The effects of oil displacement agents on the stability of water produced from ASP (alkaline/surfactant/polymer) flooding

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ABSTRACT

Alkaline/surfactant/polymer (ASP) flooding technology has been successfully used in Chinese oil fields, such as Daqing and Shengli. However, water produced from ASP flooding contains large quantities of residual chemicals (alkali, surfactant and polymer) making it a complex and stable emulsion system which is difficult to treat. The emulsion stability of water produced from ASP flooding was investigated by conducting settling experiments and measuring the oil–water interfacial properties. The experimental results showed that the addition of polymer (HPAM, hydrolyzed polyacrylamide) degrades the emulsion stability when its concentration is below 300 mg/L for the 1.2 × 107 MW polymer, and 800 mg/L for the 3.0 × 108 MW polymer. But it enhances the emulsion stability when polymer concentrations are above those levels. At low polymer concentrations, flocculation induced by the polymer on oil droplets in the produced water is the dominant factor, while at high polymer concentrations the produced water viscosity plays an important role in the emulsion stability. The adsorption of surfactant on the oil–water interface increases the zeta potentials and decreases interfacial tension, and thus remarkably enhances the emulsion stability. Furthermore, the emulsion stability is enhanced gradually with the increase of NaOH concentration up to 300 mg/L due to the increase of zeta potentials and decrease of interfacial tension, and then weakened with the further increase of NaOH concentration, which is attributed to the decreased strength of the interfacial film. A pilot experiment for the treatment of simulated water was done, and the result showed that the simulated produced water from ASP can be successfully treated by using a leaching solution of alkaline white mud.

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1. Introduction

In crude oil extraction, aqueous solutions containing large quantities of chemicals can be injected into the strata to drive the crude oil out of the ground. This process is often termed chemical flooding or tertiary oil extraction. In recent years, technologies for tertiary oil extraction have been relatively well developed. Alkaline-surfactant-polymer (ASP) flooding technology (injecting an aqueous solution containing alkali, surfactant and polymer) is an important technology in tertiary oil extraction, which has been found to enhance oil recovery by over 20% [1]. Therefore, this technology has been used in several different oil fields in China, such as Daqing and Shengli [2], to enhance oil recovery. However, the oil fields then face with new problems. Produced water is generated after dehydration of the produced liquid (a mixture of oil and water from the oil wells). And due to the use of alkali, surfactant and polymer in the injected aqueous solution in ASP flooding technology, the produced water from this process contains large numbers of residual chemicals. Thus the water produced from ASP flooding forms a complex and stable emulsion system [3] and it is more difficult to treat than that from water flooding.

In a series of articles, Deng and co-workers have investigated the properties of oil-in-water emulsions based on the Daqing crude oil [1,4–6]. Their interest has been focused on the stability of oil droplets in the produced water and treatment of the produced water using a novel crossflow oil–water separator. Furthermore, flocculation and demulsification have been conducted to remove the stable oil droplets in the produced water and Deng et al. found that demulsification was an effective method to accelerate oil–water separation for the produced water. All these studies have given us insight into the problems relating to emulsion stability and treatment of produced water from ASP flooding. However, the stabilization mechanism of produced water from ASP flooding has not been fully studied and to our knowledge, no effective technologies

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can meet the need for the treatment of produced water from ASP flooding system in oilfields.

It is now generally recognized that the zeta potentials, interfacial tension and interfacial rheology have effects on the stability of emulsions [7]. Thus, in this study, simulated produced water from ASP flooding was prepared and the effects of oil displacement agents (alkali, surfactant and polymer) on the emulsion stability was investigated in terms of oil–water separation of produced water, interfacial tension, interfacial rheology, zeta potential, bulk phase viscosity and sizes of oil droplets. Furthermore, we investigated treatment of the simulated produced water from ASP flooding using a leaching solution of alkaline white mud.

2. Materials and methods

2.1. Materials

The crude oil used in this study was obtained from the Shengli oilfield in China, with a water content of less than 0.5%, and a density of 865 kg/m³ and viscosity 50.38 mPa s, at 45 °C. The partly hydrolyzed polyacrylamide (HPAM) used as polymer was purchased from East Asiatic Company (Japan), with average molecular weights (MW) of $3.0 \times 10^6$ and $1.2 \times 10^7$, and a degree of hydrolysis of about 20–30%. The surfactant (petroleum sulphonate, WPS) was supplied by Shengli Engineering & Consulting Co., Ltd. The alkali used in this study was NaOH and of reagent grade. Other materials were all analytical reagents.

2.2. Preparation of simulated produced water

According to the underground water and discharge water qualities in the Shengli oilfield, mineral water was prepared firstly, with salt concentrations as follows (mg/L), NaCl 1600, NaHCO₃ 2600, KCl 1500, NO₃ 1200. The viscosity of the produced water was determined by NDJ-9S digital viscometer from Shanghai Balance Company (China). In the measurement, the rotor was a number one rotor, the shear rate was 60 rpm and the temperature of the produced water was kept at 45 °C constantly.

2.5. Oil–water interfacial tension and interfacial dilatational rheology measurement

Interfacial dilatational modulus ($\varepsilon$) is defined as follows:

$$\varepsilon = \frac{dy}{d \ln A},$$

where $y$ is interfacial tension and $A$ is the area of the interface. Interfacial dilatational modulus has both a real and imaginary component defined for an oscillating area as follows [10,11]:

$$\varepsilon = \varepsilon_d + i\omega\eta_d,$$

where $\varepsilon_d$ is the dilatational elasticity, $\omega$ is the frequency of the oscillations, and $\eta_d$ is the dilatational viscosity.

2.6. Determination of zeta potential of oil droplets

The zeta potential of oil droplets was measured by a Zeta plus Zeta apparatus (Shanghai, Zhongchen Company). First, 100 ml of the produced water was allowed to settle for 4 h at 45 °C in an air bath, and then 5 ml of water was taken from the beaker for zeta potential measurement.

2.7. Droplet size distribution analysis

The droplet size distribution for the produced water was measured by a laser diffraction method using a Mastersizer 2000 (Malvern Instruments Ltd., Worcestershire, UK). First, 500 ml of distilled water was added to a beaker, and heated up to 45 °C in a water bath. A proper volume of produced water containing oil droplets was then diluted to a droplet concentration of less than about 0.05 wt% (to eliminate multiple scattering effects), and gently stirred (to increase the homogeneity) prior to measurement. The initial size distribution was measured immediately after the produced water was prepared and the size distribution was measured again approximately 2 h after the preparation.

2.8. A leaching solution of alkaline white mud

Alkaline white mud, collected from the Shandong Aluminum Plant, was dried at 105 °C overnight, disaggregated, and ground to pass through a 200 mesh sieve for use. Chemical composition of the white mud was determined by X-ray fluorescence (ZSX Primus II, Rigaku Corporation) and the main metallic elements are calcium, magnesium, iron and aluminum. And we prepared a treatment agent (leaching solution of alkaline white mud) by the reaction of alkaline white mud and 1.5 mol/L dilute HCl solution and measured the solid content of the leaching solution (0.1027 g/ml, dosage of leaching solution is based on the solid content).
Experiments were conducted using a batch method by adding the leaching solution of white mud into 500-ml beakers containing 400 ml simulated water of desired concentrations (oil, alkaline surfactant and polymer) and pH 9.5. After addition of 10–20 ml leaching solution of white mud, the simulated water was stirred rapidly at 160 rpm for 1 min, followed by a slow stir phase at 40 rpm for 5 min, and then a 5 min settling time. The supernatant sample was extracted by a syringe from about 2 cm below the water surface for analysis of oil contents and COD.

3. Results and discussion

3.1. Effects of oil displacement agents on emulsion stability of simulated produced water

The emulsion stability of simulated produced water was assessed by measuring the oil concentrations after settling experiments as a function of concentrations. The oil concentration is an indicator of the emulsion stability of the simulated produced water.

3.1.1. Effects of HPAM concentration

The effects of HPAM concentrations on emulsion stability and viscosity of produced water were studied with the concentrations of NaOH and WPS at 800 mg/L and 600 mg/L, respectively, as shown in Fig. 1. HPAM is a high molecular weight polymer and can increase the viscosity of the produced water. The effect of low molecular weight (LMW) HPAM (3.0 × 10^6) on the viscosity is lower than that of high molecular weight (HMW) HPAM (1.2 × 10^7). It also can be observed that the oil concentrations of the produced water fall at first, and then go up as the HPAM concentrations increase. For HMW HPAM, Fig. 1(a) indicates that the oil concentration decreased from 510 mg/L to 486 mg/L when the HPAM concentration increased from 0 to 200 mg/L. The oil concentration then increased from 486 mg/L to 1072 mg/L when the HPAM concentration increased further from 200 to 1000 mg/L. For LMW HPAM, as shown in Fig. 1(b), the profile of oil concentrations exhibits a V-shape. When the HPAM concentration was 300 mg/L, the oil concentration reached a minimum value of 422 mg/L. When the HPAM concentration is less than 300 mg/L and 800 mg/L for HMW and LMW HPAM, respectively, the corresponding oil concentrations of produced water are less than that of 0 mg/L HPAM. These differences for HMW and LMW HPAM may be due to the difference of the produced water’s viscosities. For example, when the concentration of HMW HPAM is 500 mg/L, the viscosity is 9.9 mPa s, but the viscosity is only 1.7 mPa s when the concentration of LMW HPAM is 500 mg/L. According to the Stokes equation [12], the rising velocity of an oil droplet is in reverse proportion to the viscosity, therefore high viscosity can prevent oil–water separation. The results illustrate that HPAM degrades the emulsion stability when its concentration is low, but enhances the emulsion stability when its concentration is above 300 mg/L and 800 mg/L for HMW and LMW HPAM, respectively.

3.1.2. Effects of surfactant concentration

The effects of WPS on oil concentrations were investigated with the concentration of NaOH at 800 mg/L and HPAM (the following experiments only use LMW HPAM as material) at 200, 400 and 600 mg/L (Fig. 2). Oil concentrations of produced water increased with the increase of WPS concentration, namely the emulsion stability of the produced water is enhanced with the addition of WPS. When the concentrations of HPAM are 200, 400 and 600 mg/L, the effects of WPS on oil concentration are nearly the same. And when the concentration of WPS increases from 0 to 600 mg/L, the oil concentrations all increase by 160 mg/L for the three HPAM concentrations. The WPS surfactant can adsorb to the surface of the oil droplets, and its polar head groups extend into the water phase and the non-polar tails extend into the oil phase, so the oil–water interfacial properties may be changed [13–15]. The adsorption of WPS can enhance the emulsion stability of the produced water significantly.
3.1.3. Effects of NaOH concentration

Fig. 3 illustrates the effects of NaOH on oil concentrations of the produced water. The oil–water separation experiments were carried out with the concentrations of HPAM at 500 mg/L, and WPS at 200, 400 and 600 mg/L. It can be seen that the oil concentrations of the produced water increase initially when the NaOH concentration is increased from 0 to 300 mg/L. The oil concentrations then decrease with the further increase of NaOH concentration. When the concentration of WPS is 200 mg/L, the effect of NaOH on oil concentrations is somewhat different compared with the samples with 400 mg/L and 600 mg/L WPS. When the concentrations of NaOH increased from 0 mg/L to 300 mg/L, the increase of oil concentration is 26.6 mg/L, 74.7 mg/L and 79.8 mg/L for 200 mg/L, 400 mg/L and 600 mg/L WPS, respectively. The addition of NaOH can enhance the emulsion stability when its concentration is below 300 mg/L, but it decreases the emulsion stability when the NaOH concentration is above 300 mg/L.

3.2. Effects of oil displacement agents on oil–water interfacial properties

The emulsion stability of the produced water was determined by measuring the oil–water interfacial properties, such as interfacial tension (IFT) [16], strength of interfacial film [17–19], zeta potential [20,21] and steric stabilization [22,23]. And these parameters were affected by the residual oil displacement agents in the produced water. Therefore, we investigated the effects of the oil displacement agents on the interfacial properties to find the mechanisms of stability of the emulsions in the produced water from ASP flooding.

HPAM is a water soluble polymer, and HPAM molecules can diffuse to the oil–water interface from the aqueous phase, but the adsorption of HPAM is quite weak due to the slight interfacial activity of HPAM. Thus, HPAM concentrations effect on the oil–water interfacial properties insignificant, as shown in Figs. 4 and 5. The zeta potential increases slowly with the increase of HPAM concentration, and HPAM hardly has any effect on IFT and viscoelastic modulus (E). It can be seen from Fig. 6 that HPAM can promote the coalescence of oil droplets. The peak in the size distribution increased and shifted to larger sizes with the addition of HPAM. In addition, the tail of the drop size distribution became larger with the addition of HPAM. However, the floccu-
loration of HPAM decreased at higher HPAM concentrations, such as 600 and 1000 mg/L HPAM compared with 300 mg/L. When HPAM concentration is below 300 mg/L, the viscosity and zeta potential increase slightly, but the size of the oil droplets augments remarkably, therefore oil–water separation is accelerated and the emulsion stability is degraded with the HPAM concentration [16]. Yet at HPAM concentrations above 300 mg/L, the effects of viscosity increase gradually (Fig. 1(b)). The drainage velocity of the liquid film between adjoining oil droplets decreases due to the viscosity increase. Thus, the flocculation of HPAM decreases and the emulsion stability increases with the increasing HPAM concentration. Overall, the viscosity change of produced water due to HPAM is the main factor for the emulsion stability, while the changes in zeta potential, IFT and E are relatively insignificant. It may be related to the polymer depletion effect [24].

WPS is an anionic petroleum sulphonate surfactant and can be adsorbed to the surface of oil droplets with its non-polar tail attaching to oil phase and polar head group extending into the water phase, therefore the zeta potential increases significantly with the WPS content, as shown in Fig. 4. In addition, the interfacial properties have a great change due to the high interfacial activity of WPS. Fig. 5 shows that the IFT and elastic modulus, E, decrease with the WPS concentration. It was found that some interface active agents in crude oil, such as asphaltene, take part in the formation of the interfacial films on the crude oil–water interface, and the interfacial films possess a framework and a certain strength [25,26]. But with the addition of WPS, parts of the active agents are replaced by WPS, thus the original structure of interfacial film and the interaction force of interface molecules are changed, and IFT and E decrease. Though the addition of WPS decreases the strength of the oil–water interfacial films, lower IFT enhances the inherent stability of oil droplets and higher zeta potential increases the electrostatic repulsive force between the droplets, therefore the addition of WPS prevents their coalescence. The double electric layer effect is likely the most significant factor.

As shown in Fig. 6, the peak in the distribution decreased and shifted to smaller size when the produced water contained WPS. Thus, the emulsion stability of the produced water increases with the increase of WPS concentration.

The effect of NaOH on zeta potential is shown in Fig. 4. Zeta potential increases with NaOH concentration, but the increase is slight after NaOH concentration is above 300 mg/L. NaOH can react with acidic components of crude oil and form soap-like interfacial active components [27], that can be adsorbed to the oil–water interfacial film like the surfactant, so the zeta potential increases. But the quantity of acidic components is finite, therefore the increase of zeta potential is not obvious when NaOH concentration exceeds 300 mg/L. Also, the adsorption of soap can affect IFT and E, and the loss of interfacial active agents in the interfacial film by reacting with NaOH, so the change of interface structure is significant. As shown in Fig. 5, IFT and E both decrease with the increase of NaOH. At low NaOH concentrations (below 300 mg/L), the zeta potential increases gradually, the strength of interfacial film is comparatively high, and the low IFT enhance the stability of the oil droplet itself, thus the emulsion stability of the produced water increases with the NaOH content. At high NaOH concentrations (above 300 mg/L), the zeta potential is almost the same as that at low NaOH concentrations, but the strength of the interfacial film is greatly decreased, therefore the emulsion stability is decreased by degrees.

3.3. The pilot study for the treatment of simulated water

Given the high emulsion stability of water produced from ASP flooding, some conventional technologies have been found to be ineffective in the treatment of this water. There is a strong interaction between surfactant, polymer and hydroxides, hydroxy complex, which has been widely used in mineral floatation [28,29]. Therefore, the hydroxides and hydroxy complex produced from aqueous phase may have effect on the treatment of water produced from ASP flooding which contain some surfactant and HPAM. Then, we treated the simulated water produced by ASP flooding using a leaching solution of alkaline white mud at concentrations of alkali, surfactant and polymer of 2800, 500 and 600 mg/L, respectively. The experimental results (Fig. 7) showed that it is effective to treat the ASP flooding produced water by using a leaching solution of

Fig. 7. Effects of dosage of leaching solution of alkaline white mud on the treatment effect.

Fig. 8. The photograph of treatment effect (a) and the optical microscope image of precipitate (b).
alkaline white mud. When the dosage is above 8000 mg/L, the oil concentrations after treatment are less than 10 mg/L, which meets the demand of oilfield refill water and the COD is less than 150 mg/L. A photograph of treatment effect and an optical microscope image of the precipitate are shown in Fig. 8. We deduce that the leaching solution of alkaline white mud reacts with alkali and the other substances in the produced water including oil, WPS and HPAM, forming a co-precipitate with the reaction products. Therefore, the water and oil in the ASP flooding water are separated.

4. Conclusions

The wastewater produced from ASP flooding is a complex emulsion system with high emulsion stability, and it is difficult to treat. The effects of oil displacement agents on emulsion stability of water produced from ASP flooding are various. HPAM increased viscosity, promoted the coalescence of oil droplets, and affected the IFT and E slightly. With the increase of HPAM concentration, the emulsion stability of produced water initially decreased and then increased. At low HPAM concentration, the flocculation is the most important effect, while at high HPAM concentration; destabilization is inhibited due to the high viscosity. WPS decreases the IFT and E, and increases the zeta potentials greatly. The emulsion stability is enhanced gradually with the increase of surfactant concentration, and the double electric layer effect is the most significant factor. NaOH increases the zeta potential, and decreases IFT and E. When NaOH concentration is low, the double electric layer effect dominates, but when NaOH concentration is high, the strength of the interfacial film plays the leading role.

The leaching solution of alkaline white mud can be used for treatment of the wastewater from ASP flooding. Consequently, we are investigating the treatment mechanism of the leaching solution.

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