

Original Paper

Experimental study on the mechanism of adsorption-improved imbibition in oil-wet tight sandstone by a nonionic surfactant for enhanced oil recovery

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ABSTRACT

In recent years, production from tight oil reservoirs has increasingly supplemented production from conventional oil resources. Oil-wet formations account for a considerable proportion of tight oil reservoirs. Surfactant can change wettability and reduce interfacial tension, thus resulting in a better oil recovery. In this manuscript, a nonionic surfactant was introduced for tight oil-wet reservoirs. The oil recovery in the oil-wet sandstone due to spontaneous imbibition was 8.59% lower than that of the water-wet sandstone due to surfactant. The 0.1% surfactant solution corresponded to the highest imbibition recovery rate of 27.02% from the oil-wet sample. With the surfactant treatment, the treated core quickly changed from weakly oil-wet to weakly water-wet. The capillary force acted as the driving force and promoted imbibition. The optimal surfactant adsorption quantity in the oil-wet sandstone was observed in the sample at concentrations ranging from 0.1% to 0.3%, which also corresponded to the highest oil recovery. Analysis of the inverse Bond number N_B^{-1} suggested that the driving force was gravity for brine imbibition in the oil-wet cores and that it was capillary force for surfactant imbibition in the oil-wet cores. When the surfactant concentration was lower than the critical micelle concentration, the surfactant concentration was negatively correlated with the inverse Bond number and positively correlated with the oil recovery rate. When the surfactant concentration was higher than the critical micelle concentration, the oil recovery increased with a smaller interfacial tension. Nuclear magnetic resonance suggested that the movable pore and pore throat size in the oil-wet sample decreased from 0.363 μm in the untreated rock to 0.326 μm with the surfactant treatment, which indicated that the surfactant improved the flow capacity of the oil. The findings of this study can help to better understand the adsorption impact of surfactants on the characteristics of the oil/water and solid/liquid interfaces. The imbibition mechanism in oil-wet tight sandstone reservoirs was further revealed. These systematic approaches help to select appropriate surfactants for better recovery in oil-wet tight sandstone reservoirs through imbibition.

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1. Introduction

During long-term oilfield development, stable production becomes increasingly difficult. For the past decade, unconventional oil resources, such as tight oil, have received increasing attention as

alternative and supplemental oil resources (Kuuskraa et al., 2013; Wang et al., 2014; Zou et al., 2012). Tight oil reservoirs with large reserves have been discovered all around the world, such as those in the Bakken and Eagle Ford Formations in the United States and formations in the Ordos, Songliao, and Junggar Basins in China (Sun et al., 2019; Zhang et al., 2019).

Due to differences in reservoir lithology and sedimentary environment, among the numerous tight reservoirs, there are not only water-wet reservoirs (Yan et al., 2019) but also oil-wet reservoirs

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(Xiao et al., 2017; Yang et al., 2017) and mixed-wet reservoirs with various degrees of mixing (Xue et al., 2015). Wettability is one of the basic reservoir physical properties. Wettability has a great influence on the relative permeability of the oil and water phases, the distribution of residual oil, the flow capacity of crude oil, and the production characteristics in reservoirs (Li et al., 2017). For water-wet and neutral-wet reservoirs, water wettability is beneficial for water injection. For oil-wet tight reservoirs, it is different to recover crude oil due to the unfavorable wettability, so the oil recovery is usually very low (Tang et al., 2012).

When reservoir rock is oil-wet, crude oil adsorbs on the rock surface in a spread state. It is difficult for water flooding to mobilize the crude oil in small pores and pore throats within rocks, which leads to the retention of crude oil and the formation of residual oil (Hou et al., 2015; Mirchi et al., 2019). Compared with the pores in conventional reservoirs, the smaller pores and pore throats in tight reservoirs result in larger capillary forces. Spontaneous imbibition usually occurs in water-wet reservoirs. In oil-wet reservoirs, the capillary force in the oil-wet pores resists imbibition. Therefore, the water phase is not able to imbibe spontaneously into the formation under capillary force to displace crude oil. Therefore, spontaneous imbibition by water can rarely occur in oil-wet tight reservoirs (Morrow, 1990; Anderson, 1987).

Wettability alteration is an effective method to recovery oil from oil-wet tight reservoirs (Javanbakht and Goual, 2016; Li et al., 1998). An adsorption layer of surfactant can form on the solid surfaces of a porous medium to improve its wetting properties (Li, 2006). The molecular size of the surfactant is small, generally less than 5 nm. Compared with the tight reservoir pore throats and pores of tens of nanometers to tens of micrometers in size, surfactant molecules have no difficulty entering tight oil porous media. Surfactants also have favorable interface properties. They can reduce the interfacial tension between crude oil and water and maintain physical and chemical stability for a long time. Some surfactants have good salt tolerance. They are not sensitive to electrolytes (Jian et al., 2018; Zhang et al., 2018) and have good adaptability in reservoirs. Therefore, injecting surfactant into oil-wet tight reservoirs is an effective method of changing the wettability of tight reservoirs and further improve the effect of water flooding or water huff and puff development, as shown in Fig. 1.

There has been extensive research conducted on the use of surfactants to enhance oil recovery (Aminian and ZareNezhad, 2019). For water-wet tight reservoirs, Dai et al. (2019) used low-field nuclear magnetic resonance (LF-NMR) technology to study dynamic imbibition in water-wet sandstone with surfactants. The

behavior of water phase entry into pores of different sizes during imbibition was defined. The effect of the water flow rate in fractures on the oil saturation in pores was also revealed. For oil-wet tight reservoirs, Sheng (2017) discussed the effects of various factors on imbibition through numerical simulation and theoretical analysis. The study found that among the factors considered, initial wettability was the most important factor for imbibition. The wettability alteration by surfactant was very important for imbibition in oil-wet tight reservoirs. Low interfacial tension cannot guarantee high oil recovery by imbibition. At a certain range, the increase in oil/surfactant interfacial tension helped to increase the oil recovery. Lu et al. (2014) changed the wettability of carbonate reservoirs from oil-wet to water-wet with a surfactant, making the recovery rate reach 32% by spontaneous imbibition. Ahmadi et al. (2020) discovered that cationic surfactants displayed higher oil recovery than anionic surfactants. However, the sandstone surface is usually negatively charged. The adsorption loss of cationic surfactant is very high (Yao et al., 2008). At the same time, due to the complex mineral composition of tight oil reservoirs, anionic surfactants are also adsorbed on clay minerals with positive charges at their edges (Song et al., 2017). Therefore, in this study, a nonionic surfactant was used to reduce the adsorption capacity of the surfactant and improve the performance of surfactant imbibition.

Shirazi et al. (2020) found that a higher wettability alteration led to a higher oil recovery during the spontaneous imbibition process. LF-NMR can be used for fluid flow characterization in porous media (Guo et al., 2020). However, the interfacial properties caused by surfactant adsorption, its impact on fluid flow characterization in porous media, and the oil recovery mechanism in oil-wet tight reservoirs are not clear.

In this study, the experimental information is presented first. Then, the results are discussed, followed by the main achievements. With the nonionic surfactant of the best oil recovery during imbibition from screening experiments, the mechanism of surfactant imbibition in oil-wet tight sandstone samples was studied through systematic experiments in terms of multiple factors, including the surfactant adsorption, solid-liquid wettability, oil-surfactant interfacial tension, and driving force analyzed from the inverse Bond number, and all of them were correlated closely. During the experimental work, first, water-wet tight outcropped sandstone was treated to become oil-wet. Through spontaneous imbibition experiments, the imbibition characteristics of the water-wet and oil-wet cores were compared, and the influence of surfactant concentration on the imbibition rate and recovery in the oil-wet sandstone was analyzed. Next, the effects of surfactant on the

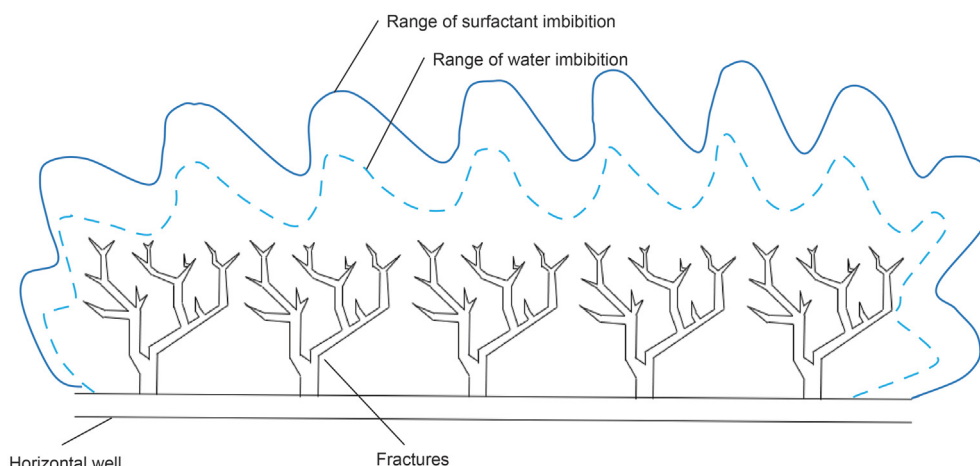


Fig. 1. Sketch of surfactant-enhanced oil recovery in oil-wet tight sandstone reservoirs.

characteristics of the solid/liquid interface and oil/surfactant interface were measured. Combined with the LF-NMR method, the cutoff value of transverse relaxation time, $T_{2 \text{ cutoff}}$ corresponding to different core sample states were determined. Then, the driving force for imbibition in oil-wet tight sandstone was analyzed. This study provides a theoretical basis for surfactant imbibition and enhanced oil recovery in oil-wet tight reservoirs.

2. Experimental

2.1. Materials

In this study, outcropped sandstone cores and quartz plates were used. The tight core samples were from the Middle Jurassic upper Shaxi Temple Formation in Sichuan, China. The permeability range of the core was 0.26–0.31 mD, and the porosity was 12.97%–14.06%.

For better observation of the oil phase, dodecane (Macklin Biochemical Co., Ltd., Shanghai, China) mixed with 2% crude oil was used as the oil phase. Crude oil has the best practicality meanings indeed. However, the composition of each crude oil is different, the polar components have a great influence on the interfacial tension (IFT) and wettability, and the visualization effect is not good in this study. However, 98% dodecane helps to provide trustworthy result to reveal the mechanism of surfactant impact on imbibition in oil-wet reservoirs. Alkyl polyglucoside was used as a nonionic surfactant (Pharmaceutical Group Chemical Reagents Co., Ltd, Shanghai, China). Other reagents, including methyl silicone oil, petroleum ether, anhydrous ethanol, NaCl, NaOH, NaHCO₃, CaCl₂, and MgCl₂·6H₂O, were all AR grade (Pharmaceutical Group Chemical Reagents Co., Ltd, Shanghai, China). Different solutions were prepared with deionized water. The composition of the formation water used in this study was adopted from the Xinjiang Jimusar Formation, with a total salinity of 23003 mg/L. The specific ionic composition is shown in Table 1.

2.2. Apparatus

A Helium porosimeter (PMI-100, Yineng, Beijing, China) was used to measure the effective porosity. A gas permeameter (ULP-613, Beijing Yineng, Beijing, China) was used to measure the Klinkenberg permeability of the cores. A goniometer (JC2000D, Powereach, Shanghai, China) was used to measure the contact angles of the oil droplets on the core chip surfaces. A high-speed centrifuge (TG18G; Xiangyi, Hunan, China) was used to measure the capillary force. An ultraviolet–visible spectrophotometer (UV5, Mettler Toledo, Greifensee, Switzerland) was used to measure the concentration of the surfactant solution. An interfacial tensiometer (TX500C, Kino, Boston, USA) was used to measure the oil/surfactant interfacial tension. An Abbe refractometer (WXZ-1, Shanghai Optical Instrument Factory, Shanghai, China) was used to measure the refractive index of the solution. A low-field nuclear magnetic resonance instrument (MacroMR12-150H-1, Niumag, Suzhou, China) was used to obtain the oil signal in the tight samples.

2.3. Procedure

The experimental procedures are illustrated in Fig. 2, and details of each step can be found in the following parts.

Table 1
Ionic composition of formation water.

Ion	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	HCO ₃ ⁻	SO ₄ ²⁻	Cl ⁻
Content, mg/L	8563	94	209	52	1486	1800	10799

2.3.1. Wettability alteration of tight sandstone

The methyl silicon oil/petroleum ether system was used to treat the sandstone cores and quartz plates, respectively. The specific steps were as follows (taking a core sample as an example):

- (1) After the cores were cut to the designed sizes, the sample ends were soaked in brine until they were clean. The purpose of this step was to prevent the cuttings from blocking the pore throat at the core ends. Then, they were dried in an oven at 110 °C for 24 h and stored in a drying box.
- (2) The dried cores were put into a petroleum ether solution with 3% (by volume) methyl silicone oil.
- (3) After 24 h, the cores were removed and placed in an oven at 175 °C for 24 h. Then, they were removed and dried for later use.

The method of the wettability alteration of the quartz plates is very similar to the above steps.

2.3.2. Core surface wettability determination

After the sandstone core chips (or quartz plates) were saturated with oil under a pressure of 20 MPa, they were placed horizontally in the sample cell containing the surfactant solution. Four microliters of oil drops were placed under the core chips through a curved syringe needle. A goniometer was used to measure the contact angles of the oil droplets on the core chip surfaces, and the angles between the tangent line of the oil/surfactant interface at the three-phase contact point and the water-solid line were analyzed to determine the surface wettability of the cores. A schematic diagram of the surface contact angle measurement of the sandstone core chips or quartz plates is shown in Fig. 3.

2.3.3. Internal wettability determination for core samples

The mineral composition of reservoir rocks is complex, and the internal wettability and surface wettability of porous media are different. The internal wettability of the treated sandstone core can be measured by the U.S. Bureau of Mines (USBM) method. The wetting phase inside the porous medium and the nonwetting phase around it display different flowing capacities under centrifugal force. The work done by the wetting phase to displace the nonwetting phase is smaller than that in the opposite case. The work done by displacement corresponds to the area enclosed by the centrifugal capillary pressure curve and saturation coordinate axis.

During the experiment, the core was first vacuumed and saturated with oil under pressure. Then, it was put into a centrifuge tube filled with water and centrifuged at 2000, 4000, 6000, 8000, and 10000 rpm for 1 h, respectively, to simulate the process of water displacing oil in the core. Then, the core was put into a centrifuge tube filled with oil, and the core was centrifuged at the same speed for 1 h to simulate the process of oil displacing water. The mass of the core was weighed, and the water saturation of the core was calculated at each speed. For the untreated sample, the wetting phase was water since the untreated rock is water-wet. For the treated sample, the wetting phase was oil since the rock was treated to be oil-wet. The centrifugal force and water saturation in the core were used to draw a capillary force curve. By comparing the areas enclosed by the capillary pressure curve and the water saturation axis during oil displacing water and water displacing oil, respectively, the internal wettability of the core can be estimated.

For both the treated and untreated samples, the native fluid is oil. During the 1st centrifuge, as the water displaces the oil at different speeds, the invading phase is water, which corresponds to an imbibition process for the untreated water-wet sample and a drainage process for the treated oil-wet sample. During the 2nd

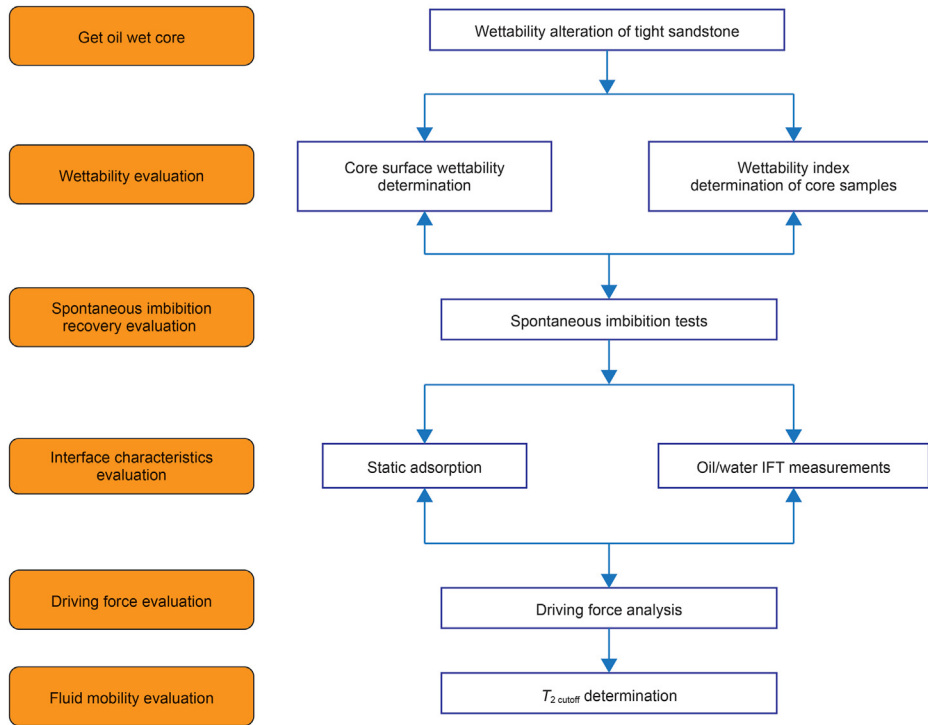


Fig. 2. Flowchart of the procedure used in this study.

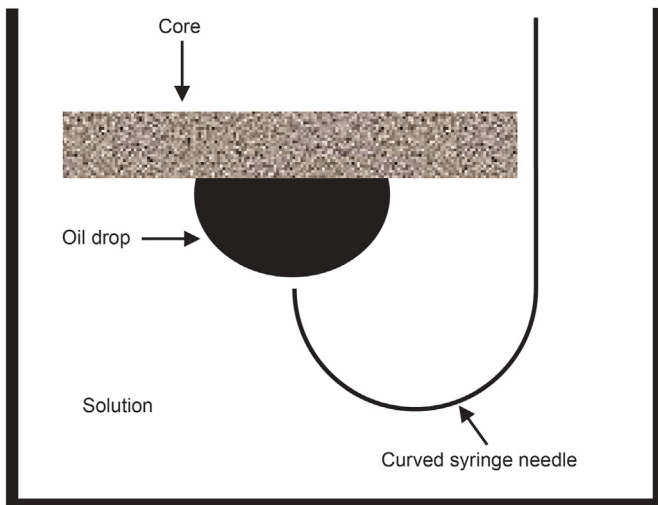


Fig. 3. Schematic diagram of the contact angle measurement.

centrifuge, as the oil displaces the water at different speeds, the invading phase is oil. This is the drainage process for the untreated water-wet sample and the imbibition process for the treated oil-wet sample.

The difference in centrifugal pressures of the two-phase fluid at different rates equals the capillary pressure. The capillary pressure during the centrifugation process was calculated according to the industry standard: SY/T5346-2005 (2005), which is as follows:

$$p_{ci} = 1.097 \times 10^{-9} \Delta\rho L \left(R_e - \frac{L}{2} \right) n^2 \quad (1)$$

where P_{ci} is the capillary pressure, MPa; $\Delta\rho$ is the density difference of two fluids, g/cm³; L is the length of the sample, cm; R_e is the

revolving radius, cm; and n is the rotation rate of the centrifuge, rpm.

Areas A_1 and A_2 enclosed by the capillary pressure curve and the water saturation axis during the process of oil displacing water and water displacing oil were calculated, and the wetting index was calculated according to the industry standard, SY/T5153-2007 (2007), which is as follows:

$$I_w = \lg \frac{A_1}{A_2} \quad (2)$$

where A_1 is the area enclosed by the capillary pressure curve and the water saturation axis during the process of oil displacing water; A_2 is the area enclosed by the capillary pressure curve and the water saturation axis during the process of water displacing oil; and I_w is the wettability index.

The USBM wettability estimation method is shown in Table 2.

2.3.4. Spontaneous imbibition tests

Spontaneous imbibition tests were conducted in Amott cells. The volumes of oil expelled from the core by surfactant at different times were directly read through the scale line on the Amott cells. The volume was recorded continuously over time. The spontaneous imbibition recovery and imbibition rate of the core at different times were calculated. Fig. 4 is the flowchart of the imbibition experiment, and the specific experimental steps were as follows:

- (1) The 10-cm long core was cut into 4 sections, and then the core powder and other debris on the core surface were fully

Table 2
Internal wettability estimation by USBM method.

I_w	$I_w < 0$	$I_w = 0$	$I_w > 0$
Wettability	Oil-wet	Intermediate-wet	Water-wet

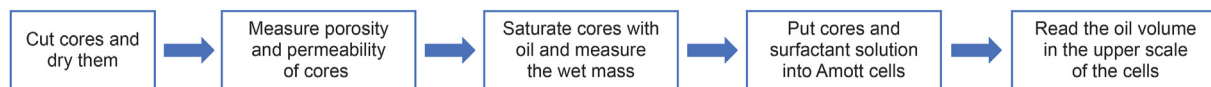


Fig. 4. Flowchart of the imbibition experiment.

cleaned with an ultrasonic cleaner. The clean cores were put in a 100 °C oven and dried for 24 h.

- (2) When the temperature of the dried cores was lower than room temperature, the dry mass of the cores was weighed. A helium porosimeter and gas permeameter were used to measure the porosity and Klinkenberg permeability of the cores.
- (3) The cores were saturated with oil after vacuum and pressurized at 20 MPa, and the wet mass was measured. Then, the well-saturated cores were immersed in oil for later experiments.
- (4) Simulated formation water was prepared and then used to prepare surfactant solutions with different concentrations. The surfactant solution and cores were heated to the experimental temperature (70 °C) in a water bath. Then, a magnetic stir bar and the cores were put into the lower part of the Amott cells, and the surfactant solution was added to the upper scale of the cells.
- (5) When the cells were put back in the water bath (70 °C), a timer was started. Through reading the oil volume in the upper scale of each cell, the imbibition recovery was calculated with a measurement accuracy of 0.005 mL. The volume was read every 5 min for the first 2 h and then every 30 min, and then the reading interval was determined according to the recovery rate. The parameters of the cores and imbibition conditions are shown in Table 3.

2.3.5. Static adsorption

The static adsorption capacity of surfactant refers to the amount of surfactant adsorbed on a certain mass of core powder under static conditions (Liang et al., 2008). The static adsorption capacities of surfactants on the surface of the original sandstone and the treated sandstone powders were measured.

First, the original and treated sandstone cores were crushed, respectively. The powders of 170–200 mesh were screened for static adsorption measurements. Core powder (1.0 g) was put into a 50 mL centrifuge cup, and then surfactant solutions of different concentrations were added with a solid/liquid mass ratio of 1:20, respectively. The centrifuge cups were placed on a magnetic stirrer for 24 h and then moved into a tabletop high-speed centrifuge. After centrifugation for half an hour at 7000 rpm, the supernatants were taken from the samples, and their equilibrium concentrations were measured by an ultraviolet–visible spectrophotometer. The equilibrium concentration refers to the concentration when adsorption reaches a very low rate or no longer occurs. When the

concentration of surfactant in the supernatant changed very little, equilibrium was considered to be reached. The static adsorption capacity was calculated by Eq. (3) (Zhang et al., 2016):

$$\Gamma = (c_0 - c)m_s/m_r \quad (3)$$

where Γ is the static adsorption capacity, mg/g; m_s is the mass of the surfactant solution, g; c_0 is the initial concentration of the surfactant solution, mg/g; c is the equilibrium concentration of the surfactant solution, mg/g; and m_r is the mass of the adsorption medium, g, which is the mass of the rock powder here.

2.3.6. Oil/surfactant IFT measurements

Surfactant solutions of different concentrations were prepared with simulated formation water, and an interfacial tension meter was used to measure the oil/surfactant IFT between the surfactant solutions and oil. According to the industry standard SY/T5370-1999 (1999), the specific steps were as follows:

- (1) Surfactant solutions of different mass concentrations were prepared. Subsequently, the oil and surfactant solution densities were measured, and the refractive index of the solution was measured with an Abbe refractometer.
- (2) The sample tube was filled with surfactant solution through a needle, and then a small oil drop was injected and maintained in the middle of the sample tube.
- (3) The speed of the sample tube was set at 6000 rpm. When the speed was stable, the length and height of the oil drop were measured by the build-in software. Then, the interfacial tension values were determined.
- (4) The length and height of the oil drops were measured every 2 min until the interfacial tension value did not change.

2.3.7. T_2 cutoff determination

The movable pore throat diameter of the core corresponds to a pore throat diameter below which fluid cannot flow within it. That is, such pore space does not contribute to the overall flow capacity of the core (Li et al., 2012). In the original sandstones and the treated sandstones, the T_2 cutoff could be measured by low-field nuclear magnetic resonance and high-speed centrifugation technology, and then the influence of wettability changes on the flow capacity of the core was evaluated.

First, the original sandstone and the treated sandstone were saturated with oil, respectively. Then, the relation of the hydrogen signal amplitude in the oil and transverse relaxation time, T_2 , was

Table 3
Parameters of the cores and experimental conditions.

Core No.	Length, mm	Diameter, mm	Porosity, %	Klinkenberg permeability, mD	Treatment	Surfactant concentration, %
L40-2	24.79	25.30	14.03	0.27	Yes	0
L40-3	24.43	25.30	14.06	0.29	Yes	0.03
L40-1-1	24.26	25.30	12.97	0.26	No	0.1
L40-1-2	24.51	25.30	12.99	0.28	No	0.1
L40-4-1	24.22	25.31	13.06	0.31	Yes	0.1
L40-4-2	24.78	25.31	13.08	0.30	Yes	0.1
L40-5	24.58	25.30	14.01	0.26	Yes	0.3
L40-6	25.46	25.30	14.05	0.27	Yes	0.5

obtained by nuclear magnetic resonance spectrum. After that, a surfactant solution was prepared with a 40% manganese chloride aqueous solution to shield the signal of the water phase. The cores were centrifuged at 2000, 4000, 6000, 8000, and 10000 rpm for 1 h each, respectively. After every centrifugation, the core mass was measured to calculate the water saturation. Simultaneously, a nuclear magnetic resonance instrument was used to obtain the oil signal in the porous medium of the core. Eq. (1) was used to calculate the capillary force at different centrifugation rates. The sum of the T_2 spectrum values of the core after centrifugation was calculated. Then, a point on the T_2 spectrum of the core saturated with oil before centrifugation was found. The sum of the T_2 spectrum values to the left of this point was equal to the sum of the T_2 spectrum values of the core after centrifugation. The abscissa value of the point was the $T_{2 \text{ cutoff}}$.

Comparing the pore throat size distribution obtained by mercury injection and the T_2 spectrum of the core saturated with oil, the $T_{2 \text{ cutoff}}$ could be converted to the lower limit of the movable pore throat diameter of the core (Wang et al., 2010).

3. Results

3.1. Wettability alteration of water-wet tight sandstone

After the quartz plate and the original sandstone core chip were dried, they were immersed in simulated formation water, and the contact angles of oil on the lower surface were measured using a goniometer. The results are shown in Fig. 5.

Fig. 5 shows that the original quartz plate and the original core were both water-wet. The contact angles of the quartz plate and the original sandstone core chip were 54.2° and 46.6° , respectively.

The dry quartz plate and the original sandstone core chip were aged in oil for 48 h, and the wettability of the oil on the surfaces of

the two was measured, respectively. The results are shown in Fig. 6.

Compared with the quartz plate and core chip before treatment, wetting alteration occurred on the surfaces of the samples. After the chips were treated with the mixed solution of methyl silicone oil and petroleum ether, the surface wettability of the quartz plate was more obvious. The contact angle of the quartz plate surface was 138.3° , which corresponded to an oleophilic surface. The contact angle of the core chip surface was 107.4° , which corresponded to a weakly oil-wet surface.

The wettability indexes of the sandstone core before and after treatment with the methyl silicone oil-petroleum ether system were measured by the USBM method. The capillary force curves of water displacing oil and oil displacing water are shown in Fig. 7.

After the treatment, the wettability index of the sandstone core was -0.96 , which indicates that the internal wettability of this porous medium was oil-wet. During the processes of water displacing oil and oil displacing water, the area enclosed by the water flooding curve and the water saturation axis is larger than the area enclosed by the oil flooding curve and the water saturation axis. This means that the work done by water displacing oil is greater than the work done by oil displacing water. Therefore, after the treatment of the methyl silicone oil-petroleum ether system, the originally water-wet sandstone core became oil wet.

X-ray diffraction analysis of the core shows that the quartz and feldspar content accounted for 78% of the total rock minerals, so the original core was water-wet. During the treatment with methyl silicone oil-petroleum ether, the components of the cyclic polydimethylsilyl, pentane, and hexane were adsorbed on the quartz and feldspar surfaces, thus changing the mineral surface wettability of the quartz and feldspar. Therefore, the measurement of the core surface contact angle indicates that it is weakly oil-wet, while the porous medium inside the core measured by the USBM method is strongly oil-wet.

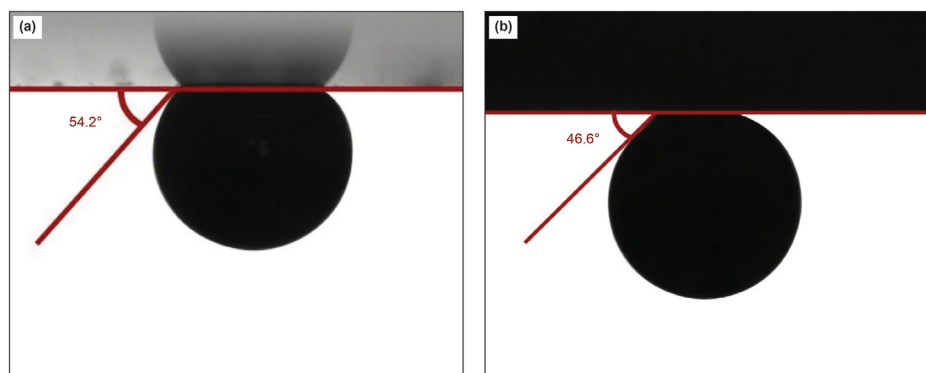


Fig. 5. Wettability of the original quartz plate and core chip. (a) Original quartz plate. (b) Original core chip.

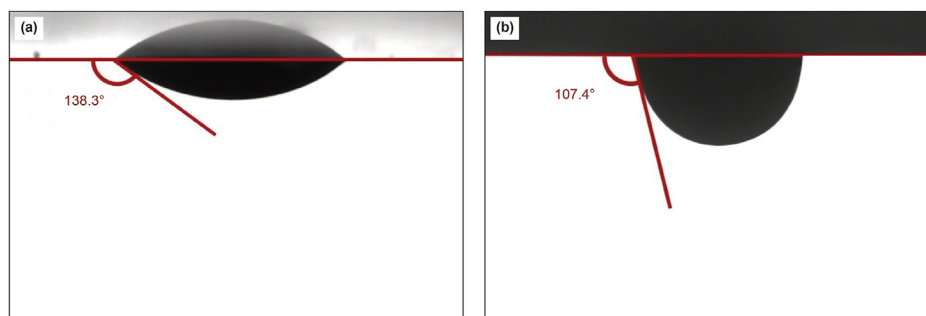


Fig. 6. Wettability of the treated quartz plate and sandstone core chip. (a) Treated quartz plate. (b) Treated core chip.

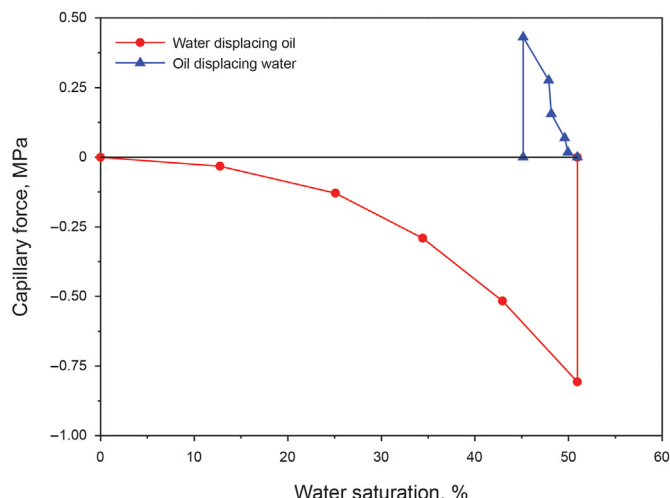


Fig. 7. Capillary force curves of the treated sandstone core sample.

3.2. Spontaneous imbibition behavior of surfactant in oil-wet tight sandstone

A surfactant solution with a mass concentration of 0.1% was prepared with simulated formation water, and the imbibition of the solution in the water-wet core and oil-wet core was observed at 70 °C. The oil recovery ratio by spontaneous imbibition with time is shown in Fig. 8.

The ratio of the volume of the displaced crude oil to the volume of the original crude oil in the core is defined as the oil recovery ratio by spontaneous imbibition. The oil recovery in the oil-wet sandstone by spontaneous imbibition was 8.59% lower than that of the water-wet sandstone with surfactant. When the oil wettability of the core was enhanced, on the one hand, the imbibition of surfactant was more difficult, and the wettability alteration was caused by only the adsorption of surfactant molecules. On the other hand, the relative permeability of the oil phase decreased, which made the flow resistance greater for the oil phase.

The curve of the oil recovery ratio by spontaneous imbibition with time can be divided into two stages. In the early stage, the imbibition recovery rate increases very fast over time. After a

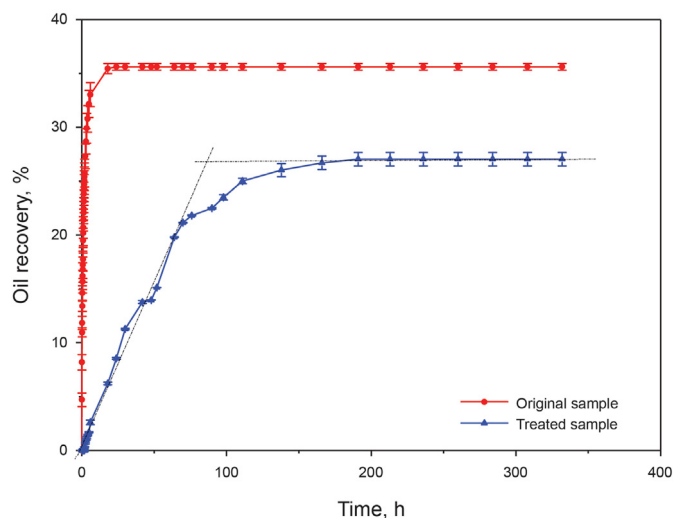


Fig. 8. Spontaneous imbibition recovery of cores with different wettabilities under surfactant solutions.

period of time, the imbibition recovery rate decreases with time, maintaining a small rate until it stabilizes (Sun et al., 2015). However, the imbibition rate of the oil-wet core was smaller than that of the water-wet core with surfactant, and the duration of the first imbibition stage was 15.23 times longer than that in the water-wet core (85.74 h for the treated sample, and 5.63 h for the original sample). Previously, this phenomenon was also observed in shale imbibition (Lakatos et al., 2011; Roychaudhuri et al., 2011, 2013). The significant difference in the imbibition rate could be explained by capillary force (Tiab and Donaldson, 2015). In the tight sandstone core, the capillary force in the small pore throats was larger, so the water phase was first imbibed into the small pores and throats, and the oil phase in the core was replaced by the imbibed water phase. Therefore, the imbibition recovery rate at the beginning of imbibition was higher. As the water saturation of the core continued to increase, the oil/surfactant interface gradually moved from the small pore throats to the large pore throats, and the capillary force decreased gradually, resulting in a significant slowdown of the imbibition rate. This phenomenon was more obvious in porous media with lower permeabilities (Ford et al., 1988).

Surfactant solutions of different mass concentrations were prepared with the simulated formation water, and the water imbibition and oil expulsion in the treated oil-wet sandstone were observed at 70 °C. The spontaneous imbibition recovery with time is shown in Fig. 9.

As shown in Fig. 9, with increasing the surfactant concentration, the spontaneous imbibition recovery in the oil-wet core first increased and then decreased. Surfactant (0.1%) displays the highest imbibition recovery rate, 27.02%. At first, the spontaneous imbibition recovery rate of oil-wet cores increased at a fast rate over a long period of time. After approximately 70 h, the rate of imbibition decreased slowly. With the progress of imbibition, the spontaneous imbibition recovery rate basically stabilized after 236 h.

3.3. Solid/liquid surface characteristics of the core samples and surfactant solution

3.3.1. Surface wettability

Oil-wet core chips were put into surfactant solutions with different concentrations to measure the surface contact angles of the cores, respectively. The results are shown in Fig. 10.

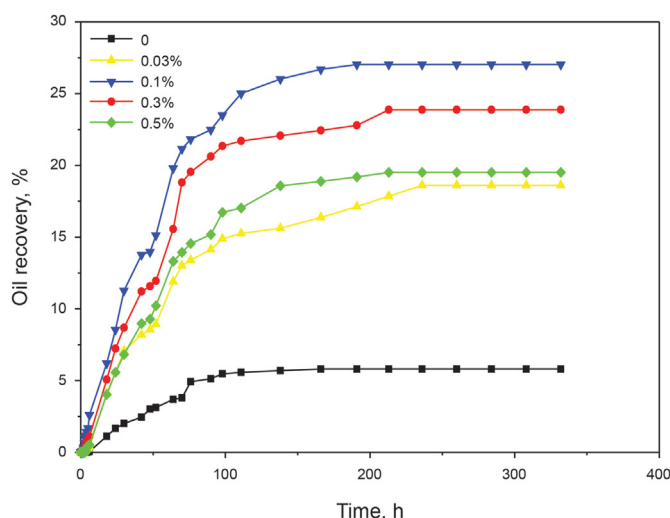


Fig. 9. Spontaneous imbibition of surfactant with different concentrations in oil-wet cores.

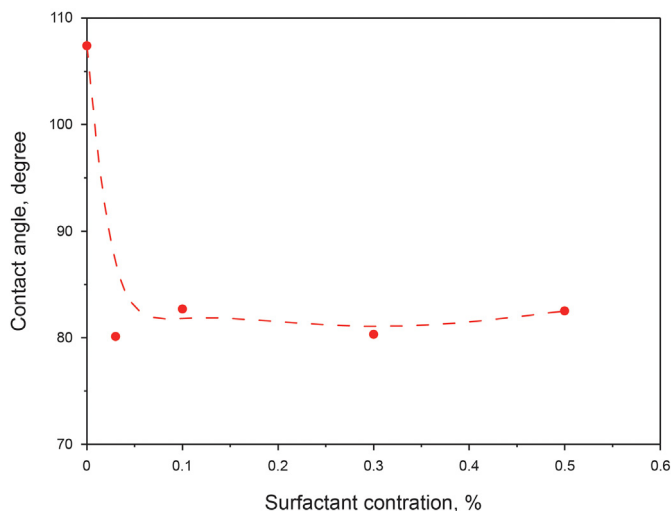


Fig. 10. Contact angles of oil-wet cores in surfactant solutions with different concentrations.

In the surfactant solutions, the surface wettability of the weakly oil-wet core (contact angle 107.4°) becomes weakly water-wet (80.1°–82.7°) very quickly. The concentration (0.03%–0.5%) of the surfactant solution has very little effect on the contact angle. Since surfactant molecules have hydrophilic head groups and hydrophobic tail chains, when the core was put into the surfactant solution, the tail chains of the surfactant molecules adsorbed onto the wall surface of the oil-wet pore throats, and the hydrophilic head groups were oriented perpendicular to the wall surface of the pore throats, causing the core surface to become hydrophilic.

The addition of surfactant changes the wettability of the pore walls in the pores and pore throats of the core. The capillary force direction is reversed, and the imbibition of the water phase is promoted, as shown in Fig. 11.

For oil-wet cores, the capillary force in the process of water imbibition results in a resistance to oil drainage and is not beneficial for imbibition. However, under the action of surfactant, the wettability of the core is reversed, and it becomes weakly water-wet. The capillary force, which is the main force for imbibition, is in the same direction as the imbibition. The capillary force becomes a positive force that drives the imbibition of the water phase, which promotes the imbibition process.

3.3.2. Static adsorption of surfactant in core samples

The change in surface wettability is due to the adsorption of surfactant on the solid surface of the core sample. The adsorption of surfactant changes the wettability of the core surface from oil-wet to water-wet. Therefore, static adsorption experiments of surfactant and core powder were carried out.

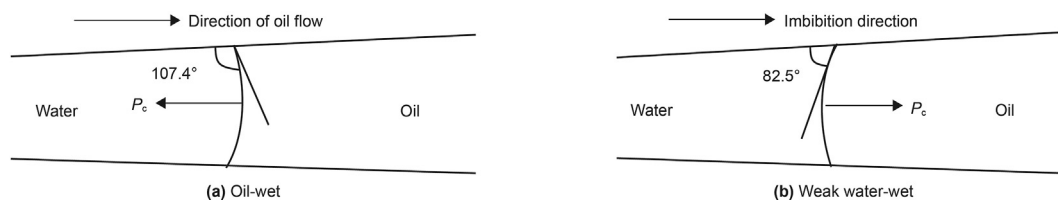


Fig. 11. Schematic diagram of the capillary force in the pore throats during imbibition. (a) Oil-wet. (b) Weak water-wet.

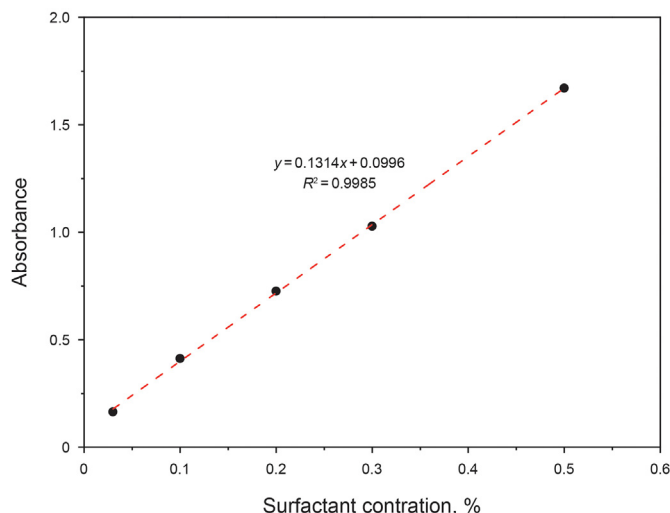


Fig. 12. Standard absorbance curve of the surfactant solutions.

(1) Establishment of a standard curve

Twenty milliliters of surfactant solutions with mass fractions of 0.03%, 0.1%, 0.2%, 0.3% and 0.5% were prepared with simulated formation water, respectively. Using simulated formation water as the reference solution, the absorbance of standard surfactant solutions with different concentrations was measured at a wavelength of 220 nm. Fig. 12 shows the relationship between the surfactant solution concentration and absorbance.

The ultraviolet absorption method was used to measure the content of surfactant in the aqueous solution. When the concentration was 0.03%–0.5%, the concentration was linearly related to the ultraviolet absorption.

(2) Static adsorption of surfactant in core samples

The core powder was first treated in a methyl silicone oil-petroleum ether system and then soaked in surfactant solutions with different mass concentrations, and the adsorption quantity was measured. The results are shown in Fig. 13.

The adsorption quantity of the surfactant on the original and treated sandstone powder increases rapidly with increasing concentration and reaches adsorption equilibrium when the concentration is 0.3%. When the surfactant concentration is small, the adsorption quantity is also small. The adsorption quantity increases sharply with increasing surfactant concentration, resulting in wetting reversal of the treated oil-wet sandstone, and the surface contact angle becomes weakly water-wet. As the surfactant concentration increases further, the surfactant continues to adsorb on the core surface, and the adsorption tends to be saturated after the first adsorption layer is formed. After that, the changes in the adsorption quantity and surface contact angle are small.

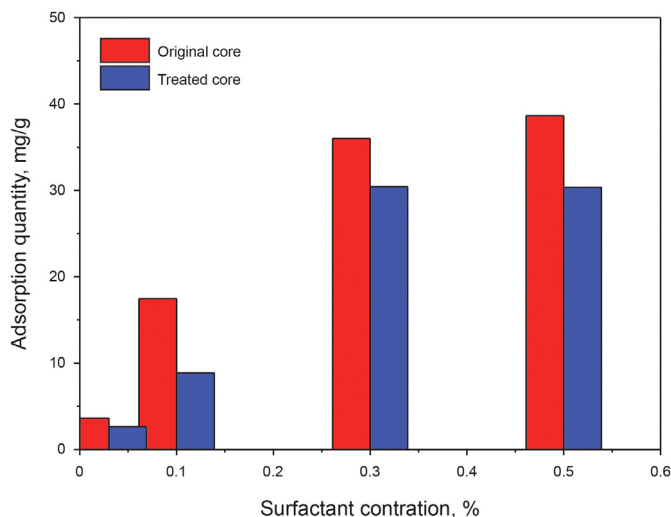


Fig. 13. Adsorption quantity of surfactant on core powders.

At the same surfactant concentration, the adsorption quantity of the surfactant on the surface of the original water-wet sandstone powder is greater than that on the treated oil-wet sandstone powder. The equilibrium adsorption quantities on the surface of the original sandstone and treated oil-wet sandstone powder are 36.1 mg/g and 30.4 mg/g, respectively. When the wettability of the sandstone changes from water-wet to oil-wet, the number of negative charges on the surface of the sandstone tends to decrease. Therefore, the adsorption of surfactant on the surface of sandstone is slightly reduced.

The adsorption quantity of surfactant with different concentrations on treated core powder is plotted against imbibition recovery in Fig. 14.

With the increased quantity of surfactant adsorbed on the core powder, the oil recovery factor increases first and then decreases slightly. There is an optimal adsorption range (8.85–30.4 mg/g), which corresponds with the surfactant concentration range of 0.1%–0.3%, and the absorption effect is the best in this range. As the surfactant adsorbs on the core surface, the core wettability changes from oil-wet to weakly water-wet. At the same time, the oil/surfactant interfacial tension is greatly reduced, which in turn

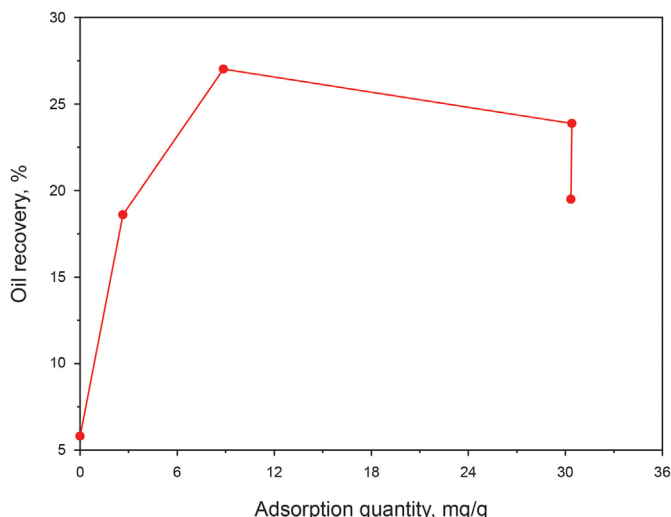


Fig. 14. The relationship between the surfactant adsorption quantity and oil recovery.

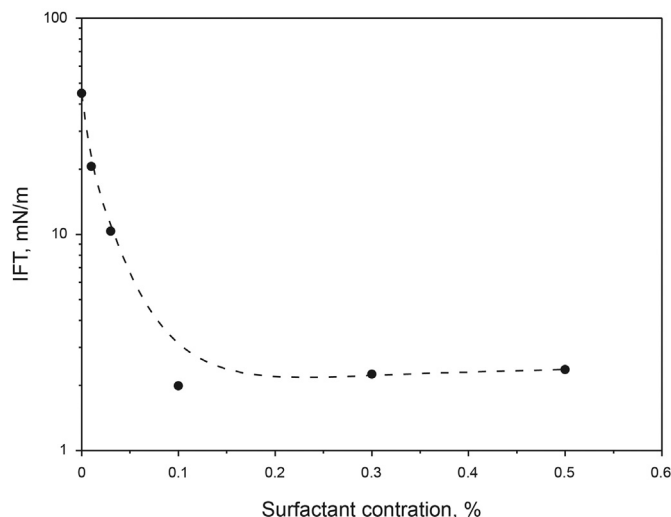


Fig. 15. IFTs between the oil and surfactant solutions at 70 °C.

increases imbibition recovery. With a further increase in the adsorption capacity (concentration exceeding critical micelle concentration, CMC), the surfactant forms micelles in the aqueous phase. The migration of micelles tends to block the pore throats, which is not beneficial to the flow of the water phase and oil phase, resulting in a slight decrease in imbibition recovery.

3.4. Oil/surfactant interfacial tension

At 70 °C, the IFTs between the oil and surfactant solutions with different concentrations were measured. The results are shown in Fig. 15.

When the surfactant concentration is low, as the concentration increases, the oil/surfactant interfacial tension decreases gradually. As the concentration of the surfactant increases further, the surfactant solution forms micelles, which reduces the number of surfactant molecules at the oil/surfactant interface, resulting in a slight increase in the IFT.

The IFT of surfactant solutions with different concentrations is plotted against the imbibition recovery, and the results are shown in Fig. 16.

As shown in Fig. 16, with decreasing surfactant IFT, the imbibition recovery increases. When the surfactant concentration is higher than the CMC, on the one hand, the surfactant micelles present in the water phase can easily block the small pores and pore throats in the core, reducing the flow channels for the oil phase and the water phase. On the other hand, after the formation of micelles, the amount of free surfactant molecules in the solution decreases, which in turn reduces the amount of surfactant at the solid/liquid interface and the liquid/liquid interface, and the surfactant/oil interfacial tension of the increases slightly. These combined effects diminish the imbibition effect, and the oil recovery decreases.

3.5. Flow capacity of oil in tight sandstone treated by surfactant

The flow capacity of fluid in porous media is affected by the wettability of the solid, the characteristics of the solid/liquid interface and the liquid/liquid interface. After the sandstone core was vacuumed and saturated with oil under a high pressure, part of the oil flowed out of the core during centrifugation. The NMR T_2 spectrums of the oil phase in the core during centrifugation at various speeds are shown in Fig. 17.

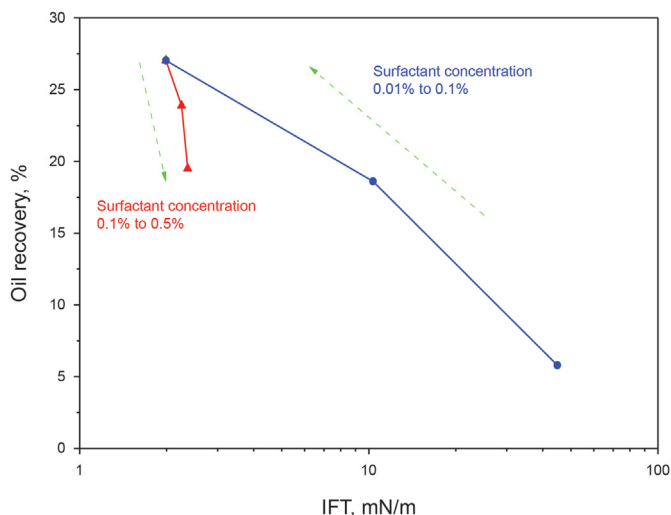


Fig. 16. Relationship between oil/surfactant IFT and imbibition recovery. Red: Surfactant concentration above the CMC. Blue: Surfactant concentration below the CMC.

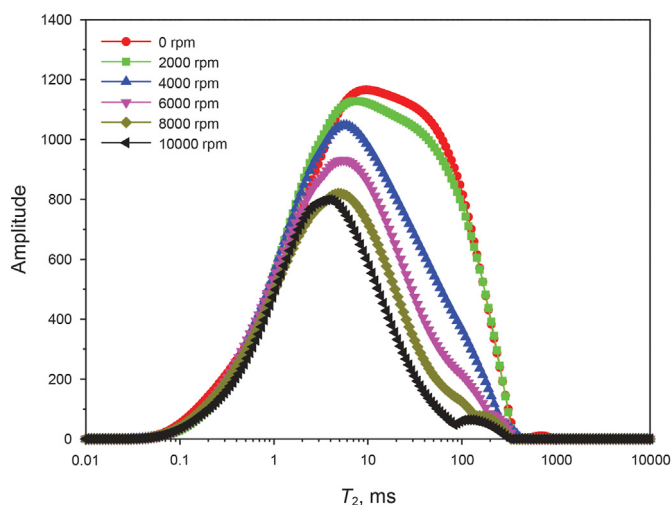


Fig. 17. NMR T_2 spectrums of the oil-wet core at different centrifugation speeds.

With increasing centrifugation speed, the oil in the large pore throats drains out gradually, but the oil in the small pore throats remains. An increase in centrifugal speed leads to an increase in the displacement pressure difference. The relaxation time of the oil in the large pore throats is long, and part of the oil phase is discharged from the core under centrifugal force. This results in a significant decrease in the signal over a long relaxation time. Another part of the oil phase is retained in the small pores, corresponding to immobile oil.

T_2 spectrums of oil phase signals in sandstone after initial and final centrifugation were plotted against the core pore size distribution, and T_2 cutoff values were calculated and plotted, as shown in Fig. 18.

In Fig. 18, the relaxation time corresponding to the T_2 cutoff of water displacing oil in the treated sandstone was 8.12 ms, corresponding to the pore throat diameter of 0.363 μm , which describes the size of the movable pore and throat (MPT). The right side of the vertical line corresponds to the flowable space of the oil phase in the pores of the sandstone core. The left side of the vertical line

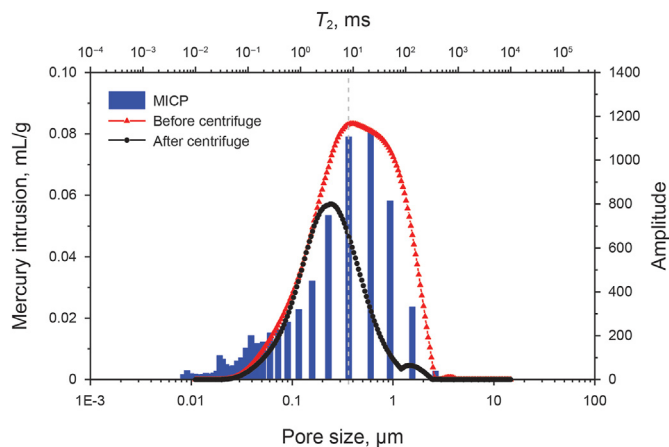


Fig. 18. Determination of T_2 cutoff of oil phase in core.

corresponds to the nonflowable pore space of the oil phase, including the clay- and capillary-bound oil phases. Under normal conditions, the oil has very little flowability. Therefore, the smaller the T_2 cutoff of the core, the more favorable the fluid flow is, and the higher the recovery factor will be.

The T_2 cutoff at various treatments was determined to obtain the impact of the fluid on the rock samples, such as water displacing oil in the original sandstone, surfactant displacing oil in the treated sandstone, and water displacing oil in the treated sandstone. The results are shown in Fig. 19.

Compared with the treated oil-wet sandstone core, the original water-wet sandstone core has a smaller MPT, and the surfactant contributes to increasing the flow capacity of the oil phase in the treated oil-wet core. For the original water-wet core, oil did not wet the pore walls. The flow resistance of the oil phase was lower than that of the water phase, and the capillary force was the driving force for imbibition, so oil flows more easily in water-wet sandstone than in oil-wet samples. Under the action of the surfactant, the wettability of the oil-wet sandstone core changes to weakly water-wet, which makes the flow resistance of the oil lower than that under oil-wet conditions. Therefore, the surfactant reduces the MPT in the oil-wet core, promotes the flow of the oil phase, and thus contributes to imbibition.

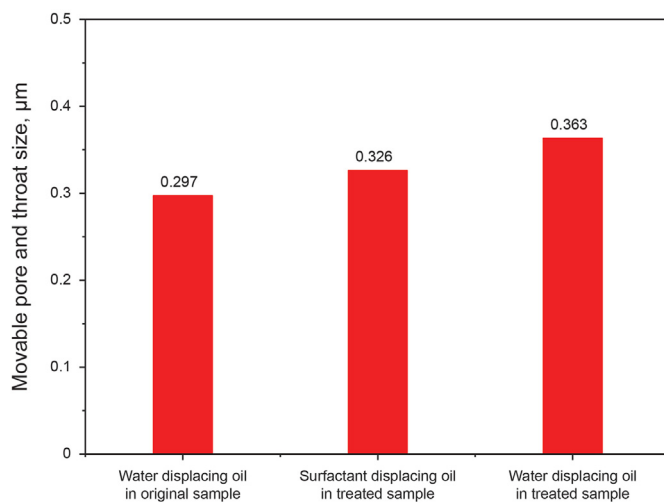


Fig. 19. MPT size corresponding to different fluids in the cores.

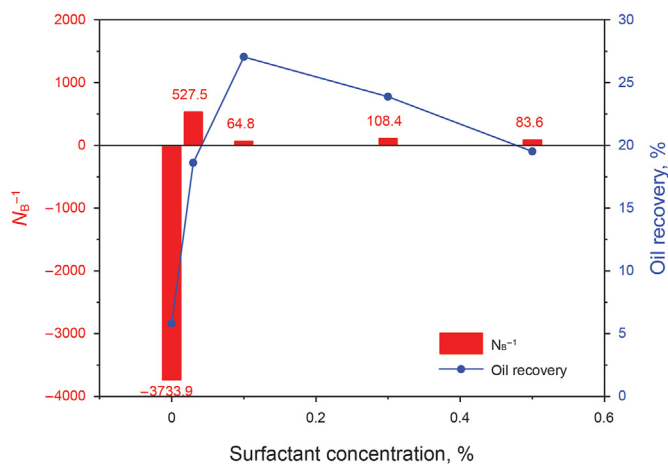


Fig. 20. Relationship between surfactant concentration, oil recovery and N_B^{-1} .

4. Discussion

The contribution and effect of capillary force and gravity force on the imbibition process are reflected by the inverse Bond number, N_B^{-1} , as expressed in Eq. (4) (Standnes et al., 2002). The relationships between the oil recovery ratio by imbibition and inverse Bond number under different surfactant concentrations were compared. The results are shown in Fig. 20.

$$N_B^{-1} = \frac{2C\sigma \cos \theta}{\Delta\rho g H} \sqrt{\frac{\phi}{k}} \quad (4)$$

where C is a constant that depends on pore geometry (for cylindrical capillaries, $C = 0.4$); ϕ is the porosity, %; k is the permeability of the sample, mD; $\Delta\rho$ is the difference in density between oil and water, g/cm^3 ; g is the gravity acceleration, cm/s^2 ; H is the core length, cm; θ is the contact angle, degree; and σ is the IFT between oil and water, mN/m.

According to Schetcher's study (Schechter et al., 1994), when $N_B^{-1} > 5$, the capillary force dominates the driving force for imbibition; when $N_B^{-1} < 0.2$, gravity dominates; when N_B^{-1} is from 0.2 to 5, the capillary force and gravity both dominate the imbibition process. Fig. 20 shows that as the surfactant concentration continues to increase, the variations in oil recovery and N_B^{-1} are not synchronized. When the surfactant concentration is 0, the inverse Bond number is smaller than 0. This shows that the direction of the capillary force is opposite to that of the water imbibition, which indicates that the driving force for imbibition at this time is gravity. This is consistent with the phenomenon of low recovery shown in Fig. 9.

When the surfactant concentration is greater than 0, the inverse Bond numbers are all greater than 5, which indicates that the capillary force dominates the imbibition process. However, the surfactant imbibition recovery and inverse Bond number do not have a linear relationship. When the concentration is 0.1%, N_B^{-1} is the smallest (64.8), but the oil recovery ratio by the imbibition is the highest. When the surfactant concentration is less than the CMC, with increasing concentration, the IFT between the oil and water decreases, which reduces the capillary force and N_B^{-1} . At the same time, the decrease in IFT improves the microscopic displacement efficiency in the pores. Thus, the capillary force has dual effects on the recovery rate. Therefore, when the surfactant concentration increases from 0.03% to 0.1%, the imbibition recovery rate increases only slightly.

When the surfactant concentration is higher than the CMC, as the concentration increases, the IFT between the oil and water increases slightly, and N_B^{-1} increases slightly. At this time, the surfactant forms micelles in the water phase, which may block the pore throats in the porous medium, thereby inhibiting the water imbibition and drainage of oil, causing the imbibition recovery rate to decrease gradually.

5. Summary and conclusions

The effect of surfactant adsorption on the spontaneous imbibition recovery of tight outcropped sandstone with different wettabilities was investigated in this study. The characteristics of the solid/liquid interface and oil/surfactant interface during surfactant adsorption were obtained. Then, the driving force for imbibition in oil-wet tight sandstones was analyzed. With the LF-NMR method, the effects of surfactant adsorption on the oil flow capacity of cores with different wettabilities were revealed. Some of the conclusions drawn are as follows:

- 1) For oil-wet tight sandstone with a wettability index of -0.96 , the oil recovery by spontaneous imbibition was 8.59% lower than that in water-wet sandstone by surfactant. With increasing surfactant concentration, the spontaneous imbibition recovery of the oil-wet core first increased and then decreased. The 0.1% surfactant solution had the highest imbibition recovery rate of 27.02%.
- 2) With the adsorption of surfactant, the core surface changed from weakly oil-wet (contact angle 107.4°) to weakly water-wet (80.1° – 82.7°) very quickly. The capillary force was converted into an imbibition force, which promoted the imbibition progress. The optimal surfactant adsorption quantity in the oil-wet sandstone corresponded to a concentration range of 0.1%–0.3%, which also corresponded to the highest oil recovery.
- 3) With a decrease in surfactant/oil IFT, the imbibition recovery increased gradually. When the surfactant concentration was lower than the CMC, the imbibition recovery was positively correlated with the surfactant concentration. When it was higher than the CMC, the oil recovery decreased with increasing surfactant concentration.
- 4) By calculating the inverse Bond number of imbibition, it was found that the driving force was gravity for brine imbibition in the oil-wet cores and that it was capillary force for surfactant imbibition in the oil-wet cores. When the surfactant concentration was smaller than the CMC, the surfactant concentration was negatively correlated with the inverse Bond number but positively correlated with the oil recovery. When the surfactant concentration was higher than the CMC, the imbibition recovery had a close relationship with the IFT and contact angle.
- 5) Through LF-NMR and centrifugation, it was found that the oil in the original water-wet sandstone core had a better flow capacity (MPT size: $0.297 \mu m$) than the flow capacity of the oil in the oil-wet core (MPT size: $0.363 \mu m$). Surfactant helps to improve the flow of the oil phase in the oil-wet core (MPT size: $0.326 \mu m$), which benefits oil production in oil-wet tight sandstone reservoirs.

Acknowledgments

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