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Laboratory Study on Active Aging Oil Plugging Agent

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Abstract: Aging oil refers to the stable emulsion of crude oil formed by emulsification that cannot be treated by conventional surfactants due to the influence of bacteria, chemical agents, self asphaltene and colloid in the process of crude oil exploitation, transportation and processing. There is a large amount of aging oil stored in the joint station of the production plant in Huabei oilfield, which not only causes certain harm to the gathering and transportation system, but also brings heavy environmental burden to the oilfield. In this study, on the basis of physical property analysis of aging oil samples, the properties of aging oil and emulsifying resin plugging agent were compared. And the water plugging principle of emulsion resin was used for reference. The system of active aging oil plugging agent is constructed by experiment. Core displacement experimental evaluation shows that active aging oil core water plugging rate was 98%, the breakthrough pressure gradient was 26 $MPa.m^{-1}$, and it has good plugging effect.

1. Introduction

Various stimulation measures in the middle and late stage of oilfield development make the composition of crude oil produced more and more complex. Aging oil is a very stable emulsion with complex components and difficulty in demulsification and dehydration. It will enter into other treatment links with the purified oil and sewage after separation, seriously damaging the oil-water separation process, affecting the quality of crude oil dehydration and the effect of oily water treatment. Current technical means cannot achieve ideal governance effect. [1-3]

After the intermediate layer of suspended oil sludge in the sewage tank and water injection tank of a jizhong oil production plant, the electric field of the electric dewatering device is unstable, and the water content of crude oil exported exceeds the standard. At the same time, a large amount of aging oil flocculent was found in sewage tank and water injection tank, which was difficult to recover and brought heavy environmental burden to the oilfield. [4-6]

This study analyzed the suspended sludge aging oil samples in sedimentation tank by experiment. And we got to know about its content and function of components. The component characteristics of aging oil and emulsion resin plugging agent were compared. On the basis of reference to the plugging principle of emulsion resin water, the experiment system of active aging oil plugging agent was constructed. Core displacement experiment confirmed that the plugging agent has good sealing performance.

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2. Aging oil sample analysis

2.1. Experimental methods
Aging oil samples were taken from suspended oil sludge in settling tanks. And water content analysis of samples was mainly conducted by GB/T/8929-2006 which was "water content determination of crude oil by distillation method". The free sewage of aged oil was filtered. The polymer content was analyzed by ultraviolet spectrophotometer. The aged oil was dissolved by petroleum ether and extracted by water. The water phase was observed under a microscope of 40 times. And the particle size was analyzed by laser particle size analyzer.

2.2. Composition analysis of aging oil samples
The moisture content was 48.4%. The colloidal asphaltene content was 61.5%. The wax content was 31.9%. The polymer content in oily sewage from sewage tank was 74.75mg/L. The polymer content in oily sewage from air-floating inlet was 65.06mg/L, which was relatively high. When the particle size was a number, the median particle size is 6.1976 micron and the average particle size is 6.9637 micron. When the particle size reference was volume, the median particle size is 14.5113 microns and the average particle size is 31.8446 microns.

2.3. Dehydration effect of conventional surfactant
The effect of demulsification and dehydration of suspended oil sludge was evaluated by using surfactants such as conventional demulsifier, paraffin remover, oil displacement agent and microbial surfactant. The on-site sewage and suspended oil sludge were stirred and mixed according to the mass ratio of 100:5. Biological decomposing agents and biological enzymes were added. And the experimental phenomena were observed after 2 days of standing at 40℃. Conventional surfactants were ineffective in dehydrating aged oils, while microorganisms had the opposite emulsifying effect on suspended sludge. [7-9]

2.4. Experimental results analysis
Suspended oil sludge is flocculated at room temperature and disappears after heating. This phenomenon indicates that flocculent adsorb waxy crude oil and collect and suspend in aqueous phase at low temperature. It is difficult to penetrate into the wax crystals to play a role at normal temperature. Suspended particles and impurities in the produced liquid also enhance the strength of oil film at oil-water interface to a certain extent, which makes it difficult to demulsify oil-water.

The colloidal asphaltene content was 61.5%, and the waxy content was 31.9% in the aging oil. The properties were similar to those of main components of emulsified resin. After mixing with water the aging oil formed a stable oil-in-water emulsion, and the viscosity reached the highest at the phase transition point of the emulsion. Rapid demulsification of the emulsion could be achieved by adding inorganic salts or alkalis. And the oil droplets stick together and formed clumps that block the water outlet.

3. Construction experiment of active aging oil plugging agent system

3.1. Experimental materials and instruments
Tween, Span, OP, C01, quaternary amine cationic surfactant, nonionic surfactant, etc. Aging oil emulsification apparatus, ultrasonic apparatus, constant temperature water bath apparatus, measuring cylinder with plug, etc.

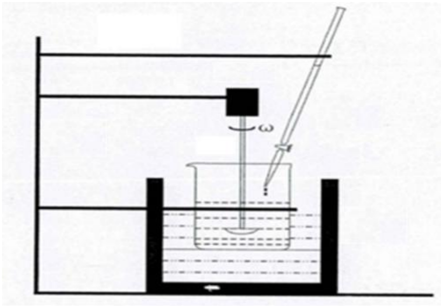

Figure 1. Emulsifying apparatus for aging oil

3.2. Experimental method

The moisture content of aging oil was determined according to GB/T/8929-2006 which was "water content determination of crude oil by distillation method". Aging oil that had been aged for a long time usually contained high moisture content. During the distillation experiment, the higher water content of the sample will affect the results. Therefore, electromagnetic stirring was carried out when the sample is heated to prevent local temperature form exploding. [10] Rheological properties of aged oil were measured by a rotary viscometer, and the shear rate was $100s^{-1}$.

3.3. Results and discussion

3.3.1. Determination of viscosity-temperature curve and water phase transition point

The aging oil was pretreated by physical ultrasonic heating dewatering method. When the dehydrated temperature of crude oil was higher than 40℃, the viscosity was significantly reduced. And the viscosity was 144mPa·s at 50℃.

The aging oil gradually formed a stable oil-in-water emulsion after mixing with water, and its viscosity increased. When the water content exceeded 52%, the emulsion viscosity decreased rapidly and changed phase.

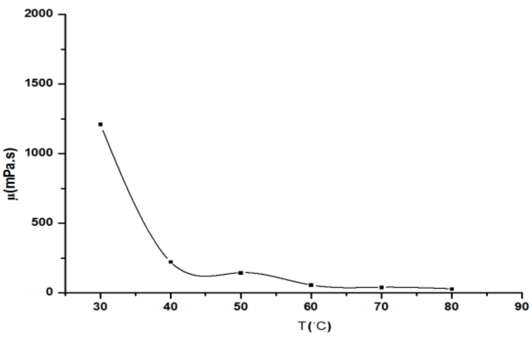

Figure 2. Viscosity-temperature diagram

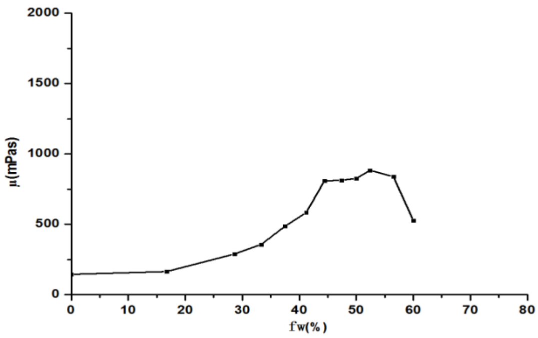

Figure 3. Emulsion water curve

3.3.2. Emulsifier screening for dehydrated aging oil

Three kinds of nonionic and two kinds of cationic surfactants were preliminarily screened. Test temperature was 50℃. Firstly, the dehydrated aging oil with a moisture content of 52% was placed in a constant temperature water bath at 50℃ for 0.5hr. Emulsifier was added to the oil sample and stirred for 5min. The emulsification and viscosity of emulsion was tested by adding different kinds of emulsifiers. The results showed that the nonionic surfactant SP had good emulsifying and viscous effect.

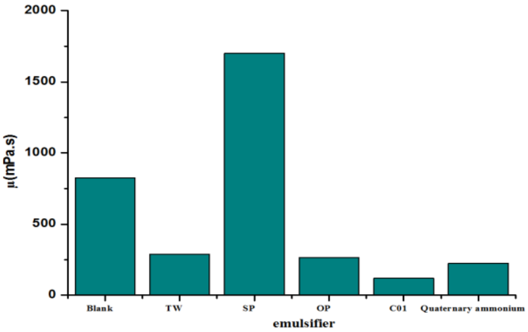

Figure 4. Emulsifying effect of different emulsifiers

3.3.3. Optimization of emulsifier concentration

After the addition of emulsifier, the water-in-oil capacity of aging oil was significantly increased. And its phase transition point was increased to 60%, as shown in figure 5. As the concentration of emulsifier increased, the viscosity of emulsion increased continuously. And the viscosity reached 1800 to 3000mpa.s when the concentration was 2% to 5%, as shown in figure 6.

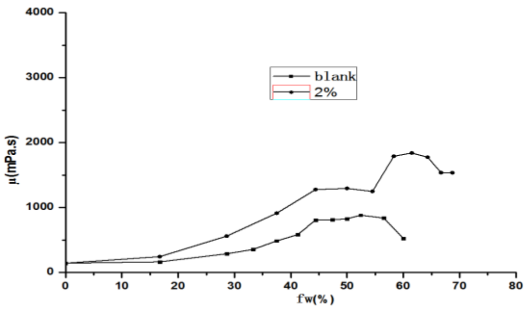

Figure 5. Phase point change after adding emulsifier

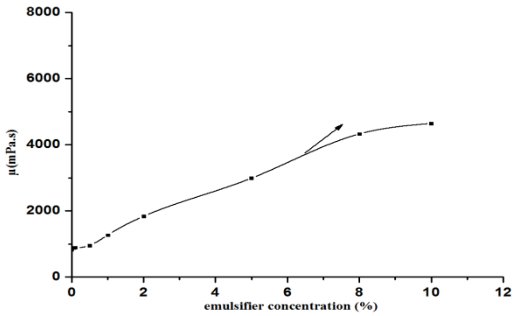

Figure 6. Viscosity of crude oil emulsion varies with the content of emulsifier

3.3.4. Demulsification experiment of active aging oil

As the emulsion is a thermodynamic unstable system, the phenomenon of partial or total separation of oil and water under certain conditions is called demulsification. First under the effect of external force, zeta potential gradually reduced as the diffusion layer gradually thinning. Droplet charge repulsion was more and more small. The droplet slowly approached, or even started to stick together and flocculate. And then the water completely flowed out between the droplets. The droplets merged and coalesced to eventually demulsify.

Active aging oil was in uniform and stable at room temperature and indoor environment. Adding some common electrolytes, such as sodium chloride, magnesium chloride, calcium chloride, aluminum nitrate, etc., can break the emulsion by reducing the electronegativity on the surface of oil beads and changing the hydrophilic and hydrophilic balance of emulsifiers, forming clumps and blocking the water outlet passage.

3.3.5. Screening of demulsifiers

According to the industry standard [11], the strength of the gel is compared according to the depth of the standard needle vertically falling into the gel. The deeper the standard needle falls, the stronger the gel. The concentration of demulsifier is optimal when the depth is the minimum. The experimental results showed that when the concentration of sodium hydroxide solution was 2% to 3%, and the concentration of calcium chloride solution was 3% to 5%, the depth of the standard needle falling into the gel was 6.5mm. And the optimal dosage of demulsifier was obtained as follows: 2% to 3% sodium hydroxide solution and 3% to 5% calcium chloride solution.

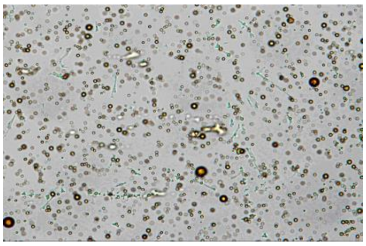

Figure 7. Microscopic image of active aged oil

4. Core displacement experiment

4.1. Instruments and materials of displacement experiment

Horizontal flow pump, hand pump, vacuum device, core gripper, intermediate container, pressure gauge, aging oil plugging agent, active aging oil and demulsifier plugging agent, clean water, artificial columnar cementing core with a permeability of 200 to 2000×10^{-3} μm^2 (diameter was 2.49cm).

4.2. Steps of displacement experiment

The single tube core displacement experiment was conducted to evaluate the breakthrough pressure, water plugging rate and erosion resistance of the core with permeability of 200 to 2000mD by two plugging agents, aging oil and active aging oil demulsification system.

Firstly, the core was flooded with water at a velocity of 2ml/min and reached the saturation state. Then, plugging agent 2PV was injected at a velocity of 0.5ml/min, and finally water flooding at 200PV. The pressure data during the experiment were recorded. The breakthrough pressure gradient and the permeability before and after core plugging were calculated, as shown in table 1.

Table 1. Single core displacement data

Core no.	Blocking adjustment type	Core permeability/mD		Water plugging rate/%	Core length/cm	Breakthrough pressure gradient/（$MPa\cdot m^{-1}$）
		Before plugging	After plugging			
1#	Aging oil	1534	89	94	8.0	10
2#	Active aging oil	1538	21	98	8.0	26

The core of #2 has a higher breakthrough pressure gradient and water erosion resistance after plugging with the active aging oil system. While the core of #1 has a lower breakthrough pressure gradient after plugging with the aging oil, and the permeability increases significantly after 90PV water flooding. Therefore, the active aging oil plugging system has good injectivity and stability.

5. Cognition and conclusion

The core water shutoff rate of the active aging oil system was 98%, and the breakthrough pressure gradient was $26MPa.m^{-1}$. It has good injection and water erosion resistance. The construction experiment of active aging oil plugging agent system proves that it not only has a good plugging effect, but also realizes the reuse of aging oil, reducing the pressure on the environment and the crude oil collecting and transporting system.

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