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Green Surfactant Made from Cashew Phenol for Enhanced Oil Recovery

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ABSTRACT: Surfactants play an important role in enhancing oil recovery (EOR). With the development of tertiary oil recovery technology and the continuous improvement of environmental protection requirements, green environmentally friendly surfactants play an important role in replacing conventional surfactants. In this paper, cardanol polyoxyethylene ether (CPE) was synthesized from natural biomass cardanol. Its structure was characterized, and its surface/interface properties, salt and temperature resistance, wettability, emulsification, and oil displacement effect were studied experimentally. The results showed that CPE had good interfacial activity and temperature and salt tolerance, which can reduce the oil−water interfacial tension to 10^{-1} mN/m. The emulsion formed by CPE had good stability. With the increase in CPE dosage, the droplet size of the emulsion decreases. The emulsion stabilized by CPE can effectively enhance oil recovery by 11.8%. Therefore, CPE not only is environmentally friendly but also has great application potential in the field of EOR.
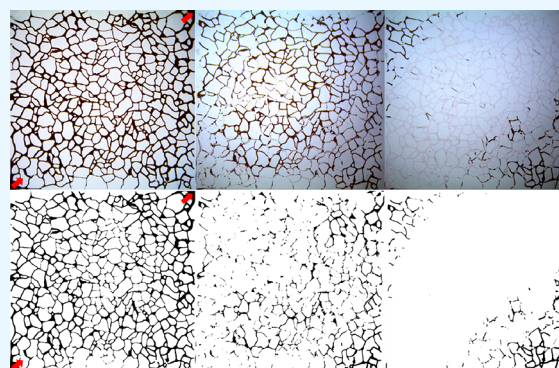
1. INTRODUCTION

Petroleum is a non-renewable fossil resource, which plays an important role in national economic development. With the rapid development of economy and technology, the consumption of petroleum is increasing and the contradiction between oil supply and demand is intensifying. In order to meet the increasing demand for petroleum, improving oil recovery has become one of the main ways to increase the output of oil fields. Chemical flooding is the main technical means to improving oil recovery.[1] Although polymer flooding is the most widely used one, with its increasing efficiency limit and the exploration and development of complex reservoirs, the surfactant, which can reduce the oil−water interfacial tension, increase the number of capillaries, and displace the residual oil, as a displacing agent for chemical flooding has become the main measure to solve the problem of slow development and decreasing production in the late stage of oil fields.[2] In short, surfactants play an important role in chemical flooding. Surfactants, whether used as a main agent or auxiliary agent, play an inestimable role in greatly enhancing oil recovery. Therefore, research on surfactants is highly valued by scientific researchers.

Surfactant is a chemical agent with high interfacial activity, which can effectively reduce the interfacial tension between oil and water, change the wettability of crude oil and rock surface with water in the reservoir, and then achieve the purpose of improving oil recovery.[3] The surfactants used for oil displacement can be classified into synthetic surfactants and natural modified surfactants. Among them, synthetic surfactants are not easy to biodegrade, which will cause damage to reservoirs, pollute groundwater resources, and destroy the ecological environment of reservoirs.[4] Development of environmentally friendly and biodegradable surfactants is urgent. Natural surfactants mainly take natural materials as raw materials and meet the requirements of use through appropriate proper modification. At present, there are many studies on rhamnolipid, modified alkali lignin, saponins, etc.[4−6] Compared with synthetic surfactants, natural surfactants are non-toxic or low in toxicity, biodegradable, harmless to the formation, and environmentally friendly.[7] In recent years, the voice of environmental protection and return to nature has become higher and higher. In order to protect the ecological environment and meet the needs of applications in various fields, countries around the world strongly advocate the use of

surfactants based on natural raw materials. In the petroleum industry, green natural surfactant has attracted much attention.

Cashew phenol is a kind of natural cashew nut shell oil refined by advanced technology. It contains a large amount of active single-component phenols and a small amount of diphenols, which can replace petroleum phenols to synthesize surfactant. Cashew phenol polyoxyethylene ether (CPE) belongs linear alkyl phenol polyoxyethylene ether, which is produced by the addition reaction of the phenolic hydroxyl group on the benzene ring of cashew phenol with ethylene oxide under the action of a catalyst. Cashew phenol is a kind of abundant and cheap renewable resource. Moreover, the product of CPE after biodegradation does not contain alkylphenol polyoxyethylene ether, which avoids the adverse impact on the environment. It is a kind of nonionic surfactant with high biodegradability and environmental friendliness. Tyman and Bruce first reported the synthesis of cardanol polyoxyethylene ether. The biodegradability of CPE, commercial t-nonyl polyethoxylate, and glucose was compared and studied.[8] The results showed that the biodegradability of CPE was similar to that of glucose.[9] Zhu et al. pointed out that the degradation rate of CPE was above 95%.[9] Zhao et al. studied the surface activity of CPE-10 and pointed out that CPE-10 had good surface activity and strong salt resistance to NaCl.[10,11] Li et al. pointed out that CPE had good wettability on low-rank coal.[12] Wang et al. pointed out that CPE had a good compounding effect with various surfactants (such as fatty alcohol polyoxyethylene ether, fatty alcohol polyoxy-ethylene ether sulfate, sodium dodecyl sulfate, Tween-60, and Tween-80).[12,13] The above studies indicate that CPE has good surface activity, biodegradability, and compounding performance. CPE is likely to have a positive effect on enhancing oil recovery. However, at present, research reports in this area are still relatively lacking.

The main purpose of this study is to explore the potential of CPE in EOR application. Therefore, first, the structure of synthesized CPE was characterized by FT-IR and ^{1}H NMR. Then, the application potential of CPE in EOR was investigated. The interfacial activity and emulsification of CPE were tested, and its oil displacement effect was evaluated. This study provides a new green natural surfactant for EOR technology.

2. MATERIALS AND METHODS

2.1. Materials. The main experimental materials used were cashew phenol ($C_{21}H_{36}O$, cashew phenol is extracted from natural cashew shell oil, technical grade, Wuhan Biet Co., Ltd.), ethylene oxide (C_2H_4O, AR, Sinopharm Chemical Reagent Co., Ltd.), acetic acid ($C_4H_8O_2$, AR, Sinopharm Chemical Reagent Co., Ltd.), and potassium hydroxide (KOH, AR, Sinopharm Chemical Reagent Co., Ltd.). All chemical reagents were used without further purification.

Two oils were used in this paper: diesel, taken from a PetroChina gas station, and crude oil, taken from an oil field in China, with a viscosity of 45 mPa·s at 60 °C.

2.2. Synthesis of CPE. The synthesis method of CPE refers to previous literature reports.[14,15] 302 g of cashew phenol was put into a high-pressure reactor, and the air in the reactor was replaced by nitrogen filling and a vacuum pumping device for four times, heated to 95 °C while stirring, and dehydrated in vacuum for 30 min. Then, nitrogen was filled into the reactor to atmospheric pressure and 0.906 g of potassium hydroxide as a catalyst was added. Under the

condition of 135 °C and 0.2 MPa, 1232 g of ethylene oxide was continuously added to the reaction within 3 h. After the reaction, it was aged for 0.5 h, cooled to 90 °C, and degassed then continued to cool to 70 °C and neutralize with 17.5 mol/ L acetic acid to pH approximately equal to 6 to obtain CPE. The synthesis route of CPE is as shown in Figure 1.
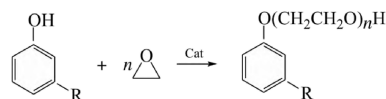

Figure 1. Schematic route of CPE.

2.3. Characterization. The synthesized CPE was characterized by FT-IR and ^{1}H NMR. A Nicolet IS5 Fourier infrared spectrometer (Thermo Fisher Scientific Shier Technology Co., Ltd.) was used to detect the infrared of the samples by the KBr tableting method, and the scanning range was 400–4000 cm^{-1}, scanning 32 times. An Avance III 400 M nuclear magnetic resonance instrument (Brook Technology Co., Ltd.) was used to measure the hydrogen spectrum with dmso-d6 solvent.

2.4. Basic Performance Test. *2.4.1. Cloud Point Measurement.* The aqueous solution of a nonionic surfactant becomes turbid when heated to a specific temperature, which is called cloud point. The cloud point of a nonionic surfactant is generally determined by heating the water bath with a concentration of 0.5–5% and observing the temperature at which it changes from clear to turbid or from turbid to clear. The determination results of these two methods are basically the same, and the difference is 0.2 °C at most. 1% of CPE solution is prepared and poured into a transparent sample bottle, a thermometer is inserted into the bottle, the sample bottle is put in a water bath and heated slowly, and the temperature is observed when it changes from clear to turbid and when it naturally cools and then changes to clear after turbid, after which these two temperatures are recorded. The measurement is repeated three times, and the average value is taken.

2.4.2. Surface Tension Measurement. The critical micelle concentration (CMC) is an important property of surfactants. When the concentration is CMC, many physical properties of the solution suddenly change and the CMC of the surfactant can be obtained by using the mutation points of these properties. The CMC can be used as a measure of the surface activity of surfactants. The smaller the CMC is, the lower the concentration of the surfactant needed to form micelles and the lower the concentration of saturated adsorption on the surface (interface). Therefore, the lower the concentration needed to change the properties of the surface (interface) and play the roles of wetting, emulsification (demulsification), solubilization, and foaming. The surface tension method is a common method to measure the CMC. The surface tension of the CPE solution was measured by the Wilhelmy plate method, and the logarithm of surface tension to CPE concentration was plotted. The concentration corresponding to the curve turning point was CMC.

2.4.3. Interfacial Tension Measurement. The interfacial tension (IFT) between the crude oil from Jiangsu Oilfield and surfactant solution was measured using a TX500 (Bowing Industry Co., Ltd.) spinning drop interfacial tensiometer at 60 °C. First, the glass tube was filled with aqueous solution. A drop of oil phase was then injected into the center of the glass

tube. Finally, the glass tube was assembled in the equipment. The IFT was measured at a constant speed (5000 rpm).[16]

2.5. Emulsification Test. The preparation of emulsion was as follows: first, a certain concentration of CPE aqueous solution was prepared, then mixed with an oil phase (diesel) at a certain oil−water volume ratio, and then stirred with a homogenizer at 10000 rpm for 3 min to obtain the required emulsion.

The type of emulsion was determined in the following: the type of emulsion was identified by dilution. The specific step was drop a prepared emulsion into water. If the emulsion droplets can spread, it is an O/W emulsion; if it cannot diffuse, it is a W/O emulsion.

The performance evaluation of emulsion includes microscopic observation, stability evaluation, and rheological evaluation. The specific operation of observing the emulsion by a microscope is as follows: 40 μL diluted emulsion sample was dropped on the slide glass, and the cover glass was carefully covered to ensure that there are no bubbles between the cover glass and the slide glass. The microstructure of the emulsion was observed by an optical microscope and the particle size of the emulsion by particle size acquisition software. The rheological property of the emulsion was measured by a Brinell viscometer. The stability of the emulsion was characterized by the water separation rate and conductivity of the emulsion, in which the water separation rate was the ratio of the volume of water separated from the emulsion to the volume of the initial water phase in the emulsion, and the conductivity was measured by a conductivity meter.

2.6. Core Flooding Experiment and Visual Micromodel Flooding Experiment. In the core flooding experiment, the core is vacuumized to obtain saturated water and saturated oil; primary water flooding is carried out until the water content reaches more than 98%; 0.5 PV emulsion is injected into the rock core; and secondary water flooding is carried out until the water content is above 98%. In the whole oil displacement experiment, the injection rate was 0.2 mL/min.

The microscopic oil displacement glass model is hydrophilic. The experimental process is as follows: the glass model is vacuumed and saturated with formation water; crude oil is saturated at formation temperature and age for 24 h; primary water flooding is carried out until no oil is produced; and the BGF stable emulsion is injected until no oil is produced. During the whole experiment, the injection rate was 2 μL/min.

3. RESULTS AND DISCUSSION

3.1. Characterization. The structure of CPE was determined by infrared spectroscopy, and the infrared spectra of raw materials were compared, as shown in Figure 2. It is shown in Figure 2 that cardanol has a stretching vibration of −OH at 3476 cm^{-1}, stretching vibration of C−H in −CH$_2$ and −CH at 2922 and 2855 cm^{-1}, and stretching vibration of the C=C bond of the aromatic ring at 1583 cm^{-1}; 1255 cm^{-1} is the C−OH stretching vibration of phenol, and 695 cm^{-1} is the aromatic ring = C−H vibration absorption. CPE exhibits a stretching vibration of −OH at 3324 cm^{-1}, stretching vibration of C−H in −CH$_2$ and −CH at 2922 and 2852 cm^{-1}, stretching vibration of benzene ring C=C at 1588 cm^{-1}, and characteristic peaks of C−O−C at 1263 and 1152 cm^{-1}, respectively. By comparing the absorption strength of the hydroxyl group and ether group, we can know that the epoxy ethylene chain is added to the phenolic hydroxyl group. The C−H stretching
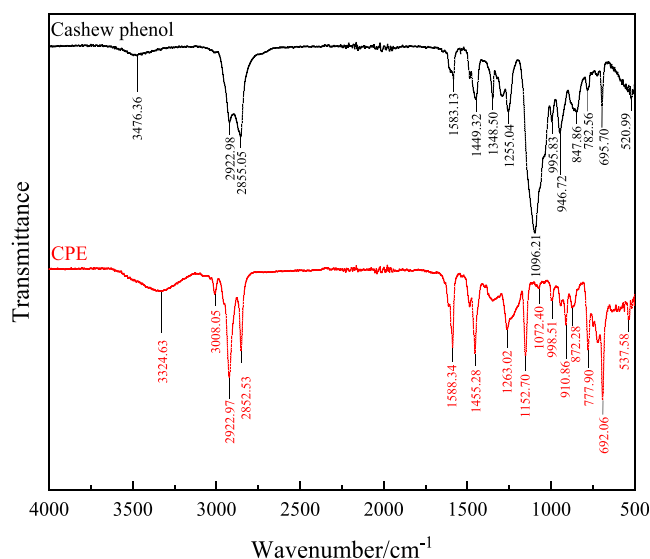

Figure 2. FT-IR spectra of cashew phenol and CPE.

vibration peak of the benzene ring is relatively reduced, which also reflects the addition of alkoxy in the original structure.

Figure 3 shows the ^{1}H NMR spectra of cashew phenol and synthetic CPE. As shown in Figure 3, CPE has a strong
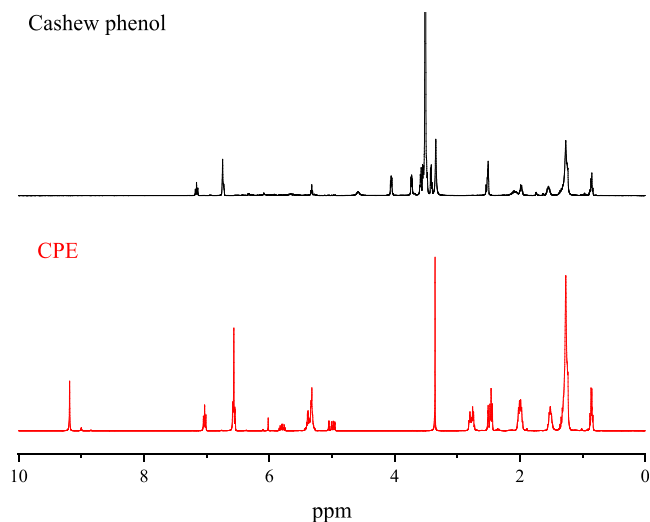

Figure 3. ^{1}H-NMR spectra of cashew phenol and CPE (dmso-d6 solvent).

absorption peak at the chemical shifts of 3.3−3.5, while the raw cardanol basically has no peak here and CPE has a strong absorption peak here. The peak at this position is the hydrogen spectrum peak in the ether group, which indicates that the ethylene oxide addition chain appears in the synthesized product CPE. According to the results of infrared spectrum analysis, the synthesized product is the target product CPE.

3.2. Basic Properties. *3.2.1. Surface Tension.* The CMC of the CPE solution was measured by the surface tension method. The variation of the surface tension of CPE with concentration is shown in Figure 4. It is shown in Figure 4 that in the initial stage, the surface tension decreases rapidly with the increase in CPE dosage. Then, with the increase in CPE dosage, the surface tension tends to be stable. It is shown in
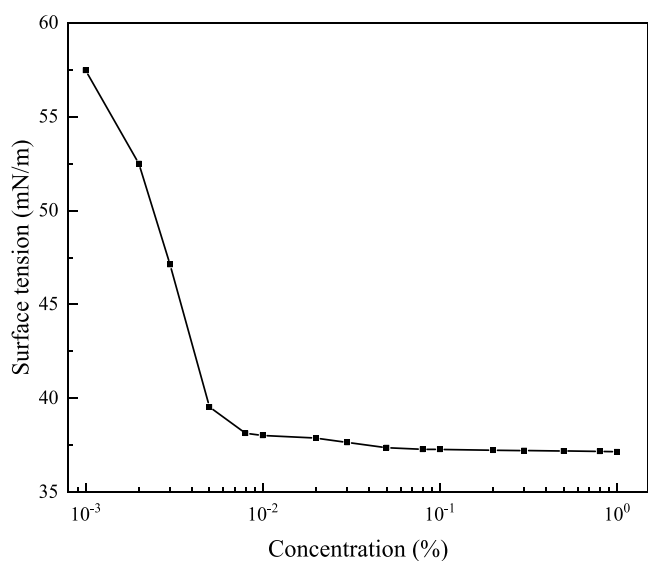
Figure 4. Surface tension of CPE at room temperature.

Figure 4 that the CMC of CPE is 0.005% and the surface tension at the CMC is 37.63 mN/m.

3.2.2. IFT. IFT is an important index to investigate the chemical flooding system. A lower IFT can greatly reduce the capillary force and effectively reduce the adhesion force of crude oil and start-up pressure gradient, thus improving the oil recovery. The dynamic IFT between CPE solution with different concentrations and crude oil is shown in Figure 5.
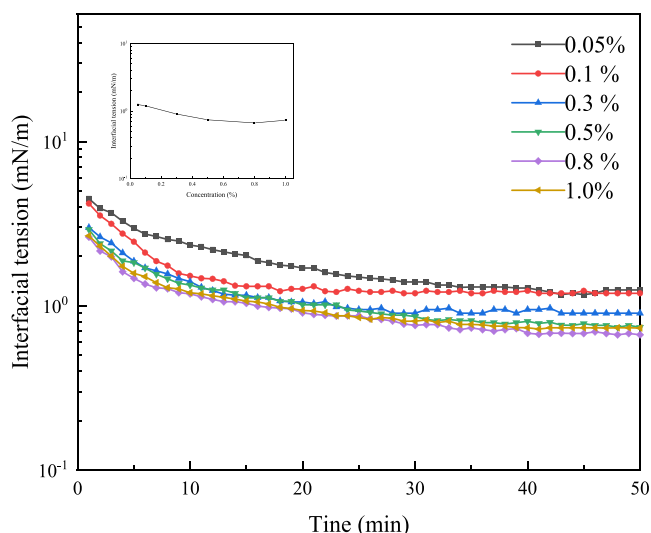

Figure 5. Interfacial tension of CPE at 60 °C.

When the concentration of the CPE solution is constant, with the increase in time, the IFT decreases rapidly and gradually stabilizes and the time to reach the equilibrium value of IFT decreases with the increase in CPE solution concentration. The IFT between CPE solution with different concentrations and crude oil is relatively small, and the IFT between 0.05 and 1.0% CPE solution and crude oil is finally stable at 1.25−0.73 mN/m. CPE can reduce the interfacial energy between oil and water, thus reducing the IFT. The decrease in oil−water IFT means that the CPE solution can overcome the cohesive force between crude oil, disperse large oil droplets into small oil

droplets, reduce the Jamin effect, and improve the flow rate of crude oil through the pore throat.

3.2.3. Temperature and Salt Resistance. The turbidity point of the solution is usually determined using a dosage of 0.5−5%. In this paper, the turbidity point of 1% CPE was tested and the results showed that its turbidity point was 81.8 °C (179.24 °F). The surface tension of 1% CPE aqueous solution was measured at different temperatures and mineralization degrees to determine its temperature and salt resistance. The results are shown in Figure 6. The results showed that the surface tension of CPE did not change significantly in the experimental temperature and mineralization degree range, indicating that it has good temperature and salt resistance.

3.3. Emulsification Properties. The emulsifying property of surfactant has a very important influence on enhancing oil recovery. Therefore, the emulsifying property of surfactant was evaluated through laboratory experiments. At room temperature, diesel oil was mixed with 0.01−1% CPE aqueous solution at a volume ratio of 1:1 and the required emulsion was prepared by high-speed stirring. As shown in Figure 7, it is a picture of the emulsion after standing for different times.

It is shown in Figure 7 that the increase in CPE dosage will affect the stability of the emulsion. After the emulsion is prepared and left standing for a certain period of time, stratification occurs, but the interface between the oil layer and the water layer is not clear enough. Therefore, the commonly used dense phase volume fraction method (that is, the stability of the emulsion is investigated according to the volume changes of the dense phase and the dilute phase during the destruction of the O/W emulsion) has great personal error. Because of the strong conductivity of the water phase, the conductivity of the O/W emulsion with water as the continuous phase is higher, while the conductivity of the W/O emulsion with oil as the continuous phase is lower because of the weak conductivity of the oil phase. Conductivity method is a common method to identify the types of emulsions, and it is also one of the research methods of emulsion destruction and transformation.[17]

The emulsion prepared in this paper was identified as O/W emulsion by the dilution method, and its stability was evaluated by the change in emulsion conductivity. The results are shown in Figure 8. As shown in Figure 8, with the increase in CPE dosage, the change in conductivity also gradually increases and the plateau where the curve begins to appear is the stable stage of emulsion. Emulsions prepared from 0.01, 0.05, and 0.1% CPE have no obvious platform at the beginning of the conductivity curve, which indicates that the emulsion prepared is unstable and will soon reach a completely stratified platform. The change of other curves starts with an obvious platform, which indicates that the prepared emulsion has a certain stable time (this phenomenon can also be observed in combination with Figure 7), but it will also delaminate, finally reaching a completely delaminated platform. The greater the amount of CPE, the longer the time to reach the fully layered platform, and the emulsion is relatively more stable, indicating that with the increase in the amount of CPE, the stability time of emulsion becomes longer.

The images of emulsions prepared by different amounts of CPE are shown in Figure 9. It can be seen that different amounts of CPE can make diesel oil emulsion into oil in water emulsion with different particle sizes. With the increase in CPE dosage, the particle size of emulsion droplets becomes smaller
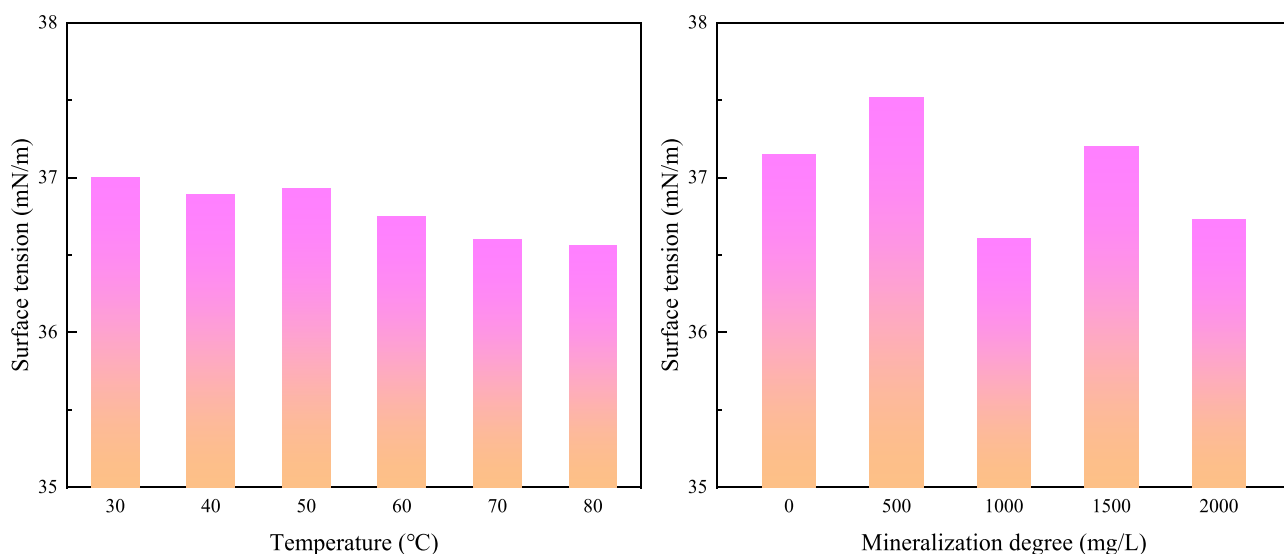
Figure 6. Temperature and salt resistance of CPE: left shows the effect of temperature on surface tension, right shows the effect of mineralization on surface tension.
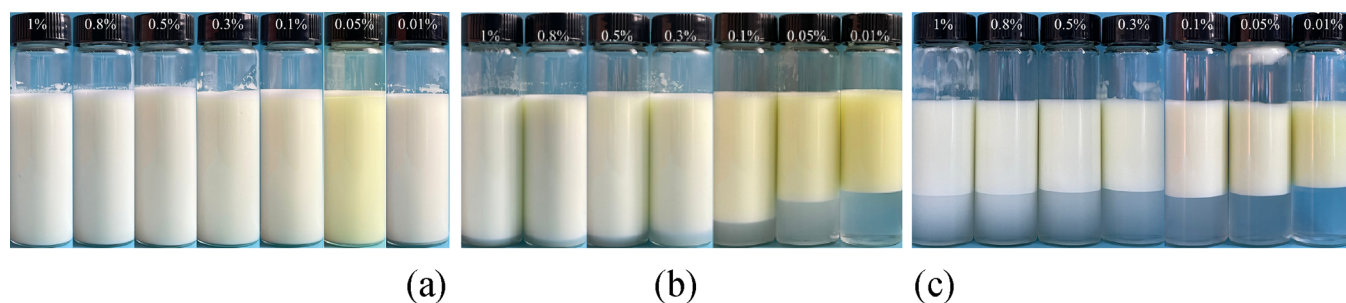

Figure 7. Appearance diagram of the emulsion with different dosages of CPE: (a) preparation is completed; (b) standing for 4 h; (c) standing for 48 h.
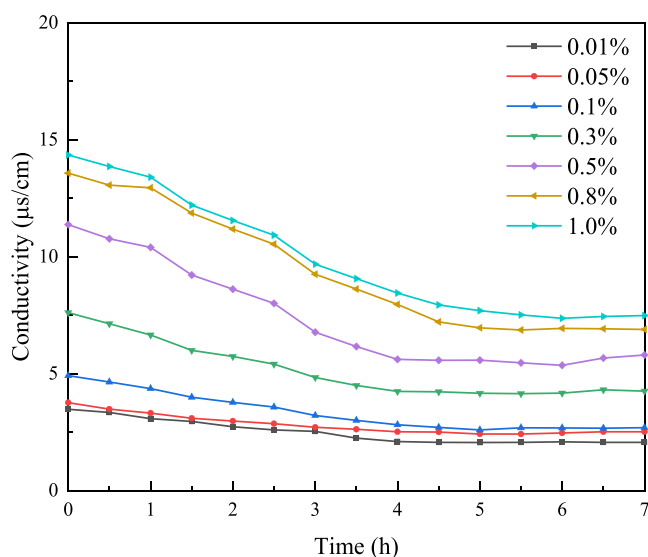

Figure 8. Conductivity of CPE at room temperature.

Figure 9. Microscopic images for emulsions made by CPE.

and more uniform, which means that the emulsion is becoming more stable.[18] The average size of emulsion droplets was measured, and the results are shown in Figure 10. The results in Figure 10 reflect the size distribution of the emulsion well. The results show that the dosage of CPE exceeds 0.5% and the droplet size of emulsion becomes smaller and weakened.
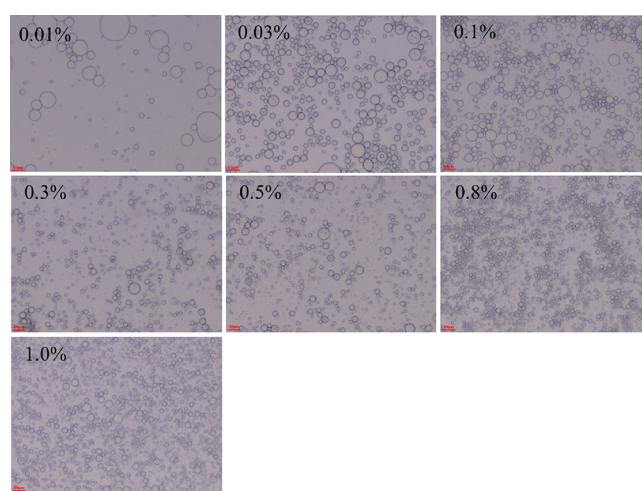
3.4. Emulsion Flooding Experiments and Mechanisms.

At 60 °C, the oil-in-water emulsion prepared by CPE was tested in the rock core with permeability of 1.5 μm^2. The viscosity of the displaced crude oil is 56 mPa·s, the mass fraction of CPE is 0.3%, and the volume ratio of water to oil is 1:1. The displacement pressure, water cut, and recovery ratio change with the injection volume in the process of emulsion flooding, as shown in Figure 11. During the initial water
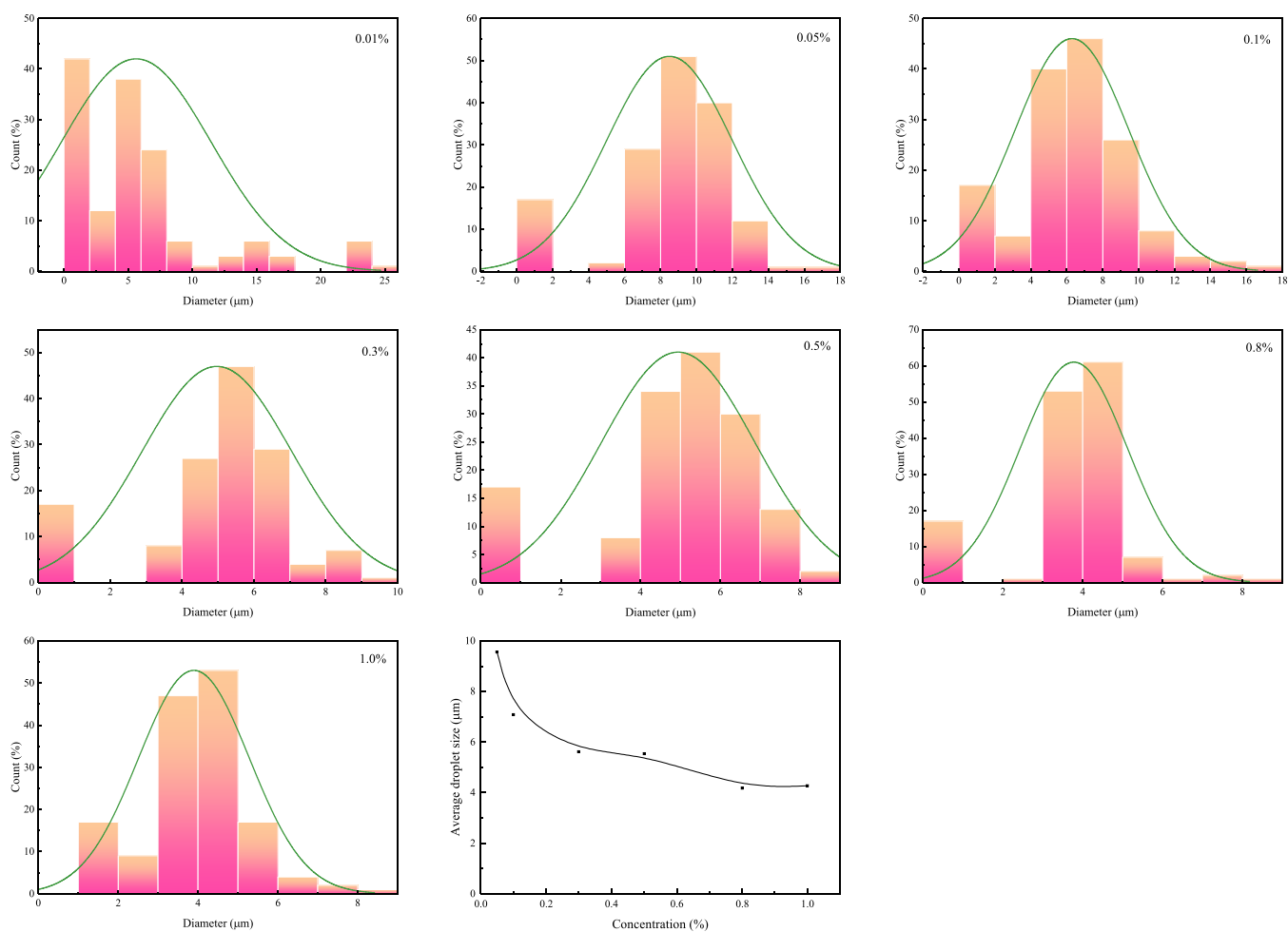
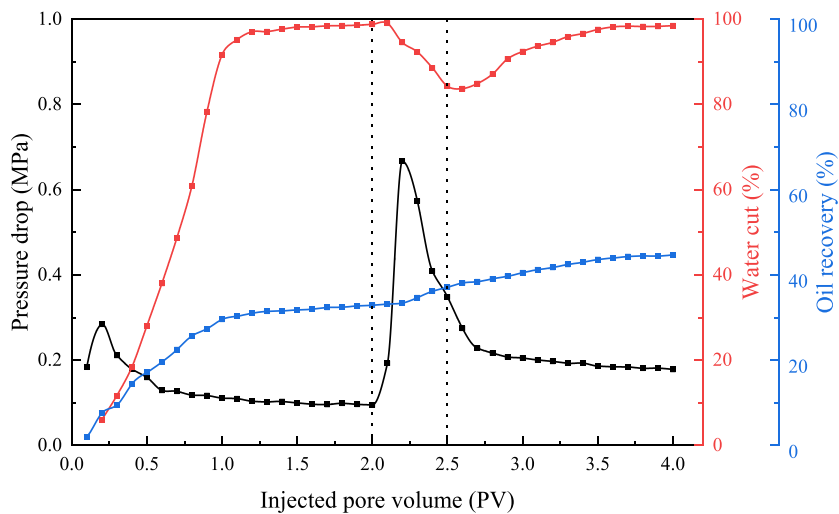**Figure 10.** The droplet size of emulsion prepared by CPE.

Figure 11. Results of the core flooding experiment at 60 °C.

flooding, the injection pressure rises rapidly to a small peak. When the injected water reaches the core outlet, that is, after the breakthrough of water flooding, the injected water flows along the path with low resistance due to the formation of a water channel, which leads to the rapid decrease in the injection pressure and the end of the initial water flooding. At this time, the recovery degree of water flooding is only 39.7%.

Then, 0.5 PV of oil-in-water emulsion slug was injected. After the emulsion was injected, the injection pressure began to rise and the pressure also reached a peak at this time. This is because the oil-in-water emulsion can block the water flow channel through the Jamin effect, increasing the flow resistance, significantly reducing the water cut in the stage, and the subsequent injected fluid will turn to the unaffected
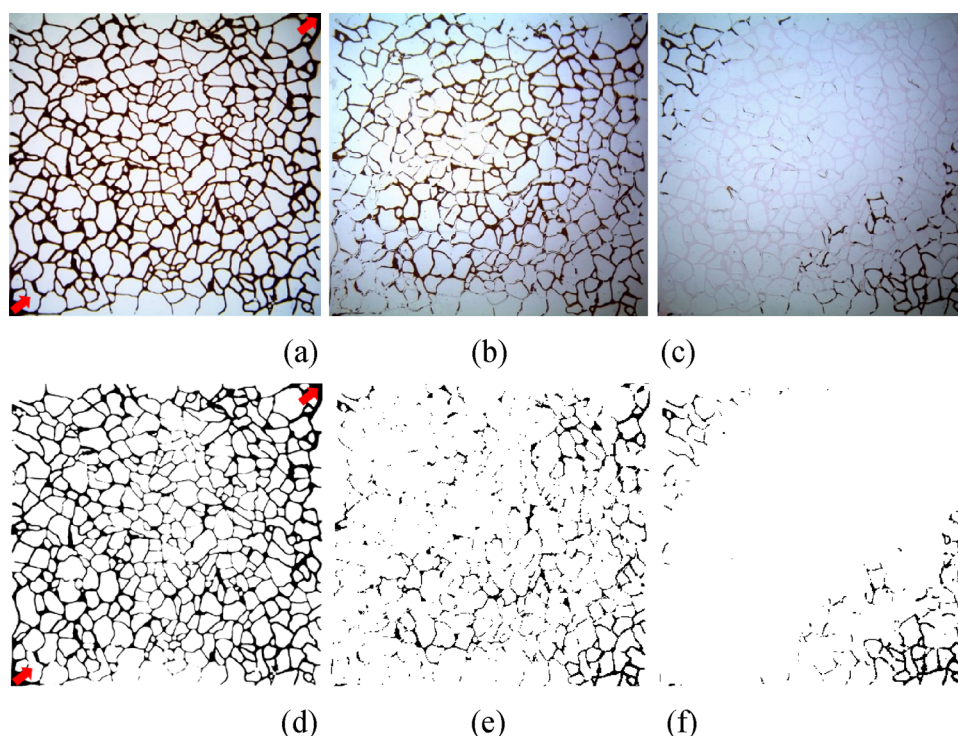
Figure 12. Microscopic visualization images of the distribution of residual oil, (a) saturated oil (original image), (b) initial water flooding (original image), (c) emulsion flooding (original image), (d) saturated oil (image after treatment), (e) initial water flooding (image after treatment), and (f) emulsion flooding (image after treatment).

area, thus increasing the sweep coefficient. When 0.5 PV of emulsion is injected, the emulsion completely and effectively blocks the water flow channel formed by initial water flooding, so that the later injected water enters the unaffected area. It is shown in Figure 11 that the recovery rate increases rapidly and the water content decreases. The final recovery degree of emulsion flooding can be increased by 11.8% on the basis of water flooding, and the total recovery degree can reach 44.7%.

In order to further study the mechanism of enhanced oil recovery by CPE emulsion, oil displacement experiments were carried out by using the microscopic visualization model; the distribution of residual oil in each stage is shown in Figure 12. Figure 12a,d shows the distribution of saturated oil in the model; Figure 12b,e shows the residual oil model after water flooding, which forms a dominant channel; and Figure 12c,f shows the model diagrams after subsequent emulsion system flooding. Compared with Figure 12e,f, it can be clearly seen that the emulsion system can displace and produce most of the residual oil in the dominant channel on the basis of water flooding, the swept area and oil displacement efficiency have been improved, and the effect of increasing oil recovery is remarkable. The reason is that, in the process of emulsion droplets flowing, some of them will block the water channel formed in the early stage and some of them will converge and form an oil belt. The Jamin effect produced by the trapping of emulsion droplets increases the displacement pressure, and the subsequent fluid enters the unaffected area during water flooding, thus increasing the swept area. Smaller droplets squeeze and scrape oil laterally to improve oil washing efficiency and reduce residual oil saturation.

4. CONCLUSIONS

CPE was prepared from cashew phenol, and its molecular structure was determined by infrared spectroscopy and proton nuclear magnetic resonance spectroscopy. CPE has the ability to reduce the surface IFT to 37.63 mN/m, and the IFT between oil and water can be reduced to 10^{-1} mN/m, and it has good temperature and salt resistance. CPE has strong emulsification ability and can form stable oil-in-water emulsions with a small droplet size and uniform distribution, which can increase oil recovery by 11.8% with a good oil displacement effect. Therefore, CPE can be used as an efficient and environmentally friendly oil displacement agent and emulsifier.

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Notes
The authors declare no competing financial interest.

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