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Research Article

Preparation and Performance of an Oil-Soluble Polyethylene Wax Particles Temporary Plugging Agent

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In order to solve the problem of low strength, low permeability recovery rate, and narrow suitable temperature range of the oil-soluble temporary plugging agent, the idea of preparing the oil-soluble polyethylene wax particles temporary plugging agent (OPPTA) using polyethylene wax as the main raw material was proposed. Emulsifier and stabilizer were selected, and the dosage of emulsifier, stabilizer, and stirring speed was optimized. The method for preparing OPPTA was formed. The temporary plugging performance of OPPTA for the natural core was studied. The plugging rate and the recovery rate of core permeability after plugging removal of OPPTA were 99.9% and 90.1%, respectively. The temporary plugging performance of OPPTA was better than that of a conventional oil soluble temporary plugging agent W-11.

1. Introduction

When a workover operation is performed in a reservoir with fractures or dominant percolation channels, in order to prevent the workover fluid entering the reservoir to form a blockage, the temporary plugging agent is used to temporarily block the fractures or the dominant percolation channels [1–3]. After the completion of the workover, the temporary plugging agent loses the plugging effect, and the permeability of the fractures and the dominant percolation channels can be restored, so as to realize the protection of the reservoir [4–12].

At present, the temporary plugging technology mainly includes the selective temporary plugging, crude oil thickening temporary plugging, shielding temporary plugging, and so on. The oil-soluble temporary plugging agent is a selective temporary plugging because it can be dissolved in oil and taken out of the reservoir during plugging removal, the permeability of the reservoir is recovered, and the damage to the reservoir is small. Therefore, the oil-soluble temporary plugging agent has a good application prospect. The oil-soluble resin temporary plugging agent is mainly made of petroleum resin products, such as oil-soluble phenolic resin, rosin and rosin modifier, and polyurethane, with higher melting point. This kind of material is insoluble in water, but it has good solubility in oil, so it is less harmful to the reservoir. However, the price of this kind of material is higher, and it is difficult to be popularized and applied on a large scale. Because of the brittleness and softening point of this kind of material, the temporary plugging effect for the oil wells with high temperature, fracture, or dominant percolation channel is usually not ideal, and the plugging strength needs to be further strengthened. In addition, the melting point of the oil-soluble temporary plugging agent is generally fixed at present. So its application range is small [13–15].

Polyethylene wax (PEW), also known as high molecular wax, has the following characteristics: (1) high melting point, (2) miscible with crude oil at high temperature, (3) controllable particle size, (4) high hardness, and (5) stable chemical properties. Because the melting point of polyethylene wax varies with the molecular weight, the temporary plugging can be carried out at different reservoir temperatures by controlling the molecular weight of polyethylene wax [16–23]. However, the existing production technology can not produce the oil-soluble polyethylene wax particles temporary plugging agent (OPPTA) which can meet the temporary plugging demand of the reservoir with fractures or dominant percolation channels.

2. Experimental

2.1. Reagents and Instruments. Polyethylene wax (PEW-95, PEW-110, PEW-125, PEW-140, and PEW-160; median particle size 2 mm) was purchased in Yangzhou New Material Co., Ltd. The melting points of PEW-95, PEW-110, PEW-125, PEW-140, and PEW-160 were 95°C, 110°C, 125°C, 140°C, and 160°C, respectively. Span-60 (S-60), Tween-20 (T-20), sodium hexadecyl sulphate (AS-1), sodium dodecyl sulphate (SDS), two methyl silicone oil, partially hydrolyzed polyacrylamide (HPAM, molecular weight 25 million, hydrolysis degree 20%), and guar gum (TR) were purchased from the Sichuan Kelong Chemical Reagent Factory, and all the reagents were analytically pure and were not purified before use. Hydrochloric acid of mass concentration of 10% and hydrofluoric acid of mass concentration of 10% were purchased from Sichuan Kelong Chemical Reagent Factory. The oil-soluble temporary plugging agent W-11 and stabilizer A-1 were all self-made in the laboratory.

The ion compositions of the formation water and injection water are shown in Table 1.

The parameters of natural cores are shown in Table 2.

The natural cores with the artificial fracture are shown in Figure 1.

The main instruments were the particle size analyzer, multifunction core displacement test device, electronic balance, microscope, and German IKA top mechanical stirrer.

2.2. Performance Evaluation of Polyethylene Wax

2.2.1. Oil Solubility of Polyethylene Wax. 100 g polyethylene wax PEW-95 and 300 ml two methyl silicone oil were added to the beaker with a capacity of 1000 ml. The quality of undissolved PEW-95 at 75°C, 85°C, and 95°C was measured. The oil soluble rate of polyethylene wax was calculated by the following formula:

$$y = \frac{m_1 - m_2}{m_1} \times 100, \quad (1)$$

where y is the oil soluble rate of polyethylene wax (%), m_1 is the quality of the added polyethylene wax (g), and m_2 is the quality of undissolved polyethylene wax (g). According to the above methods, the oil-soluble rate of polyethylene wax PEW-110, PEW-125, PEW-140, and PEW-160 was determined at 10°C below the melting point, 20°C below the melting point, and melting point, respectively.

2.2.2. Acid Solubility of Polyethylene Wax. 100 g PEW-95 and 300 ml hydrochloric acid with a mass concentration of 10% (or the mixed solution of hydrochloric acid and hydrofluoric acid, hydrochloric acid with a mass concentration of 10%, and hydrofluoric acid with a mass concentration of 3%) were added to a beaker, and then, the mixture was stirred at room temperature for 24 h. The quality of insoluble polyethylene wax was determined. The acid solving rate of polyethylene wax was calculated by the following formula:

$$Y = \frac{M_1 - M_2}{M_1} \times 100, \quad (2)$$

where Y is the acid solving rate of polyethylene wax (%), M_1 is the quality of polyethylene wax added (g), and M_2 is the quality of insoluble polyethylene wax (g).

Table 1: Ion compositions of injected water and formation water.

Ion	$Na^+ + K^+$	Ca^{2+}	Mg^{2+}	Cl^-	SO_4^{2-}	HCO_3^-
Injected water ion content (%)	0.0616	0.0058	0.0032	0.0420	0.0103	0.1117
Formation water ion content (%)	0.2235	0.0081	0.0020	0.2798	0.0069	0.1380

2.3. Preparing Method of OPPTA. OPPTA was prepared through the following method:

(1) A certain amount of the emulsifier and 38 g polyethylene wax (PEW-95) were added to a flask and heated until polyethylene wax was completely melted.

(2) A certain amount of the stabilizer, 0.6 g dispersants T-20, and 58.8 g injected water were added to a beaker and stirred until complete dissolving, and then the mixture solution was heated to the temperature of the polyethylene wax melting point.

(3) At a certain stirring speed, 10 g mixture solution was dripped into the melted polyethylene wax, and the stirring time was 2 minutes. Then, the rest of the mixture solution was added to the melted polyethylene wax, and the stirring time was 20 minutes. The products were cooled at room temperature.

2.4. Optimization of the Emulsifier and Its Dosage. The effects of different emulsifiers (S-60, T-20, AS-1, and SDS) on the particle size and stability of OPPTA were studied. The dosage of the emulsifier was 2 g. The stirring speed was 800 r/min. OPPTA was prepared in accordance with the above preparation method, and no stabilizer was used.

After the emulsifier was selected, the influence of emulsifier dosage on the particle size and stability of OPPTA was studied by changing the dosage of the emulsifier.

2.5. Optimization of Stirring Speed. The influence of stirring speed on the particle size and stability of OPPTA was studied. Emulsifier was 2 g S-60. At different stirring speeds, OPPTA was prepared in accordance with the above preparation method, and no stabilizer was used.

2.6. Optimization of the Stabilizer and Its Dosage. The influence of the stabilizers (HPAM, TR, and A-1) on the particle size and stability of OPPTA was studied. Emulsifier

Table 2: The parameters of natural cores with the artificial fracture.

Core number	Length (cm)	Diameter (cm)	Porosity (%)	Pore volume, PV (ml)	Permeability (mD)	Temporary plugging agent
NP-7	5.76	2.51	18.1	5.2	421.3	OPPTA
NP-8	5.41	2.52	18.7	5.0	415.7	W-11

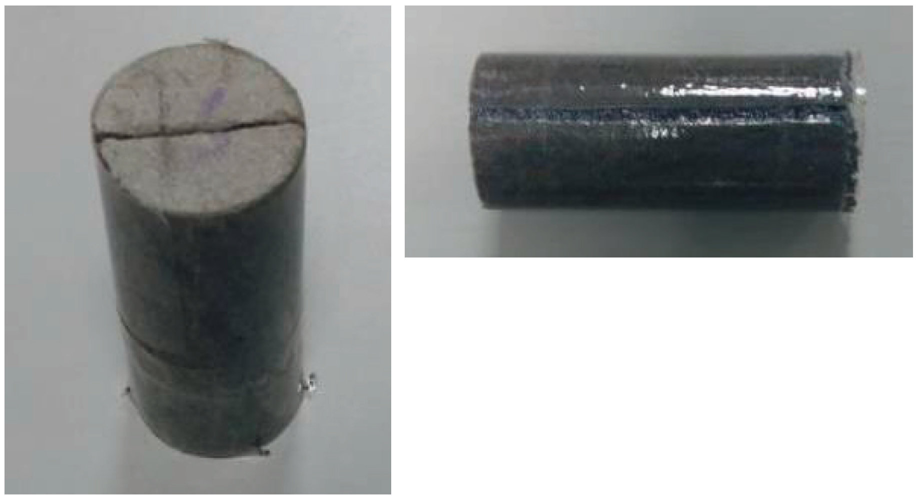

Figure 1: The natural cores with the artificial fracture (NP-7).

was 2 g S-60, and the stirring speed was 900 r/min. The dosage of the stabilizer was 0.5 g. OPPTA was prepared in accordance with the above preparation method.

After the stabilizer was selected, the influence of stabilizer dosage on the particle size and stability of OPPTA was studied by changing the dosage of the stabilizer.

2.7. Temporary Plugging Ability of OPPTA. The natural core with artificial fracture was used to study temporary plugging performance of OPPTA. The experimental procedures for determining the temporary plugging ability of OPPTA on the core were as follows [24, 25]:

(1) 100 g OPPTA was prepared using PEW-95 according to the above preparation method. The emulsifier was 2 g S-60, the stirring speed was 900r/min, and the stabilizer was 0.6 g A-1.

(2) At 65°C, the formation water was injected into the natural core at the rate of 0.2 ml/min until the injection pressure was basically stable; OPPTA was injected into the core positively at the rate of 0.2 ml/min, and the volume of OPPTA was 6 pore volume (PV); and the injected water was injected positively into the core until the injection pressure was essentially smooth.

(3) At 65°C, the formation water was injected into the core in the reverse direction at the rate of 0.2 ml/min until the injection pressure was basically stable; the kerosene was injected in the reverse direction into the core at the rate of 0.2 ml/min until the injection pressure was basically stable; and the formation water was injected in the reverse direction into the core at the rate of 0.2 ml/min until the injection pressure was basically stable.

The permeability of the core could be calculated by the Darcy formula in different experimental process. The plugging rate of OPPTA in the core was calculated by Formula (3), and the permeability recovery rate after plugging was calculated by Formula (4):

$$D = \frac{K_b - K_a}{K_b} \times 100, \tag{3}$$

$$Q = \frac{K_r}{K_b} \times 100, \tag{4}$$

where D is the plugging rate (%), Q is the permeability recovery rate (%), K_b is the core permeability before OPPTA injection (mD), K_a is the core permeability after OPPTA injection (mD), and K_r is the core permeability after plugging removal (mD).

3. Results and Discussion

3.1. Oil Solubility of Polyethylene Wax. The oil solubility of polyethylene wax with different melting points at different temperatures had been studied. The results are shown in Table 3. Polyethylene wax had good oil solubility. When the temperature was lower than its melting point 10 or 20 degrees centigrade, the oil solubility could reach above 97%. The melting point of polyethylene wax was related to molecular weight. Polyethylene wax with different melting points could be obtained by controlling the molecular weight of polyethylene wax during its production process. Thus, temporary plugging and plugging removal could be realized

Table 3: The results of oil solubility of polyethylene wax.

PEW	PEW-95			PEW-110			PEW-125			PEW-140			PEW-160		
Temperature (°C)	75	85	95	90	100	110	105	115	125	120	130	140	140	150	160
Undissolved mass (g)	1.52	0.37	0.01	0.98	0.35	0.03	1.31	0.46	0.01	1.72	0.62	0.01	1.64	0.22	0.01
Oil soluble rate (%)	97.97	99.56	99.99	98.91	99.65	99.97	98.75	99.60	99.99	98.57	99.52	99.99	98.83	99.85	99.99

for different temperature reservoirs. Good oil solubility was the key performance of polyethylene wax in the preparation of the temporary plugging agent.

3.2. Acid Solubility of Polyethylene Wax. The solubility of polyethylene wax in different acid solutions was studied. The results are shown in Table 4. The acid dissolution rate of polyethylene wax in different acid solutions was less than 1%. Hydrochloric acid and soil acid had less acid corrosion on polyethylene wax. The polyethylene wax had no reaction with the acid solution, indicating that polyethylene wax could be used for temporary plugging protection of the reservoir, when the acid workover fluid was used. This phenomenon could be explained by the very stable chemical properties of polyethylene wax at room temperature and atmospheric pressure.

3.3. Optimization of the Emulsifier and Its Dosage. The influence of the emulsifier on the particle size of OPPTA was shown in Table 5. The emulsifying effect of S-60 was the best in the four kinds of emulsifiers. The OPPTA had a particle size of 51.5 μm and a stable time of 28 hours.

The influence of emulsifier S-60 dosage on the particle size and stability time of OPPTA was shown in Figure 2. With the increase of emulsifier S-60 dosage, the median diameter of OPPTA gradually was decreased and the stability time was prolonged. When the emulsifier S-60 dosage was increased to 2%, the particle size of the temporary plugging agent was 51.5 μm, and the stability time was 28 hours. Subsequently, increasing the amount of S-60 had little influence on the particle size and stability time of the temporary plugging agent.

3.4. Optimization of Stirring Speed. The influence of stirring speed on the particle size of OPPTA was shown in Figure 3. With the increase of stirring speed, the dispersion effect of polyethylene wax was better, and the particle size of OPPTA became smaller. When the stirring speed was 900 r/min, the corresponding particle size was 50.7 μm, and the stability time was 28.8 h.

3.5. Optimization of the Stabilizer and Its Dosage. The influence of stabilizers on the particle size of OPPTA was shown in Table 6. The stability effect of A-1 was the best in the three kinds of stabilizers. The particle size of OPPTA prepared by A-1 was 50.2 μm, and the stability time was 36 hours.

The influence of stabilizer A-1 dosage on the particle size and stability time of OPPTA was shown in Figure 4. The

Table 4: The results of acid solubility of polyethylene wax.

PEW	Acid	Undissolved mass (g)	Acid dissolution rate (%)
PEW-95	/	100	/
	10% HCl	99.3	0.70
	10% HCl + 3% HF	99.1	0.91
PEW-110	/	100	/
	10% HCl	99.5	0.50
	10% HCl + 3% HF	99.2	0.81
PEW-125	/	100	/
	10% HCl	99.4	0.60
	10% HCl + 3% HF	99.1	0.91
PEW-140	/	100	/
	10% HCl	99.7	0.30
	10% HCl + 3% HF	99.1	0.91
PEW-160	/	100	/
	10% HCl	99.4	0.60
	10% HCl + 3% HF	99.2	0.81

Table 5: Influence of the emulsifier on particle size and stability time of the temporary plugging agent.

Emulsifiers	S-60	T-20	AS-1	SDS
Median particle size (μm)	51.5	56.9	79.2	97.3
Stable time (h)	28	24	20	12

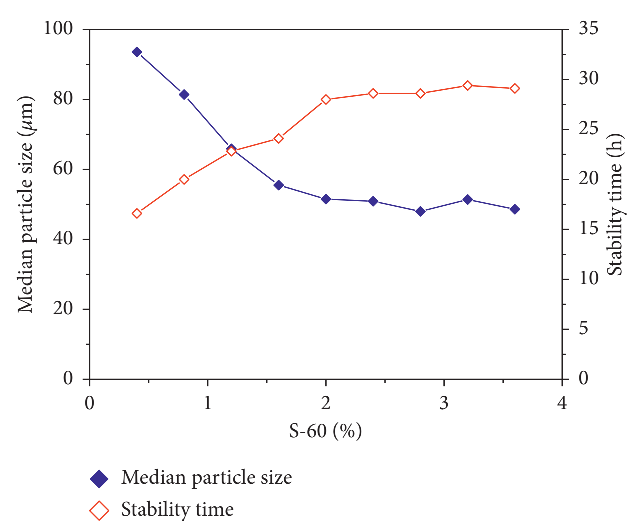

Figure 2: Influence of emulsifier S-60 dosage on particle size and stability time of the temporary plugging agent.

effect of stabilizer A-1 dosage on the median diameter of OPPTA was small, and the median particle size was kept at about 50 μm. When the amount of stabilizer A-1 was

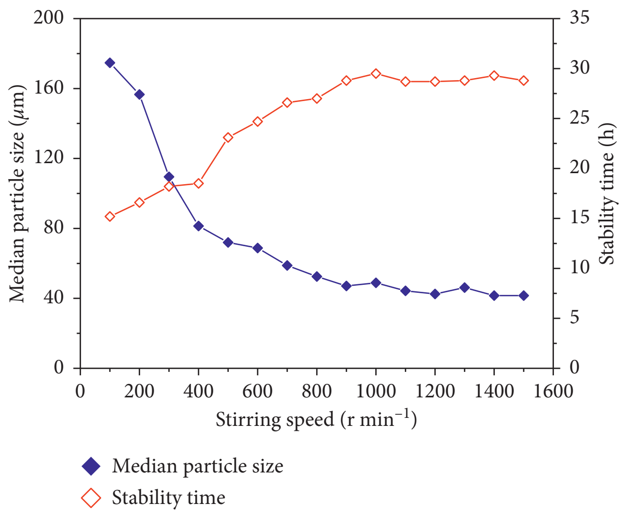

Figure 3: Effect of stirring speed on particle size and stability time of the temporary plugging agent.

Table 6: Influence of the stabilizer on particle size and stability time of the temporary plugging agent.

Stabilizers	HPAM	TR	A-1
Median particle size (μm)	52.2	51.4	50.2
Stability time (h)	32	33	36

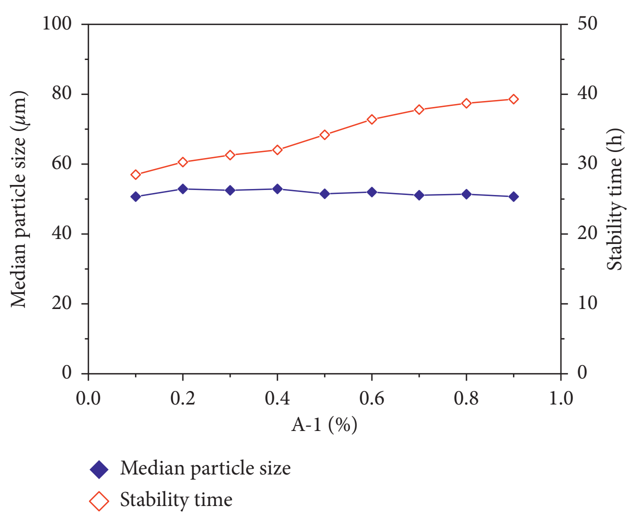

Figure 4: Influence of stabilizer A-1 dosage on particle size and stability time of the temporary plugging agent.

increased to 0.6%, the stability time of the temporary plugging agent reached 36 hours, and it could meet the requirements of conventional workover operation. Considering the cost and injection of the temporary plugging agent, the optimum dosage of stabilizer A-1 was 0.6%.

3.6. Preparing Method of OPPTA. 2 g S-60 and 38 g polyethylene wax were added to a flask and heated until polyethylene wax was completely melted. 0.6 g A-1, 0.6 g dispersants T-20, and 58.8 g injected water were added to

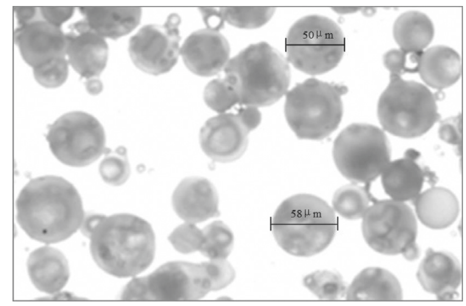

Figure 5: The micromorphology of OPPTA at room temperature.

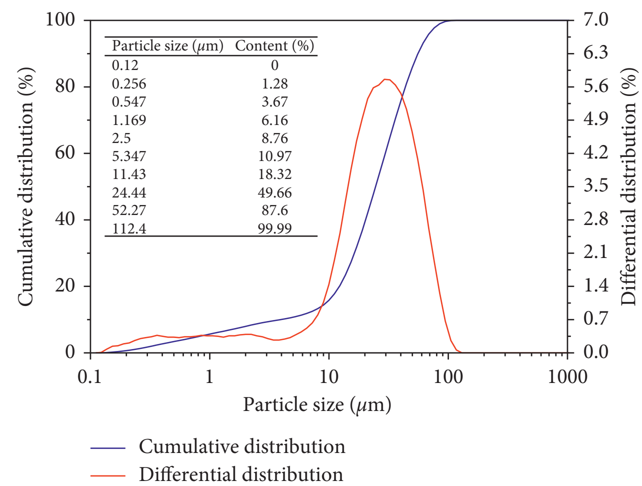

Figure 6: The particle size distribution diagram of OPPTA at room temperature.

a beaker and stirred until dissolved completely, and then the mixture solution was heated to the temperature of the polyethylene wax melting point. At the stirring speed of 900 r/min, 10 g mixture solution was dripped into the melted polyethylene wax, and the stirring time was 2 minutes. Then, the rest of the mixture solution was added to the melted polyethylene wax, and the stirring time was 20 minutes. The products were cooled at room temperature.

The micromorphology of OPPTA was observed under a microscope at room temperature, as shown in Figure 5. The particle size distribution diagram of OPPTA was obtained using the laser particle size analyzer at room temperature, as shown in Figure 6. The experimental results showed that OPPTA was polyethylene wax microspheres with good sphericity. Microspheres with a diameter of 11.43 to 52.27 μm accounted for 69.28%.

3.7. Temporary Plugging Effect of OPPTA on Core. As shown in Figure 7, when the permeability of the core was measured by water injection, the oil-soluble polyethylene wax particles temporary plugging agent was injected. The injection pressure continued to rise, and the injection pressure increased to 1.1 MPa when the 6PV OPPTA was injected. Then, the positive injection was carried out, and the pressure remained at about 0.49 MPa. The core corresponding permeability was 0.6 mD, and the plugging rate was 99.9%. In the process of

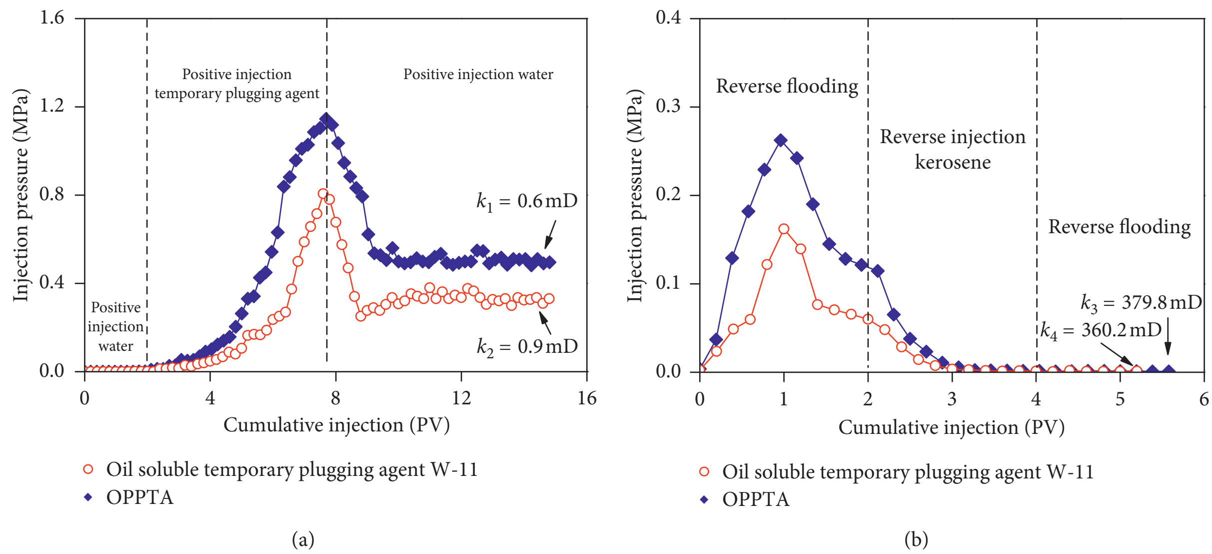

Figure 7: Temporary plugging effect of the temporary plugging agent on the crack: (a) sealing process; (b) plugging removal process.

plugging removal, reverse water injection was first carried out, and the injection pressure was kept at about 0.12 MPa; using kerosene to remove plugging, the injection pressure reduced to about 0.0015 MPa. In the reverse flooding process, the injection pressure basically decreased to the pressure level of the forward water flooding. The recovery rate of core permeability of OPPTA reached 90.1% after plugging removal. Under the same conditions, the plugging rate of the temporary plugging agent W-11 was 99.8%, and the recovery rate of core permeability after plugging removal was 86.6%. The experimental results showed that the plugging rate and the recovery rate of core permeability of OPPTA were higher than that of W-11 under the same experimental conditions. These experimental data confirmed that the OPPTA had a better temporary plugging effect.

4. Conclusion

The findings of the study are as follows:

(1) A method for preparing OPPTA using polyethylene wax as the main raw material was put forward, which solved the problems of low plugging strength of the existing temporary plugging agent and low recovery rate of core permeability after plugging removal.

(2) Compared with the conventional oil-soluble temporary plugging agent W-11, the plugging rate and the recovery rate of core permeability after plugging removal of OPPTA were higher, which indicated that it had a better temporary plugging effect.

(3) By changing the preparation conditions, a series of OPPTA with different particle sizes could be prepared to meet the temporary plugging requirements of different reservoirs.

Data Availability

No data were used to support this study.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

Acknowledgments

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