

Quantitative Characterization Method of Microscopic Remaining Oil Distribution and Its Application in Laboratory Displacement Experiments

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Abstract

The study of microscopic remaining oil is of great significance for the effective development of reservoirs after water flooding. An observation and quantitative characterization method of occurrence states of remaining oil on pore scale is proposed in this manuscript. Core columns are frozen immediately after the displacement experiment with liquid nitrogen freezing technology, and the samples are ground to a thickness of 0.05 mm under frozen conditions. Distributions of oil and water in pores are observed with the technology of ultraviolet fluorescence microphotography. The remaining oil content of different types is quantitatively calculated by analysis of characteristic parameters of the core image. Quantitative analysis of the laboratory displacement experimental results indicated that the average oil recovery reaches over 48% after water flooding. The main types of the remaining are throat state, cant state, thin film on pore surface, cluster state, interparticle adherence state and particle adherence state. Their relative contents account for 1.84%, 3.07%, 37.42%, 5.83%, 27.91% and 24.23% of total remaining oil reserves, respectively. Among them, the remaining oil in a thin film on pore surface, remaining oil of interparticle adherence, and remaining oil of particle adherence with high content are the development targets after water flooding. Based on determining the type and distribution characteristics of the microscopic remaining oil, the mechanism and influential factors of different types of microscopic remaining oil are analyzed, and the exploitation method for different types of remaining oil is proposed. This study is of great significance for guiding the development of remaining oil after water flooding and improving enhanced oil recovery.

Keywords: Microscopic Remaining Oil, Occurrence States, Distribution Characteristics, Quantitative Characterization, Displacement Experiment.

Introduction

Previous studies indicated that the recoverable oil reserve only accounted for 35% of the total oil reserves and that approximately 65% of the total oil reserves remained in the reservoir [1]. Since most oilfields in China have entered the ultra-high water cut stage of development, complex geologic conditions and reservoir heterogeneities of continental oilfields aggravate the complexities of distributions of the remaining oil. They increase the difficulties for oil development [2-5]. After polymer flooding, the remaining oil saturation is low, and the remaining oil is highly dispersed. To improve oil recovery rate at ultra-high water cut stage, it is an urgent task to study the occurrence state and distribution characteristics of microscopic remaining oil [6-8]. Currently, many researchers have concentrated their studies on microscopic remaining oil, greatly improving the deficiencies of conventional methods

and constantly introducing new techniques and methods [9-19]. Techniques of core analysis and microscopic simulation model have played important roles in these new methods. The former technology can not accurately describe the distribution characteristics of crude oil after displacement since coring ranges and numbers constrain it, and it can damage the original state of the remaining oil during core sample preparation. Under ordinary fluorescence, the interface of oil and water and rock is not clear, and there is a large error in the statistics of oil-water content. The latter technology can observe the dynamic process of oil displacement. Still, there is a significant difference between simulation models and real cores, leading to insufficient understanding of problems such as distribution characteristics and types of microscopic remaining oil [20-23].

Effective technical instruments are needed to meet these challenges above and further study the distribution,

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occurrence state and quantitative characterization method of microscopic remaining oil. In this study, first, the technique of frozen sectioning is applied to cut and grind samples in the temperature range of minus 30 °C to minus 177 °C and to ensure the original state of fluids in pores during sectioning. Secondly, the pore properties of rocks are identified by a polarizing microscope, and an ultraviolet fluorescence filter is selected by a fluorescent microscope to distinguish oil-water contact. Analysis software of remaining oil of independent development is applied to extract information of saturation and occurrence state of the remaining oil. The distribution state of the remaining oil in the pores is identified by observing the fluorescence images. Finally, the microscopic distribution of the remaining oil is quantitatively characterized based on the obtained results. Compared with previous methods, this method maintains the original state of oil and water and rock in samples and provides a clear oil and water interface.

Materials and Methods

Materials

The target layer of this study is the second segment of the Saertu Formation (Member S_{II}) in the Nansan Area of the Saertu Oilfield. Member S_{II} is a sandstone reservoir with good porosity and permeability. The porosity and permeability values are 27.0%-32.5% and 1150 mD-1700 mD, respectively. The sandstone type is fine feldspathic sandstone.

A set of five cores samples from Member S_{II} formation were selected for laboratory water flooding physical simulation, and then cores were evacuated for 4 hours. Afterwards, they were heated in a thermostat at 45 °C for 24 hours, and the permeabilities of cores were measured. The core is saturated with the formation water to measure the pore volume. The type of formation water is sodium bicarbonate, and its salinity is 6000 mg/L. Then the core is saturated with simulated oil with a density of 0.87 g/cm³ and viscosity of 8 MPa.s. The simulated oil is formed by mixing the crude oil in Member S_{II} and kerosene. At the temperature of 45 °C, injected water is used to flood the oil at the flow rate of 0.3-0.4 ml/min. The water cut is 98% after water flooding, and the multiple displacement volume is 10 PV (Table 1). The cores are taken from the holders, and then they are put in liquid nitrogen to freeze quickly and preserved to be studied.

Methods

Preparation of Frozen Samples

To study the fluid characteristics of cores micropores,

structures of pores and characteristics of fluid should be observed simultaneously. Moreover, pore structures as well as the position and state of the fluid in the pores, should be maintained real and stable. Therefore, there are three problems for us to be solved: (1) the detection equipment are required to detect inorganic minerals as well as oil and organic matters in pores at the same time in high power observation, (2) original states of minerals and oil in pores should be maintained during sample preparation and (3) interface of oil and water. The frozen sample preparation method that can maintain the original oil state in pores will be introduced as follows.

Multi-layer sectioning is carried out in one core segment to prepare thin sections for frozen molding and SEM images. Hence, oil and water distributions in cores, as well as pore structures, can be analyzed at the same time. Figure 1 shows the interception position of the core after freezing.



Fig. 1 Interception position of frozen core after laboratory displacement experiment.

The purpose of preparation in the temperature range of minus 30 °C to minus 177 °C is to maintain the solid forms of water and oil during grinding and to ensure the original positions of oil and water in pores. Samples after frozen preparation could be applied in observation of oil and water distribution by ultraviolet fluorescence microscope.

Preparation of frozen molding includes sectioning procedures, cementing with glue, polished sectioning, sticking and grinding sections. Samples are kept in liquid nitrogen for frozen preservation before sectioning. Then frozen sections are taken out to cut into flat end faces by frozen slicer covering the development area of fractures, holes and pores as far as possible (Figure 1). After that, the sectioned sample is put in an environment of 5 °C for air drying and cemented in a vacuum with glue 502. Rock end faces after cementing is polished by a polisher. Then they are stuck to glass sections with glue 502, and the rock section with the thickness of 5 mm is cut after the consolidation of rock and glass. Sections are first roughly ground to 0.10 mm, then to 0.06~0.07 mm, and finally finely ground to 0.04~0.05 mm without cover glass.

Table 1 Basic data of water flooding

Sample No.	Porosity(%)	Air permeability(mD)	Oil saturation(%)	Multiple of displacement (PV)	Water flooding recovery (%)
1	32.2	1237	58.8	10	50
2	27.66	1190	67.77	10	48.72
3	32.5	1506	58.17	10	46.15
4	31.56	1673	67.29	10	48.28
5	30.73	1590	70.74	10	47.46

Image Acquisition Method of Microscopic Remaining Oil

An ultraviolet fluorescence microscope is applied to acquire images of the microscopic remaining oil. The fluorescence microscope adopts a high-pressure mercury lamp that emits vita light as the light source. A digital camera of low illumination is applied to acquire images. Sections put below the camera go through focusing, exposure, and color balance processes. Furthermore, oil in core sample will produce fluorescence when it is illuminated by vita light. The relationship between distribution of oil and rock structure is identified by observing positions of fluorescent oil and detrital minerals (feldspar, quartz).

The original states of distribution of oil and water are maintained after frozen molding. The thickness of the section is less than 0.05 mm, which avoids the shielding of particles and fluorescent disturbance. Ultraviolet fluorescence is applied to excite core samples to collect fluorescence information of all bands.

The detrital minerals show black color without fluorescence, the oil shows yellowish-brown fluorescence, and the water shows blue fluorescence. The interface of oil and water can be clearly distinguished.

This method has the following advantages on studying remaining oil: (1) identifying rock particles and interface of oil and water in pores clearly and (2) integrating mutual analysis software of computer images and quantitatively analyzing types of remaining oil and contents of the water in pores.

Quantitative Analysis Method of Microscopic Remaining Oil Determination Method of Remaining Oil

Oils with different compositions have different fluorescent characteristics, intensities and colors [24]. Therefore, oil compositions can be identified by fluorescence colors [25]. Conventional experiment observation results suggest that fluorescent colors should reflect the evolution of organic matters, i.e. fluorescent colors will turn to ignites → yellow → orange → blue → blue-white (blue shift) as the organic matters evolve from low to high maturity [26-27]. Goldstein also suggests that the fluorescent colors of oil inclusion turn to brown → crocus → light-yellow → blue → blue-white as oil quality becomes lighter [28-29]. With the increase in the small molecule content and maturity, its fluorescence will show a blue shift. The wave length of the main peak of the optical spectrum will decrease; otherwise, the wave length of the main peak of the optical spectrum will increase [30-31].

By the excitation of vita light, saturated hydrocarbon does not fluoresce; aromatic hydrocarbon exhibits blue-white; non-hydrocarbon usually exhibits yellow, crocus, orange, brown; bitumen exhibits red, reddish-brown or even pitchy. Water does not fluoresce in a fluorescent microscope, but water in pore with the micro dissolution of aromatic hydrocarbon will exhibit light blue. Water and oil can be distinguished by fluorescent colors, and the relationship of oil compositions and fluorescent colors is shown in Table 2 [32]. Therefore, oil and water in rock pores can be identified and distinguished according to the fluorescence color characteristics of oil compositions and water.

Table 2 The relationship between colors and compositions

Bitumen composition	Colors
Aromatic hydrocarbon	blue, blue-white, light blue-white
Oleaginous Bitumen	yellow, yellow-white, light yellow-white, green-yellow, light green-yellow, yellow-green, light yellow-green, green, light green, blue-green, light blue-green, green-blue, light green-blue
Colloidal Bitumen	mainly orange, brown-orange, light brown-orange, light orange, yellow-orange, light yellow-orange
Asphaltene Bitumen	mainly brown, brown, light brown, orange-brown, light orange-brown, yellow-brown, light yellow-brown
Carbonaceous bitumen	no luminescence (totally black)

Pore structures of rocks are observed by a polarizing microscope. Then, the distribution range of remaining oil and water in pores and fluorescent oil colors are described roughly by observation in a fluorescent microscope.

Process of Image Analysis

Process of image analysis is shown in Figure 2. Image shooting: colored CCD video camera with high sensitivity is

selected to collect digital image signals of R, G and B. Color alternation: since color spaces of R, G and B are linear, i.e. people feel different brightness, chromaticity and saturation from equivalent R, G and B in different regions and it is unfavorable for image division. Therefore, the mathematical model of R, G and B and H, V and C (brightness, chromaticity and saturation) is built up to realize the transformation from R, G and B to H, V and C.

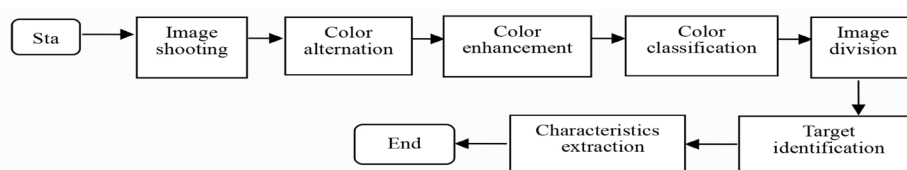


Fig. 2 Flow chart of software analysis of remaining oil.

Color enhancement: square enhancement and histogram equalization are applied. Valley points of R, G and B curves are selected as a coefficient to enhance space square.

Color classification: values of R, G and B, and peak value of H curve are utilized to classify colors.

Image division: the targets are extracted by image colors.

Characteristics extraction: suitable gray thresholds are selected by gray histogram to separate targets from the background and calculate parameters of oil area, water area and types of remaining oil.

Characteristics Extraction of Remaining Oil and Water

In microscopic fluorescent images of the remaining oil (Fig. 3a), the yellow-brown part is oil, and the blue part is water. The purpose of image division is to extract the oil (yellow-brown) and water (blue) from the rock body and run subsequent analyses.

The threshold method is conventional for image division. After division (Fig. 3b), oil and water are characterized by purple and green colors.

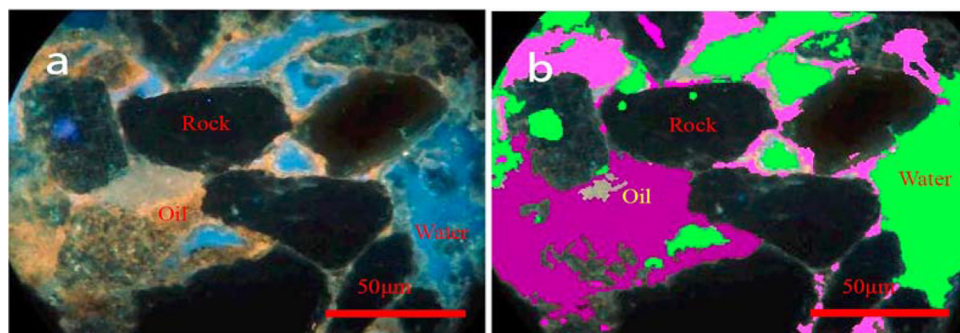


Fig. 3 Characteristics extraction image of microscopic remaining oil: (a) original fluorescent image, (b) image after division.

After identifying microscopic remaining oil individually, the remaining oil can be extracted. In an obtained binary image, target pixels are calculated to get the size and shape of the remaining oil. In the following, digital image methods of size and shape analysis of remaining oil are going to be introduced (the unit of the following calculation is a pixel):

- (1) oil area: statistics of amounts of foreground pixel corresponding to the area of remaining oil;
- (2) water area: statistics of amounts of foreground pixel corresponding to the area of water;
- (3) perimeter: statistics of amounts of foreground pixel of remaining oil boundary;
- (4) types of remaining oil: statistics of amounts of foreground pixel corresponding to this type of remaining oil;
- (5) oil/water ratio: the ratio of oil area to water area;
- (6) ratio of remaining oil type: ratios of different types of the remaining oil in the total remaining oil.

Results and Discussion

Analysis of the Main Types of Microscopic Remaining Oil

The microscopic remaining oil is quantitatively analyzed after the frozen samples are sectioned and prepared into thin sections. The fluorescence images analysis results of core samples are shown in Fig. 4, in which blue, yellow and black represent water, oil and rock particles, respectively. The remaining oil in micropores is classified into three types: (1) remaining oil of bound state which is adhered to the mineral particle surface, including states of thin film on pore surface (Fig. 4a) and particle adherence (Fig. 4b), (2) remaining oil of semi-bound state which is in the outer layer of the bound state, including cant state (Fig. 4d) and throat state (Fig. 4e), and (3) remaining oil of free state which is far from pore surface, including states of the cluster (Fig. 4f) and interparticle adherence (Fig. 4c).

Analysis of Microscopic Remaining Oil Content

Quantitative analysis of experimental results is given in Table 3. The average oil recovery reaches over 48% after water flooding. The content of remaining oil of bound state after water flooding accounts for over 60% of total remaining oil reserves. Of this state, thin film on pore surface and state of particle adherence account for 37.42% and 24.23%, respectively. The content of remaining oil of semi-bound state accounts for over 4% of total remaining oil reserves, of which cant state and throat state account for 3.07% and 1.84%, respectively. The content of remaining oil of free state accounts for over 30% of total remaining oil reserves, of which state of interparticle adherence and cluster state account for 27.91% and 5.83%, respectively.

As shown in Figure 5, among them, the content of remaining oil of thin film on pore surface after water flooding accounts for 37.42%, the content of remaining oil of interparticle adherence accounts for 27.91%, and the content of remaining oil of particle adherence accounts for 24.23%. These three types of remaining oil with high content are the development targets after water flooding.

Advantages of Frozen Molding

Compared with non-frozen sample preparation and effects of the ordinary fluorescent microscope, the thickness of a fluorescent section of ordinary fluorescent microscopic imaging is 1 mm. It is difficult to distinguish pores and particles because of the particle upper and lower shielding when it is smaller than 1 mm. Ordinary fluorescent microscopic imaging applies blue light excitation, and it causes unclear contact between oil and water due to oil showing yellow-brown fluorescence and water showing yellow fluorescence. Preparation at normal temperature will damage the initial distributions of oil and water.

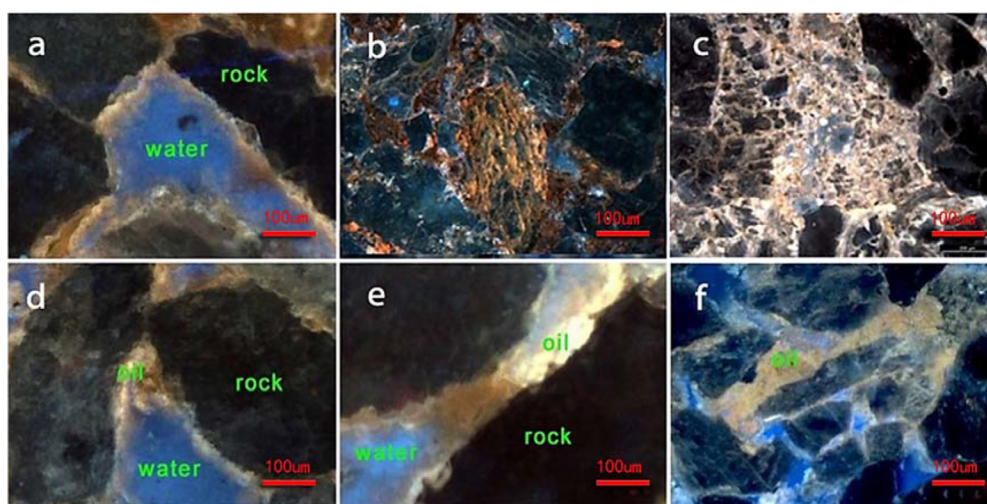


Fig. 4 Fluorescent images of microscopic remaining oil after water flooding: (a) state of thin film on pore surface adhering to particle surface of petrogenetic mineral in the form of thin-film, (b) state of particle adherence adhering to particle surface of petrogenetic mineral in tiling and dissemination forms, (c) state of interparticle adherence distributing in interparticle argillaceous matrix or area with high content of clay minerals, (d) cant state occurring in the secret corners of complex pore spaces with one side adhering to contact angle of particle and the other side in open space of the free state, (e) throat state occurring in tiny throats connected to pores, and (f) cluster state occurring in pore space in forms of a cluster, lump and oil droplet.

Table 3 Types of microscopic remaining oil and their relative content.

State of thin film on the pore surface	State of particle adherence	Cant state	Throat state	Cluster state	State of interparticle adherence
37.42	24.23	3.07	1.84	5.83	27.91

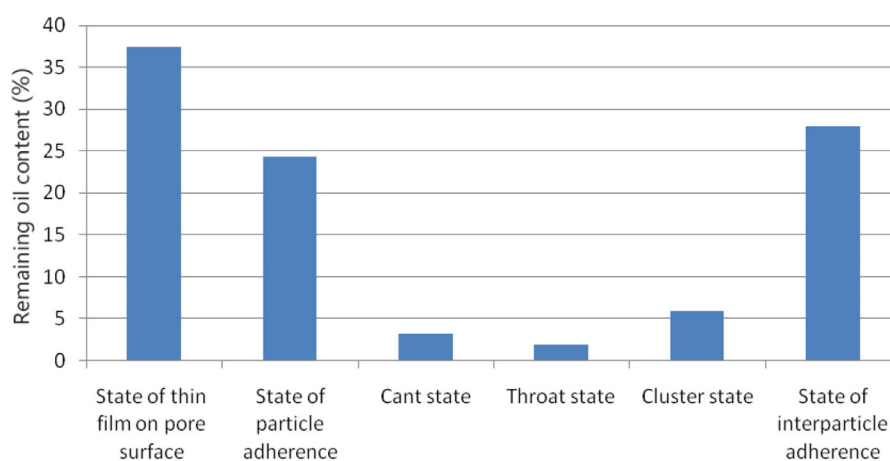


Fig. 5 Distribution of the relative content of microscopic remaining oil.

The conventional fluorescent analyzing method has the disadvantage of unclear distinguishing oil, water and minerals (Fig. 6a). Frozen section preparation maintains the initial distribution of oil and water. It avoids upper and lower shielding of particle and fluorescent disturbance because section thickness is less than 0.05 mm. Ultraviolet fluorescence is applied to excite the frozen section to collect fluorescent information of all bands. Oil-water contact can be distinguished because rock shows black color without fluorescence, oil showing yellow-brown fluorescence and water showing blue fluorescence (Fig. 6b). Therefore, the

saturation of remaining oil, oil area, water area and contents of different types of remaining oil can be calculated by computer-based image analysis method.

Forming Mechanism of Microscopic Remaining Oil

Oil in pores is affected by different forces such as displacement force, particle surface adherence force, and capillary force during the displacement and migration process. When the driving force is over-resisting, tiny oil droplets in pores can break the restrains. When resisting force is overdriving, the remaining oil is left in pores [33-34].

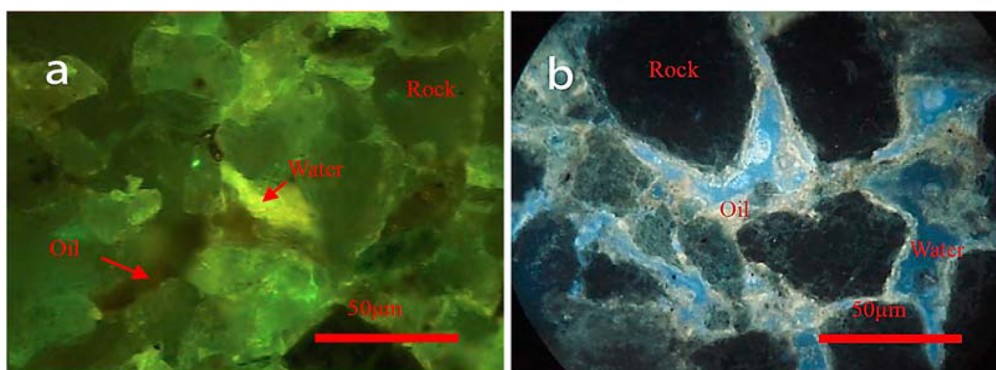


Fig. 6 Comparison of fluorescent effects: (a) distribution characteristics of oil and water and rock of ordinary fluorescence, and (b) distribution characteristics of oil and water and detrital minerals of frozen section preparation.

Six ways of forming remaining oil are summarized as follows: (1) Remaining oil caused by pore structures and heterogeneity of throat: when rock particles are nonhomogeneous, fluids produce dominant pathway in high capacity channels, and round flow appears. The remaining oil of cluster state is formed in low capacity channels because of the insufficient

driving force (Fig. 7a), and the remaining oil of cant state is formed in corners of complicated space of pores (Fig. 7b). The larger the particle size difference of sandstone is, the more favorable it is to form the remaining oil of cluster state and corner state.

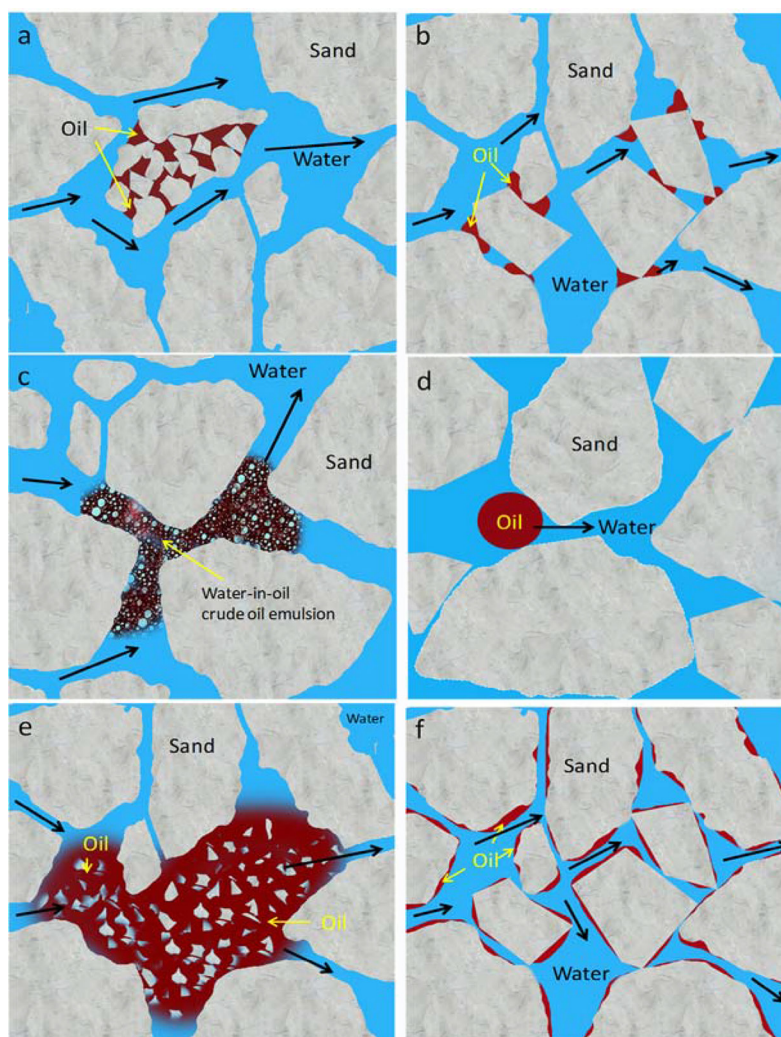


Fig. 7 Forming mechanism of different types of microscopic remaining oil: (a) forming mechanisms of remaining oil of cluster state caused by pore structures and heterogeneity of throat, (b) forming mechanisms of remaining oil of corner state caused by pore structures and heterogeneity of throat, (c) forming mechanisms of remaining oil of cluster state oil caused by alteration of oil viscosity, (d) forming mechanisms of remaining oil of throat state caused by Jamin effect, (e) forming mechanisms of remaining oil of interparticle adherence state caused by clay minerals, and (f) forming mechanisms of remaining oil of thin film on pore surface caused by adherence force of particle surface.

(2) The remaining oil is caused by alteration of oil viscosity: different degrees of emulsification appear during oil migration in pores. When the water-in-oil emulsion is formed, the remaining oil of the cluster state is formed with the increase in viscosity of oil and resisting force of migration (Fig. 7c).

(3) Remaining oil caused by Jamin effect: when the connectivity of pores is lower, throats among pores become narrower. The contour capillary force of two ends of oil droplets becomes resisting force when oil droplets pass due to radius difference between porous channel and throat. When passing through throats with a small radius, oil droplets must be stretched, and their shapes must be changed, which consumes a part of the energy, slows the movement of oil droplets and produces remaining oil of throat state (Fig. 7d). The smaller the ratio of permeability to porosity is, the more the proportion of fine throat in the rock is, and the higher the content of remaining oil of throat state is.

(4) Content of clay minerals and dispersed occurrence are the main factors that affect the microscopic distribution of remaining oil: XRD analysis shows that feldspar, quartz and clay minerals account for 42.4%, 53.7% and 3.9% of total mineral content, respectively. The content of kaolinite in the clay minerals is 77%, and most kaolinite is distributed in the pores of the rock (Fig. 8).

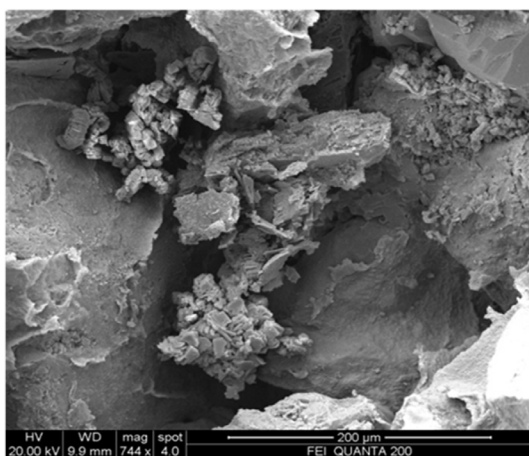


Fig. 8 SEM image of kaolinite: kaolinite is a kind of clay mineral, its chemical formula is $\text{Al}_2[\text{Si}_2\text{O}_5](\text{OH})_4$ and it belongs to the silicate mineral of the triclinic system with layer structure. As the alteration product of Feldspar, Kaolinite mainly exists in the intergranular pores in the form of pore filling, which is in the shape of pages, worms, and accordions. With the loose intercrystalline structure, kaolinite can easily move with fluid under the scouring, dividing pores and plugging throat, so it is an important velocity-sensitivity mineral.

The kaolinite is a velocity sensitivity mineral, flaky crystal aggregate of kaolinite suffers mechanical and chemical flushing and denudation of injected water, and crystal skeletons of aggregate are damaged to form tiny particles [20], which locally concentrates with oil by driving of fluids for reservoir development. Interparticle adherence remaining oil is formed with the alteration of oil viscosity and decrease of mobility (Fig. 7e). The higher the kaolinite content is, the more favorable it is to form the remaining oil of the interparticle adherence state.

(5) Remaining oil of thin film on pore surface caused by

adherence force of particle surface: for fine-grained feldspar sandstone, because the mineral particles have oil wetting characteristics, the residual oil adsorbed by the particles can not be displaced by a shear force generated by the injected water. In such a case, the remaining oil of thin film on the pore surface is formed. The thickness of oil film varies with different lithology of rock particles of porous channels, particle sizes and contact ways (Fig. 7f).

(6) Remaining oil of particle adherence state caused by capillary force: feldspar particles in sandstone are easily dissolved by water to form micropores, and oil in micropores receives holding force from the capillary force, and it cannot be displaced by injected water to form remaining oil of particle adherence state.

The above analysis shows that the formation and distribution of microscopic remaining oil are influenced by pore structure, detrital mineral type, particle size, clay mineral content, and physical properties. The important types of remaining oil after water flooding are the state of thin film on pore surface, particle adherence state and interparticle adherence state, and their content is high. Among them, the remaining oil of thin film on the pore surface is mainly distributed on the surface of the porous channel of oil-wet mineral particles. This type of remaining oil can be developed by applying surfactant to reduce surface tension. The remaining oil of particle adherence is mainly distributed in dissolution pores of feldspar and flaky minerals. This type of remaining oil can be developed by increasing injection pressure and sweeping volume. The remaining oil of interparticle adherence is mainly distributed in pores of open states with high argillaceous matrix or clay minerals (kaolinites). This type of remaining oil can be developed by injecting polymer and surfactant.

Conclusions

According to the study which has been carried out, the obtained results are as follows:

(1) This paper proposes an observation and quantitative characterization method of occurrence states of microscopic remaining oil using the technology of liquid nitrogen freezing and ultraviolet fluorescence microphotography. Compared with previous methods, this method can quantitatively describe the remaining oil and water distribution in pores, maintain the original state of oil and water and rock in samples, and provide a clear oil-water interface.

(2) The main types of the remaining oil after water flooding include throat state, cant state, state of thin film on pore surface, cluster state, interparticle adherence state and particle adherence state. Among them, the remaining oil of thin film on pore surface, the remaining oil of particle adherence, and the remaining oil of interparticle adherence are important types. Their relative contents account for 37.42%, 24.23%, and 27.91% of total remaining oil reserves, respectively. These three types of remaining oil with high content are the development targets after water flooding.

(3) The forming of the microscopic remaining oil is influenced by pore structure, particle size, detrital mineral type, clay mineral content and physical properties of sandstone. Based on analyzing the formation mechanism and distribution of remaining oil, exploitation methods of three important types of remaining oil are put forward. Among

them, the remaining oil of thin film on pore surface can be developed by applying surfactant to reduce surface tension. The remaining oil of particle adherence can be developed by increasing injection pressure and sweeping volume, and the remaining oil of interparticle adherence can be developed by injecting polymer and surfactant.

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