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Effect of pore structure characteristics on imbibition recovery of shale with different fabric facies

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Introduction

Spontaneous imbibition refers to the process by which wettable fluid displaces non-wettable fluid from the matrix pores without pressure. According to the shale oil reservoir in Huanghua Depression of Bohai Bay Basin, Zhao Xianzheng[1] et al. believed that shale in deep basino-lacustrine facies has the characteristics of high-frequency laminar structure, and divided it into laminar and massive fabric facies modes. Some scholars have studied the imbibition characteristics of shale with different fabric facies. Huang Ruizhe[2] et al. carried out a study on the impact of shale fabric characteristics on spontaneous imbibition, believing that pore structure and mineral composition are important factors affecting spontaneous imbibition. Makhanov[3] and Ghanbari[4] et al. studied the influence of rock fabric on shale water phase imbibition, and found that the water phase imbibition rate and water absorption rate of samples parallel to bedding were higher than those perpendicular to bedding. At present, the study of shale imbibition mainly focuses on using volume method or weighing method to optimize the parameters of imbibition experiment, especially the relationship between imbibition efficiency (recovery factor) and imbibition time. Although some scholars have used nuclear magnetic resonance (NMR) technology to reveal the imbibition characteristics of shale, few have revealed the law and characteristics of crude oil production in microscopic pores during the imbibition of fracturing fluid from the perspective of shale pore structure. In this paper, low-temperature nitrogen adsorption (LTNA) experiment was used to characterize pore structure parameters, and the conversion coefficient between pore diameter and relaxation time (T_2) was calibrated. Finally, spontaneous imbibition experiment of fracturing fluid based on nuclear magnetic resonance technology was carried out to study the characteristics of crude oil production in microscopic pores during imbibition of shale with different fabrics.

Experimental samples and experimental process

Experimental samples

The core samples used in the experiment are shown in FIG. 1. It can be found that shale with laminar fabric facies (L, K) is more well-stratified than that with massive fabric facies (E). Physical properties of shale core samples are shown in Table 1. The average porosity and permeability of laminar cores are higher than those of massive cores.

Experimental device

The spontaneous imbibition experimental device is shown in Figure 2, and the main instruments include: beaker, bracket, suspension line and low-field NMR instrument.

Experimental steps

1) Make slick water fracturing fluid, and 18 wt.% $MnCl_2\ 4H_2O$ was added to remove the nuclear magnetic signal in water.

2) After soaking and washing for 10 days, the shale samples were dried in an oven at 110℃ for 48 hours. The core dry weight was measured and the core T_2 substrate signal was obtained by NMR.

3) The core was vacuumized and saturated with 30MPa crude oil under pressure for 20 days. The wet weight of the core was measured, and the core was tested again by NMR T_2 spectrum.

4) The core is placed in a beaker filled with fracturing fluid (slick water) for spontaneous imbibition.

5) After spontaneous imbibition for a period of time, the core was taken out of the slick water and its NMR T_2 spectrum was measured. Then the experiment was completed by repeating step 4 until the core no longer produced oil and the NMR T_2 spectrum remained basically unchanged.

Fig. 1 Experimental samples

Table 1 Physical properties of shale core samples

Sample	Porosity /%	Permeability /mD	TOC /%	Fabric
L	5.87	0.04914	1.80	laminar
E	1.93	0.00010	2.96	massive

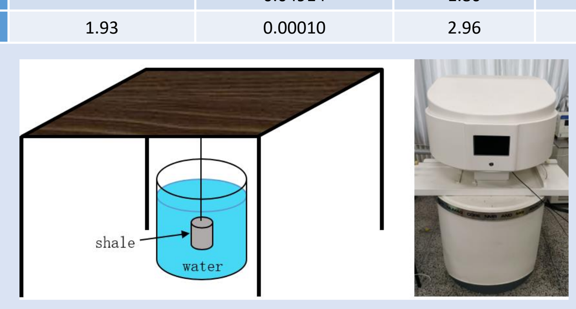


Fig. 2 Schematic diagram of spontaneous imbibition experiment and NMR apparatus

Calibrate the conversion coefficient between T_2 and pore diameter

To reveal pore size distribution of shale by NMR, it is necessary to accurately Calibrate the conversion coefficient between T_2 and pore diameter. In porous media, the relationship between transverse relaxation time of fluid and pore radius can be expressed as:

$$\frac{1}{T_2}=\rho\frac{S}{V}=\rho\frac{F}{r} \qquad (1)$$

Where, T_2 is the transverse relaxation time, ms; ρ is the surface relaxation rate, nm/ms; S is pore surface area, nm2; V is pore volume, nm3; F is pore shape factor; R is the pore radius, nm.

According to Formula (1), the transverse relaxation time T_2 of crude oil in pores is positively correlated with pore size, which can be expressed as:

$$d = CT_2 \qquad (2)$$

Where, d is pore diameter, nm; C is the conversion coefficient, nm/ms.

According to LTNA pore volume cumulative distribution curve (FIG. 3) and NMR T_2 spectrum amplitude cumulative distribution curve (FIG. 4), the pore diameter when the cumulative pore volume percentage is 50% and the relaxation time when the cumulative amplitude percentage is 50% can be substituted into Formula (2) to calculate the conversion coefficient. The calculated parameters and results are shown in Table 2. The relaxation time T_2 can be converted into pore diameter by the conversion coefficient, and then the calibrated T_2 spectrum can be used to reveal the pore size distribution characteristics of shale.

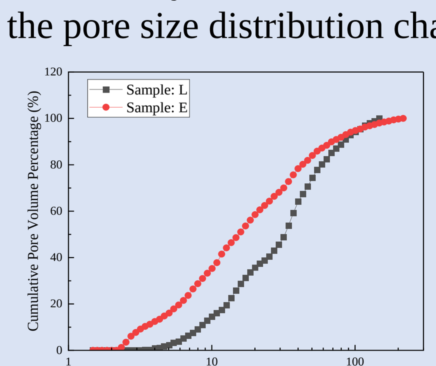


Fig. 3 Cumulative distribution curve of pore volume by LTNA method

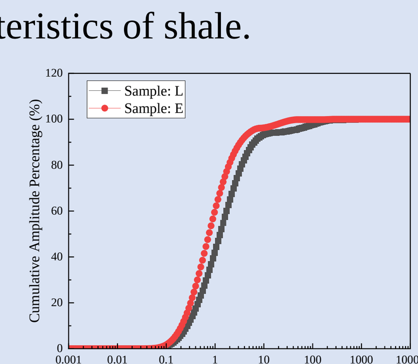


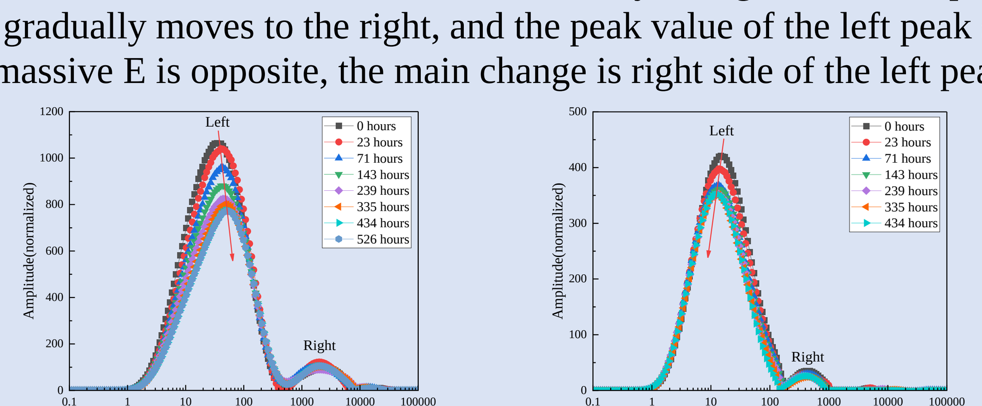
Fig. 4 Cumulative distribution curve of NMR T_2 spectra amplitude

Table 2 Conversion coefficient between T_2 and pore diameter

Sample	T_2 /ms	Pore diameter /nm	Conversion coefficient /(nm·ms⁻¹)	Fabric
L	1.2458	31.7924	25.5180	laminar
E	0.7663	15.9409	20.8013	massive

Characteristics of T_2 spectrum variation during imbibition

The change of NMR T_2 spectrum (with substrate signal removed) during spontaneous imbibition is shown in FIG. 5. It can be found that the laminar L mainly changes in the left peak and the left side of the left peak gradually moves to the right, and the peak value of the left peak gradually moves to the right. The change of massive E is opposite, the main change is right side of the left peak.



(a) laminar - L；　　(b) massive - E

Fig. 5 Image of NMR T_2 spectra changes

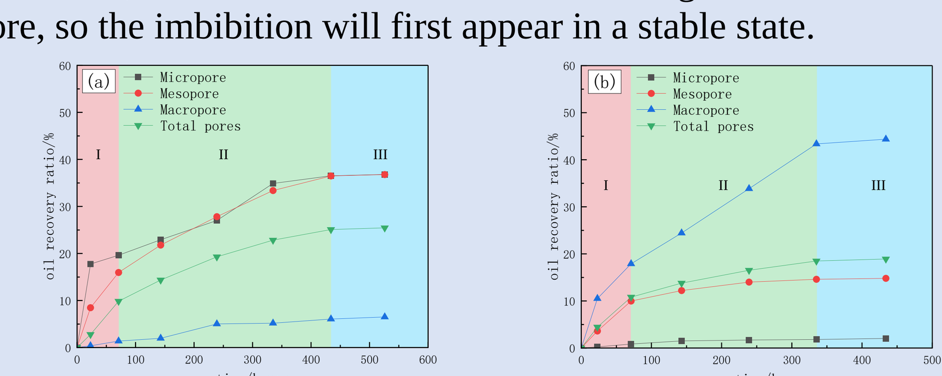
Oil production characteristics of different pores

The oil recovery can be calculated by the ratio of the change value of T_2 spectrum area in NMR to the spectrum area before imbibition. In order to study the oil recovery of pores with different sizes, the oil recovery ratio of micropores, mesopores, macropores and total pores can be calculated separately by formula (3). The calculation formula is as follows:

$$E=\frac{S_0-S}{S_0} \qquad (3)$$

Where, E is the recovery degree; S0 is T_2 spectral area before imbibition; S is the T_2 spectral area after imbibition.

The variation of oil recovery ratio in micropore (<2 nm), mesopore (2~50 nm) and macropore (>50 nm) is shown in Figure 6. It can be found that the change of oil recovery ratio can be divided into three stages: rapid imbibition stage (I), transition stage (II) and stable stage (III). According to FIG. 6(a) and (b), compared with laminar cores, oil recovery ratio curve of massive cores entered into a stable stage earlier, and the imbibition process ended earlier. This is because the pore permeability of massive shale is small, and it is difficult for the fracturing fluid to imbibition and replace the crude oil in the pore, so the imbibition will first appear in a stable state.



(a) laminar - L；　　(b) massive - E

Fig. 6 The variation of oil recovery with time in different size pores

Comparison of oil recovery ratio in microscopic pores

The comparison diagram of oil recovery ratio of different pores is drawn, as shown in FIG. 7. In addition, the contribution of different pores to shale oil recovery ratio (total pores) is calculated as shown in Table 3. According to FIG. 7, there are differences in the oil recovery ratio in different pores during imbibition. Oil recovery of spontaneous imbibition in laminar core: micropore = mesopore> total pores > macropore. Oil recovery in massive core: micropore < mesopore < total pores < macropore. It can be found that the oil recovery of the total pores is close to that of the mesopore. This is because the recovery of shale oil mainly depends on the production of mesoporous, and the contribution of mesoporous to the total pores oil recovery can reach more than 60% (Table 3). The volume of micropore and macropore is relatively small, so it can not play a great advantage in the imbibition process, and its contribution is also low.

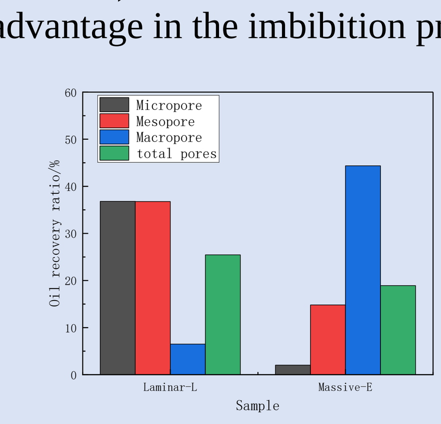


Fig. 7 Comparison of oil recovery of different size pore

Table 3 Contribution of different pores to shale oil recovery

Sample	Micropore /%	Mesopore /%	Macropore /%	Fabric
L	0.54	89.90	9.56	laminar
E	0.15	63.82	36.03	massive

Conclusion

1) The imbibition process of shale oil reservoir can be divided into three stages: rapid imbibition stage, transition stage and stable stage. Compared with laminar cores, the oil recovery ratio curve of massive cores enters into a stable stage earlier and the imbibition process ends earlier.

2) There are differences in the oil recovery ratio in different pores during imbibition. Oil recovery of spontaneous imbibition in laminar core: micropore = mesopore > total pores > macropore. Oil recovery in massive core: micropore < mesopore < total pores < macropore.

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