New insight to experimental study on pore structure of different type reservoirs during alkaline-surfactant-polymer flooding

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Abstract

Recently, as the demand for fossil and renewable energy continuously increases, enhancing oil recovery has become one of the key methods to meet the increased requirement. However, most of the oilfields are facing serious problems, including formation heterogeneity and low recovery factor. Therefore, further analysis is required to study the distribution of remaining oil and how to enhance oil recovery effectively. In this study, the core samples of different reservoir types were employed and characteristics of pore structure were measured by a high-pressure mercury porosimeter. The recovery factor and distribution of remaining oil with different reservoir types were determined by core flooding experiments and nuclear magnetic resonance tests. According to the results, the heterogeneity of pore structure becomes weaker as the permeability of the reservoir increases. The recovery during different periods improved as the core permeability increased. The distribution of remaining oil in different pore sizes has an obvious difference. The contribution of the recovery factor is highest in small pores and mesopores for type II reservoir while is greatest in mesopores and macropores for type III reservoir. These results can provide theoretical and technical support for further enhancing oil recovery.

KEYWORDS
alkaline/surfactant/polymer flooding, distribution of remaining oil, enhance oil recovery, nuclear magnetic resonance test, pore structure

1 | INTRODUCTION

In recent years, most of the oilfields have entered the middle and later stage with serious problems, including formation heterogeneity and low recovery factor.1-3 As the demand for fossil and renewable energy continuously increases in the world,4 crude oil plays a critical role in the maintenance of energy consumption nowadays.5,6 The Global Energy Review 2021 predicts that global energy demand has grown by 4.6% in 2021. Global oil demand is expected to increase by 5.4 million barrels a day from the same period last year, up 6% from a year
earlier. However, it is more difficult for most of the oilfields to enhance oil recovery due to long-time water flooding.\textsuperscript{7–9} Up to the present time, there is still a significant portion of residual oil unrecovered in the reservoir.\textsuperscript{10,11} Additionally, the scatter of remaining oil is another serious problem that restricted the development of oil recovery.\textsuperscript{12,13} To further enhance oil recovery, some efficient methods are needed for oil production.\textsuperscript{14}

In the past decades, numerous methods were conducted to improve the reservoir condition and oil recovery.\textsuperscript{15,16} Among them, chemical flooding, especially ASP flooding, has become one of the most effective methods, which were widely used in China’s mature oilfields. It has been reported that the recovery rate of Daqing Oilfield increases more than 20% since ASP flooding has been put into industrial scale application.\textsuperscript{17–19} In the ASP flooding system, polymer can increase the solution viscosity to improve the mobility ratio. Surfactants can reduce the interfacial tension (IFT) of oil/water so that the remaining oil in small pores can be mobilized. Moreover, alkali can form in suit soaps by reacting with crude oil, which can emulsify the oil droplets and reduce the surfactant adsorption in the reservoir. The synergy work of all additives used in slug can reduce the cost and increase the efficiency significantly.\textsuperscript{20–23} Therefore, ASP flooding has been widely used and become an efficient method for a conventional reservoir to enhance oil recovery.\textsuperscript{24–26}

However, due to long-time water and ASP flooding, the reservoir becomes more heterogenous with prominent interlayer contradictions although there is still a large amount of residual oil that exists, which cannot be swept.\textsuperscript{27} As most of the reservoirs in China are continental sandstone reservoirs with different geological conditions,\textsuperscript{28} One of the greatest challenges is how to keep stable production for different types of reservoirs.\textsuperscript{29,30} To study the properties of different reservoirs, PetroChina has divided the reservoir into five types according to the permeability and porosity. Detailed classification methods are shown in Table 1. Therefore, the development based on the feature of reservoir and pore structure is one of the most effective ways to further enhance oil recovery. Especially, for the reservoir of types IV and V, which have lower porosity and permeability, and are considered tight oil. Most scholars have expressed that ASP flooding is not suitable for reservoirs with lower porosity and permeability like types IV and V reservoirs. As a result, we only conducted this study with these three kinds of reservoirs.

Nowadays, many scholars focus on the development effect of pore structure. The accurate identification of pore structure and the remaining oil are essential to enhance oil recovery for the mature conventional oilfield.\textsuperscript{31,32} Numerous attempts were conducted to describe and analyze the reservoir as well as residual oil.\textsuperscript{33,34} High-pressure mercury injection experiment can observe the distribution and proportion of pore throat of different sizes.\textsuperscript{35} CT scanning as well as scanning electron microscopy (SEM) can provide a visual micromodel to observe the pore structure from a microscopic view.\textsuperscript{36,37} Although the casting of thin sections can provide the displacement mechanism in porous media, the distribution of the fluid in the pore throat of different sizes cannot be given.\textsuperscript{38} However, in term of these visual models, the pore structure of core samples may damage and the characteristics of samples are sensitive, which affect the accuracy of the result.\textsuperscript{39} Recently, nuclear magnetic resonance (NMR) tests have become one of the most applied methods to evaluate the pore structure.\textsuperscript{39,40} Furthermore, the NMR result curve can quantitatively give out the content of remaining oil in different pore sizes. Numerous studies have been conducted on pore structure by NMR tests in unconventional reservoirs.\textsuperscript{41,42} Nevertheless, this method has not been used in the evaluation of conventional reservoirs. Besides, the pore radius calculated by NMR test is referred as an empirical formula, which lead the result inaccuracy easily.

Hence, the main objective of this study was to study the distribution of residual oil. In particular, further analysis is required to enhance the oil recovery with heterogenous reservoirs after secondary oil recovery. In this study, core samples of different reservoir types were employed and characteristics of different pore structures were measured by a high-pressure mercury porosimeter. Then the ASP flooding experiments were conducted to compare the effect on recovery factors of different permeability. At last, the NMR test was performed after each period of flooding experiments to identify the distribution of remaining oil in different pore sizes. The results can provide theoretical and technical support for further enhancing oil recovery.

\section*{2 \hspace{1em} MATERIALS AND METHODS}

The materials and methods used in the study are shown in this section.

\begin{table}[h]
\centering
\caption{Classification of reservoir}
\begin{tabular}{|l|l|l|}
\hline
Type & Porosity (%) & Permeability (10^{-3} \textmu m^2) & Evaluation \\
\hline
I & >25 & >1000 & Best \\
II & 20–25 & 100–1000 & Good \\
III & 15–20 & 10–100 & Middle \\
IV & 10–15 & 1–10 & Bad \\
V & 5–10 & 0.1–1.0 & Worst \\
\hline
\end{tabular}
\end{table}
2.1 Materials

Chemical agents: partially hydrolyzed polyacrylamide (HPAM) with a molecular weight of 1900 million. Petroleum sulfonate with an effective content of 42%. Na₂CO₃ was used as the alkali agent. The formula of the ASP system is 0.3%alkylbenzene sulfonate + 1.2% NaOH + 2% HPAM, which is widely used in Daqing Oilfield.

Oil: The oil used in the experiment was prepared by mixing Daqing crude oil with jet fuel according to the viscosity of Daqing crude oil (9.3 mPa s) at the reservoir temperature.

Water: The water used in the experiment was obtained from Daqing Oilfield with a total salinity of 4672 mg/L.

Core: The cores with different permeabilities were provided by a block of Daqing Oilfield. All the experiment samples should conform to the characteristic of different reservoir types.

Temperature: The oil flooding experiments were conducted at a reservoir temperature of 45°C.

2.2 Experimental method

2.2.1 Distribution of pore-throat size and distribution

The pore-throat distribution tests were conducted on the core samples before the experiment. A high-pressure mercury porosimeter was used to measure the size of the pore-throat. The distribution and microstructure of pore samples with different permeability were measured by SEM. The distribution probability of different pore radii can be calculated according to the capillary pressure curve combined with mercury volume.

2.2.2 NMR test

The NMR test was carried out after each period of core flooding experiments by an NMR spectrometer. The pore structure can be analyzed after the oil is saturated. Then the identification of oil and water in the core samples can be obtained after water and chemical flooding.

2.2.3 Core flooding procedure

The schematic diagram of the core flooding experiment is shown in Figure 1. The displacement device includes an ISCO pump, intermediate vessels, and a core holder. The experiments were conducted in a thermostat oven to keep the temperature at 45°C. Details of the flooding procedure are as follows:

1. Dry core samples for 24 h and then measure their dimension and weight.
2. Put core samples in the high-pressure mercury porosimeter to obtain the distribution of pore throat. Then the scanning electron microscope tests can be conducted to obtain the microstructure of core samples.
3. Water saturated: Vacuum the core slugs for 10 h before the cores are saturated with stimulated water. Then measure core samples in an NMR spectrometer after water saturation and centrifuge to obtain the initial $T_2$ spectrum.
4. Oil saturated: Oil was injected into core samples at a rate of 0.1 ml/min until water appears at the outlet and then kept in the thermostat to age the oil for 1 day. Then measure core samples using an NMR spectrometer to obtain the $T_2$ spectrum.
5. Water flooding: Inject stimulated water at a rate of 0.1 ml/min until the content of water is 100% at the outlet. Then measure core samples using an NMR spectrometer to obtain the $T_2$ spectrum.
6. ASP flooding: Inject 0.7 PV ASP slug as well as stimulated water at a rate of 0.1 ml/min until the content of water is 100% at the outlet. Then measure core samples using an NMR spectrometer to obtain the $T_2$ spectrum.
3 | FUNDAMENTAL THEORY

3.1 | Pore radius conversion theory is based on NMR

According to the NMR relaxation mechanism, the transverse relaxation time $T_2$ in a uniform magnetic field is:

$$\frac{1}{T_2} = \frac{1}{T_{2B}} + \rho_2 \frac{S}{V} + \frac{D(yGT_g)^2}{12},$$

where $\frac{1}{T_{2B}}$ is the bulk relaxing time, $\rho_2 \frac{S}{V}$ nuclear magnetic resonance, and $\frac{D(yGT_g)^2}{12}$ is the diffusion relaxing time.

When only one fluid exists, the bulk relaxing time and the diffusion relaxing time can be ignored. As a result, the result is affected by surface relaxing time. The formula can be simplified as follows:

$$\frac{1}{T_2} = \rho_2 \frac{S}{V},$$

where $\frac{S}{V}$ is the specific surface of the pore. For spherical pores, $\frac{S}{V} = \frac{3}{r}$. For columnar throat, $\frac{S}{V} = \frac{2}{r}$. The surface relaxation rate $\rho_2$ can be approximated as a constant for the same rock sample.

3.2 | Relationship between $T_2$ spectrum and remaining oil

The area surrounded by the $T_2$ spectrum and signal amplitude in the NMR result curve reflects the volume of fluid in the pores, and the relationship is shown as follows:

$$v_0 = \alpha S(T_2),$$

where $V_0$ is the fluid volume, $S(T_2)$ is the area of the $T_2$ spectrum curve and the total NMR signal amplitude, $\alpha$ is the coefficient conducted by experiment.

The area difference of the two curves represents the change of fluid volume, and the formula of the remaining oil volume is:

$$\Delta V = v_1 - v_2 = \alpha S_1 - \alpha S_2.$$

Then the recovery factor $\eta$ can be given as follows:

$$\eta = \frac{\Delta V}{V_1} \times 100\% = \frac{S_1 - S_2}{S_1} \times 100\%.$$

4 | RESULTS AND DISCUSSION

4.1 | Basic properties of experiments

4.1.1 | Core sample properties

Basic properties, including permeability, porosity, and classification of core samples are shown in Table 2.

4.1.2 | Fluid properties

The IFT of ASP solution is $5.7 \times 10^{-3}$ mN/m, which would be considered the ultralow IFT. Additionally, the viscosity of the ASP slug is 59.5 mPa.s.

4.2 | Results of pore-throat distribution based on a scanning electron microscope (SEM)

The difference in microstructure of core samples was compared by SEM. SEM results of core samples with different core samples are shown in Figure 2.

From Figure 2 we can see that as the core permeability becomes higher, the pore size of core samples becomes larger and interpore connectivity becomes stronger.

4.3 | Results of pore-throat distribution based on high-pressure mercury

We can obtain the capillary pressure curves and pore-throat distribution maps through a high-pressure

| Core sample | Square (cm²) | Length (cm) | Permeability ($10^{-3}$μm²) | Porosity (%) | Type |
|-------------|-------------|-------------|-----------------------------|-------------|------|
| L-3         | 4.91        | 10          | 50.5                        | 23.2        | III  |
| L-2         | 484.9       | 26.3        | I                           |             |      |
| L-1         | 925.8       | 27.4        | I                           |             |      |
mercury injection experiment. The results are plotted in Figure 3 and some details are shown in Table 3.

The results demonstrated that there is a direct relationship between the distribution of pore throat and permeability. As permeability increases, the peak of pore distribution moves to the right, the distribution of large pores increases, and the heterogeneity of pore structure becomes weak. For the core sample of L-3, the frequency between 4 and 6.3 μm is the highest and the permeability contribution is mainly at 6.3 μm. Moreover, the highest distribution frequency of L-2 and L-1 are increased to 10–16 μm. In addition, it is important to point out that there exists the size of pore-throat at 40 and 63 μm, respectively, which only appear in the L-1 core. It is apparent from this table that the contribution of coarse pore throat to permeability is more obvious.

The capillary pressure curve above illustrates that the distribution of pore throat becomes wider and scatter as permeability increases. Equally important, thin throat is mainly distributed in type III reservoir while thick throat always appears in type II and type I reservoir.

Table 3 illustrates the characteristic parameters of pore structure. As mentioned above, the maximum and mean pore radius increased as the permeability of cores increased. The sorting coefficient represents the uniformity of the pore-throat distribution. It can be found that the sorting coefficient is positively correlated with permeability. Moreover, the structure coefficient usually characterizes the degree of pore curvature and detour. The results showed that there is no close relation between structure coefficient and different reservoir types.

4.4 | Results of NMR tests

The core samples after centrifugal and water saturated were measured by NMR tests. The results of the NMR spectrum are shown in Figure 4.
The curves of pore structure distribution. (A) Curve of L-1, (B) curve of L-2, and (C) curve of L-3.
According to the NMR spectrum result, after water is saturated, the distribution of fluid in different pores scale can determine the porosity of different pore sizes. From the graph above, we can see that the cumulative porosity of L-1 is 27.4%, while the cumulative porosity of L-2 and L-3 are 26.3% and 23.2%, respectively. A closer inspection of the chart shows that the content of bound water is 7%, which appeared to be unaffected by reservoir type. The differences in cumulative porosity mainly appeared to range from 1 to 500 ms by $T_2$ relaxing time. The maximum $T_2$ relaxation time increases according to the permeability increases. The most interesting aspect is that the $T_2$ relaxation time of the L-1 core fluctuates in the range of 700–2000, which is different from that in L-3 as well as L-2. It also proves that there are macropores in L-1, which do not exist in other core samples.

### 4.5 Conversion of $T_2$ relaxation time and pore-throat radius

#### 4.5.1 Conversion result

In this study, the result of the NMR and high-pressure mercury injection experiment is depicted in one chart. The composite curve is shown in Figure 5. According to the result of the conventional mercury experiment, the minimum and maximum pore throats are 0.016 and 63 μm, respectively. The maximum and minimum relaxation times obtained by the NMR test are 2.115 and 353.93 ms, respectively. The relationship between relaxation time ($T_2$) and pore radius ($r$) is shown in Figure 6.

#### 4.5.2 Pore-scale classification

It can be seen in the NMR result curves that the distribution of remaining oil was in the pore size of 0.1–200 μm. As a result, the pore throats are divided into the following four categories according to fluid distribution, the classification details are shown in Table 4.

| Number | Core | Permeability ($10^{-3} \mu m^2$) | Parameters | Mean pore radius (μm) | Sorting coefficient | Structure coefficient |
|--------|------|---------------------------------|------------|----------------------|---------------------|----------------------|
| 1      | L-3  | 80.0                            | Maximum pore radius (μm) | 13.09 | 3.16 | 2.56 | 1.77 |
| 2      | L-2  | 475.1                           | Mean pore radius (μm)    | 26.29 | 5.71 | 4.11 | 0.74 |
| 3      | L-1  | 927.6                           | Sorting coefficient     | 75.00 | 8.35 | 5.38 | 1.29 |

#### 4.5.3 Results of pore-throat distribution

According to the above method, the distribution of fluid in different pore sizes can be calculated by the $T_2$ relaxing time after water is saturated. The details of fluid distribution are summarized in Table 5.

From this data, we can see that the pore volume of L-1 in macropores is obviously larger than those in L-3 and L-2. The pore size of L-2 mainly consists of mesopores and small pores. Moreover, the proportion of micropores in L-3 is the highest. The correlation between pore size and permeability appears to be in close relation to the higher permeability due to the higher proportion of the pore with large size.

### 4.6 Core flooding results

To investigate the oil recovery of ASP flooding with different permeability, core samples of different reservoir types were used to conduct flooding experiments. The results, including oil recovery, pressure, as well as water cut, are presented in Figure 7. Since the study focuses on the recovery of different permeability, the formula of different experiments is the same. Some details and brief descriptions are summarized below to show the outcomes of the experiment.

The performance of the flooding experiment with type III is shown in Figure 7A. During the period of water flooding, water cut increased rapidly when the injection volume reached 1.5 PV. The water recovery is 38.3% at this stage. Then ASP flooding was carried out until the water cut reached 100%. In this period, the water cut decreased obviously and recovery improved. Additionally, the pressure only has a tiny change after ASP flooding. The cumulative recovery is 56%.

Figure 7B presented the flooding result of the core sample from the type II reservoir. During water flooding, the injection pressure was unchanged and 42% original oil in place (OOIP) was achieved before
The ASP slug was injected. The final recovery improved by 25.5% OOIP.

As shown in Figure 7C, the injection pressure significantly increased after water as well as ASP injection. Water cut decreased after ASP flooding. After subsequent water flooding, pressure decreased and then became stable. The final recovery is 62.7%.

The recovery of different core samples was compared in Figure 8. It is apparent from this chart that ASP flooding can significantly improve recovery factors on the
basis of water flooding. Among them, it has the best effect on the core sample of type III.

**4.7 Analysis of remaining oil mobilization**

**4.7.1 Mobilization of remaining oil of different pore size**

The NMR curves of different permeability at different periods during the flooding experiments are depicted in Figure 9. The recovery factor of different pore sizes is analyzed in Figure 10. ASP flooding has the best effect on type III reservoir. These results can help us further study the distribution of the remaining oil. As can be seen from the NMR result, there are three obvious wave crests in the curve. The remaining oil in different pore sizes decreases obviously after water flooding. It can also certify that the pore size of core sample with different permeability has a increasing tendency after ASP flooding. However, there was a significant difference in the distribution of remaining oil among different pore sizes.

From the graph above, we can see that the oil production was greatest in smallpores for the L-3 core sample. As the micropores are mainly concentrated around 0.1 \( \mu \)m, ASP flooding not only cannot enlarge the sweep degree of the micropores but also emulsify the remaining oil in smallpores into micropores, which has a bad effect on the recovery factor. The contribution of the recovery factor is highest in smallpores and mesopores for the L-2 core sample. What is interesting in this chart is that the proportion of recovery contribution in smallpores is higher after waterflooding while is lower after ASP flooding compared with mesopores. It can also confirm that ASP flooding can enlarge the sweep volume of micropores. Under the synergistic action of ASP flooding, surfactant and alkali can enter the micropore to reduce the interfacial tension and improve washing efficiency in smallpores. For the L-1 core sample, the oil production was greatest in mesopores and macropores with a proportion of 43.5% and 41.77%. During the experiment, the ASP flooding system can emulsify the remaining oil in smallpores into oil droplets but cannot affect the remaining oil in the micropores. As a result, the recovery factor of type III reservoir cannot be improved in micropores.

Meanwhile, to verify the accuracy of experimental results, the recovery factor of the flooding experiment and the NMR test are compared. The result is presented in Table 6.

From this data, we can see that the result of the NMR test and flooding experiment did not differ. In general, the results testify that the recovery factor calculated by the NMR test is accurate and reasonable.

**4.7.2 Distribution of remaining oil after ASP flooding**

The distribution of remaining oil with different core samples after ASP flooding can be obtained according to the NMR experiment. The results are shown in Figure 11.

From the data in Figure 11, it is apparent that the percentage of remaining oil is highest in smallpores in type III reservoir as well as micropores in type II reservoir after ASP flooding. For type I reservoir, the remaining oil most exists in mesopores. As a result, in tertiary oil recovery, the critical problem is how to efficiently develop and reduce the remaining oil of different types of reservoirs. For example, we can use nanofluid as a displacement agent to enhance the oil recovery in type III reservoir.
In this study, an NMR interpretation method combined with the result measured by a high-pressure mercury porosimeter was proposed. The effect of recovery factors with different reservoir types and the distribution of remaining oil were determined by core flooding experiments and NMR tests. The main findings and conclusions are as follows:

1. There is a direct relationship between the distribution of pore throat and permeability. The pore radius increased and the heterogeneity become weaker as the permeability of the reservoir increased. The sorting coefficient is positively correlated with permeability while the structure coefficient has no close relation to reservoir permeability.

2. The pore throats can be divided into four categories according to the fluid distribution conducted by NMR tests combined with high-pressure mercury injection. The correlation between pore size and permeability appears to be in close relation to higher permeability due to the higher proportion of the pore with large size.

3. The recovery is improved as the core permeability increases. ASP flooding can significantly improve the recovery factor on the basis of water flooding. Among them, it has the best effect on the type III reservoir.

4. ASP flooding plays a significant effect on different kinds of reservoirs. Also, the oil remaining in

**FIGURE 7** Core flooding performance with different permeability. Flooding experiment curve of (A) L-1, (B) L-2, and (C) L-3.

**FIGURE 8** Comparison of recovery factors with different types of reservoir

5 | CONCLUSION

In this study, an NMR interpretation method combined with the result measured by a high-pressure mercury porosimeter was proposed. The effect of recovery factors with different reservoir types and the distribution of remaining oil were determined by core flooding experiments and NMR tests. The main findings and conclusions are as follows:

1. There is a direct relationship between the distribution of pore throat and permeability. The pore radius increased and the heterogeneity become weaker as the permeability of the reservoir increased. The sorting coefficient is positively correlated with permeability while the structure coefficient has no close relation to reservoir permeability.

2. The pore throats can be divided into four categories according to the fluid distribution conducted by NMR tests combined with high-pressure mercury injection. The correlation between pore size and permeability appears to be in close relation to higher permeability due to the higher proportion of the pore with large size.

3. The recovery is improved as the core permeability increases. ASP flooding can significantly improve the recovery factor on the basis of water flooding. Among them, it has the best effect on the type III reservoir.

4. ASP flooding plays a significant effect on different kinds of reservoirs. Also, the oil remaining in
FIGURE 9  The NMR result of different permeability. (A) NMR curve of L-1, (B) NMR curve of L-2, and (C) NMR curve of L-3. NMR, nuclear magnetic resonance.

FIGURE 10  Recovery factors of different pore sizes

TABLE 6  Comparison of recovery factor

| Recovery factor (%) | L-3 | L-2 | L-1 |
|---------------------|-----|-----|-----|
| Flooding experiment | 38.3| 17.3| 42  |
| Total               | 55.6| 67.5| 72.7|
| NMR test            | 38.43| 15.77| 43.86|
| Total               | 54.1| 68.1| 72.04|

FIGURE 11  The distribution of remaining oil
different pore sizes is different. The oil production was greatest in smallpores for the L-3 core sample. The contribution of the recovery factor is highest in smallpores and mesopores for type II reservoir. For type III reservoir, oil production was greatest in mesopores and macropores.

(5) The percentage of remaining oil distribution is highest in the smallpores of type III reservoir as well as micropores of type II reservoir after ASP flooding. For type I reservoir, the remaining oil most exists in mesopores. In a further study, the critical problem is how to efficiently develop and reduce the remaining oil for different types of reservoirs.

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CONFLICT OF INTEREST
The authors declare no conflict of interest.

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