Retraction

Retraction: Laboratory research of wettability and heterogeneity effect on microscopic residual oil distribution in tight sandstone cores during CO$_2$ flooding (IOP Conf. Ser.: Earth Environ. Sci. 208 012065)

Baoquan Zeng, Hui Xu, Tailai Qu, Haixia Xu, Shucheng Wu, Jing Wang and Muzhen Zhang

PetroChina Research Institute of Petroleum Exploration and Development, Beijing, China.

Published 16 June 2021

This article has been retracted by IOP Publishing in light of clear evidence that some content in this article have been taken from another source (Xiao P 2018 'Laboratory study heterogeneity impact on microscopic residual oil distribution in tight sandstone cores during CO2 immiscible flooding', https://www.tandfonline.com/doi/full/10.1080/15567036.2019.1582732) without permission or reference. In line with the Committee for Publication Ethics (COPE) this has been investigated in accordance with COPE guidelines and it was agreed the article should be retracted.

Retraction published: 16 June 2021
Laboratory research of wettability and heterogeneity effect on microscopic residual oil distribution in tight sandstone cores during CO₂ flooding

Baoquan Zeng*, Hui Xu, Tailai Qu, Haixia Xu, Shucheng Wu, Jing Wang and Muzhen Zhang

PetroChina Research Institute of Petroleum Exploration and Development, Beijing, China.

*Corresponding author e-mail: 3162606142@qq.com

Abstract. Based on physical simulation experiment and nuclear magnetic resonance (NMR) measurements, the effect of wettability and pore throat heterogeneity upon oil recovery efficiency during CO₂ flooding, were conducted to investigate how crude oil residing in different sized pore are produced by immiscible CO₂ flooding schemes in tight sandstone cores. Experimental results indicate that water wet cores lead to the highest final recovery factor in comparison with intermediate wet cores and weak oil wet cores. The recovery factor difference in clay micro pore is mainly because of the heterogeneity, while the difference in medium pore and large pore is affected by asphaltene precipitation phenomenon. Therefore, it is important to enhance the recovery in the clay micro pores, and focus on the cores permeability reduction caused by asphaltene precipitation. Based on this investigation on the residual oil saturation in pore throats subjected to immiscible CO₂ flooding, it is possible to design an optimum flood scheme which suits for the microscope pore throat characteristics for a given tight oil reservoir. Keywords: asphaltene precipitation; microscope pore throat structure; CO₂ flooding; tight sandstone reservoirs; nuclear magnetic resonance

1. Introduction

Energy demand and greenhouse gas emission reduction are two significant problems confronting the economic development in China [1]. The large amount of unconventional resources especially tight oil has drawn more attention recently. However, the threshold pressure gradient, non-Darcy flow behavior, poor physical properties and low porosity, low permeability and low pressure may elevate the difficulty of field development [2, 3]. Currently, CO₂ flooding has shown favorable recovery potential from either laboratory investigations or pilot applications [4-6].

Nuclear magnetic resonance has been recently developed and become a popular and effective tool of providing realistic assessment of the amount of fluid that can flow in the porous media. A distribution of decay times, called transverse relaxation times (T2), can be used to describe the distance between crude oil and core pore surface. Details of the NMR principles can be found in the following papers.

The major purpose of this study is to elucidate the dependence of residual oil in a microscopic level on the following factors: (1) wettability; (2) microscopic pore throat heterogeneity. In this paper, seven core samples are obtained from Changqing oilfield in China, we have conducted core-flood tests, NMR
test and constant rate mercury intrusion tests and investigated the injection process, recovery degree and residual oil saturation distribution under different injection schemes. The distribution of remaining oil in different pore throats was analyzed by NMR, and the reason of different residual oil saturation was elucidated.

2. Experimental set-up and procedures

2.1. Materials
Oil samples and sandstone core samples are collected from the layer of Chang 8, which located in the southern district of Ordos basin. The average porosity is 10.65%, and average permeability is 0.4 mD, belonging to extra-low permeability reservoirs. Properties of core samples used in the experiments are listed in Table 1. The crude oil used in this experiment is a dehydrated crude oil with viscosity of 3.2 cp and at reservoir conditions of 70 °C and 18 Mpa. A synthetic brine has a salinity equals 61237mg/L, and viscosity of 0.5 cp at 70 °C. CO2 with a purity of 99.99% is supplied by Beijing Gas Supply Station (China).

| NO. | Permeability /mD | Porosity /% | Pore and Throat Type | Wettability type |
|-----|------------------|-------------|----------------------|------------------|
| 1-1 | 0.375            | 5.60        | Heterogeneity        | Weak Oil Wet     |
| 2-1 | 0.589            | 16.10       | Homogenity           | Intermediate Wet |
| 3-1 | 0.566            | 15.10       | Homogenity           | Water Wet        |

2.2. Experimental Setup
As shown in Figure 1, the experimental apparatus is mainly composed of five parts: injection system, displacement system, production system, pressure control system and data acquisition system. In the injection system, the simulated brine water, formation oil and CO2 are stored in container. In the production system, a backpressure regulator is used to set the outlet pressure. The produced CO2 is measured by a gas flow meter. The data acquisition system is used to record the displacement pressure and flow rate.

![Figure 1](https://example.com/flood_test_flow_diagram.png)

**Figure 1.** Flood test schematic flow diagram

NMR testing can detect the hydrogen nucleus signal to analyze the pore fluid distribution by tuning the instrument frequency to hydrogen nucleus magnetic resonance frequency. Since there is no nuclear magnetic signal in the brine water which is treated by MnCl2 solution, the nuclear magnetic signal would be originated from the oil in the core. Therefore, the signals of crude oil and water can be differentiated. A constant speed mercury injection setup is used for conducting the mercury intrusion measurements. The maximum injection pressure is 6.2MPa corresponding to a pore throat radius of 0.12 μm and a
mercury injection rate of 0.00005 mL/min. The mercury intrusion measurements can be used to measure pore throat radius distribution of the core.

2.3. Experimental Procedure
Experimental procedure is briefly described as follows. (1) Firstly, Air permeability of core samples from different layers are measured with the flow meter method. Then after being vacuumed for 24 h, the core samples is saturated with simulated brine water under a pressure of 12.00 MPa for 12 h, the porosity of each core sample is determined by dividing the difference between dry weight and wet weight by its volume. Then the core sample saturated with brine is placed in the NMR apparatus for testing its transverse relaxation time (T2) spectrum. (2) To eliminate the hydrogen signals of water in the core, the core sample is first displaced with 1.5 times the pore volume (PV) of Mn2+ solution (15000 mg/L), and the sum of signal is usually should kept below the 1% of saturated water core. (3) NMR T2 spectrum is measured after the core was saturated with the oil till the initial oil saturation is achieved. (4) The core samples are displaced under CO2 immiscible injection schemes, and the NMR T2 spectrum is measured again at the end of the displacement. The slim tube test is a wide used standard method to measure MMP in the petroleum industry, through the slim tube test we can determine the MMP is 28.5MPa.

2.4. Relationship between T2 Response and Pore Throat Radius
Total T2 response of fluids in porous media can be described with the following Equation

\[ T_{2,\text{surface}} = \frac{1}{\rho_2 F_s} r_c \]  

(1)

Where \( \rho_2 \) is the surface relaxivity (μm/ms), \( F_s \) is the dimensionless shape factor of a pore, and pore radius, \( r_c \) (μm). When a given core is considered, its surface relaxivity (\( \rho_2 \)) and shape factor (\( F_s \)) can be assumed to be constant. Thus, after obtaining the constant \( \rho_2 \) and \( F_s \), the T2 spectrum of NMR can be eventually converted into the distribution curve of pore throat radius.

3. Results and discussion
There exists no large difference in pore size, and the pore exhibits the normal distribution mainly in the 100-150 μm. Unlike pore distribution, there is a huge difference in the throat distribution. Weak oil-wet cores have the narrowest throat distribution, changing from 0.2-0.6 μm and the throat peak radius slightly increases along with an increasing permeability. Nevertheless, as for the intermediate wet cores and water wet cores, if the permeability is greater than 0.3×10-3μm2, the peak radius of throat will increase from 0.5 μm to 0.6 μm. At the same time, the distribution range of throat radius becomes wider obviously, changing from 0.3-1.0 μm to 0.3-2.5 μm, 0.2-0.8 μm to 0.2-4.2 μm respectively. We can infer that under the same conditions of permeability, the water wet cores have the strongest heterogeneity followed by intermediate wet cores and weak oil wet cores.

As is shown in the Figure 2, T2 distribution curves of saturated water (black curve) can reveal the distribution of pore throats in cores. T2 distribution curves under saturated oil condition (red curve) can reveal the distribution features of pores saturated with oil, and the black section reflects the distribution of irreducible water in pores. T2 distribution curves under residual oil condition (blue curve) can reveal...
the distribution of residual oil in pores, and the red section reflects distribution of the produced oil in pores. During the core flood process, the CO\textsubscript{2} fluid can go into the cores to displace the original oil, therefore, the signal of oil decreases, and this is proportional to the displaced quantity of CO\textsubscript{2} fluid. Therefore, study of T2 distribution allows us to analyze and compare the microscopic change of original oil in pore under different injection schemes. Methodology used to determine the recovery degree of pore throats falling from 0.1ms-1000ms, give the area (S\textsubscript{red} + S\textsubscript{blue}) stand for the initial oil content contained in different pore throats, and the area S\textsubscript{blue} stand for the residual oil, the following formula is used to determine the recovery degree in different pore throats.

There is certain corresponding alternate relation between T2 distribution and pore throat distribution in the sandstone sample. The relaxation time of T2 is converted into pore radius using the modified linear relation through the equation 1. Pore throats with radius (<1μm) would correspond to the size of clay micro pore, while pore throats with radius more than (>10μm) belong to the large pore. The medium pore is between them (1-10μm).

The initial original oil in all the core samples are mainly distributed in 1-10μm and >10μm pore throats, then followed by the range of <1μm radius. The weak oil wet cores #1-1, #1-2 have highest oil saturation in clay micro pore fall between 55.33% and 85.77% and total oil saturation with an average value 82.7%, followed by intermediate wet cores (54.73%) and water wet cores (28.18%). When oil has been injected in porous medium, there exists capillary pressure P\textsubscript{c}. Oil can displace water only if the driving force overcomes the capillary pressure. The oil as wetting phase in the weak oil wet cores obvious can be easily displaced into the clay micro pore. For another, the oil as non-wetting phase have greater resistance force when displacing the wetting phase (water) in the intermediate wet and water wet cores. Accordingly, weak oil cores have the highest original oil saturation.

![Figure 2 - Schematic diagram for calculating the recovery degree of pore throats with NMR technique](image)

The measured NMR amplitude distribution as a function of pore size under various conditions for the core samples are plotted in Figure 3. On the whole, the water wet core yield a favorable recovery factor (55.33%) compared the intermediate wet core (49.30%) and weak oil wet core (42.39%). During the immiscible CO\textsubscript{2} flooding experiment, The heterogeneity cores #2-1 and #3-1, after the CO\textsubscript{2} flooding, the residual oil was mainly stayed in the clay micro pore, only 8.73% and 14.27% of crude oil has been recovered from smaller pore throats with radius less than 1μm. It is easier for bigger pore throats to cause the viscous fingering in immiscible CO\textsubscript{2} flooding, and the viscous fingering leads to the channelling and early breakthrough of the displacing phases, which reduces the effective sweeping area and a larger degree of pore throat heterogeneity lead to more residual oil in clay micro pore. But when it comes to homogeneity core #1-1, it possesses a higher content of smaller pore throats and a lower
heterogeneity exists in its pore throats, with relatively larger percolation resistance, then CO₂ would intrude into small pore throats, improving the recovery degree of the clay micro pore, thus the average 42.97% of crude oil can be brought out by CO₂ flooding way. It is interesting to note that, the oil wet cores give the worst recovery factor and leave the maximum residual oil in the medium pore and large pore. It is speculated that some precipitated asphaltenes are left in the medium pore and large pore, and the light hydrocarbons are extracted from the original crude oil due to strong light-hydrocarbons extraction by CO₂ flooding. Figure 4 shows the respective compositional analysis results and grouped carbon number distributions of the produced oil and the original crude oil. It can be clearly seen that the oil wet core have more light hydrocarbons (C8-C14), which lead to higher mole percentage of more heavy hydrocarbons remaining in the core. We can infer that permeability reduction because of the hydrocarbons precipitated in the narrow throat radius, it may be the crucial factor for lowest recovery factor in the medium pore and large pore.

Figure 3. Distribution of pore size for different cores under different conditions with NMR technique

Figure 4. Compositional analysis results of the original crude oil and the produced oil collected in test
4. Conclusion
Through the constant-rate mercury injection method, it is found from core samples with the similar permeability, average pore size is in a range of 100-150μm and pore throat radius of homogeneity cores is in a range of 0.2-0.6 μm. Heterogeneity cores throat radius is in a range of 0.2-4.2 μm. By employing the core flood and NMR relaxometry measurements, we investigated how crude oil residing in different sized pore is recovered by different injection scenarios in different wettability and heterogeneity cores, and quantify the recovery factors and residual oil distribution in different sized pore. Relatively speaking, after immiscible CO₂ flood in heterogeneity cores, more oil has been produced from moderate and bigger pore throats and the residual oil is mainly present in smaller pore throats due to microscopic pore throat heterogeneity. Because of the hydrocarbons precipitated in the narrow throat radius, it may be the crucial factor for lowest recovery factor in the medium pore and large pore.

Acknowledgments
The work was supported by National Technology Major Project of China (2017ZX05013-001).

References
[1] Bachu, S., CO₂ storage in geological media: role, means, status and barriers to deployment, Progress in Energy and Combustion Science. 34(2008) 254-273.
[2] Ming Q, Wang. R. C., Characteristics and influencing factors of movable fluid in ultra-low permeability sandstone reservoir, Acta Petrolei Sinica. 4(2008)10-16.
[3] Hoffman, B. T., Comparison of various gases for enhanced recovery from shale oil reservoirs, In SPE Improved Oil Recovery Symposium. Society of Petroleum Engineers, 2012.
[4] Arshad, A., Al-Majed, A. A., Menouar, H., Muhammadain, A. M., & Mtawaa, Carbon dioxide (CO₂) miscible flooding in tight oil reservoirs: A case study, In Kuwait International Petroleum Conference and Exhibition, Society of Petroleum, 2009.
[5] Ren, B., Xu, Y., Ren, S., Li, X., Guo, P., & Song, X., Laboratory assessment and field pilot of near miscible CO₂ injection for IOR and storage in a tight oil reservoir of Shengli Oilfield China, In SPE Enhanced Oil Recovery Conference. Society of Petroleum Engineers, 2011.
[6] Ren, B., Ren, S., Zhang, L., Chen, G., & Zhang, H., Monitoring on CO₂ migration in a tight oil reservoir during CCS-EOR in Jilin Oilfield China, Energy. 98(2016)108-121.