

Influence of Surfactant and Weak-Alkali Concentrations on the Stability of O/W Emulsion in an Alkali-Surfactant–Polymer Compound System

Jie Zhang, Dangke Ge, Xiaoyan Wang, Wei Wang, Dandan Cui, Guangyu Yuan, Keliang Wang, and Wei Zhang*



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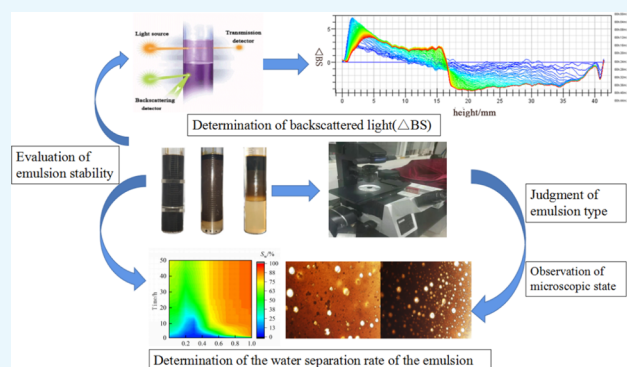
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ABSTRACT: Emulsions have emerged as advanced materials for wide industrial applications because of their unique properties. In the actual application in oilfields, emulsions can significantly enhance oil recovery. In the present study, the stability test shows that the concentrations of a surfactant and alkali and salinity have a great influence on the stability of the emulsion, but the addition of excessive chemical agents may adversely affect the emulsion stability. The addition of excessive alkali causes the phase inversion behavior of the emulsion to be discovered, which is also the main reason for the destabilization of the oil-in-water emulsion. Rheological experiments reveal that the emulsion produced by the chemical-flooding fluid is a pseudoplastic fluid, and the apparent viscosity decreases with the increase of the shear rate. Core-flooding experiments were conducted to study the effect of the emulsion stability on enhanced oil recovery, and the results indicate that the system with a better emulsion stability has higher oil recovery and displacement pressure.



1. INTRODUCTION

Emulsions play an important role in numerous industrial applications, for example, cosmetics,¹ inorganic powder material synthesis,² food,³ and petroleum industries.^{4–7} Surfactants, alkalis, and polymers have been widely used for achieving enhanced oil recovery (EOR) using chemicals. Alkali–surfactant–polymer (ASP) flooding is a technology for EOR that emerged in the 1980s. ASP flooding makes full use of the synergistic effect between an alkali and a surfactant by reducing the interfacial tension (IFT) between oil and water to ultralow^{8,9} and by wettability alteration.¹⁰ The main role of the polymer is to increase the viscosity of the displacement agent so that it can increase the sweep volume of the ASP composition. Many laboratory experiments and oil field tests have gradually found that the emulsification of crude oil in a reservoir plays an important role in enhancing oil recovery.¹¹ One of the important reasons is that the ASP composition emulsifies with the crude oil and carries crude oil out of the formation during the migration process,^{12,13} and the emulsion droplets plugging the pores play a role in profile control because of the “Jamin effect” when the emulsion droplets migrate to the pores with a pore diameter smaller than the droplet diameter. Stability of the emulsion is determined by different factors such as the nature of the interfacial film,

continuous phase viscosity, oil-water-ratios, salinity, and temperature. Alkali and surfactant concentrations and salinity are the main factors influencing the stability of the emulsion in the ASP composition because they can affect the nature of the interfacial film and the viscosity and salinity of ASP system. Poor emulsion stability caused by the coalescence of droplets has limited their use in EOR.¹⁴ Thus, it is necessary to study the various factors of emulsion stability.

Emulsions could be divided into two phases over time through creaming, coalescence, flocculation, or Ostwald ripening.¹⁵ The occurrence of creaming or sedimentation of drops is caused by the density difference between the dispersed and continuous phases. The rate of droplet coalescence and flocculation depends on several factors such as the volume fraction of the dispersed phase, solid content, surfactant type and concentration, and droplet diameter.¹⁶ The viscosity of the

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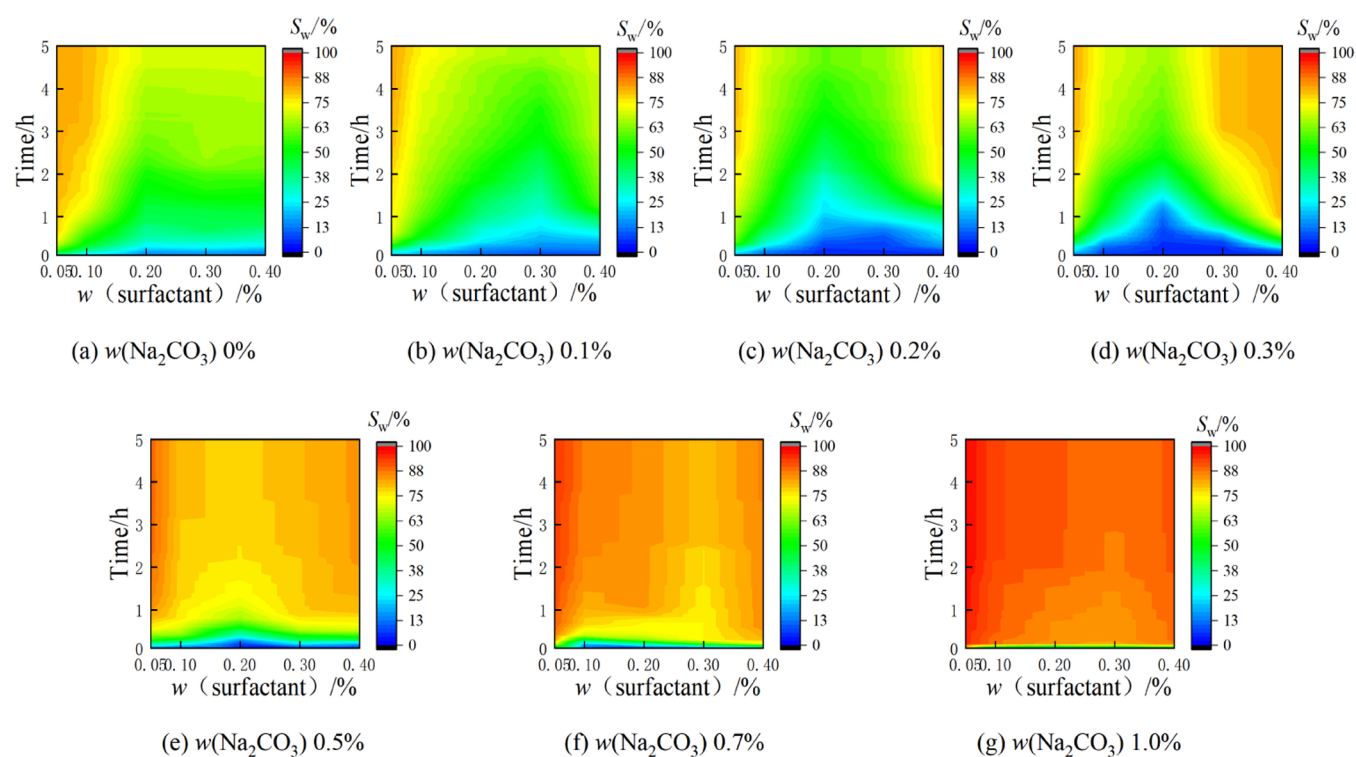


Figure 1. Water-separation rate of the emulsion in various alkali concentration systems. The degree of separation of water from the emulsion is indicated by the difference in color. Blue indicates a lower water-separation rate, while red indicates a higher water-separation rate. [From (a–g), the alkali concentration increases from 0 to 1.0 wt %].

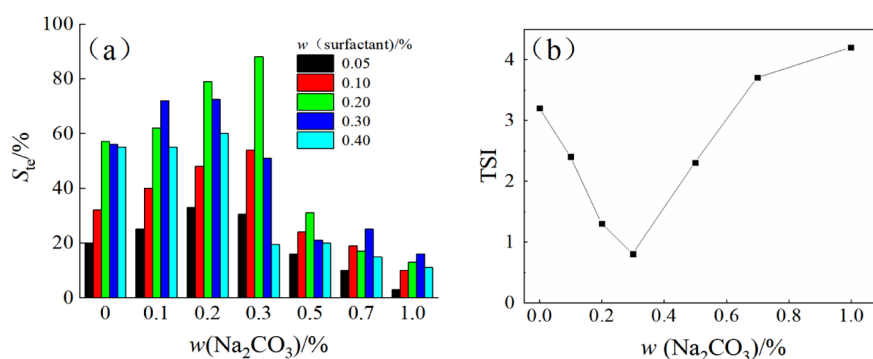


Figure 2. (a) Stability index of emulsion at different alkali concentrations. (b) Turbiscan Stability Index (TSI) of emulsion at different alkali concentrations (the surfactant concentration is 0.3 wt %).

interfacial film appears to be the most important factor to enhance emulsion stability. Highly viscous interfacial films slow down the rate of interfacial film drainage during the coalescence of the droplets by providing a mechanical barrier to coalescence. It has been proposed that when two droplets approach each other, they deform and a planar film is formed between them.¹⁷ When the thinning of the interfacial film between approaching drops reaches a critical thickness, the coalescence phenomenon will take place. A majority of publications discussed about the factors affecting the emulsion stability, but the research on the effect of sodium carbonate on the stability of emulsions and the effect of high surfactant concentration on the stability of emulsions are still relatively limited. Therefore, this study mainly investigates the effect of the sodium carbonate content and the surfactant concentration on the stability of emulsions. The experimental results will

have a certain guiding significance for optimizing the amount of alkali and surfactant rationally in oil fields.

2. RESULTS AND DISCUSSION

2.1. Effects of Alkali Concentrations on Stability of Emulsion. Alkali mainly reacts with the acidic hydrogen in the fractions of the crude oil and this reaction form petroleum acid soap with interfacial activity.¹⁸ These interfacial active components have a positive influence on the emulsion stability through adsorption onto the oil–water interface. In order to study the effect of alkali on the emulsion, a series of ASP composite systems with different alkali concentrations were prepared. As seen from Figure 1, there are different water-separation rates in different ASP composite systems. The areas of the “low” water-separation rate of the heatmap increased with the alkali concentration when the concentration of Na_2CO_3 was less than 0.3 wt %. The optimal alkali

concentration for emulsion stability was 0.3 wt %. As the Na_2CO_3 concentration increased to 0.5 wt % and above, the overall water-separation rate of the system was relatively high and the area of the “low” water-separation rate of the heatmap decreased until it disappeared.

It can be seen from Figure 2 that in a system where the alkali concentration was less than 0.5%, the stability of the emulsion increased with the alkali concentration. The main reason is that the crude oil has a relatively high acid value. The addition of an appropriate amount of alkali can react with acidic hydrogen in the crude oil to form interfacial active components. It is worth pointing out that these active components together with the added surfactant molecules absorb on the oil–water interface to increase the strength and thickness of the interface film. At the same time, sodium ions compress the double layer on the oil–water interface within a low range of alkali concentration, weakening the electrostatic repulsion of the ionic surfactant adsorbed on the interface.¹⁹

Compared with the system without alkali, S_{te} was lower and the TSI was higher when the alkali concentration was 0.5, 0.7, and 1 wt %, respectively. The addition of excessive alkali led to an increase in the water-separation rate of emulsions and the destabilization of the emulsion obviously. This is explained by the fact that the effect of “electrolyte” on the stability of the emulsion appears with the increase of alkali concentration. The increase of the number of sodium ions in the system compresses the double layer on the oil–water interface extremely, and thins the thickness of the oil–water interface film. Meanwhile, the gathering of sodium ions at the interface of the emulsion drops acts as a “charge shielding”, which reduces the electronegativity of the droplets and weakens the repulsion between the droplets. As a result, small drops are more likely to coalesce into larger drops, resulting in the destabilization of emulsion. In addition, the density difference between oil and water may increase when excessive Na_2CO_3 is added to the system. According to the Stokes rule, the phase-separation speed of the emulsion is directly related to the oil–water density difference. The addition of excessive alkali may cause an increase in the emulsion phase-separation speed.

Additionally, phase inversion of the emulsion with excessive alkali concentration was observed. The type of freshly prepared emulsion was oil-in-water when the alkali concentration was 0.7 and 1 wt %. However, the type of emulsion observed using a light microscope after a period of standing was water-in-oil. The phase inversion was also a cause of the sharp increase in the water-separation rate of the emulsion. Typical microscopic images of water-in-oil emulsion are shown in Figure 3.

According to the dense packing principle of solid geometry, when the volume fraction of the inner phase reaches the dense packing fraction of the spherical particles, which is 74%, the phase inversion could occur with the increase of the volume

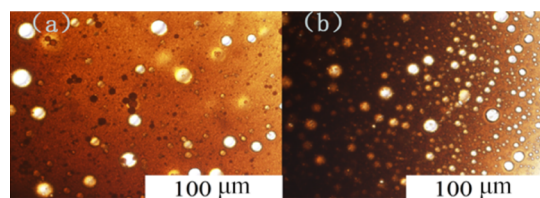


Figure 3. Microscopic image of water-in-oil emulsion with high alkali concentrations [alkali concentrations of (a) and (b) are 0.7, 1 wt %, respectively].

fraction of the inner phase of the emulsion. The volume fraction of inner phase increases when the concentration of alkali is higher. On the one hand, the density difference between the oil phase and the water phase of the emulsion increases. On the other hand, because of the uneven emulsion drop size distribution at high alkali concentrations, the probability of sedimentation and coalescence increases. As shown in Figure 4, both the water-separation rate and the oil-

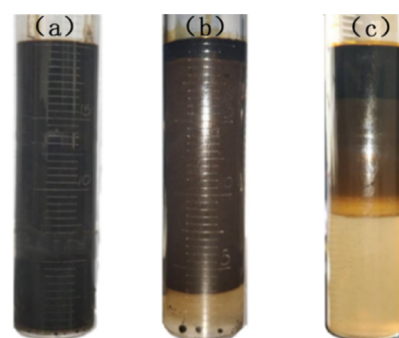


Figure 4. Photographs of the increase in the volume fraction of the emulsion inner phase. (a) Freshly prepared emulsion. (b) Emulsion after standing for 2 h. (c) Emulsion after standing for 5 h.

separation rate of the emulsion increase, the oil-separation rate is significantly lower than the water-separation rate. Therefore, the inner phase volume fraction of the emulsion increases until phase inversion occurs. In addition, the double layer was compressed by sodium ions when the alkali concentration increased to above 0.7 wt %.^{20–22} Repulsive forces between the polar groups reduced and formed clusters with hydrophilic heads sequestered at the center.²³ They are also the reason for the phase inversion of the emulsion.

2.2. Effects of Surfactant Concentrations on the Stability of Emulsion. Surfactant acts as an emulsifier in the process of emulsification and the concentration of the surfactant is directly related to the emulsification and stability of the emulsion. To investigate the effect of the surfactant concentration on the stability of emulsion, stability experiments at different surfactant concentrations were carried out. The change of the water-separation rate of the ASP composition in the range of 0.05–0.4 wt % of the surfactant concentration was analyzed. As shown in Figure 5, when the surfactant concentration was lower than 0.3 wt %, the area of the “low” water-separation rate of the heatmap increased with the surfactant concentration. When the surfactant concentration increased to 0.3 wt % and above, the area of the “low” water-separation rate of the heatmap decreased. Therefore, the optimal surfactant concentration for the best emulsion stability is 0.3 wt %.

It can be seen from Figure 6 that the performance of emulsion stability with the increase of the surfactant concentration was first increased and then decreased. The emulsion stability increased with the increase of surfactant concentration when the surfactant concentration was less than 0.3 wt %. The reason for enhanced stability of the emulsion is that the number and density of surfactant molecules adsorbed on the oil–water interface increase and the arrangement of surfactant molecules becomes tighter with the increase of the surfactant molecules, thus the strength and thickness of the oil–water interface film increase. However, there was a tendency for the stability of the emulsion to decrease when

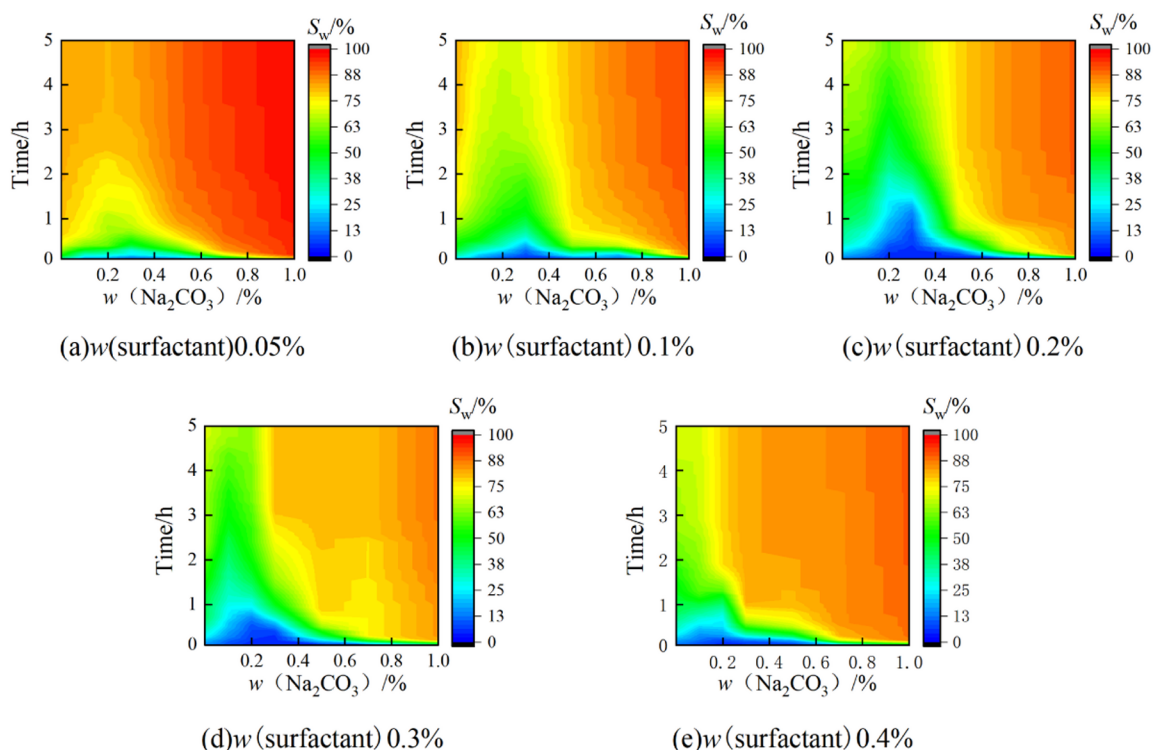


Figure 5. The water-separation rate of the emulsion in different surfactant concentration systems. The degree of separation of water from the emulsion is indicated by the difference in color. Blue indicates a lower water-separation rate, while red indicates a higher water-separation rate. [From (a–e), the surfactant concentration increases from 0.05 to 0.4 wt %.]

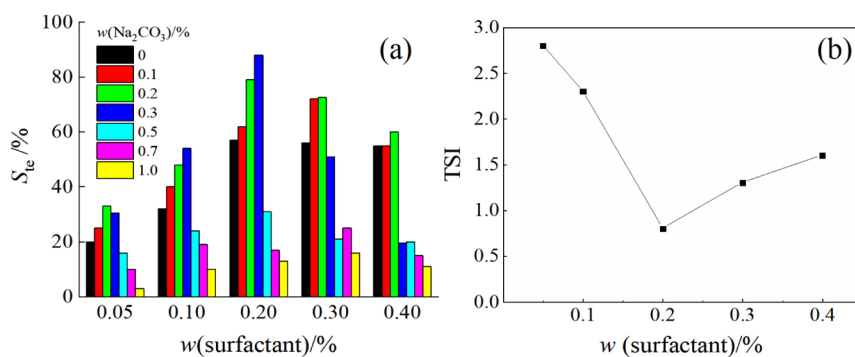


Figure 6. (a) Stability index of the emulsion at different surfactant concentrations. (b) TSI of emulsion at different surfactant concentrations (the alkali concentration is 0.3 wt %).

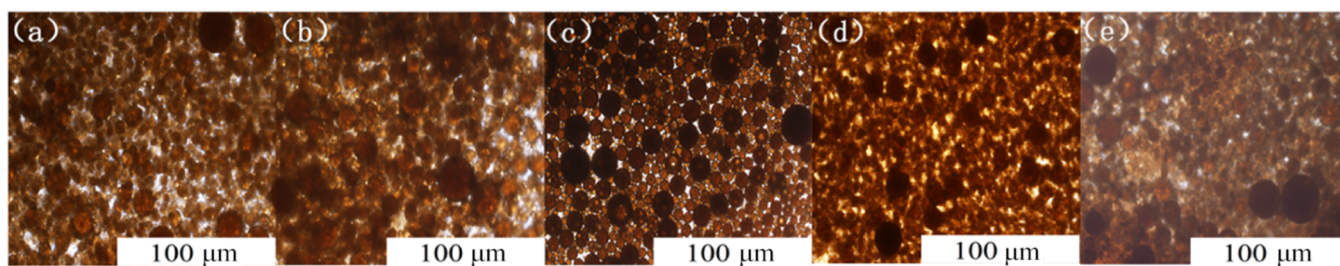


Figure 7. Microscopic images of emulsions with different surfactant concentrations. [From (a–e), the surfactant concentration increases from 0.05 to 0.4 wt %.]

the concentration of the surfactant increased continually. This is because that the surfactant molecules in the bulk phase can form micelles, which have a “solubilization” effect on the surfactant molecules on the interface after the concentration of surfactant reaches critical micelle concentration.²⁴ The effect of

micelle solubilization is not conducive to adsorption of surfactant molecules on the interface, resulting in a decline in the strength of the interface film.

As shown in Figure 7, the emulsion drop size became smaller and emulsion drop distribution became more uniform with the

increase of the surfactant concentration. The drop size distribution of the emulsion was relatively more uniform and the drop size was smaller when the surfactant concentration was 0.2 wt %, showing the law of the water-separation rate of the emulsion coincides with the results of the emulsion drop size and the emulsion drop distribution.

2.3. Effects of NaCl on the Stability of Emulsion. To determine the effect of the NaCl content on the stability of crude oil emulsion, experiments were performed on the samples prepared at different NaCl contents. Table 1 shows

Table 1. Variation of the Water-Separation Rate of Emulsion with Different NaCl Contents

NaCl content (wt %)	water-separation rate at different time					
	time					
	0.5 h	1 h	2 h	3 h	4 h	5 h
0.1	5.4	8.5	14.1	17.5	19.7	23.6
0.3	3.6	5.7	9.6	12.5	14.1	16.8
0.5	2.8	4.3	7.7	11.0	12.4	13.4
0.8	4.6	6.7	11.3	14.9	18.2	21.4
1.0	5.7	8.8	14.8	18.2	21.3	25.9
1.2	6.6	9.3	16.4	19.6	24.4	27.2
1.5	7.2	12.3	17.4	22.3	28.7	34.6

the variation of the water-separation rate of the emulsion with time at different NaCl contents. It can be observed that the water-separation rate of the emulsion first decreased and then increased with the increase of the NaCl content. The optimal salinity of emulsion stability observed was 0.5 wt % in this study. The increase of the water-separation rate at low NaCl contents may be because of the effect of the electrolyte. The electrolyte promotes the distribution of the surfactant from the water phase to the interface phase, so that the surfactant is easily adsorbed on the oil–water interface, which reduces the oil–water IFT, and is beneficial to the stability of the crude oil emulsion. As evident from Figure 8, an increase in the water-separation rate of the emulsion was observed with an increase of the NaCl contents. When the NaCl content is higher, the charge density of the oil–water interface and the thickness of the double layer of the emulsion drop are reduced because of the ionic effect. As a result, the strength of the interface film is reduced and the repulsive force between the droplets is weakened, thus leading to the coalescence of the drops.

2.4. Rheology Study. The rheology study demonstrates the nature of the fluid, which is important in the stability

analysis of emulsions. The viscosity of emulsion is a function of shear rate and is closely related to the concentration of the surfactant and alkali. To investigate the effect of concentration of the surfactant and alkali on viscosity of the emulsion, a rheological behavior study was carried out at different surfactant and alkali concentrations. Figure 9a,b shows the dependence of apparent viscosity on the shear rate with different surfactant and alkali concentrations. A pseudoplastic behavior was shown with increase of the shear rate. The viscosity decreased with the increase of the shear rate, which is of great significance in oilfield applications.²⁵ The reduction of viscosity with an increase in the shear rate may be attributed to disentanglement of emulsion droplets and their arrangement along a flow streamline.²⁶ Other reasons for reduction in viscosity may be because of the thermodynamic movement of emulsion drops and the rupture of the base fluid structure of emulsion. An increase from 6.1 to 7.6 mPa·s was observed when the surfactant concentration increased from 0.1 to 0.3 wt %. As the alkali concentration increased from 0.1 to 0.3 wt %, the viscosity also increased from 5.7 to 7.6 mPa·s. The increase in viscosity was because of a double layer formed around the emulsion drop.²⁷ The viscosity increase may also be because of the emulsion drop interaction. The interaction between the emulsion drops may be caused by an increase in the number of emulsion drops and a decrease in the size of the emulsion drops with the increase of surfactant and alkali concentrations. A decrease in the viscosity of the emulsion was observed when the surfactant concentration was 0.4 wt % and the alkali concentration was 0.5 wt %, which was related to the formation of micelles and the effect of the electrolyte on double layer.

2.5. Influence of Emulsion Stability on EOR. Based on the stability analysis of the emulsion in the first three sections, the chemical-flooding experiments were carried out. The purpose of this experiment was to investigate the influence of the emulsion stability on EOR. The flooding experiments were performed on sandstone cores by injection of chemical-displacing fluids with 0.5 wt % NaCl to investigate the effect of the alkali and surfactant concentrations on EOR at optimal salinity. Figure 9 shows the different pressure response of chemical-displacing fluids injected into the sandstone cores. The displacement pressure was relatively higher during the process of entire chemical-displacing fluid injection in a ternary system with good emulsion stability. The pressure curve increased simultaneously with the increase of pore volume (PV) of displacing fluids injected. It was found that the produced fluid has been emulsified when the pressure

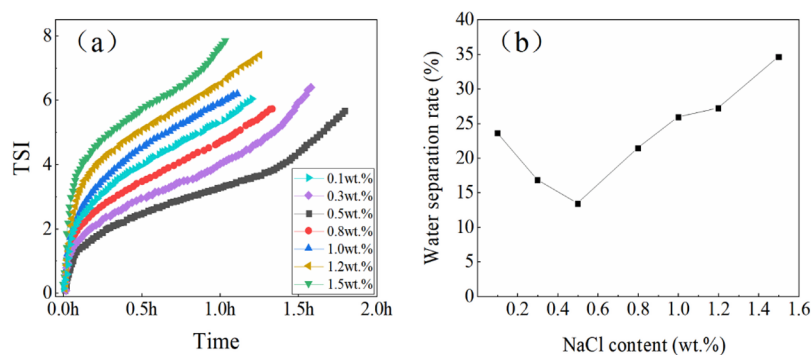


Figure 8. (a) TSI curves of emulsions with different NaCl contents. (b) Water-separation rate curve of emulsion with different NaCl contents after 5 h.

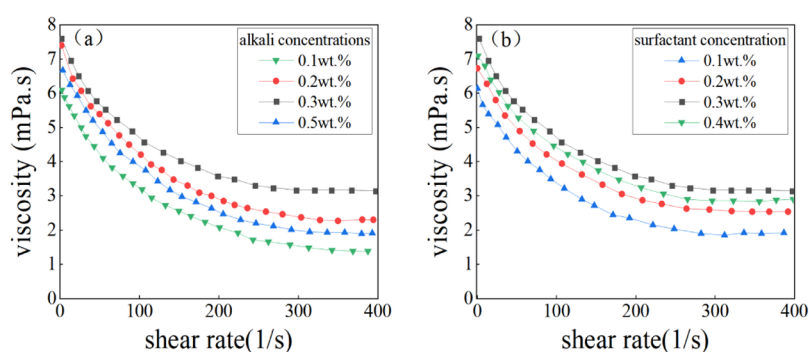


Figure 9. (a) Viscosity variation of emulsions with the shear rate at different alkali concentrations (0.1, 0.2, 0.3, and 0.5 wt %). (b) Viscosity variation of emulsions with the shear rate at different surfactant concentrations (0.1, 0.2, 0.3, and 0.4 wt %). Emulsions show pseudoplastic nature with an increase of the shear rate.

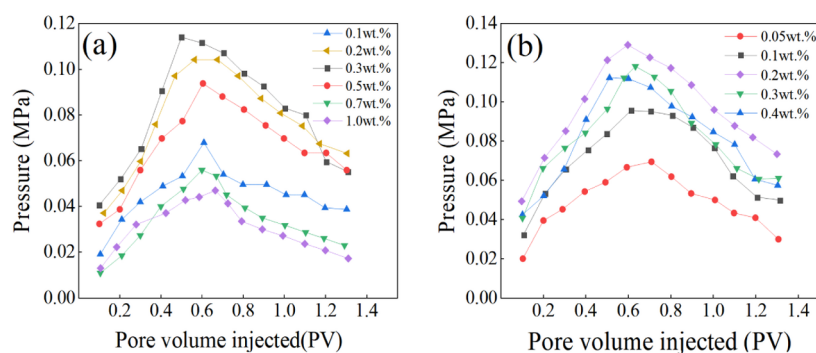


Figure 10. The injection pressure changes as the PV of displacing fluids injected increases. (a,b) Response to pressure at different alkali and surfactant concentrations, respectively.

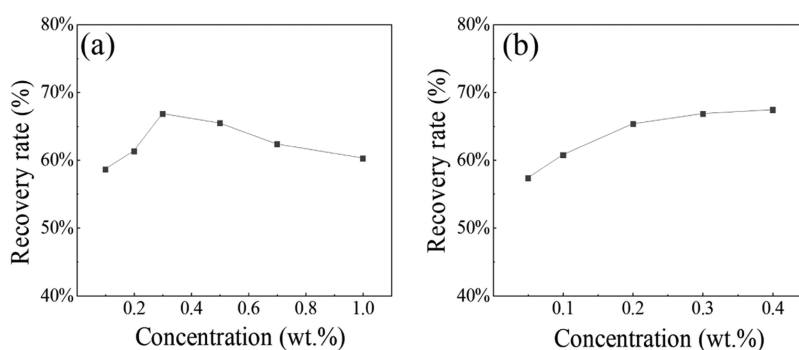


Figure 11. Recovery rate at different alkali (a) and surfactant (b) concentrations.

Table 2. Ionic Composition and Concentration of Experimental Water

ion type	cation (mg/L)				anion (mg/L)			total salinity
	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	HCO ₃ ⁻	CO ₃ ²⁻	Cl ⁻	
salinity	2669.3	21.5	14.1	2.4	1026.8	141.1	1547.2	5458.1

gradually increased. This phenomenon fully illustrates that the emulsified emulsion drops caused an increase in pressure of the migration in the porous medium. The sandstone core permeabilities were roughly in the same range. However, the system with good emulsion stability had higher pressure compared with other systems. This was because the system with good emulsion stability played a more obvious control and displacement role in the core, thus the displacement pressure was increased. In addition, the results of the chemical-flooding recovery rate were also consistent with the results of emulsion stability. The recovery rates at different concen-

trations of alkali and surfactant are shown in Figure 10. Both experimental results fully illustrated that the concentrations of alkali and surfactant had an effect on the stability of the emulsion, which in turn had an indirect effect on the recovery rate (Figure 11).

3. EXPERIMENTAL SECTION

3.1. Materials. Partially hydrolyzed poly-acrylamide (HPAM) was obtained from PetroChina Daqing Refining and Chemical Company. It is a water-soluble polymer with a bulk density of 900–1200 kg/m³. HPAM has a high molecular

weight of 1.9×10^7 g/mol with 20.2% hydrolysis. An anionic surfactant, which is named BHS-01A was used in the experiment, it was provided by Dagang Oilfield, its effective content is 40%. Analytical pure sodium carbonate (Na_2CO_3) with an effective content of 98% was used as the alkali in the ASP composite system. The oil mixed with kerosene and dehydrated crude oil from Dagang Oilfield was used as the oil phase in the emulsion preparation. The experimental water was obtained from an injection station of the Dagang Oilfield. Ionic composition and concentration of experimental water are listed in Table 2.

3.2. Emulsion Preparation. Prior to emulsification, the oil was heated at 58°C and shaken to ensure a homogeneous mixture for sampling. Emulsions were prepared by a volume of oil and a known composition of the ASP composite system aqueous solution, constantly 1/1 (v/v), a total of 60 mL placed in a 100 mL beaker, with an IKA T25 ultra TURRAX homogenizer (Germany) at 2000 rpm for 5 min.

3.3. Emulsion Properties. The type and microscopic morphology of emulsions were evaluated by using a dilution method. A drop of the emulsion was dropped on a glass slide covered with a small amount of experimental water to observe the dispersion of the diluted emulsion. The type of emulsion is characterized by the dispersion of emulsion droplets. Emulsion droplets that are quickly dispersed are considered as the oil-in-water (O/W) type, otherwise they are considered as the water-in-oil (W/O) type. In addition, the distribution and geometry of emulsion droplets were observed with an Olympus IX73 microscope (Japan) fitted with a sCMOS digital camera.

3.4. Stability Characterization. Two methods were used to characterize the stability of the emulsion, namely, stability performance index (S_{te}) and TSI.

3.4.1. Stability Performance Index. 20 mL of the prepared emulsion was poured into a colorimetric tube and placed in a 58°C constant temperature cabinet. The stability of emulsion was characterized by the relative volume of emulsion constituents, defined as the ratio of the water-separation volume to the total volume of water. The water-separation volume of the emulsion was recorded every 1 h to calculate the water-separation rate. The stability performance index (S_{te}) of different emulsion systems was measured using the water-separation rate (S_w) of emulsion at the same time interval. In this experiment, S_w of the emulsion is selected for 5 h, and S_{te} of the emulsion is calculated, where $S_{te} = 1 - S_w$. The higher the ratio, the greater is the emulsion stability.

3.4.2. Turbiscan Stability Index. Samples with different concentrations of surfactant and alkali were scanned for 1 h, respectively. The curve of the backscattered light along the height of the glass test bottle containing the sample was obtained at different scanning times (Turbiscan LAB expert, France), and the variation of backscattered light per unit time was calculated from the scan pattern. According to the principle of multiple light scattering, the absolute value of the amount of backscattered light change per unit time determines the stability of the emulsion. The smaller the absolute value of TSI, the more stable the emulsion.

3.5. Rheology Study. The apparent viscosity measurements of the emulsions at different surfactant and alkali concentrations were carried out in the shear rate range of $0\text{--}500\text{ s}^{-1}$ using an advanced TA Discovery HDR-3 Rheometer (USA). The rheology study gives information about the nature of fluid (shear thinning or shear thickening). The resistance produced between the rotator and the fluid is different at

different shear rates, so the viscosity value is also variable. The viscosity value is generated because of the rotation of the rotator in the cup.

3.6. Flooding Experiment. Core-flooding experiments were conducted using a high-temperature and -pressure core-flooding apparatus to investigate the effect of the alkali and surfactant concentrations on EOR at optimal salinity. The experimental flooding setup consists of a Teledyne ISCO displacement pump (USA) used in the flooding apparatus, a sand pack holder, and measuring cylinders for the collection of the produced fluids. The control and measuring system consists of a computer and two pressure sensors. A pressure of 500 psi was applied to saturate the sand pack holder with brine for the calculation of absolute permeability and then crude oil was flooded using a Teledyne ISCO pump to displace the water until no further water flowed out. The initial oil saturation (S_{oi}) and irreducible water saturation (S_{wirr}) were determined by the volume of water displaced. For oil recovery, first the core was flooded with brine at a constant flow rate of 0.4 mL/min until no further oil was produced. Second, chemical-flooding slug was injected followed by the injection of brine. All core-flooding experiments were carried out at 58°C which resembles the temperature of the reservoir.

4. CONCLUSIONS

The effects of the surfactant, alkali, and NaCl on the stability of emulsions were successfully investigated in this study.

- (1) In the ASP composition with the same surfactant concentration, the emulsion stability first increases and then decreases with the increase of alkali concentration. The reason for the increase in emulsion stability is that a little alkali can interact with acidic hydrogen in crude oil to produce interfacial active components. The reason for the decrease in the stability of the emulsion is that the addition of excessive alkali exhibits the effect of salinity. At the same time, the phase inversion of the emulsion occurs at a higher alkali concentration, reducing the stability of the emulsion.
- (2) In the ASP composition with the same alkali concentration, the emulsion stability first increases and then decreases with the increase of the surfactant concentration. The thickness and strength of the oil–water interface film increase as the surfactant concentration increases. The stability of the emulsion decreases when the surfactant concentration increases to 0.4 wt %. This is because of the formation of “micelles” in the bulk phase, which is not conducive to the adsorption of surfactant on the oil–water interface, leading to the destabilization of the emulsions.
- (3) The NaCl content has an important effect on the stability of the emulsion. NaCl can promote the adsorption of the surfactant on the oil–water interface, while a high concentration of NaCl reduces the charge density and thickness of the double layer of the emulsion drop.
- (4) The rheological behavior manifests a decrease of viscosity with the increase of the shear rate, exhibiting a pseudoplastic fluid behavior. The viscosity performance of the emulsion and stability performance at different alkali and surfactant concentrations are the same, that is, apparent viscosity increases and then

decreases with the increase of surfactant and alkali concentrations.

- (5) The core displacement experiment verifies that the ASP compound system with better emulsion stability has high oil recovery and high displacement pressure.

AUTHOR INFORMATION

Corresponding Author

Wei Zhang – School of Petroleum Engineering, Northeast Petroleum University, Daqing, Heilongjiang 163318, China; orcid.org/0000-0002-9015-3611; Email: 1023922552@qq.com

Authors

Jie Zhang – Production Technology Research Institute, PetroChina Dagang Oilfield, Tianjin 300450, China
Dangke Ge – Production Technology Research Institute, PetroChina Dagang Oilfield, Tianjin 300450, China
Xiaoyan Wang – Production Technology Research Institute, PetroChina Dagang Oilfield, Tianjin 300450, China
Wei Wang – Production Technology Research Institute, PetroChina Dagang Oilfield, Tianjin 300450, China
Dandan Cui – Production Technology Research Institute, PetroChina Dagang Oilfield, Tianjin 300450, China
Guangyu Yuan – Production Technology Research Institute, PetroChina Dagang Oilfield, Tianjin 300450, China
Keliang Wang – School of Petroleum Engineering, Northeast Petroleum University, Daqing, Heilongjiang 163318, China

Complete contact information is available at:

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Notes

The authors declare no competing financial interest.

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