DOI 10.1007/s12182-013-0254-9

A study of the mechanism of enhancing oil recovery using supercritical carbon dioxide microemulsions

Dong Zhaoxia[1, 2, 3]*, Li Yi[1, 2], Lin Meiqin[1, 2, 3] and Li Mingyuan[1, 2, 3]

[1] Enhanced Oil Recovery Research Institute, China University of Petroleum, Beijing 102249, China
[2] Beijing Key Laboratory for Greenhouse Gas Storage and Enhanced Oil Recovery, Beijing 102249, China
[3] Key Laboratory of Enhanced Oil Recovery, CNPC, Beijing, 102249, China

Abstract: Supercritical carbon dioxide ($scCO_2$) microemulsion was formed by supercritical CO_2, H_2O, sodium bis(2-ethylhexyl) sulfosuccinate (AOT, surfactant) and C_2H_5OH (co-surfactant) under pressures higher than 8 MPa at 45 ºC. The fundamental characteristics of the $scCO_2$ microemulsion and the minimum miscibility pressure (MMP) with Daqing oil were investigated with a high-pressure falling sphere viscometer, a high-pressure interfacial tension meter, a PVT cell and a slim tube test. The mechanism of the $scCO_2$ microemulsion for enhancing oil recovery is discussed. The results showed that the viscosity and density of the $scCO_2$ microemulsion were higher than those of the $scCO_2$ fluid at the same pressure and temperature. The results of interfacial tension and slim tube tests indicated that the MMP of the $scCO_2$ microemulsion and crude oil was lower than that of the $scCO_2$ and crude oil at 45 ºC. It is the combined action of viscosity, density and MMP which made the oil recovery efficiency of the $scCO_2$ microemulsion higher than that of the $scCO_2$ fluid.

Key words: Supercritical carbon dioxide, microemulsion, MMP, enhancing oil recovery

1 Introduction

Supercritical CO_2 ($scCO_2$) is one of the environmentally friendly and nontoxic fluids. It has been widely used for many industry processes because of its low critical temperature, moderate critical pressure and low price (Eckert et al, 1996; Yu et al, 2006; Chattopadhyay and Gupta, 2001; Kalogiannis et al, 2005; Kikic et al, 1997; Matson et al, 1987; Zhao et al, 2011).

To realize a win-win situation of enhancing oil recovery and CO_2 emission reduction, injection of released CO_2 into reservoirs is becoming an important way of beneficial CO_2 utilization (Shen and Yang, 2006; Roper et al, 1992; Grigg and Siagian, 1998; Christensen et al, 1998; Langston et al, 2003). There are two types of CO_2 flooding: miscible flooding and immiscible flooding. For miscible flooding, there is a stable flooding zone formed, and the microscopic displacement efficiency is higher than 90%. Meanwhile for immiscible flooding, the displacement efficiency is low. Most reservoirs in China are continental depositional ones; the minimum miscibility pressure (MMP) for CO_2 flooding is higher than the formation fracture pressure, so miscible flooding cannot be achieved, which results in a low displacement efficiency. If the MMP may be controlled lower than the formation fracture pressure, CO_2 would be miscible with crude oil, and then the displacement efficiency would be significantly improved.

Nowadays, the commonly-used method to decrease MMP is to add hydrocarbon gases into the CO_2 (Bon and Sarma, 2005; Yuan et al, 2004). However, this method is difficult to apply in the reservoirs with few hydrocarbon gases. Moreover, the hydrocarbon gases injected into the reservoir may separate from CO_2, which makes the MMP of CO_2 flooding increase. This means that the miscible flooding cannot be achieved.

The $scCO_2$ reverse microemulsion method is a combination of supercritical technology and microemulsion technology (Liu et al, 2001; Luo et al, 2005; Zielinski et al, 1997; Zhang et al, 2009; Hutton et al, 1999; Heitz et al, 1997; Eastoe et al, 1996). Surfactant molecules are dissolved in the $scCO_2$ fluid, spontaneously forming nanoscale aggregates in the $scCO_2$ microemulsion is widely used in many industrial processes, such as chemical reaction, extraction and synthesis of nano-particles (Sun et al, 2001; Holmes et al, 1999; Kane et al, 2000; Ohde et al, 2005), but there is no reports about using $scCO_2$ microemulsion for enhancing oil recovery.

For the microemulsion system, $scCO_2$ is the continuous phase and the surfactant molecules are dissolved in the $scCO_2$ fluid which makes it is possible for the microemulsion

*Corresponding author. email: dzx@cup.edu.cn
Received September 19, 2012

to be miscible with crude oil under relatively low pressure and decrease the MMP of CO_2 and crude oil during CO_2 flooding. As a result a high efficiency of oil recovery could be obtained. It is known that the critical pressure of CO_2 is 7.32 MPa and the critical temperature is 31.1 ºC (Zhu and Xu, 2006). The experimental pressure in this work is higher than 9 MPa at 45 ºC, which means that the CO_2 is in a supercritical state. The MMP of $scCO_2$ microemulsion and Daqing crude oil is determined by a slim tube test and an interfacial tension meter, and the mechanism of $scCO_2$ microemulsion for enhancing oil recovery is also discussed.

2 Experimental

2.1 Materials and apparatus

Carbon dioxide (purity > 99.95wt%) was purchased from the Beijing Jinggao Gases Industry Company. Sodium bis(2-ethylhexyl) sulfosuccinate (AOT) (purity > 96wt%) was provided by the ACROS Company, USA. Absolute ethanol (C_2H_5OH) was of guaranteed reagent grade. The crude oil was taken from the Daqing Oilfield.

A high-pressure PVT cell and a high-pressure interfacial tension meter were provided by the Sanchez Technologie Company, France. A SYLDJ-2 high-temperature high-pressure falling sphere viscometer was made by the China University of Petroleum (East China).

2.2 Determination of the density of the $scCO_2$ microemulsion

For the $scCO_2$ microemulsion, the contents of both AOT and H_2O were quite low, so the effect of AOT and H_2O on the molar volume of the microemulsion was neglected. The molar percentages of CO_2 and C_2H_5OH in the microemulsion were 86.7% and 13.3%, respectively in all experiments. The surfactant AOT, C_2H_5OH, H_2O and CO_2 were all injected into the PVT cell and the weight of this system was 95.30 g. The pressure of the PVT cell was set at a specified value, then the volume of this system was recorded every 30 min. When the volume had been constant for 60 min (three successive recordings), the system was considered to have reached equilibrium, and the final volume was then recorded. The same steps were repeated at different pressures. The density values of the $scCO_2$ microemulsion at different pressures were calculated based on the volume data of the system. The experimental temperature was kept at 45 ºC.

2.3 Determination of the viscosity of the $scCO_2$ microemulsion

The viscosity of the $scCO_2$ microemulsion was measured with a falling sphere viscometer at 45 ºC. The viscometer was adapted to measure the viscosity of high-pressure fluids.

2.4 Determination of minimum miscibility pressure (MMP)

2.4.1 Interfacial tension measurement

The interfacial tension values between the $scCO_2$ microemulsion (and $scCO_2$) and crude oil were measured with a high-pressure interfacial tension meter at 45 ºC. The MMPs of the $scCO_2$ microemulsion (and $scCO_2$) and crude oil were calculated from interfacial tension values.

2.4.2 Slim tube test

A slim tube test is commonly used to estimate the MMP of a given injection solvent and reservoir. A schematic diagram of the slim tube test is shown in Fig. 1. The slim tube was a long coiled tube filled with 100 mesh fine sand. The tube was 19 m long and 6.2 mm in diameter, with a pore volume of 222.5 mL. The entry of the slim tube was connected to an intermediate container with a piston. The bottom of the container was connected to a constant speed pump. The gas in the intermediate container was pushed into the slim tube by the piston, which was driven by the pump. A back-pressure valve was installed at the exit end of the slim tube to control the pressure. The high-pressure parts of the apparatus were kept in a thermostat. A graduated cylinder was used to collect and accurately measure the volume of the crude oil displaced by the injection fluid. The slim tube was cleaned and then dried for 10 hours at 45 ºC. After being dried the slim tube was saturated with crude oil. Crude oil was injected at a rate of 0.2 mL/min. The minimum miscibility pressure of the $scCO_2$ system and crude oil was determined based on the oil recovery. The test was conducted at 45 ºC. An amount of AOT (mass percentage 5.03×10^{-3}%-15.16×10^{-3}%), C_2H_5OH

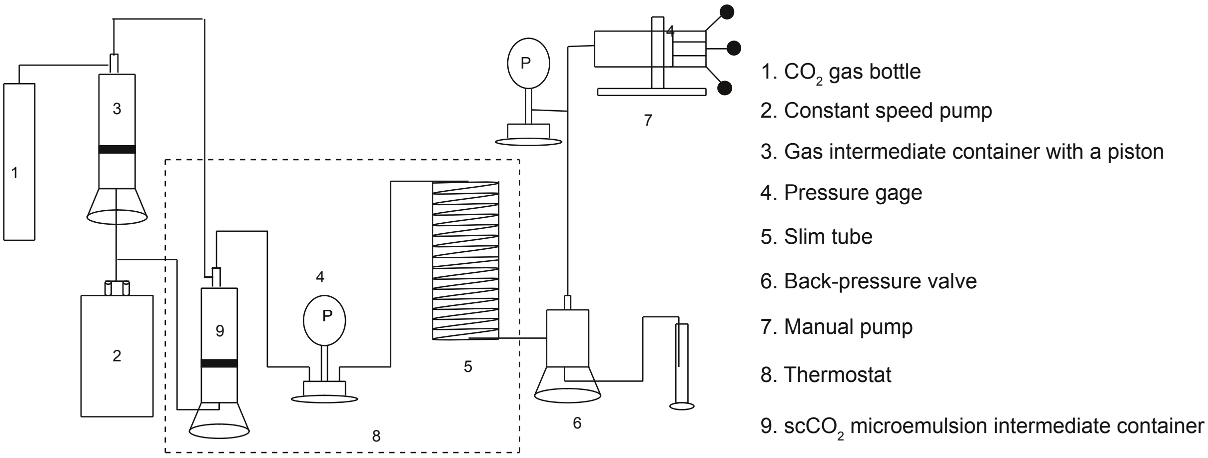


Fig. 1 The equipment for the slim tube test

(mass percentage 13.76%), H_2O (mass percentage 0.41%-1.61%) and CO_2 (mass percentage 86.23%) were added in $scCO_2$ microemulsion intermediate container and the pressure was set above 8 MPa. There was also a stirrer in the container to assist the formation of the microemulsion. After the $scCO_2$ microemulsion was formed, it was injected into the slim tube under a specified pressure.

3 Results and discussion

3.1 Formation of the $C_2H_5OH/scCO_2$ microemulsion

The initial conditions for preparing the $scCO_2$ microemulsion were as follows: the volume of the visualization PVT cell was set to 105.6 mL, the temperature was set at 45 ºC and the final pressure was 19.0 MPa. A specified amount of AOT, C_2H_5OH (mass percentage 13.76%), H_2O and CO_2 was added. Then the piston of the PVT cell was pushed to increase the pressure of the system up to 19.0 MPa. The transparent and homogeneous phases of the fluid would be formed which could be observed in the visualization PVT cell (Fig. 2).

Fig. 2 indicated that when the pressure was 3 MPa, the system containing CO_2, AOT, H_2O and C_2H_5OH was heterogeneous at 45 ºC. It can be seen that there were two phases in the system. This indicated that the system of CO_2, AOT, H_2O, and C_2H_5OH would not form a microemulsion at 45 ºC. When the pressure was 19.0 MPa, a transparent and homogeneous phase was observed in the PVT cell at 45 ºC. This indicated that a stable $scCO_2$ microemulsion was formed. Our previous research (Cui, 2009) suggested that the cloud point of the $scCO_2$ microemulsion was 8.0 MPa at 45 ºC, which indicated that the microemulsion would be formed when the pressure higher than 8.0 MPa. $ScCO_2$ was the continuous phase of the microemulsion and H_2O was the dispersed phase. Nanoscale aggregates of AOT molecules were formed in the $scCO_2$ microemulsion. In the $scCO_2$ phase, the oleophilic ends of AOT molecules expanded in the non-polar CO_2 phase, the polar ends aggregated and formed hydrophilic inner cores, and the water molecules were solubilizing in the cores, the AOT (surfactant) and C_2H_5OH (co-surfactant molecules) were adsorbed on the interface, forming a stable interfacial film. Therefore, an $scCO_2$ microemulsion was formed which was optically transparent and thermodynamically stable.

(a) Heterogeneous (3.0 MPa) (b) Homogeneous (19.0 MPa)

Fig. 2 The phase transition of supercritical CO_2 microemulsion

3.2 Viscosity of the $scCO_2$ microemulsion

The fluidity of the displacing phase decreases with an increase in the viscosity and the mobility ratio of the displacing fluid to crude oil thereby decreases, so fingering of the displacing phase may be reduced and the sweep efficiency improved; consequently oil recovery is enhanced. Therefore, if the viscosity of the $scCO_2$ microemulsion is higher than that of the $scCO_2$ fluid, this will improve the oil recovery of CO_2 flooding. The viscosities of the $scCO_2$ fluid and the $scCO_2$ microemulsion were measured and the results are shown in Table 1. Within the range of the experimental pressure, the viscosity of the $scCO_2$ microemulsion was 36%-49% higher than that of the $scCO_2$ fluid. This is beneficial to oil recovery. Table 1 also indicated that and the viscosity difference between the $scCO_2$ microemulsion and the $scCO_2$ fluid increased with pressure. This may be due to existence of a "micro-pool" structure; a substance structure similar to a high-molecular-weight compound dissolved in water. This structure may cause an increase in the flow resistance inside the fluid, which is indicated by the increase in the apparent viscosity. Meanwhile, the fluid was continuously compressed with increasing pressure, making the intermolecular distance decrease and the number of micelles in the unit fluid volume increase. The rise in the micelle concentration resulted in increasing bulk viscosity.

Table 1 The viscosities of the $scCO_2$ fluid and the $scCO_2$ microemulsion at different pressures and 45 ºC

Pressure MPa	Viscosity, mPa·s		Percentage increase %	Viscosity difference mPa·s
	$scCO_2$ fluid	$scCO_2$ microemulsion		
9.0	343.18	510.18	48.66	167.00
11.0	485.60	660.62	36.04	175.02
13.0	567.26	752.80	32.71	185.54
15.0	632.51	879.62	39.13	247.11

3.3 Density of the $scCO_2$ microemulsion

The densities of the $scCO_2$ fluid and the $scCO_2$ microemulsion at different pressures and 45 ºC are shown in Fig. 3. The density of the $scCO_2$ microemulsion was higher than that of the $scCO_2$ fluid at the same pressure. When the pressure was higher than 10.0 MPa, the density of the $scCO_2$ microemulsion was 0.8036 g/cm^3, almost twice that of the $scCO_2$ fluid at 0.4336 g/cm^3. This is mainly because of the addition of C_2H_5OH, AOT and H_2O. The density difference between these two systems was larger when the pressure was relatively low, and the difference decreased with an increase in pressure. The density of the $scCO_2$ microemulsion was the weighted average of all the substances in the system, meanwhile the proportion of CO_2 in the system was quite high, up to 86.7%. When the pressure increased, the density of CO_2 increased, meanwhile the densities separately of C_2H_5OH, AOT and H_2O were considered to be constant. Therefore, the density difference between the $scCO_2$ microemulsion and the $scCO_2$ fluid decreased continually with increasing pressure.

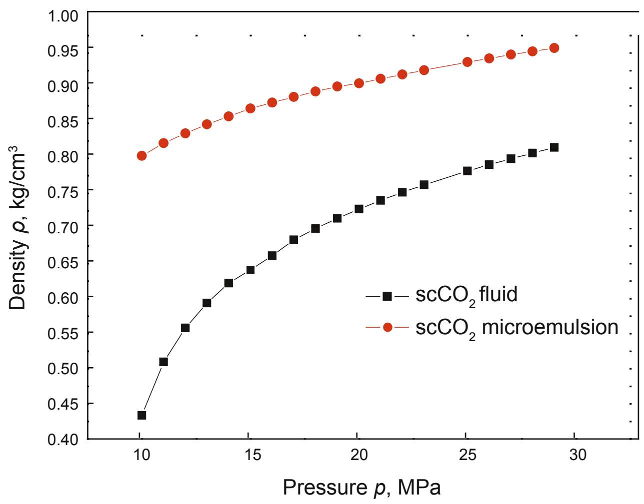


Fig.3 Density of the $scCO_2$ microemulsion and the $scCO_2$ fluid at 45 ºC

3.4 The MMP of the $scCO_2$ microemulsion and Daqing crude oil

The interfacial tension between the $scCO_2$ microemulsion (and the $scCO_2$ fluid) and the crude oil at 45 ºC was determined by the pendant drop method. The result is shown in Fig. 4. When CO_2 was completely miscible with the crude oil, the interfacial tension between them was zero, but at this time the value of the interfacial tension could not be measured with an IFT meter and only the gradual diffusion of crude oil into the CO_2 phase was observed. Therefore, the pressure at zero IFT was obtained by an extrapolation method, namely the minimum miscibility pressure (MMP). The images of the miscible phase of the $scCO_2$ fluid/the $scCO_2$ microemulsion with Daqing crude oil are shown in Figs. 5 and 6.

The interfacial tension between the $scCO_2$ and the crude oil was linear with pressure. A fitting expression, σ=17.92–0.73p, with a correlation coefficient of 0.9942 was obtained and used to describe the relationship between the interfacial tension and the pressure for the $scCO_2$ and

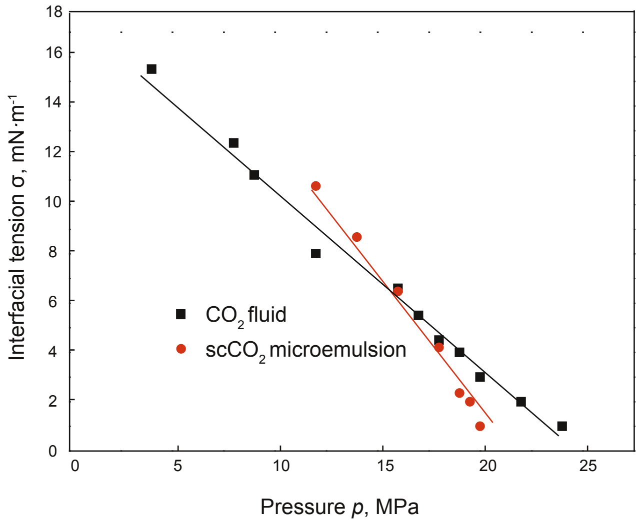


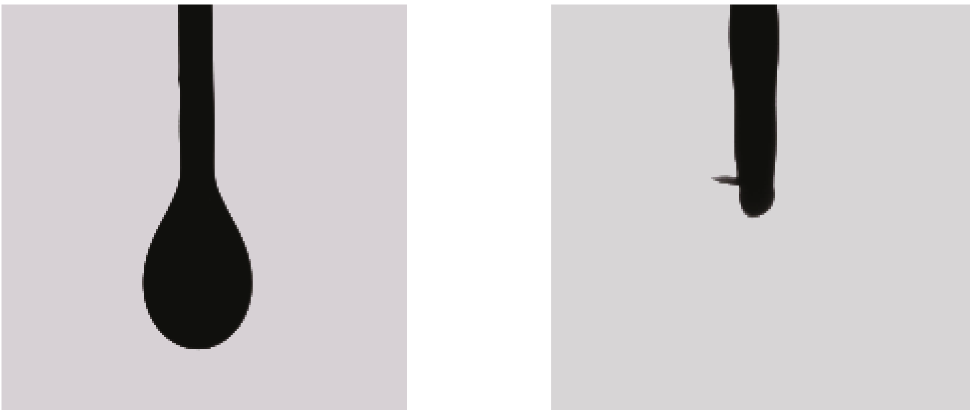
Fig. 4 The interface tension between CO_2 and crude oil versus pressure

crude oil, where σ was the interfacial tension and p was the pressure. When the pressure was equal to 24.55 MPa, the interfacial tension was zero, which meant the MMP of the CO_2 and crude oil was 24.55 MPa. In the experimental process, the pressure increased gradually, when the crude oil totally dissolved in the CO_2 phase, it was considered that CO_2 was completely miscible with crude oil; the actual measured pressure was 25.6 MPa.

Similarly, the relationship between the interfacial tension and the pressure for the $scCO_2$ microemulsion and crude oil could be expressed as follows: σ=21.08–0.96p (with a correlation coefficient of 0.9890). The calculated MMP of the $scCO_2$ microemulsion and crude oil was 22.02 MPa. The actual measured MMP of the $scCO_2$ microemulsion and crude oil was 23.1 MPa. This is to say the MMP between the $scCO_2$ microemulsion and crude oil was lower than that between $scCO_2$ and crude oil.

The light hydrocarbon component in the crude oil is easily dissolved in the CO_2 phase. When the pressure reaches a certain value, the light hydrocarbon is dissolved in the CO_2, and then the content of the light hydrocarbon in the CO_2 phase increases, which gives the CO_2 some properties of a rich gas; as a result it is easier for CO_2 to become miscible with crude oil. For the $scCO_2$ microemulsion, AOT (surfactant) and C_2H_5OH (co-surfactant) dissolved in the CO_2 phase are conducive to reducing the interfacial tension between CO_2 and crude oil, thereby making the light and heavy hydrocarbon components of crude oil miscible with $scCO_2$. Consequently the MMP of the $scCO_2$ microemulsion and crude oil decreases significantly.

The oil recoveries of $scCO_2$ flooding and $scCO_2$ microemulsion flooding were measured separately with slim tube tests. The results are shown in Fig. 7. In CO_2 flooding, if the oil recovery reaches 80% at gas breakthrough or the final oil recovery reaches 90%, the flooding pattern is considered miscible (Yang, 1998). The final oil recovery increased with increasing pressure, but the rate of increase of recovery was quite slow when the pressure was higher than the MMP. Therefore, the MMP may be determined by the relation between oil recovery and pressure, which means that the



(a) 6.2 MPa (b) 25.6 MPa

Fig. 5 Images of oil drops in the $scCO_2$ fluid at 45 ºC

(a) 12 MPa (b) 23.1 MPa

Fig. 6 Images of oil drops in the supercritical CO_2 microemulsion at 45 ºC

miscible conditions could be determined by analyzing the turning point in the recovery curve. When the pressure was lower than that of the turning point, the oil recovery increased sharply with pressure, but when the pressure was higher than the turning point, there was a slight change of oil recovery and the curve of oil recovery versus pressure was nearly horizontal. Fig. 7 indicated that when the temperature was 45 ºC, the MMP for CO_2 flooding was 23.8 MPa; for the $scCO_2$ microemulsion flooding, it was 22.7 MPa. This is to say the MMP of the $scCO_2$ microemulsion and crude oil was lower than that of the $scCO_2$ and oil.

3.5 Mechanism of $scCO_2$ microemulsion for enhancing oil recovery

The results of slim tube tests showed that the oil recovery of the $scCO_2$ microemulsion flooding was higher than that of the $scCO_2$ flooding at the same pressure. Except for the lower interfacial tension between the $scCO_2$ microemulsion and crude oil, the viscosity of the $scCO_2$ microemulsion was significantly higher than the $scCO_2$ fluid. The fluidity of the displaced phase (oil) is not changed, so the mobility ratio of the displacing phase to the displaced phase decreases, and the relative flow rate also decreases. Therefore, the area swept by the displacing phase increases before breakthrough and the oil recovery is enhanced. In addition, the density of the $scCO_2$ microemulsion is higher than that of the $scCO_2$ fluid; this may prevent fluid overlap and reduce viscous fingering, and delay and minimize the potential for fluid breakthrough.

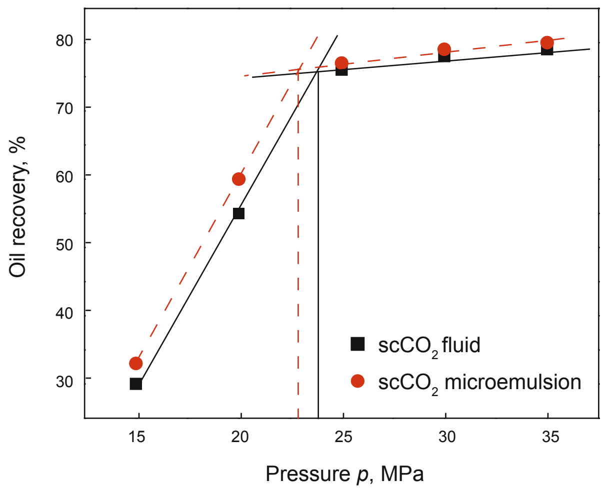


Fig. 7 Oil recovery at different pressures and 45 ºC

4 Conclusions

1) Based on the pendant drop method, the miscibility minimum pressure (MMP) of $scCO_2$ and Daqing crude oil was 24.55 MPa at 45 ºC, and the MMP of the $scCO_2$ microemulsion and Daqing crude oil was 22.02 MPa. Based on the slim tube tests, the MMP of $scCO_2$ and Daqing crude oil was 23.8 MPa at 45 ºC, and the MMP of the $scCO_2$ microemulsion and Daqing crude oil was 22.7 MPa. The MMP significantly decreased for the $scCO_2$ microemulsion compared with $scCO_2$.

2) The density and viscosity of the $scCO_2$ microemulsion were both higher than those of the $scCO_2$ fluid, and the MMP with crude oil was lower. The $scCO_2$ microemulsion could not only improve the sweep efficiency, but also decrease the MMP, thereby achieving higher oil recovery by $scCO_2$ microemulsion flooding compared with CO_2 flooding.

Acknowledgements

The authors are grateful for financial support from the National Natural Science Fund (50904073) and the CNPC Science and Technology Innovation Fund (2008D-5006-02-06).

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