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Demulsification of Emulsions Exploited by Enhanced Oil Recovery System

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ABSTRACT

Experimental data are presented to show the influence of the enhanced oil recovery system's components, alkali, surfactant, and polymer, on the demulsification and light transmittance of the water separated from the emulsions. Among which, the effects of surfactants, polyoxyethylene (10) alkylphenol ether (OP-10) and sodium petroleum sulfonate (CY-1) on emulsion stability, are the strongest of any component, the effects of polymer, hydrolytic polyacrylamide (HPAM) 3530S, on emulsion stability are the weakest. This research also suggests a possible emulsion minimization approach, which could be implemented in refineries utilizing microwave radiation. Compared with conventional heating,

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microwave radiation can effectively enhance the demulsification rate by an order of magnitude and increase the light transmittance of the water separated from the emulsions. The demulsification efficiency may reach 100% in a very short time under microwave radiation.

Key Words: Water-in-oil emulsion; Demulsification; Alkali; Surfactant; Polymer; Microwave radiation.

INTRODUCTION

Alkali-surfactant-polymer has been successfully applied to certain oil fields as an enhanced oil recovery system. The drive fluid possesses higher viscosity, and further, it can enlarge, affecting volume during flooding oil. It can also decrease interfacial tension and enhance oil exploitation efficiency by about 15% to 20%. Furthermore, a great deal of water can be saved through the use of this system. However, the emulsions exploited by the enhanced oil recovery system are plagued by a subsequent difficulty in the demulsification of crude oil emulsions, connected to the oil recovery.[1] For economic and operational reasons, it is necessary to completely separate the water from the crude oil before transporting or refining it. Minimizing the water levels in the oil also reduces pipeline corrosion and maximizes pipeline usage.[2] Kim et al.[3] presented the demulsification of water-in-crude oil emulsions by a continuous electrostatic dehydrator. The separation rate of water from the simulated crude oil increased along with the applied field, frequency, demulsifier concentration, temperature, and contact time. As the applied field increased up to 2.5 kV/cm, the separated percentage increased up to 90%. Bailes et al.[4] presented the effect of air sparging on the electrical resolution of water-in-oil emulsions. A novel process was described in which the resolution of a stable water-in-oil emulsion was augmented by the simultaneous use of pulsed DC electric fields and mild bubbling of the emulsion with air. Emulsion resolution increased steadily with increasing air flow rate until a maximum value was reached after which, the extent of phase separation started to fall rapidly with further increase in the air flow rate. The electrical resolution was best (79% resolution) when the ratio of air to emulsion flow rate (volumetric) in the coalescer was about 120:1.

Microwave dielectric heating is quite different from conventional heating because it can dissipate heat inside the medium and rapidly raise the energy of the molecules. By means of microwave dielectric heating, more molecules become energized, resulting in superheating and higher reaction rates.[5,6]

The concept of microwave demulsification was first introduced by Klaila[7] and Wolf[8] in their patent applications. Klaila conducted several other field tests after his patent,[9] and the results were encouraging. Later, Fang et al.[10] presented a demulsification model for 1:1 and 3:7 water-in-oil systems under microwave radiation. The experimental results illustrated that the percentage of water separated from the emulsions was higher than 80% under certain conditions. Liu[11] compared microwave radiation demulsification with demulsification by gravity sediment, demulsification by chemicals, and demulsification by conventional heating. The microwave radiation process increased both the speed and efficiency of the demulsification. The present study set out to examine the effects of alkali, surfactant, and polymer on the demulsification and light transmittance of water separated from the emulsions. Our results undoubtedly proved that microwave radiation is a very effective method for demulsification of emulsion exploited by enhanced oil recovery system. Compared with conventional heating, microwave radiation can effectively enhance the demulsification rate by an order of magnitude and increase the light transmittance of the water separated from the emulsions.

EXPERIMENTAL

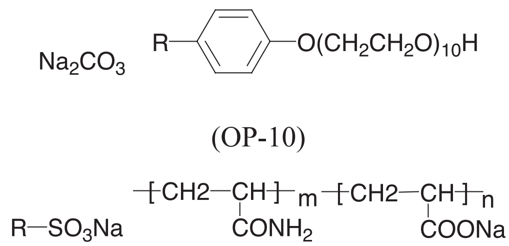
Model System

Oil Phase

The crude oil with water and gas removed came from the Shengli Oil Field in Shandong, China. The oil had an acid content of 2.98 mg of KOH/g, a density of 0.9518 g/mL at 25°C, a 32.5% (wt) resin composition, and a 4.2% (wt) asphaltene content. Kerosene was processed by flash chromatography to remove aromatic hydrocarbons. The model oil was made by mixing the crude oil and kerosene (1 wt:5 wt) to the make sample prepared and observed easier. Silica gel (diameter 120 to 200 mesh) was heated for 10 h at 110°C in a muffled oven, then cooled in a desiccator.

Aqueous Phase

The sodium carbonate used in this study was an analytical-grade reagent. The nonionic surfactant, polyoxyethylene (10) alkylphenol ether (OP-10), was obtained from Shanghai Auxiliary Manufactory (Shanghai City, P.R. China). Hebei Shijiazhuang Xinji Petroleum Chemical Plant (Shijiazhuang City, P.R. China) provided the anion surfactant, sodium petroleum sulfonate (CY-1). The polymer, hydrolytic polyacrylamide (HPAM) 3530S, had a molecular weight of 1500 to 1800, a hydrolytic degree of 28% to 30%, and was acquired from Pfizer. The molecular structure of alkali-surfactant-polymer is:



(OP-10)

(CY-1) (HPAM)

Preparation of Emulsions

Emulsions were prepared by using 3.0000 g model oil and 2.0000 g flooding oil agent in a 10-mL test tube with a stopper (length 135 mm,

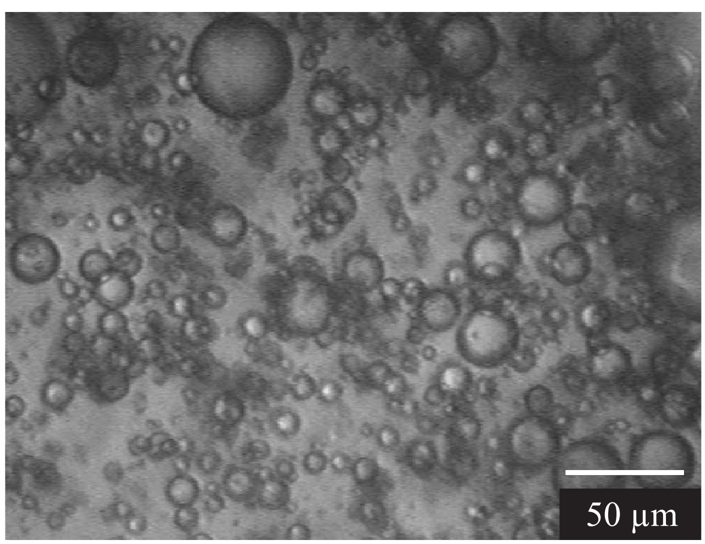


Figure 1. Optical microscopy photographs of the emulsion state. Experimental conditions: 1) The photograph of the emulsion was taken after 10 min with a camera installed on an optical microscope at a magnification of 400 at 15°C. 2) Flooding oil agent: 0.3 wt% Na_2CO_3, 0.04 wt% OP-10, 0.04 wt% CY-1, and 0.01 wt% HPAM.

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diameter 16 mm). The phases were mixed by a hand shaking motion for 200 times at a frequency of roughly 2 times/sec. The ratio of components of the emulsion was 3.0000 g model oil:0.0020 to 0.0120 g agent:1.9880 to 1.9980 g water. The photograph of the emulsion was taken after 10 min with a camera installed on an optical microscope at a magnification of 400 at 15°C.

Demulsification

The demulsification experiments were performed by using either oil-bath heating at 90°C or microwave irradiation (850 w) at 2450 MHz. The demulsification efficiency was evaluated by measuring the volume of water separated from the emulsions as a function of time. The amount of resolved water is the most appropriate gauge of demulsification in water-in-crude oil emulsions because coalescence of the droplet phase is the limiting step in the demulsification process.[12,13] The data reported for the experiments are an average over five runs.

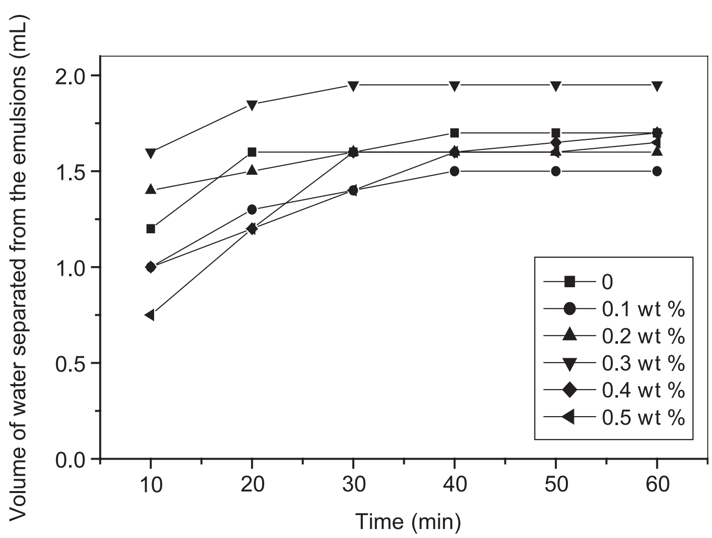

Figure 2. Effect of Na_2CO_3 on demulsification with conventional heating. Experimental conditions: 1) Demulsification was carried out in an oil-bath at 90°C. 2) Concentration of OP-10 was 0.04 wt%, concentration of CY-1 was 0.04 wt%, and concentration of HPAM was 0.02 wt%.

Measurement of Temperature

The temperature was measured by a mercury thermometer. The mercury thermometer was inserted in the under part of the samples promptly when the samples were taken out of an oil-bath or a microwave oven.

Measurement of Light Transmittance

The samples for measuring the light transmittance of the water separated from the emulsions were prepared by using either oil-bath heating for 60 min at 90°C followed by sedimentation for 30 min at room temperature or microwave irradiation (850 w) for 210 sec at 2450 MHz followed by sedimentation for 86 min at room temperature. The water separated from the emulsion was removed by an injector. The light transmittance of the water was measured by a model 200-10 spectrophotometer (Hitachi Corporation, Japan) with a double-beam light path at 620 nm, using distilled water as a reference. The light transmittance of distilled water was assumed to be 100%. The data reported for the experiments are an average over five runs.

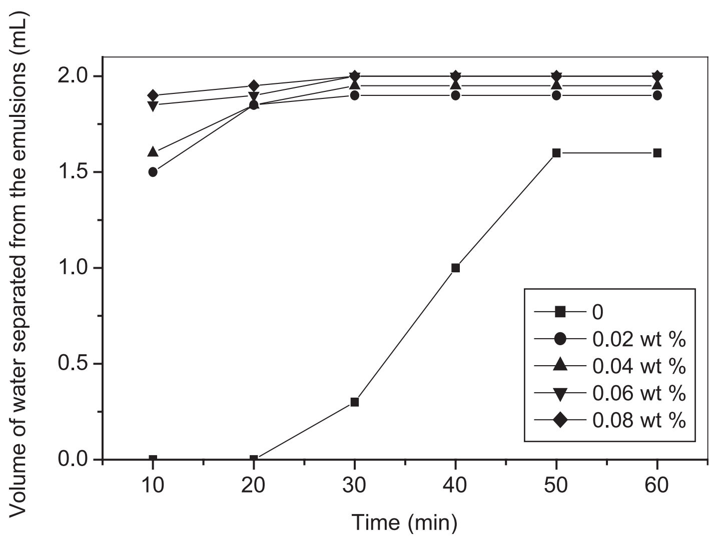

Figure 3. Effect of OP-10 on demulsification with conventional heating. Experimental conditions: 1) Demulsification was carried out in an oil-bath at 90°C. 2) Concentration of Na_2CO_3 was 0.3 wt%, concentration of CY-1 was 0.04 wt%, and concentration of HPAM was 0.02 wt%.

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RESULTS AND DISCUSSION

The emulsion state is shown in Figure 1. It is proven, by using methylene blue, that alkali-surfactant-polymer stabilizes water-in-oil emulsions.

Demulsification with Conventional Heating

The results of the demulsification with conventional heating are plotted in Figures 2–5.

Figure 2 illustrates that the existence of sodium carbonate is advantageous to the stabilization of emulsions when the concentration of sodium carbonate is below 0.2 wt%. This is because of the fact that sodium carbonate quickly moves to the crude oil–water interface and rapidly reacts with the petroleum acids in the crude oil to produce surface-active substances after the flooding oil agent contacts the oil phase. The surface-active substances can delay the coalescence between droplets to stabilize the emulsions. This factor dominates others, affecting the stability of the emulsions when the concentration of sodium carbonate is below 0.2 wt%. However, the electrolyte, excess sodium carbonate, plays a key role in

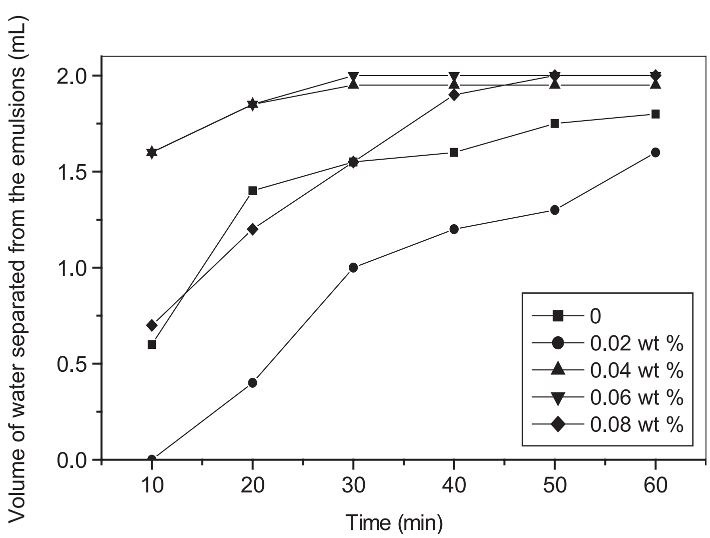

Figure 4. Effect of CY-1 on demulsification with conventional heating. Experimental conditions: 1) Demulsification was carried out in an oil-bath at 90°C. 2) Concentration of Na_2CO_3 was 0.3 wt%, concentration of OP-10 was 0.04 wt%, and concentration of HPAM was 0.02 wt%.

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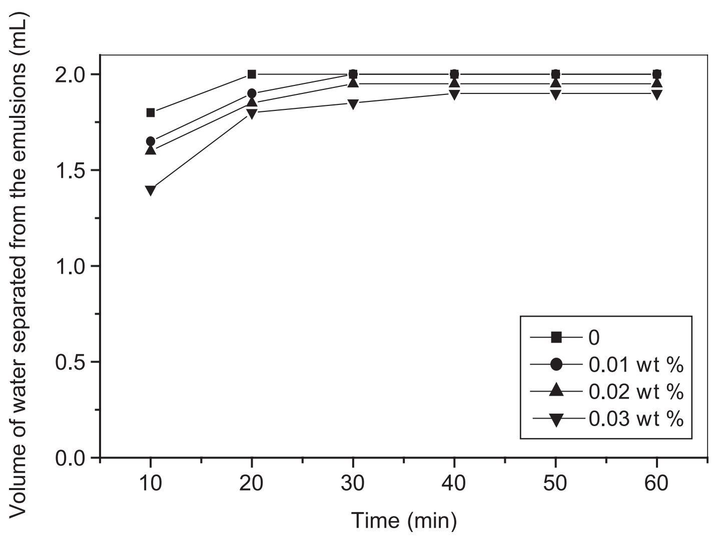

Figure 5. Effect of HPAM on demulsification with conventional heating. Experimental conditions: 1) Demulsification was carried out in an oil-bath at 90°C. 2) Concentration of Na_2CO_3 was 0.3 wt%, concentration of OP-10 was 0.04 wt%, and concentration of CY-1 was 0.04 wt%.

affecting the stability of the emulsions when the concentration of sodium carbonate is increased to 0.3 wt%. The electrolyte can destroy the double-charge layers of the emulsions to enhance the demulsification efficiency. When the concentration of sodium carbonate is increased to 0.4 wt% or higher, sodium carbonate can promote anion surfactants to enter the oil phase from the aqueous phase.[14] This effect will be advantageous for the stabilization of water-in-oil emulsions.

Table 1. Effect of Na_2CO_3 on demulsification under microwave radiation.

Time (sec)	Volume of water separated from the emulsions (mL)					
	0	0.1 wt%	0.2 wt%	0.3 wt%	0.4 wt%	0.5 wt%
210	1.80	1.70	1.85	2.00	1.90	1.90

Experimental conditions: 1) A domestic 2450-MHz microwave oven was used, and demulsification was carried out under microwave irradiation (850 w). 2) Concentration of OP-10 was 0.04 wt%, concentration of CY-1 was 0.04 wt%, and concentration of HPAM was 0.02 wt%.

Table 2. Effect of OP-10 on demulsification under microwave radiation.

Time (sec)	Volume of water separated from the emulsions (mL)				
	0	0.02 wt%	0.04 wt%	0.06 wt%	0.08 wt%
210	1.70	2.00	2.00	2.00	2.00

Experimental conditions: 1) A domestic 2450-MHz microwave oven was used, and demulsification was carried out under microwave irradiation (850 w). 2) Concentration of Na_2CO_3 was 0.3 wt%, concentration of CY-1 was 0.04 wt%, and concentration of HPAM was 0.02 wt%.

Figure 3 shows that the demulsification efficiency increases significantly when a small quantity of OP-10 (0.02 wt%) exists in the system. The volume of water separated from the emulsions increases from 0 to 1.50 mL in an oil-bath, heating at 90°C for 10 min. However, increasing the concentration of OP-10 from 0.02 wt% to 0.08 wt%, only increased the volume of water separated from the emulsions from 1.50 to 1.90 mL. This is due to the fact that OP-10 can stabilize oil-in-water emulsions, so it will reverse water-in-oil emulsions.

The demulsification is most difficult when the concentration of CY-1 is 0.02 wt%, giving a volume of water separated from the emulsions of 1.60 mL in an oil-bath, heating at 90°C for 60 min. The demulsification efficiency increases with the concentration of CY-1 after the concentration of CY-1 reaches 0.04 wt%. The content of CY-1 increases in the aqueous phase with the concentration of CY-1. It is believed that the excess CY-1, as an electrolyte, can destroy the double-charge layers of the emulsions, reducing the repulsion between water droplets, enhancing the demulsification efficiency.

It is evident that the volume of water separated from the emulsions decreases with the concentration of HPAM. HPAM can increase the viscosity

Table 3. Effect of CY-1 on demulsification under microwave radiation.

Time (sec)	Volume of water separated from the emulsions (mL)				
	0	0.02 wt%	0.04 wt%	0.06 wt%	0.08 wt%
210	1.85	1.85	2.00	2.00	2.00

Experimental conditions: 1) A domestic 2450-MHz microwave oven was used, and demulsification was carried out under microwave irradiation (850 w). 2) Concentration of Na_2CO_3 was 0.3 wt%, concentration of OP-10 was 0.04 wt%, and concentration of HPAM was 0.02 wt%.

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Table 4. Effect of HPAM on demulsification under microwave radiation.

Time (sec)	Volume of water separated from the emulsions (mL)			
	0	0.01 wt%	0.02 wt%	0.03 wt%
210	2.00	2.00	2.00	1.95

Experimental conditions: 1) A domestic 2450-MHz microwave oven was used, and demulsification was carried out under microwave irradiation (850 w). 2) Concentration of Na_2CO_3 was 0.3 wt%, concentration of OP-10 was 0.04 wt%, and concentration of CY-1 was 0.04 wt%.

of the system, decreasing the rate of film thinning between coalescing emulsion droplets. HPAM can also promote the viscoelastic character of the interfacial films. All these effects complicate the demulsification.

Comparing the data from different components of conventional heating, it is clear that the effects of surfactant on emulsion stability are the

Table 5. Sample temperature.

Flooding oil agent (wt/wt)	Sample temperature (°C)	
	Conventional heating	Microwave radiation
0.04% OP-10, 0.04% CY-1, and 0.02% HPAM	68.7	90
0.5% Na_2CO_3, 0.04% OP-10, 0.04% CY-1, and 0.02% HPAM	69	98.5
0.3% Na_2CO_3, 0.04% CY-1, and 0.02% HPAM	69	93.6
0.08% OP-10, 0.3% Na_2CO_3, 0.04% CY-1, and 0.02% HPAM	68.5	94.5
0.3% Na_2CO_3, 0.04% OP-10, and 0.02% HPAM	69	92
0.08% CY-1, 0.3% Na_2CO_3, 0.04% OP-10, and 0.02% HPAM	68.7	95.7
0.3% Na_2CO_3, 0.04% OP-10, and 0.04% CY-1	68.6	94.1
0.03% HPAM, 0.3% Na_2CO_3, 0.04% OP-10, and 0.04% CY-1	68.9	94

Experimental conditions: 1) The samples were prepared by using 3.0000 g model oil and 2.0000 g flooding oil agent. 2) The sample temperature was measured after 60 min or 210 sec in an oil-bath at 90°C or under microwave irradiation. 3) Room temperature 17°C.

strongest of any component. The volume of water separated from the emulsions increases from 0 to 1.90 mL or from 0 to 1.60 mL, with a change in concentration of OP-10 or CY-1, respectively, from 0 to 0.08 wt% in an oil-bath, heating at 90°C for 10 min. The effects of polymer on emulsion stability are the weakest. The volume of water separated from the emulsions only increases from 1.40 to 1.80 mL with a change in concentration of HPAM from 0 to 0.03 wt%.

Demulsification Under Microwave Radiation

The microwave field effects on demulsification are shown in Tables 1–4. Table 1 illustrates the effect of Na_2CO_3 on demulsification under microwave radiation. Compared to conventional heating, microwave dielectric heating can enhance the demulsification rate by an order of magnitude and increase the demulsification efficiency. For example, when the concentrations of Na_2CO_3 are 0.1 wt% or 0.4 wt%, the volumes of water separated from the emulsions using microwave radiation for 210 sec,

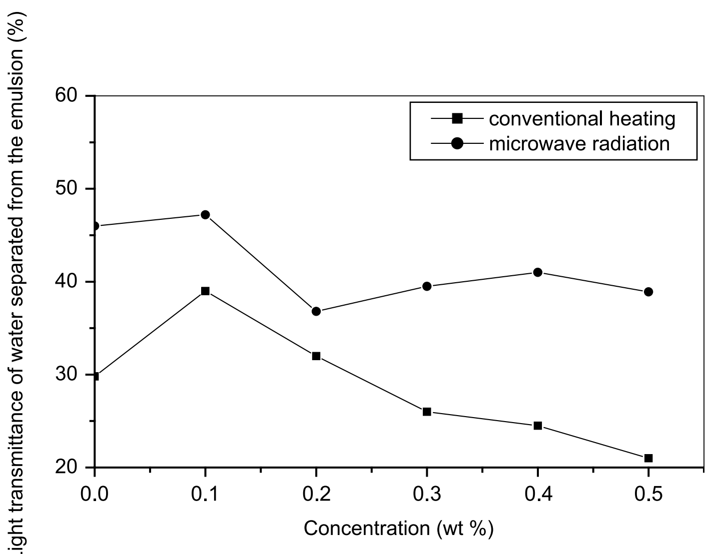

Figure 6. Effect of Na_2CO_3 on the light transmittance of water separated from the emulsion. Experimental conditions: 1) Demulsification was carried out in an oil-bath at 90°C for 60 min. 2) A domestic 2450-MHz microwave oven was used, and demulsification was carried out under microwave irradiation (850 w) for 210 sec. 3) Concentration of OP-10 was 0.04 wt%, concentration of CY-1 was 0.04 wt%, and concentration of HPAM was 0.02 wt%.

are 1.70 mL or 1.90 mL, respectively. However, the volumes of water separated from the emulsions are 1.50 mL and 1.70 mL using an oil-bath at 90°C for 60 min. Similar results are obtained when comparing the effect of OP-10, CY-1, and HPAM on demulsification by conventional heating with that of microwave radiation.

Table 5 shows temperatures of the samples. The sample temperatures reached 90°C or higher using microwave irradiation for 210 sec. However, the sample temperatures only reached about 69°C under oil-bath heating at 90°C for 60 min.

It is obvious that microwave radiation is a very effective method for demulsification of the emulsions exploited by the enhanced oil recovery system. On the one hand, the temperatures of the samples are higher under microwave radiation than those with conventional heating. On the other hand, it is believed that the electromagnetic field formed by the microwave can neutralize the zeta potential by disturbing the ordered arrangement of the electrical charges surrounding the water droplets.[8] Without the zeta

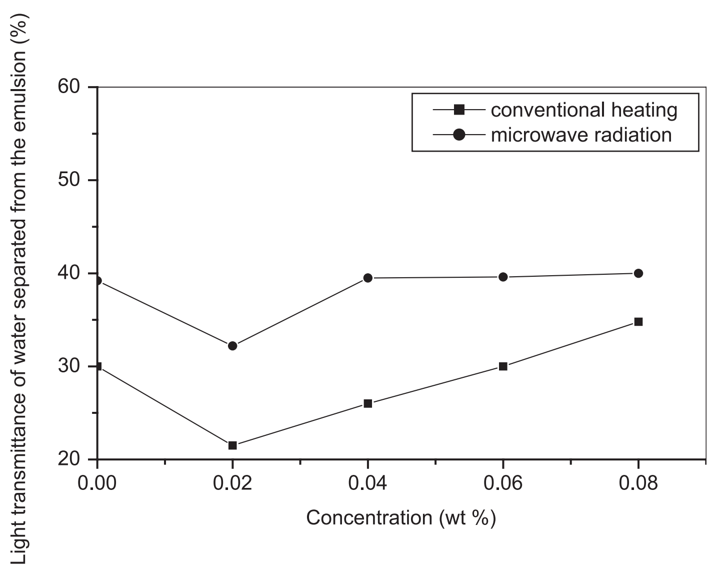

Figure 7. Effect of OP-10 on the light transmittance of water separated from the emulsion. Experimental conditions: 1) Demulsification was carried out in an oil-bath at 90°C for 60 min. 2) A domestic 2450-MHz microwave oven was used, and demulsification was carried out under microwave irradiation (850 w) for 210 sec. 3) Concentration of Na_2CO_3 was 0.3 wt%, concentration of CY-1 was 0.04 wt%, and concentration of HPAM was 0.02 wt%.

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potential, water droplets will move freely downward due to gravitational force. Furthermore, when the water droplets collide with each other in their downward motion, coalescence takes place, speeding up the emulsion separation. In addition, the dielectric constant and loss angle of water are greater than that of oil, causing water to absorb more energy than oil.[5] Water droplets expand under the internal pressure, making the interface film thinner. Consequently, the mechanical strength of the interface film becomes weaker and more easily broken. These effects all ease the demulsification process.

Investigations were also carried out on the light transmittance of the water separated from the emulsions, and the results obtained are shown in Figures 6–9. These experiments were performed both under conventional heating and microwave radiation.

Figure 6 illustrates that with conventional heating, the light transmittance of the water separated from the emulsions is highest (39.4%) at a Na_2CO_3 content of 0.1 wt%. After which, the value decreases with

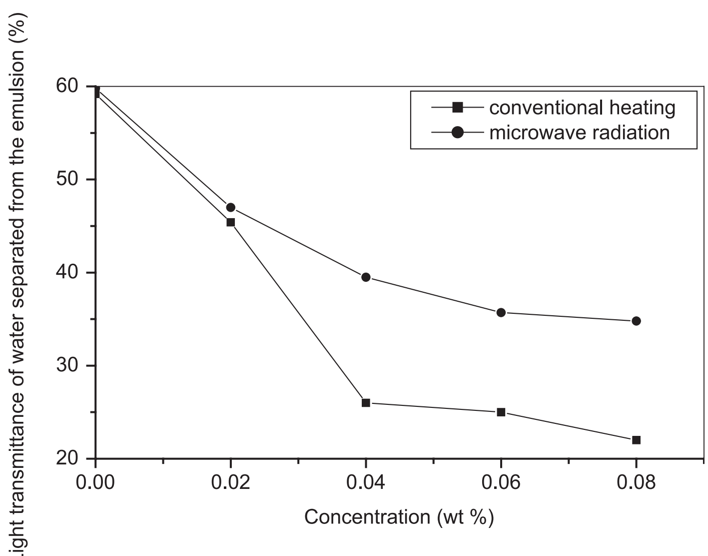

Figure 8. Effect of CY-1 on the light transmittance of water separated from the emulsion. Experimental conditions: 1) Demulsification was carried out in an oil-bath at 90°C for 60 min. 2) A domestic 2450-MHz microwave oven was used, and demulsification was carried out under microwave irradiation (850 w) for 210 sec. 3) Concentration of Na_2CO_3 was 0.3 wt%, concentration of OP-10 was 0.04 wt%, and concentration of HPAM was 0.02 wt%.

increasing Na_2CO_3 concentration. Under microwave radiation, the light transmittance of the water separated from the emulsions is the highest (47.2%) when the Na_2CO_3 content is 0.1 wt%, and lowest (36.8%) when the Na_2CO_3 content is 0.2 wt%.

Figure 7 demonstrates that the light transmittance of the water separated from the emulsions is the lowest under both conventional heating and microwave radiation (respectively, 21.5% and 32.2%) OP-10 content of 0.02 wt%. Then, the value increases with the concentration of OP-10. However, the light transmittance does not significantly change after the concentration of OP-10 reaches 0.03 wt% under microwave radiation.

Figure 8 depicts that the light transmittance of the water separated from the emulsions decreases with increasing CY-1 concentration. This can be explained by the low light transmittance of the CY-1 solution.

Figure 9 illustrates that the light transmittance of the water separated from the emulsions decreases with increasing HPAM concentration. When the concentration of HPAM is high, with a subsequent high viscosity of the system, more oil and HPAM remain in the water.

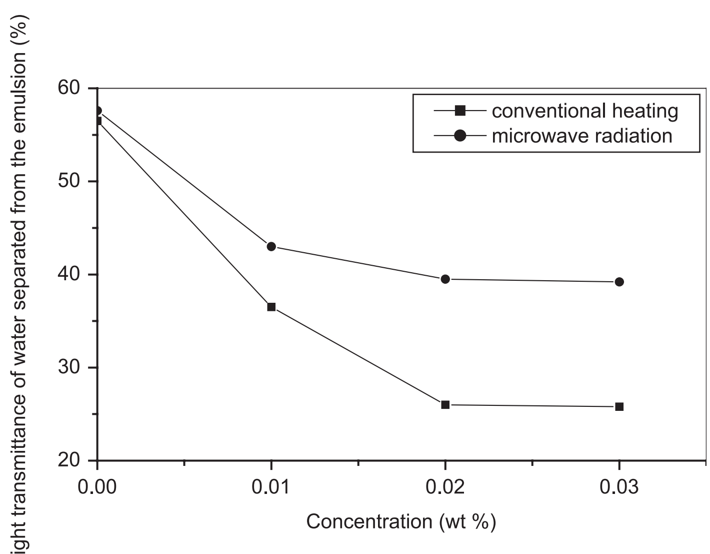

Figure 9. Effect of HPAM on the light transmittance of water separated from the emulsion. Experimental conditions: 1) Demulsification was carried out in an oil-bath at 90°C for 60 min. 2) A domestic 2450-MHz microwave oven was used, and demulsification was carried out under microwave irradiation (850 w) for 210 sec. 3) Concentration of Na_2CO_3 was 0.3 wt%, concentration of OP-10 was 0.04 wt%, and concentration of CY-1 was 0.04 wt%.

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It is obvious from Figures 6–9 that the light transmittances of the water separated from the emulsions under microwave radiation are higher than those under conventional heating. When water-in-oil emulsions are heated under microwave radiation, two phenomena simultaneously take place. The first phenomenon is the localized superheating of the emulsions in the presence of ions. The second is the reduction of viscosity.[11] The higher temperature and lower viscosity make the coalescence process easier, resulting in faster separation. Consequently, the light transmittance of the water separated from the emulsion is higher under microwave radiation than conventional heating. Hence, microwave demulsification can benefit oil recovery processes and reduce waste oil generation.

CONCLUSION

The results from this study support the idea that the demulsification of water-in-oil systems is affected by the alkali, surfactant, and polymer. The effects of the alkali, surfactant, and polymer on demulsification efficiency and the light transmittance of the water separated from the emulsions are different because their properties are very different. Among which, the effects of surfactants, polyoxyethylene (10) alkylphenol ether (OP-10) and sodium petroleum sulfonate (CY-1), on emulsion stability are the strongest of any component. The effects of polymer, hydrolytic polyacrylamide (HPAM) 3530S, on emulsion stability are the weakest. This research also suggests a possible emulsion minimization approach, which could be implemented in refineries utilizing microwave radiation. Compared with conventional heating, microwave radiation can effectively enhance the demulsification rate by an order of magnitude and increase the light transmittance of the water separated from the emulsions. The demulsification efficiency of the water-in-oil emulsions may quickly reach 100% under microwave radiation.

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