. This was because the Y622–2 solution leads to the greatest reduction in adhesion work, and this further rationalizes the highest imbibition efficiency.

#### 4. CONCLUSIONS

This study systematically studied the efficiency and mechanism of individual surfactant, binary surfactant mixture, and ternary surfactant mixture to improve oil recovery with Yumen sandstone outcrops. The major conclusions are put in a nutshell as follows:

- (1) Surfactant solution effectively reduced the interfacial tension between the oil and solutions. The negatively

charged anionic surfactant molecules can form a monolayer at the interface, resulting in a higher capability in IFT reduction. Furthermore, the addition of strong base-weak acid salt would decrease the interfacial tension between oil and solutions more efficiently, reducing the IFT value from 0.071 to 0.015 mN/m.

- (2) Anionic surfactants had better wettability alteration capabilities compared to amphoteric surfactants and the polymer. Moreover, the synergism between the anionic surfactant and nonionic surfactant favors the reduction of the contact angle. In addition, the introduction of strong base-weak acid salt increased the repulsion between oil and rock, which resulted in stronger water-wetness more efficiently.
- (3) The ternary surfactant mixtures Y622–2 had the highest capability to both increase the water-wetness and decrease the IFT, causing the greatest decrease in adhesion work value and leading to the most significant imbibition recovery.

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## Notes

The authors declare no competing financial interest.

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