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Preparation and evaluation of sodium oleate-derived multifunctional surfactants as oilfield chemicals

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ABSTRACT

In this paper, sodium oleate (SO) was modified by the reaction with formaldehyde to prepare SO-1 to SO-4. And then surface tension, foaming ability and oil displacement ability were carried out to evaluate the applicability as oilfield chemicals. On this basis, the microstructural observation of the microstructure of the synthesized surfactant foam was observed by microscopy. The decay mechanism of different surfactant foams, the droplet structure was revealed. In the relationship between stability, the interfacial tension evolution reduces the ability of oil-water interfacial tension. The results show that SO-1 to SO-4 can significantly reduce the oil-water interfacial tension at a concentration of 1.0%. When the concentration is 0.5%, SO-1 can reduce the oil-water interfacial tension to 0.001 mN/m, and 1.0% SO-1 can reduce the oil-water interfacial tension to 0.0001 mN/m. The foaming ability evaluation shows that the optimal foaming concentration of SO-SO-4 is 0.4%, and the temperature has little effect on the foaming ability. The oil displacement ability evaluation is the evaluation of the viscoelasticity of the surfactants with different concentrations. The results show that the oil displacement efficiency of the new surfactants is higher than that of SO, and the SO-2 oil displacement ability is the best. 0.3% SO-2 displays the best oil displacement efficiency of 30%, indicating that the synthesis of SO-series surfactants can be greatly improved. The above experimental results all indicate that they have an excellent ability to reduce the oil-water interfacial tension, and they can be used in the oil and petrochemical industry for oil production and have certain practical uses.

Keywords: Sodium oleate; Surface interfacial tension; Foaming; Emulsification; Oil displacement

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1. Introduction

Surfactants play a key role in oil and gas field production and act as performance additives in many formulations. Currently, in the gas well drilling process, a certain amount of surfactant solution is sometimes added to the drilling fluid to make the drilling fluid foamy, and the pressure generated is slightly less than the formation pressure. Anionic surfactants are among the oldest, most productive, and most widely used surfactants [1]. The various functions of surfactants are manifested mainly in the properties of the surface, the liquid–liquid interface and the liquid–solid interface of the liquid, where the surface properties of the liquid are of utmost importance. Sodium oleate (SO) is an

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anionic surfactant whose hydrophilic group is a carboxyl group. The surface-active part after ionization in water is a fatty acid anion and the fatty acid salt surfactant is the earliest anionic surfactant that has been developed in history. Sodium oleate is widely used in petroleum and petrochemical production processes, such as drilling, oil recovery, and collection and transportation due to its unique structure and wide range of sources. However, problems such as non-resistance and difficulties exist in practical applications in the field.

Based on the above understanding, sodium oleate is the main component, and formaldehyde having different molar ratios is introduced into the aldol reaction [2], and one or two methylol groups are introduced on the α carbon of the carboxyl group for the purpose of increasing the polarity group and adjusting the hydrophilic-lipophilic balance, which affects surface activity. Therefore, with the introduction of the methylol group, the foaming and emulsifying ability of sodium oleate are enhanced, especially the ability to lower the oil-water interface. The above experimental structure shows that the synthesized product has an excellent ability to lower the oil-water interfacial tension. The petroleum and petrochemical industries can be used in oil recovery operations and have certain practical uses.

2. Experimental setup

2.1. Materials

Sodium oleate was purchased at the Xi'an Chemical Reagent Factory, methanol and potassium bromide was purchased from the Tianjin Tianli Chemical Reagent Group, formaldehyde was purchased at the Chengdu Kelon Chemical Reagent Factory, and crude oil samples were obtained from the oil field.

2.2. Synthesis of multifunctional surfactants

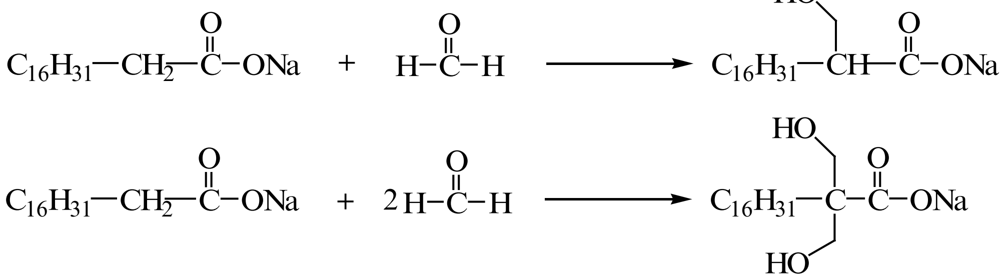
The proportion (molar ratio) is called formaldehyde and sodium oleate. Sodium oleate was placed in a 250 mL three-necked flask. 50 mL of methanol was added as a solvent. At the reflux temperature, formaldehyde was added dropwise to the flask to control the drop rate of 1 d/s. After the addition was completed, the reaction was carried out for 3 h, and the reflux temperature was measured at 68°C. After completion of the reaction, the solvent was distilled off under reduced pressure to obtain a product. The reaction formula is shown in Fig. 1. The ratio of sodium oleate to formaldehyde can be controlled to obtain different products. Compositions with a ratio of sodium oleate to formaldehyde of 1:0 are labeled SO, 1:1 is labeled SO-1, 1:2 is labeled SO-2, 1:3 is labeled SO-3, and 1:4 is labeled SO-4.

2.3. Surface tension test conditions

According to the National Standard GB/T 22237-2008 of the People's Republic of China, the method for determining the surface tension of surfactants was analyzed. In the static method, move the sample container so that the plate contacts the liquid. In order to avoid the measurement error caused by buoyancy, the lowest part of the plate should be at the same height as the liquid level. Record the current reading, which is proportional to the surface tension. In order to measure whether the flat plate is fully wetted, the flat plate can be contacted with the liquid, the current data can be recorded, and then the container containing the liquid to be measured can be raised for 2 mm and then lowered to the original height. The lowest part of the flat plate is at the same height as the liquid level, at this time, the reading of the instrument should be the same as the initial reading. Surfactant solutions of 0.0001%, 0.001%, 0.01%, 0.03%, 0.05%, 0.1%, 0.3%, 0.5% (mass fraction) of deionized water were used to determine the surface tension of each surfactant solution by the hanging-ring method.

2.4. Test conditions for interfacial tension

The method for determining the interfacial tension of surfactants is in accordance with the National Standard of the People's Republic of China GB/T 6541-1986 (1991). The interfacial tension is determined by a horizontal platinum wire measuring ring pulling the platinum wire ring from the liquid surface with high interfacial tension, that is, the force required to pull the platinum wire ring upward from the water-oil interface. When calculating the interfacial tension, the measured force is corrected by an empirical measurement coefficient, which depends on the force used, the density of oil and water, and the diameter of the ring. The measurement is carried out under strict and standardized non-equilibrium conditions, that is, the measurement is completed within 1 min after the formation of the interface. 0.5%, 1% (mass fraction) of the surfactant solution was prepared with deionized water, and kerosene was used as the internal phase by the rotary drop method.



$$C_{16}H_{31}-CH_2-\overset{O}{\overset{\|}{C}}-ONa \quad + \quad H-\overset{O}{\overset{\|}{C}}-H \longrightarrow C_{16}H_{31}-\overset{HO}{\overset{|}{CH}}-\overset{O}{\overset{\|}{C}}-ONa$$

$$C_{16}H_{31}-CH_2-\overset{O}{\overset{\|}{C}}-ONa \quad + \quad 2\,H-\overset{O}{\overset{\|}{C}}-H \longrightarrow C_{16}H_{31}-\underset{HO}{\underset{|}{\overset{HO}{\overset{|}{C}}}}-\overset{O}{\overset{\|}{C}}-ONa$$

Fig. 1. The chemical equation for the synthesis of surfactants.

2.5. Test conditions for foaming capacity

The foaming ability of surfactants was tested in accordance with the National Standard GB/T 7462-1994 of the People's Republic of China. The measurement process is to keep the solution flowing down until the horizontal plane drops to 150 mm scale, and record the outflow time. All measurements where the difference between the outflow time and the arithmetic mean of the observed outflow time is greater than 5% shall be ignored, and abnormal long time indicates the presence of air bubbles in the metering tube or plug. Foam volume was measured at 30 s, 3 min and 5 min after the flow stopped. If there is a depression in the center above the bubble, record the readings based on the arithmetic mean between the center and the edge. Repeat the measurement to obtain at least 3 times of error within the allowable range. First, the best foaming concentration of the surfactants was tested by agitation. The experimental temperature was at room temperature, the volume of the solution was 100 mL, the stirring rate was 7,000 rad/min, and the stirring time was 3 min. Immediately after the stirring was stopped, the formed foam was poured into a 500 mL measuring cylinder, and the stopwatch was periodically recorded for foam volume and foam half-life (the precipitation time of 50 mL of water). The foaming ability of the surfactants at different temperatures was determined by a Roche foam apparatus.

2.6. Microstructure analysis of foam

The microstructure of the surfactants was observed by an optical microscope. Polarized light was chosen as the light source [3].

2.7. Test conditions for emulsifying ability

The emulsifying properties of the surfactants were tested according to the National Standard GB/T 6369–2008. Add 25 mL distilled water of specified temperature into three separators respectively, then add 0.2 g of newly prepared emulsifier and oil mixture respectively, weigh to 0.001 g, add 25 mL of distilled water, fix the separators on the horizontal shaker, shake for 2 min, place them vertically on the bracket, stand for 30 s, put 30 mL of emulsion layer solution down in the beaker, mix evenly, and suck 10 mL with the pipette, Transfer it to another separating funnel, extract it several times with about 50 mL of trichloromethane, and collect the extracting liquid in a 50 mL volumetric flask to the scale. If it is found that the extraction liquid is turbid, anhydrous sodium sulfate can be added for dehydration to make the solution brown and transparent. At the wavelength of R = 400 nm, trichloromethane was used as the reference liquid to determine the optical density value of the extraction liquid in three volumetric flasks. According to the optical density value, the corresponding content was found from the working curve, and the emulsifying capacity of the emulsifier was obtained by adding the oil amount. 0.1%, 0.2% (mass fraction) of surfactant solution was prepared, and 10 mL of the above solution and 10 mL of crude oil were measured and placed in a 25 mL plugged test tube and later placed in a water bath, with an experimental temperature being set to 45°C at a constant temperature for 10 min. After removal, it was shaken 100 times to form an oil-in-water emulsion, which was then placed in a water bath, and the volume of the precipitated water was recorded every 5 min until the change was small, and the water dissolution rate was recorded [4]. The formula for calculating the water separation rate is like Eq. (1).

$$\text{Water splitting rate} = \frac{\text{Amount of precipitated water}}{\text{Amount of added water}} \times 100 \quad (1)$$

2.8. Test conditions for oil displacement capacity

The dry core sand and the experimental oil were evenly mixed in a ratio of 7:1 (volume ratio), aged at 40°C for 24 h, cooled to room temperature, and placed in a reserve dryer. The core sand of the experimental oil was weighed with an 8 g electronic balance and pressed into thin sheets with a four-column press. The surface-active agent of 100 mL with different concentrations of surfactants (0.2%, 0.3%, 0.6%, 1.0%, mass fraction), the same concentration (0.3%, mass fraction) and different synthetic proportions (SO, SO-1, SO-2, SO-3, SO-4) were added to the oil displacement meter, and the oil displacement meter was placed at 50°C. In a constant temperature water bath, the data was recorded and the displacement efficiency was calculated until the displacement volume (static) remained unchanged [5].

3. Results and discussion

3.1. Surface tension evaluation

Surfactant solutions having mass fractions of 0.0001%, 0.001%, 0.01%, 0.03%, 0.05%, 0.1%, 0.3% and 0.5% were prepared with deionized water, respectively. The surface tension of each solution was measured by the hanging-ring method. The results are shown in Fig. 2. As shown in Fig. 2, with the increase in concentration, the surface tension of the SO-SO-4 solution first decreases rapidly, and then slowly decreases, with an inflection point in the middle. This is

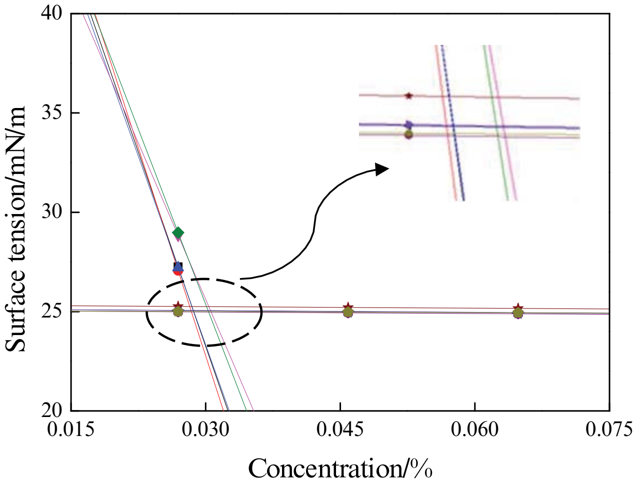


Fig. 2. Surface tension pattern of SO to SO-4.

because their molecules can be aligned in an aqueous solution. Within a certain range, at higher concentrations, more molecules accumulate on the surface of the solution and the corresponding surface tension is lower [6,7]. However, when the surface of the liquid is completely covered, the concentration in the solution will increase and the corresponding counterions will accumulate to form micelles. At this time, the surface tension of the solution tends to be stable.

The above data was processed to obtain the critical micelle concentration (CMC) value of SO-SO-4, which is listed in Table 1. From this result, it can be seen that the difference between the critical micelle concentrations of the SO-series surfactants is small. Since the concentration of the liquid surfactant is too high, the surface tension tends to be stable.

3.2. Interfacial tension evaluation

The interfacial tension test was performed on SO, SO-1, SO-2, SO-3 and SO-4. First, 0.5% and 1.0% SO-SO-4 were tested at 30°C to reduce the oil-water interfacial tension. The experimental results are shown in Fig. 3. The oil phase used in the experiment was kerosene. Kerosene contains more light components, less heavy components such as naphthenes and aromatic hydrocarbons, and the carbon chain length is 11–17, while SO-SO-4 molecules have a hydrophobic carbon number of 17–19, which is close to the carbon number of kerosene and achieves the "appropriate carbon number of the oil phase" necessary to reduce the interfacial tension between the solution and the light oil to an ultra-low value [8]. When the concentration of the solution reaches the adsorption saturation at the interface between kerosene and water, the upper interface between kerosene and water will be closely arranged, and the interfacial tension will reach the lowest value. In Fig. 3, it can be seen that SO-3 and SO-4 have poor ability to reduce the oil-water interfacial tension when the concentration is 0.5 at 30°C, and the SO-SO-2 oil-water interfacial tension is an order of magnitude smaller than both. When the concentration is 1.0%, the interfacial tension of SO decreases slightly with time, only from 0.08 mN/m to about 0.04 mN/m. Compared to SO, the interfacial tension of the other four surfactants at the beginning is less than SO, which is ultimately reduced by one order of magnitude, and SO-1 is even reduced by two orders of magnitude. It shows that SO-1-SO-4 can effectively reduce the oil-water interfacial tension at this temperature relative to SO. At the same time, when hydroxymethyl is introduced into the SO molecule, the ionicity of the SO itself is weakened, which greatly reduces the interfacial tension between the oil and water [9,10].

3.3. Foaming ability test

The foaming ability of SO, SO-1, SO-2, SO-3 and SO-4 was tested, and the experimental results are shown in Fig. 4. It can be seen in Fig. 4 that as the concentration increase, the volume of the foam produced by SO-SO-4 first increases sharply, and after 0.2%, the growth rate slows down. Then, after reaching 0.4%, it begins to decrease again. The half-life of the foam first increases sharply, and then the growth rate becomes slower and eventually stabilizes [11]. Considering the foam volume and half-life, the optimum foaming concentration of the SO-series surfactant should be 0.4%. At this concentration, the SO-1-SO-4 foam volume is higher than that of SO, and the half-life is not much different from the SO.

Table 1
Critical micelle concentration and surface tension pattern at 0.5% of SO-SO-4

Surfactant name	CMC value (%)	0.5% surface tension (mN/m)
SO	0.0286	24.369
SO-1	0.0284	24.418
SO-2	0.0287	24.379
SO-3	0.0303	24.664
SO-4	0.0303	24.685

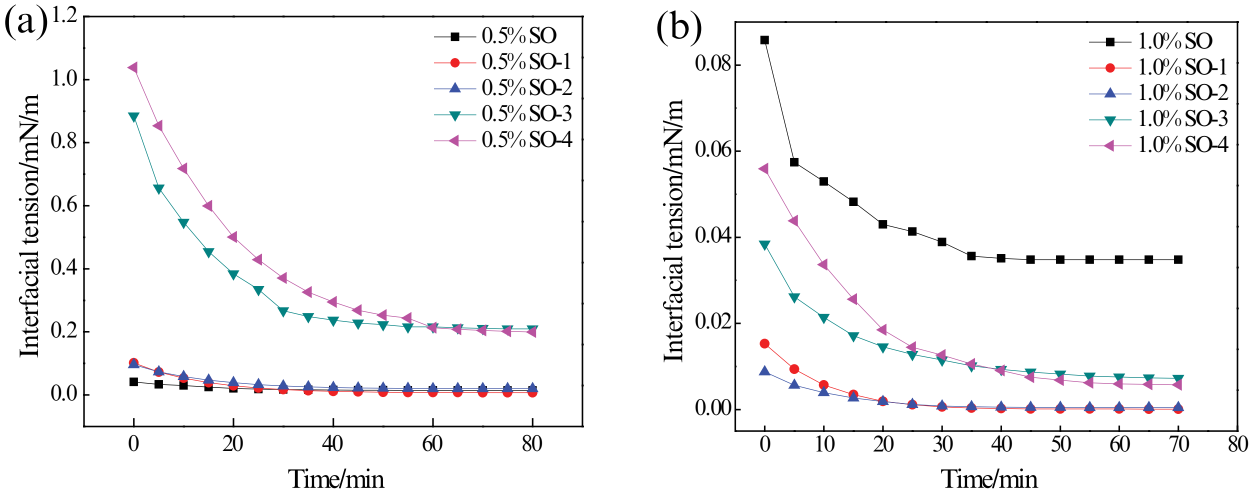


Fig. 3. Interfacial tension pattern of different SO-SO-4 concentrations 0.5 wt.% (a) and 1.0 wt.% (b).

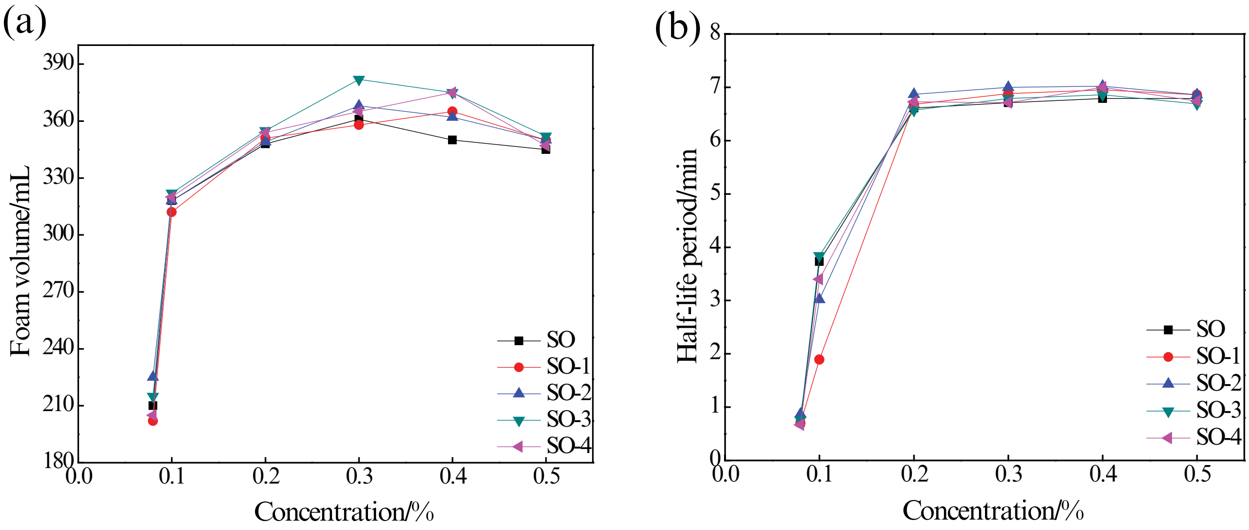


Fig. 4. Foaming ability tests for SO-SO-4 foam volume (a) and half-life period (b).

3.4. Microstructure analysis of foam

Optical microscopy was used to observe the change in the microstructure of the SO-SO-4 foam with time. Polarized light was selected as the light source. To better explore the foaming ability of various surfactants, the foam characteristic value is introduced. When the Φ is in the range of 0.52–0.74, the moisture content of the foam is larger than that of the wet foam, and the foam is spherical or ellipsoidal. As the liquid in the foam precipitates continuously, the phi increases, and when it is greater than 0.74, it becomes dry foam. The foam is generally polygonal, and the SO-SO-4 foam values at different times are shown in Table 2.

As can be seen from Fig. 5 and Table 2, the foam shape of SO-SO-4 is ellipsoidal when it is 0 min, but since its value is close to 0.74, the inner wall of the foam is polygonal. Just because the foam has just formed and the water content is more, the outer wall appears to be elliptical. At 10 min, the shape of SO-3 and SO-4 foam changes into obvious polygons. The wall thickness of the foam has become thinner, and the thickness of SO-2 foam wall is thickest; that of SO-4 is the second and that of SO-1 is the thinnest. Generally speaking, the thicker the foam wall, the better the mechanical strength of the foam, hence the better the stability. In addition, the stability of the foam is also related to the viscosity [12], and the viscosity of the foam is also related to its characteristic value. Such as Φ is the foam characteristic value, η is the foam viscosity, and η_0 is the initial viscosity of the foam in the formula.

$$\eta = \eta_0 \left(1.0 + 4.5\,\Phi\right) \quad \left(0.52 < \Phi < 0.74\right) \tag{2}$$

$$\eta = \eta_0 \left[\frac{1}{1 - \Phi^{1/3}}\right] \quad \left(\Phi > 0.74\right) \tag{3}$$

The stability of the foam increases as the viscosity increases over a certain range. It can be seen from the above formula that the viscosity of the wet foam is linearly related to the characteristic value of the foam, and the dry foam is exponentially related to the Φ value. The viscosity of the two increases with increasing Φ value. In combination with Table 2, it can be seen that the value of SO-1-SO-4 is greater than SO when 0 min, and SO-2 > SO-4 > SO > SO-3 > SO-1 when 10 min; its foam viscosity and foam stability are also ranked as SO-2 > SO-4 > SO-1. Theoretically, when the foam is generated, the energy (surface energy) of the system will increase correspondingly with the increase in the surface area of the liquid. When the foam is destroyed, the energy of the system will decrease correspondingly. Therefore, from the energy point of view, low surface tension is beneficial for foam formation. Combined with the results of surface interfacial tension test and foaming ability test at 30°C, it can be found that the smaller the surface interfacial tension of surfactants, the stronger the foaming ability; that is, the lower surface interfacial tension is beneficial for foaming. However, it does not guarantee that the generated foam has good stability. Low surface tension can help stabilize the foam only when the surface of the liquid is capable of forming a surface film with certain intensity [13,14].

Table 2
The "Φ" values of SO-SO-4 foam at different times

Φ value	0 min	10 min
SO	0.706	0.889
SO-1	0.714	0.881
SO-2	0.714	0.893
SO-3	0.709	0.885
SO-4	0.711	0.891

3.5. Emulsification test

The emulsifying abilities of SO, SO-1, SO-2, SO-3 and SO-4 were tested. The experimental results are shown in Fig. 6. The amount of water precipitation rate the

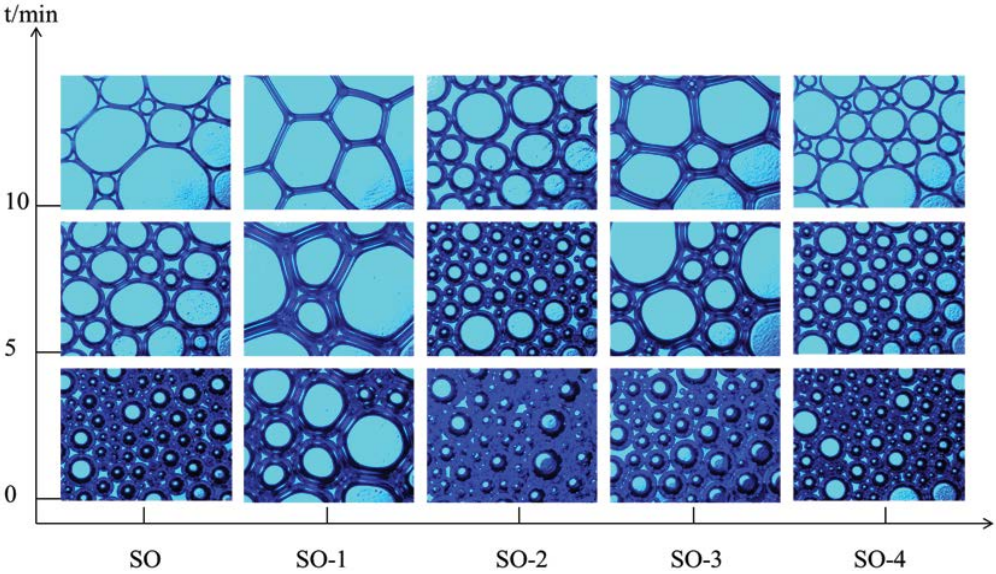


Fig. 5. Variation of foam microstructure with time for SO-SO-4 (magnification was 40 times in 0, 5 and 10 min).

emulsifying ability of surfactants [15,16]. The emulsions may coalesce, resulting in phase separation. The water precipitation rate is shown in Table 3. As shown in Fig. 6, when the surfactant concentration is 0.1%, the precipitation rate of SO-1 and SO-2 is obviously faster than that of SO, and the final amount of precipitated water is also more than that of SO. SO-4 precipitates water at the same rate as SO, but the final amount of water is greater than SO, indicating that SO-1, SO-2, SO-4 have lower emulsifying capacity than SO. However, the emulsifying capacity of SO-3 is much higher than that of SO, and no water has been precipitated during the experimental period. When the surfactant concentration is 0.2%, the precipitation rate of SO-1 and SO-4 is obviously faster than that of SO, and the final precipitation amount is also more than that of SO, indicating that the emulsifying ability of SO-1 and SO-4 is lower than SO. Similar conclusions can be drawn from the water analysis rate data in Table 3. Meanwhile, it can be seen from the water analysis data in Table 3 that with the increase in concentration, the water absorption of the emulsions formed, except SO-2, SO, SO-1, SO-3 and SO-4, increases. The reason for this phenomenon may be that, in a certain range, an increase in the mass fraction of the surfactant leads to an increase in the particle diameter of the emulsion, while the density difference exists between crude oil and water [17]. Increasing the diameter of the particles will accelerate the separation of crude oil and water. In addition, when the crude oil droplets in the emulsion float to the surface of the liquid, some of the droplets will aggregate into clusters. At this time, increasing the dosage of surfactant will increase the self-aggregation phenomenon between these already aggregated droplets, which in turn leads to deterioration of emulsion stability [18,19].

Table 3
Syneresis rate of emulsions made by SO-SO-4 at different concentrations

Rate of water evolution	0.1%	0.2%
SO	35	40
SO-1	45	45
SO-2	52	24
SO-3	49	37
SO-4	41	47

3.6. Oil displacement ability

The oil displacement test was carried out on SO, SO-1, SO-2, SO-3 and SO-4. The experimental results are shown in Fig. 7.

As shown in Fig. 7a, the oil displacement effect of SO with different concentration indicates the concentration is an important fact on the oil displacement efficacy. As the concentration rises to 0.3%, the efficiency is much higher than that of 0.1%, and 0.5% is also higher than that of 0.3%. Considering the cost performance, 0.3% was chosen for further research. Fig. 6b shows the oil displacement effect of SO as well as its derived surfactants, and the efficiency was summarized in Table 4, which shows that the displacement efficiency of So, SO-1, SO-3 and SO-4 has been improved at 50°C. SO-2 has the best displacement efficiency of 30%, indicating that the synthesis of SO-series surfactants can be greatly improved. The reduction of oil-water interfacial tension and surface tension can effectively improve oil recovery [20,21].

4. Conclusion

A series of surfactants derived from SO were synthesized with sodium oleate and formaldehyde. Their surface tension and the CMC values of each surfactant were measured. With the concentration of 0.5%, SO-1 can reduce the oil-water interfacial tension to 0.001 mN/m, and 1.0% SO-1

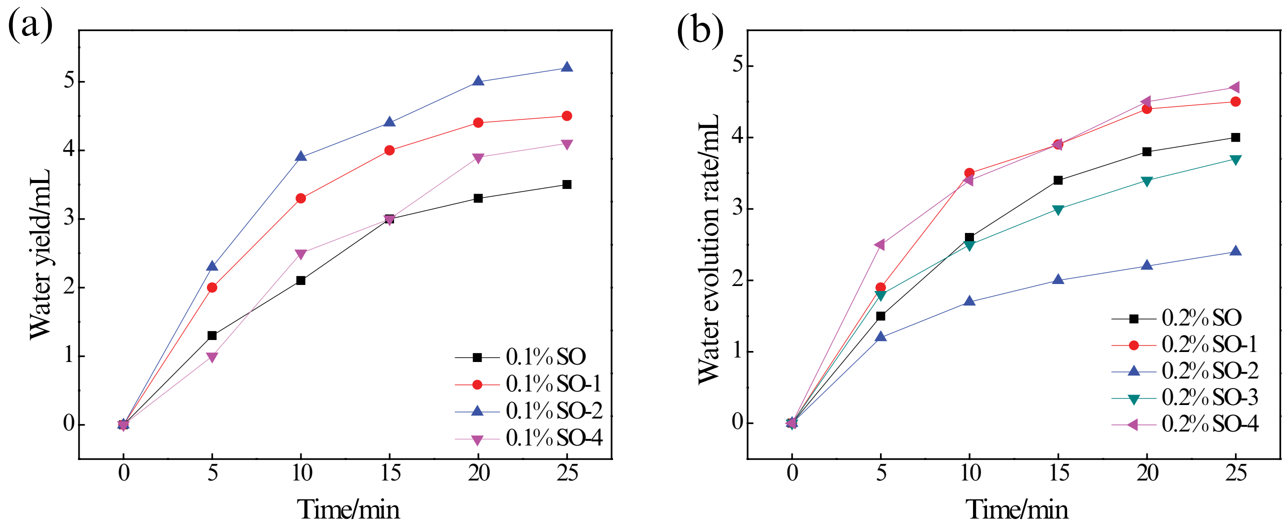
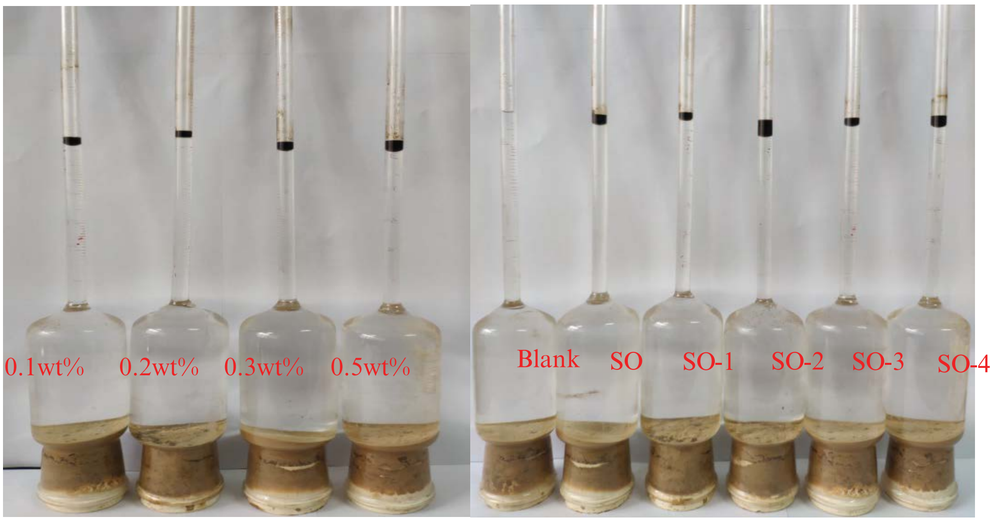


Fig. 6. Emulsification ability for different SO-SO-4 concentrations 0.1 wt.% (a) and 0.2 wt.% (b).



(a) (b)

Fig. 7. Oil displacement effect of the SO with different concentrations (a) and different surfactants (0.3 wt.%) (b).

Table 4
Oil displacement rate with a concentration of 0.3%wt. at 50°C

Name	Oil displacement rate
SO	15%
SO-1	10%
SO-2	30%
SO-3	15%
SO-4	25%

can reduce the oil-water interfacial tension to 0.0001 mN/m. The foaming ability evaluation shows that the foam produced by the synthesized surfactants had higher stability, but the foaming ability was generally affected by inorganic salts, and SO-1 has the best performance. And the optimal foaming concentration of SO-SO-4 is 0.4%, and the temperature has little effect on the foaming ability. The oil displacement ability shows that the efficiency of the new surfactants is higher than that of SO, and the SO-2 is the best, with an efficiency of 30%. In conclusion, the experimental results show that the surfactant has a good ability to reduce the interfacial tension between oil and water, so it has a certain practical application value for the oil industry.

Acknowledgments

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