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Research paper

Quantitative analysis method of oil occurrences in tight reservoir

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ABSTRACT

Combining NMR, high-speed centrifugal experiment, high pressure mercury injection and physical simulation experiment technologies, a new quantitative analysis method of oil occurrences in tight reservoir is put forward and applied to two tight oil blocks. Research shows that percentage of free oil is low (about 18%) in tight reservoir, while adsorbed oil is high (about 43%). There is a positive correlation between percentage of free oil and permeability; Free oil in reservoirs greater than 0.1mD are approximately 26% and 35% in block A and B respectively, while in reservoirs less than 0.1mD are only about 12% and 17%. Free oil content controlled by micron-throats is low (only about 4% and 5% in block A and B respectively). Free oil is mainly controlled by Nano-throats. The amount of adsorbed oil on surface of larger pore-throats is the same as amount in small pore-throat in block A reservoir, while adsorbed oil in block B mainly occurs in small pore-throats. Amount of adsorbed oil on surface of larger pore-throat in block A reservoir greater than 0.1mD is lower than reservoir less than 0.1mD, while occurrence law of adsorbed oil in small pore-throat is opposite.

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More and more attention is paid to unconventional oil and gas in China (Wei et al., 2019). China is rich in tight oil resources with broad development prospects (Jia et al., 2012; Zou et al., 2012a,b; Wu et al., 2016; Zhang et al., 2016; Yang et al., 2013; Guo et al., 2016; Wu et al., 2015). The pore structures of tight oil reservoirs are complex such as high proportion of nanopore throat, large specific surface area and poor connectivity (Yao et al., 2013; Bai et al., 2013; Zhao et al., 2015; Huang et al., 2018; Ge et al., 2016; Ghanizadeh et al., 2015; Yang et al., 2012; Lin et al., 2019, 2018), and the interaction of solid–liquid and liquid–liquid have a great influence on the fluid seepage (Li et al., 2016; Zhao et al., 2015; Ghanizadeh et al., 2015; Wang et al., 2015a,b; Yang et al., 2014, 2007; Wang et al., 2009; Yu et al., 2012; Loucks et al., 2009; Law and Curtis, 2002; Yang et al., 2009; Mullen, 2010; Zhong et al., 2012; Yao et al.; Quan et al., 2011; Li et al., 2006). The pores of oil existing are small and the occurrence law is complex. The proportion of adsorbed oil is high, which lead to the poor seepage ability of oil and big effect of boundary layer (Zou et al., 2012a,b; Li et al., 2015a,b; Yang et al., 2009; Wang et al., 2015a,b; Huang, 1998; Sun et al., 2011; Xu et al., 2007; Fei, 2010). The tight oil development faces great challenges such as conventional

Nomenclature

D	Diameter, cm
L	Length, cm
K_g	Gas permeability, mD
S_{HgN}	Percentage of pore volume controlled by throats of below 50 nm under high pressure mercury injection in core, %
S_o	Oil saturation in irreducible water, %
S_{oA}	Percentage of total adsorbed oil, %
S_{oC}	Percentage of saturated oil at 1 MPa centrifugal force after centrifugation in saturated oil core
S_{oF}	Percentage of total free oil
S_{oFm}	Percentage of free oil controlled by micron-throats, %
S_{oN}	Oil in pore space controlled by throats smaller than 50 nm, %
ϕ_g	Gas porosity, %

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water injection is difficult to develop, rapid production decline and low recovery; and the mode of occurrence and mechanism of producing tight oil are not clear. It is of important guiding significance clarifying oil micro-occurrence state and proportion on understanding tight oil reservoir, carrying out in-depth studies on oil production and selecting a reasonable development mode. Zhongyuan et al. (2018), Uth et al. (2016) and Dastvareh and Azaiez (2017) studied liquid flow in nano- or micro-sized circular tubes. Wei et al. (2014) analyzed influencing factors on water-oil displacement. Datta et al. (2014) studied fluid flow mechanism from a three-dimensional porous medium. De Paoli et al. (2016), Rokhforouz and Akhlaghi Amiri (2017) and Jafari et al. (2017) studied flow behavior of fluids in heterogeneous porous media. CT and SEM are common methods for visualization study on fluid occurrences (Wang et al., 2015a,b; Yang et al., 2014; Gong et al., 2015; Guo et al., 2013; Li et al., 2012). Zhao et al. (2015) studied CO₂ and oil-immiscible and miscible flow processes in porous media using NMR micro-imaging. Gong et al. (2015) analyzed the main occurrence modes and pore types of crude oil in tight oil reservoirs in Songliao Basin. Wang et al. (2015a,b) combined NMR and nano-CT, studied the occurrence forms of crude oil in tight oil reservoirs. It was considered that the main occurrence forms of crude oil in tight oil reservoirs are emulsion and film. Xiaobing et al. used NMR to find that macroporous, mesoporous, microporous and nanoporous in Yanchang Formation of Ordos Basin were well developed. By using NMR and CT, Wenxue et al. obtained the intuitive three-dimensional pore and throat maps of Chang 7 reservoir in Ordos.

Due to the small pore-throat of tight oil reservoirs, CT and SEM have some shortcomings in studying oil occurrences in tight reservoir. CT analysis has strict requirements on the samples (cores with a diameter of 1 mm correspond to a minimum resolution of 400 nm). The representativeness is restricted and the characteristics of oil occurrence in pore throats with a diameter of smaller than 400 nm cannot be observed, while the interpretation of oil and water is empirical. The SEM observation field is small, the representativeness of the results is restricted, and it has strict requirements on the operators. NMR (Nuclear Magnetic Resonance) is widely used in the study of fluid occurrence. The diameter of samples analyzed is generally 2.5 cm or 3.8 cm. Compared with small cores for CT, The representativeness of 2.5 cm diameter sample is relatively satisfactory and it can reflect the fluid occurrence in full-size pore throats. Based on technologies such as NMR, high-speed centrifugal experiment and high pressure mercury injection, the authors have established a quantitative analysis method on oil occurrences in tight reservoir which could obtain the oil proportion under different occurrences in cores quantitatively. This method can not only effectively distinguish the content of free and adsorbed oil in core, but also distinguish the throat control of free oil and the proportion of oil in different adsorbed states. The result provides a theoretical basis for effective development of tight oil and selecting a rational development mode.

1. Quantitative analysis method on oil occurrences in tight reservoir

1.1. Experimental principle

When the core saturated fluid is centrifuged at high speed, the fluid in core is forced out under the centrifugal force against the capillary pressure. In this study, high speed centrifugation experiment complete by core centrifuge of PC-18 (max rotation speed of about 18000 rpm). The greater centrifuge speed, the greater centrifugal force, the greater capillary pressure that can be overcome, and the smaller throat-controlled fluid can be centrifuged. The centrifugal force is inversely proportional to the

throat radius of rock: The smaller the centrifugal force is, the larger the throat radius is; Whereas the larger the centrifugal force is, the smaller the throat radius is. When the core saturated fluid is single-phase water or light oil, the NMR T_2 relaxation time of the fluid in pores of core corresponds to the state of fluid occurrence. Small T_2 relaxation time corresponds to the boundary fluid in small or large pores which is in close contact with solid surface, while large T_2 relaxation time corresponds to the bulk phase fluid in large pores which is not in close contact with solid surface. The bulk phase fluid is free. Under external force, part of the boundary fluid is also movable, and in a free state: the larger the force, the larger the movable part; the smaller the force, the smaller the movable part. The remaining fluid in the core is immovable and adsorbed. Therefore, it is feasible to combine high-speed centrifugation with NMR to quantify the proportion of free oil to adsorbed oil in the core.

1.2. Experimental steps

Firstly, quantitative analysis of free oil was carried out by combining NMR with high-speed centrifugal experiment (see Fig. 1). The specific steps are as follows: first, wash off oil from core, and test gas porosity and gas permeability after drying core in oven, then make the core saturated with kerosene. Test the T_2 spectrum (NMR experiment is completed using a Reccore-04 model core NMR analyzer provided by Institute of Flow and Fluid Mechanics, Chinese Academy of Sciences; The selection of main measurement parameters in NMR test fully considers the rock characteristics of the reservoir, and the waiting time is 3000 ms, the echo time is 0.6 ms, and the number of echoes is 2048); then make the core dried again, vacuumized and pressurized heavy water with a saturated salinity of 20000 mg/L (there is no hydrogen element in heavy water and no signal is generated during NMR detection. Therefore, the T_2 spectrum measured in each state is the T_2 spectrum of oil in this state). According to oil industry standards (SY/T 5345-2007), select an appropriate displacement pressure. Use the conventional oil physical simulation experiment system to displace water with oil and establish a core saturated oil with irreducible water, and test T_2 spectrum under this state. Then carry out gas displace oil centrifugal experiment under a pressure of 0.1 MPa and 1 MPa respectively (using PC-18 core centrifuge). After each centrifugation, the core was tested T_2 spectrum; Compare the T_2 spectra under different states, and calculate oil proportion in different types of occurrences (Fig. 2).

After centrifugation under pressure of 1 MPa, the adsorbed oil in the core can be divided into two parts. One is the adsorbed oil in small pores (with a throat radius of smaller than 50 nm), and the other part is the adsorbed oil on large pore-throat surface (with a throat radius of greater than 50 nm). In order to further obtain the contents of adsorbed oil in small pore-throat and on larger pore-throat surface quantitatively, a quantitative analysis method for adsorbed oil in different types of occurrences is designed. The steps are as follows: first, saturate the dried core with kerosene oil to test T_2 spectrum under this state; then carry out gas displace oil centrifugation with 1 MPa centrifugal force, and test the T_2 spectrum under this state; compare the T_2 spectra before and after centrifugation, and calculate the total percentage of adsorbed oil in the core S_{OC} (this value is the sum of oil controlled by pore-throats smaller than 50 nm S_{ON} and the adsorbed oil on the core throat surface controlled by throats larger than 50 nm S_{OM}); and then carry out high pressure mercury injection tests for the core or the parallel sample of the core to obtain core throat distribution (high pressure mercury experiment is completed using Poremaster 60 automatic mercury porosimeter which minimum test throat is up to 2 nm), and calculate the percentage of pore volume of throats smaller than



Fig. 1. Core experimental system of nuclear magnetic resonance and high speed centrifugal.

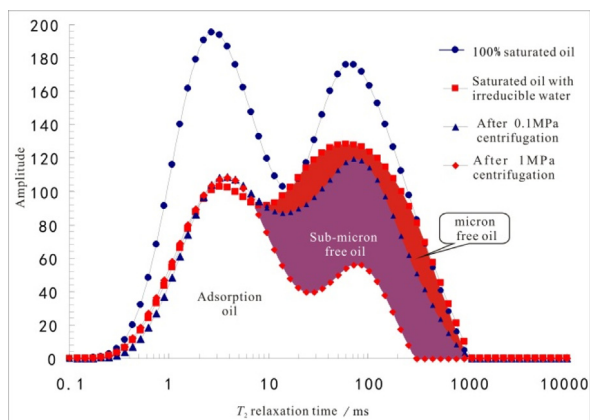


Fig. 2. Schematic diagram of quantitative analysis on oil occurrence in core.

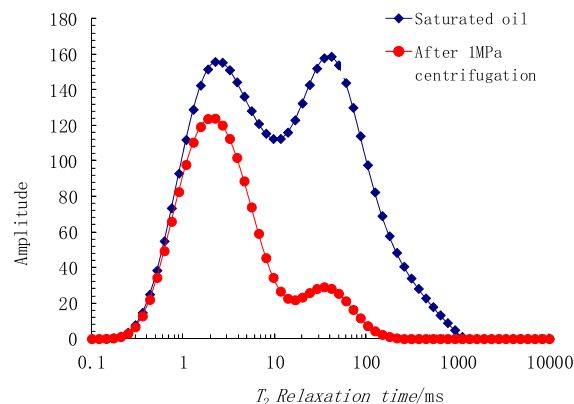


Fig. 3a. T_2 spectra of core saturated oil and after 1 MPa centrifugation.

50 nm in the core S_{HgN} ; S_{OC} minus S_{HgN} is S_{OM} (as after the gas displace oil centrifugation, the throats smaller than 50 nm in the oil saturated core are filled with oil, S_{ON} equals to S_{HgN}). See Fig. 3.

1.3. Quantitative analysis of free oil and adsorbed oil

It can be seen from Fig. 2 that the oil separated by centrifugation is free oil, corresponding to the area between the T_2 spectrum of core irreducible water with saturated oil and the T_2 spectrum after centrifugation under 1 MPa force (red/purple). A lot of theories and experiments show that the fluid in pore throats below 50 nm is in an irreducible state and 50 nm can be used as a threshold to distinguish the fluids in free state from the adsorbed state (Zou et al., 2012a,b; Li et al., 2015a,b; Guo et al., 2013; Li et al., 2012). Since the centrifugal force has a relationship with the throat radius (the radius of the rock throat is 500 nm and 50 nm for the gas displace oil centrifugation force of 0.1 MPa and 1 MPa respectively). Utilizing the gas displace oil centrifugation experiment with different centrifugal force, the free oil can be divided into free oil controlled by micron-throats (red) and free oil controlled by Nano-throats (with a radius of around 50 ~ 500 nm) (purple). The percentage of free oil can be used to characterize reservoir potential by conventional development mode. The larger the free oil proportion controlled by micron-throats, the smaller difficulty in conventional development; vice versa, the larger the free oil proportion controlled by Nano-throats, the larger the fluid seepage resistance, the higher-pressure gradient required for development and the larger difficulty in conventional development.

Fig. 4 shows the residual oil distribution (nano-CT) after gas flooding in a tight core. From the figure, it can be seen that the residual oil mainly exists in the isolated macropore, the

surface of macropore and micron (in the form of film) and the nanopore (because of the resolution, the CT image of the sample can only see the occurrence of crude oil in the throat larger than 1 μm). Using CT technology, the remaining oil can only be qualitatively analyzed, and cannot quantitatively obtained for each type of residual oil. The higher the resolution of CT, the lower the diameter requirement of the test sample and the worse the representative of the sample (the diameter of the test sample is 2.5 mm). With method this study, the amount of oil adsorbed by different types of cores can be quantitatively obtained, and the larger sample diameter (2.5 cm) is more representative.

The production mechanisms of adsorbed oil on larger pore-throat surface and in small pore-throat are quite different. In case of water driving development, water can reach the adsorbed oil on the large pore-throat surface. The method of increasing the displacement pressure or changing the oil–water interface force can be used to developed this type adsorbed oil. Whereas for the adsorbed oil in small pore throats, water cannot reach it, only the imbibition action can be used to displace it to large pore throats and then recovered through other means.

Based on the above analysis method, the adsorbed and free oil can be effectively distinguished. The free oil controlled by micro-throats and Nano-throats can be quantitatively analyzed, and the amount of adsorbed oil on the large pore-throat surface and in small pore-throat can be quantitatively obtained, providing a basis for evaluation on the occurrence and production of tight oil.

2. Quantitative test results and analysis of oil occurrences in tight reservoir

According to the above methods, 14 tight cores taken from two blocks of Triassic chang 7 tight oil reservoir in Ordos basin

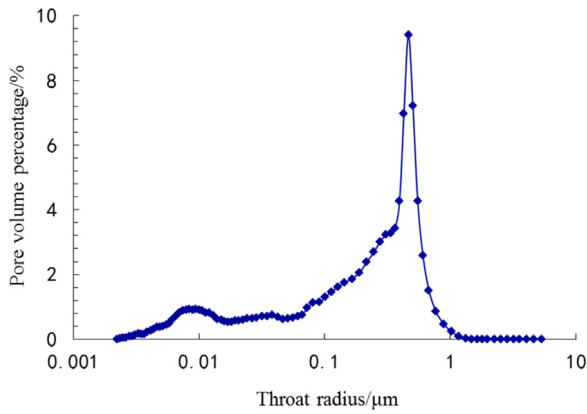


Fig. 3b. Core pore-throat distribution from high pressure mercury.

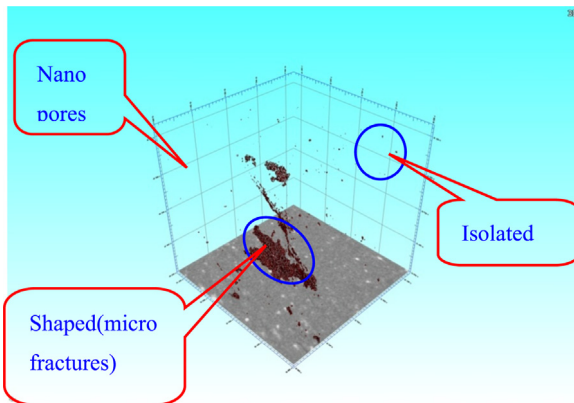


Fig. 4. Distribution of residual oil after gas displacement in a core.

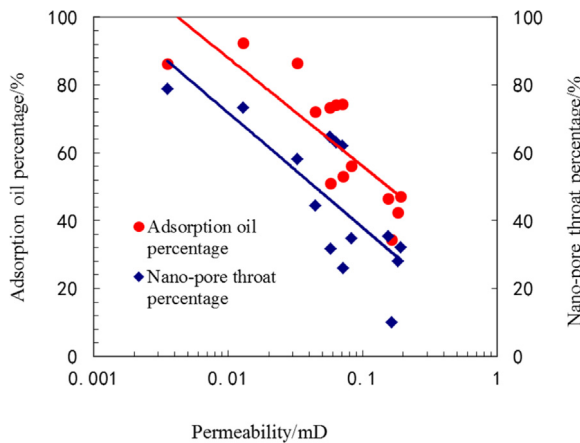


Fig. 5. Percentage of adsorbed oil, percentage of Nano-pore throats and permeability in 14 pieces of cores.

China are carried out quantitative analysis of oil occurrence. The lithology of the core is sandstone. Results are shown in Figs. 5, 6, 7 and Table 1. Gas porosity of 14 cores is 3.35%–11.24%, 8.72% on average, and gas permeability is $(0.0035\text{--}0.19) \times 10^{-3} \mu\text{m}^2$, $0.085 \times 10^{-3} \mu\text{m}^2$ on average (Table 1). See the statistics on permeability from analysis results in Table 2.

It can be seen from Figs. 6, 7, Tables 1 and 2 that: (1) oil saturation in tight oil reservoirs is high. The oil saturation of block A and block B with irreducible water is 57.39% and 67.69% respectively, but the content of free oil are low, which are 17.49% and 19.79%

Table 2

Statistics on permeability from experimental results of 14 pieces of cores.

kg (mD)	kg (mD)	Ar	S _o (%)	S _{oF} (%)		S _{oA} (%)	
				S _{oFm} (%)	S _{oFsm} (%)	S _{oN} (%)	S _{oM} (%)
<0.1	0.044	A	51.81	2.26	9.86	15.30	24.39
	0.056	B	65.59	4.70	11.93	36.86	12.10
>0.1	0.18	A	66.70	7.29	19.14	23.68	17.93
	0.16	B	78.20	5.41	30.20	36.71	10.88

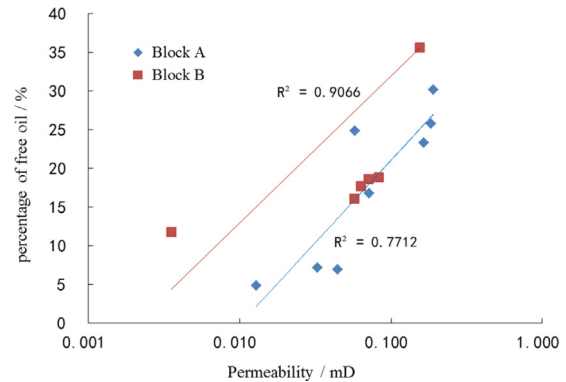


Fig. 6. Comparison of percentage of free oil and permeability.

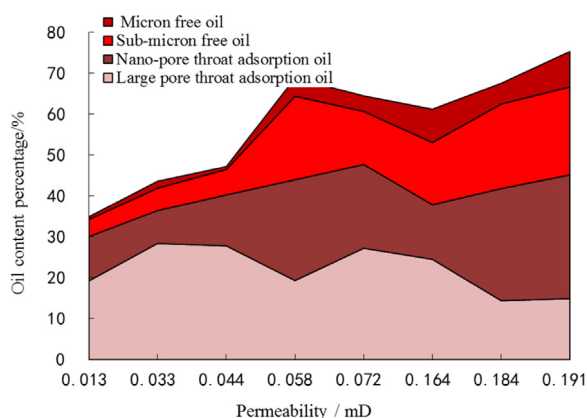
in block A and block B respectively. The contents of adsorbed oil is high, which are 39.90% and 47.90% respectively in block A and block B; (2) There is a positive correlation between free oil percentage and permeability. With the decrease of permeability, the percentage of free oil in reservoir decreases. The percentages of free oil in reservoirs greater than 0.1 mD are 26.43% and 35.62% in block A and block B respectively, and those in reservoirs less than 0.1 mD are only 12.12% and 16.63% respectively; (3) The content of free oil controlled by micron-throats is low, only 4.15% and 4.82% in block A and block B respectively. The lower the permeability, the less free oil controlled by micron-throats. Free oil is mainly controlled by Nano-throats. It is 13.34% and 14.97% in block A and block B respectively controlled by Nano-throats; (4) The adsorbed oil proportion on larger pore-throat surface (21.96%) is comparable to that in small pore-throat in block A reservoirs (18.44%). While the adsorbed oil on larger pore-throat surface in block B is less, which is 11.90%, and proportion of adsorbed oil in small pore-throat is more, which is 36.00%; (5) There are great differences in the occurrence of adsorbed oil in reservoirs with different permeability in block A. The adsorbed oil proportion on larger pore-throat surface in reservoirs greater than 0.1 mD is lower than that reservoirs smaller than 0.1 mD, whereas the adsorbed oil proportion in small pore-throat in reservoirs greater than 0.1 mD is smaller than that reservoirs smaller than 0.1 mD.

In the summary, the free oil proportion in tight oil reservoirs is low and free oil is mainly controlled by Nano-throats, which determines the inevitable conventional water flood development difficulties, such as poor effect and low yields. The adsorbed oil proportion in tight oil reservoirs is high, and it is necessary to adopt unconventional means to develop them effectively. In addition, in-depth studies should be carried out on a rational development mode for adsorbed oil in different types of occurrence.

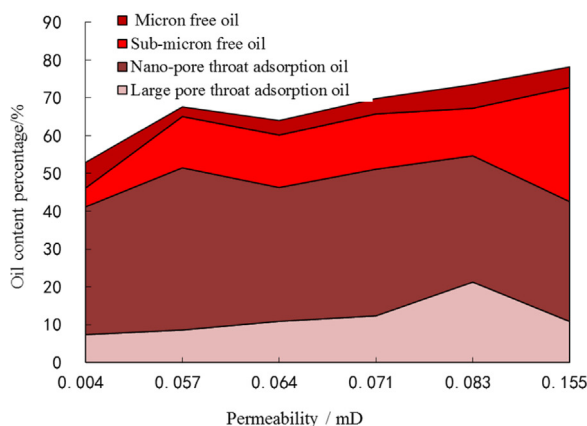
Nevertheless, there are some limitations in this study. In this study, core experiments cannot take into account the impact of large natural fractures in the actual formation. In addition, the wettability of actual reservoir is more complex, and this study can only carry out experiments on specific wettability cores.

Table 1
Statistics on the quantitative analysis results of oil occurrence in 14 pieces of cores.

No	Ar	L (cm)	D (cm)	φ_g (%)	kg (mD)	S_o (%)	S_{OFm} (%)	S_{OFsm} (%)	S_{OF} (%)	S_{OA} (%)	S_{OC} (%)	S_{HgN} (%)	S_{OM} (%)	S_{ON} (%)
1	A	3.22	2.52	8.26	0.013	34.93	0.67	4.17	4.85	30.08	92.41	73.17	19.24	10.84
2	A	3.23	2.52	10.33	0.033	43.60	1.68	5.48	7.16	36.44	86.45	58.07	28.38	8.06
3	A	3.25	2.52	5.69	0.044	47.19	0.71	6.22	6.93	40.26	72.14	44.35	27.79	12.47
4	A	3.30	2.52	6.84	0.058	68.83	4.43	20.43	24.86	43.97	51.03	31.71	19.33	24.64
5	A	3.25	2.52	10.85	0.072	64.48	3.79	13.00	16.79	47.69	53.08	25.88	27.21	20.49
6	A	3.28	2.52	9.60	0.16	61.23	8.15	15.21	23.36	37.87	34.43	9.92	24.50	13.36
7	A	3.32	2.52	11.03	0.18	67.55	5.06	20.69	25.76	41.80	42.38	27.98	14.40	27.39
8	A	3.26	2.52	10.55	0.19	71.33	8.66	21.52	30.19	41.14	47.05	32.17	14.87	30.27
9	B	3.13	2.53	3.35	0.0035	52.96	6.81	4.95	11.76	41.19	86.15	78.78	7.38	33.82
10	B	3.13	2.53	8.70	0.057	67.62	2.54	13.58	16.12	51.51	73.24	64.64	8.60	42.90
11	B	3.20	2.53	11.24	0.064	64.03	3.86	13.89	17.75	46.28	74.01	63.12	10.89	35.40
12	B	3.15	2.53	9.83	0.071	69.81	4.03	14.64	18.67	51.14	74.37	62.01	12.36	38.78
13	B	3.16	2.53	6.98	0.083	73.54	6.27	12.57	18.84	54.70	56.03	34.75	21.28	33.42
14	B	3.13	2.53	8.87	0.16	78.20	5.41	30.20	35.62	42.59	46.34	35.46	10.88	31.71



Block 1



Block 2

Fig. 7. Percentage of oil with different types of occurrences in cores.

3. Conclusion

Combining NMR, high-speed centrifugal experiment, high pressure mercury injection and physical simulation experiment techniques, a new quantitative analysis method of oil occurrences in tight reservoir is put forward and applied to two tight oil blocks in Ordos Basin. Percentage of free oil and adsorption oil obtained quantitatively. Percentage of free oil is low (17.49% and 19.79% in block A and block B respectively), and percentage of adsorbed oil is high. There is a positive relationship between

free oil percentage and permeability. The free oil controlled by micro-throats and nano-throats can be quantitatively analyzed. The content of free oil controlled by micron-throats is low, only about 4.15% and 4.82% in block A and block B respectively, and free oil is mainly controlled by Nano-throats. The amount of adsorption oil in different types can be quantitatively obtained.

The characteristics that free oil proportion in tight oil reservoirs is low determine that the conventional waterflood development have many difficulties, such as poor effect and low yield. The adsorbed oil proportion in reservoirs is high, and reasonable development mode for adsorbed oil in different types of occurrence should be carried out.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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