Quantitative Investigation of Water Sensitivity and Water Locking Damages on a Low-Permeability Reservoir Using the Core Flooding Experiment and NMR Test

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ABSTRACT: Production of oil and gas energy is often greatly hindered by reservoir formation damage, particularly the occurrences of water sensitivity and water locking damages on a low-permeability reservoir. For the purpose of this paper, a formation damage assessment methodology combining core flooding experiment and NMR (nuclear magnetic resonance) $T_2$ relaxation tests is performed and applied to quantitatively determine water sensitivity/water locking damage on sandstone oil formation. XRD tests are used to analyze the mineral composition of cores. Core flooding experiments are designed to simulate the two damages and determine the permeability reduction. NMR tests are introduced to compare water saturation before and after flooding through rock cores, calculate the porosity damage and changes of the pore size, and analyze the mechanism of water sensitivity and water locking damages. Also, SEM experiments are used to determine the pore morphology before and after damage. Low-permeability sandstone rock cores cored from the Jilantai reservoir are assessed through this whole set of experiments. The results demonstrate that the permeability and porosity of core samples strongly decrease with the occurrence of water sensitivity/water locking damage, reflecting that the Jilantai reservoir has strong water sensitivity and is prone to be damaged by water locking. Compared with the previous formation damage assessment ideas, much attention is given to the microchanges of cores after damage, and using fluorinated oil instead of kerosene can help observe the distribution of water in rock core samples after each flooding by the NMR $T_2$ spectra.

1. INTRODUCTION

As the global demands on energy and depleting conventional reservoirs rise, reservoir production targets are moving toward an increasingly challenging direction, usually exhibiting low permeability.1−5 However, the reservoirs with low permeability that are commonly characterized by the tiny size of the pore throats, high content of clay minerals, high capillary pressure, and high flow resistance, which more easily cause external formation damage during drilling and hydraulic fracturing operation, particularly water sensitivity and water locking damages because of the physical and chemical properties and the hydrodynamic effect of working fluids, exhibit a certain kind of difficulty in development.1−8

Reservoir formation damage can result in substantial production capacity and economic loss; hence, understanding the underlying mechanisms of damages and then implementing corresponding protective measures on the target reservoir formation layer with low permeability so as to minimize the formation damage have very important significance for the construction of productivity and the discovery of target formations.1−9

Traditionally, the standard water flooding experiment is the most popular way to assess water sensitivity and water locking.10,11 Although this approach is able to determine the damage degree, it cannot directly and visually reflect the damage process and mechanism. Microscopy scan, X-ray, and other microstructure and chemical analyses are methodologies used for analyzing the micromechanism of sensitivity damage, but there are still disadvantages, such as the rock core sample, which has to be destroyed for testing only then to observe its microstructure, and these methodologies cannot quantitatively investigate damage effects on water saturation and permeability.12−14

NMR is an efficient way to detect the pore structure and oil and water distributions in reservoir rocks by analyzing the relaxation time of reservoir fluids, which causes no damage on rock cores.15−17 It is great to investigate fluid flow within porous media in the petroleum industry. From NMR results, more information about fluid saturation and accumulation of
fluid in rock pores, fluid features, and rock core structure characteristics can be obtained, and it happens that water sensitivity and water locking damages are related to the water saturation in rock pores. Hence, NMR can be applied in the investigation of water sensitivity and water locking damages on a low-permeability sandstone oil reservoir.

Inspired by the aforementioned research studies, this paper details the quantitative investigation of water sensitivity and water locking damages. The permeability damage and irreducible water/residual oil saturation of three rock core samples cored from Jilantai low-permeability sandstone field were measured by core fluid flooding experiments, and the change of pore size in the core samples and porosity damage were measured by NMR T2 relaxation measurements. Fluorinated oil was used to displace kerosene in the common flooding experiment so that the quantitative distribution of water saturation can be correlated by comparing the difference in the T2 spectra after each flooding for the three core samples. This work therefore helps understand the assessment method and mechanisms of water sensitivity and water locking damages and encourage further studies to develop mature anti-water locking and anti-water sensitivity agents. The formation damage evaluation method can also be applied to assess other types of porous media. The experimental results presented in this paper have been applied to the production of Jilantai oilfield.

2. THEORY

2.1. Analysis of Water Locking and Sensitivity Damage Mechanism. 2.1.1. Water Sensitivity Damage. When the injecting fluid contacted with the reservoir fluid and rock minerals, physicochemical reaction would happen and the pore throat would be narrowed and blocked once the salinity of injecting fluid was lower than that of the reservoir fluid, leading to the reduction of permeability and reservoir damage. Generally, water sensitivity damage is caused by hydration, swelling, migration, and dispersion of clay minerals; therefore, the crack, pore, and throat sizes decrease while the flow resistance increases, leading to the reduction of permeability (cf. Figure 1). In addition, the shrinkage of pores will lead to the increase in capillary pressure and irreducible water saturation. Therefore, the occurrence of water sensitivity damage further aggravates the degree of water locking damage.

2.1.2. Water Locking Damage. The essence of water locking damage is that the water phase occupies different flowing spaces, which impedes the flow of the oil phase and leads to the reduction of oil permeability. The main underlying mechanism of water locking damage is the detention effect of the capillary pressure. When the external aqueous phase fluid is injected into the reservoir, the water saturation Sw of the reservoir will increase. Once the external aqueous phase fluid is sucked into the fine pore throat, building up a curved surface of the concave oil phase in the oil/water interface, and the immovable water saturation exceeds the initial water saturation, there will be capillary resistance in the reservoir (cf. Figure 2).

Figure 1. Plugging of the reservoir pore caused by water sensitivity damage.

Figure 2. Capillary resistance in pores of reservoir rocks.
continuous oil flow into oil droplets. When these droplets flow to the smaller throat, an additional resistance will be produced by the oil droplets due to deformation, which can be expressed by \( \text{eq } 2 \). The as-produced damage to the reservoir is also called the Jamin effect \(^{35-36} \) (cf. Figure 3).

\[
P_{\sigma} = \frac{2\sigma}{(\frac{1}{r_1} - \frac{1}{r_2})}
\]

where \( \sigma \) is the interfacial tension, \( r_1 \) is the pearl bubble back-end meniscus radius, \( r_2 \) is the pearl bubble front meniscus radius, and \( P_{\sigma} \) is the additional resistance of oil droplets.

Once water locking damage occurs, the fluid in the reservoir needs to overcome not only the conventional frictional resistance of fluid flowing but also the capillary resistance and additional resistance of the Jamin effect to flow into the wellbore, or the resistance would keep the water in the rock and hinder the rock permeability from being recovered. \(^{37,38} \)

### 2.2. NMR Test Principles

NMR test signals come from hydrogen atoms. More hydrogen atoms lead to stronger signals. \(^{39} \) However, it is difficult to distinguish the signals from water and oil phases because there are hydrogen atoms in both water and hydrocarbons. Therefore, fluorinated oil was selected to replace kerosene in the core flooding experiment because there is no hydrogen atom in it and it is cheap compared to \( \text{D}_2\text{O} \) so that signals from the oil flood phase cannot be obtained. \(^{40,41} \) Due to the hydrogen atoms only existing in the water phase and residual crude oil, the NMR signals are related to the water saturation of core samples. Hence, the change of water saturation and, further, the pore throat radius is as follows, \(^{39} \)

\[
\frac{1}{T_2} \approx \rho_2 \frac{S}{V}
\]

where \( T_2 \) is the NMR relaxation time (ms), \( \rho_2 \) is the interfacial relativity determined by the mineral constituents and surficial properties of the pores (in./s), \( S \) is the specific pore surface area (in.\(^2\)), \( V \) is the volume of the pore (in.\(^3\)), and \( S/V \) is the specific pore surface area per volume, which is inversely correlated with the pore diameter, which is in 1/in.; it can also be calculated by the following formula, \(^{40} \)

\[
\frac{S}{V} = \frac{F_2}{r}
\]

where \( F_2 \) is the dimensionless shape factor of the pore, and \( r \) is the pore throat radius (in.). Eventually, \( T_2 \) can be expressed by,

\[
T_2 = \frac{1}{\rho_2 F_2}
\]

With assumptions for \( \text{eqs } 4 \) and \( 5 \), the bulk relaxation time and the diffusion relaxation time are both neglected; \( F_2 \) is determined by comparing the \( T_2 \)-weighted average value of the right part of the \( T_2 \) spectrum with the average pore radius from constant-rate fluid injection.

It can be seen from \( \text{eq } 5 \) that the pore throat radius is directly proportional to \( T_2 \).

### 3. RESULTS AND DISCUSSION

#### 3.1. Quantitative Analysis for the Permeability Damage Degree of Cores

Regarding the damage index for the evaluation of the damage degree of water sensitivity/water locking damage, here, \( \text{eq } 6 \) is defined, \(^{43} \)

\[
I = \frac{(K_i - K_f)}{K_i} \times 100\%
\]

where \( K_i \) is the initial permeability, and \( K_f \) is the damaged permeability. According to the \( I \) value, the damage degree due to water sensitivity and water locking can be evaluated, with the evaluation criteria shown in Table 1. It can be seen from \( \text{eq } 6 \) that when the invasive fluid blocks up the whole pore, \( K_f = 0 \) and \( I = 1 \), indicating that the reservoir is subject to the greatest damage degree, while when the invasive fluid is completely drained, that is, \( K_i = K_f \) and \( I = 0 \), demonstrating that the damage on the reservoir is fully removed. Hence, when the \( I \) value is close to 1, the damage degree is more severe. \(^{28} \)

The permeability damage degrees of three core samples obtained from the core flooding experiment are shown in Table 2.
934 is the unit conversion factor, $A$ is the cross-sectional area of the liquid passing through the rock (in.$^2$), and $Δp$ is the pressure difference before and after the liquid passes through the rock (psi).

As can be found from Table 2, the core permeability decreases after damage. The calculated water sensitivity damage degree of core no. 1 is 63.33$, the calculated water locking damage degree of core no. 2 is 71.79$, and the damage degree of core no. 3 with both of the two damages reaches up to 92.02$. So, the Jilantai reservoir rocks have strong water sensitivity, and once the injection fluid was designed inappropriately, the water locking damage would be strong. The results indicate that reservoirs near the wellbore that are damaged by water sensitivity and water locking will suffer dramatic permeability loss, and oil production will also be affected.

### 3.2. Quantitative Analysis for the Irreducible Water/Residual Oil Saturation of Cores

The no. 2 and no. 3 cores saturated with brine were all first forward-flooded with fluorinated oil, and the remaining water in the core could be defined as the initial water saturation of the core. Then, the two cores were backward-flooded with water, and the remaining oil in the core could be defined as residual oil. After the third flooding, the cores were flooded with fluorinated oil again, and the water that remained in the core could be called irreducible oil. The movable water/fluorinated oil that flooded out was measured by a 5 cc pipette with an accuracy of 0.01, and the water/oil saturation of cores after each flood was calculated (cf. Table 3).

| Table 3. Quantitative Results of Core No. 2 and No. 3 |
|-----------------------------------------------|
| core no. | no. 2 | no. 3 |
|-----------------|-------|-------|
| porosity, %      | 13.06 | 13.04 |
| pore volume, cc   | 3.21  | 3.20  |
| movable water that flooded out in the first flood, cc | 2.10  | 2.10  |
| movable fluorinated oil that flooded out in the second flood, cc | 1.50  | 1.48  |
| movable water that flooded out in the third flood, cc | 0.55  | 0.30  |
| initial water saturation, % | 34.58 | 34.38 |
| residual oil saturation, % | 18.69 | 19.38 |
| irreducible water saturation, % | 64.17 | 71.25 |

As can be seen in Table 3, the movable water volume that flooded out is around 1.50 cc, and the pore volume of core no. 2 is about 3.21 cc, so the initial water saturation is about 34.58$. Then, the core is backward-flooded with brine, simulating the injecting process, and the movable oil volume that flooded out is nearly 1.50 cc. With the increase in water saturation, because of the existence of irreducible water, the resistance of oil–water two-phase flow in core pores is very large. Finally, the core is forward-flooded with fluorinated oil, which simulates the process of reservoir fluid flowing into the wellbore. The movable water volume that flooded out is around 0.55 cc, so the irreducible water in the core pore now is about 2.06 cc, and the irreducible water saturation increases to nearly 64.17$. The analysis of core no. 3 is the same.

### 3.3. NMR $T_2$ Spectra

Figures 4–6 illustrate the $T_2$ spectra for core nos. 1–3 flooded with different schemes.

From eq 5, it can be seen that the pore throat radius is directly proportional to $T_2$, meaning that the amplitude of $T_2$ at faster relaxation times represents NMR test signals in small pores, while the amplitude of $T_2$ at slower relaxation times refers to signals in larger pores. To understand the $T_2$ spectra more easily, the $T_2$ spectra are divided into four segments combining the $T_2$ characteristics of cores, which are 0–1, 1–10, 10–100, and 100–1,000 ms. The $T_2$ spectrum distributions of the four segments for core nos. 1–3 are listed in Table 4.

As can be observed from Figures 4–6 and Table 4, with the core samples dried, there are still weak NMR signals in the $T_2$ spectra of all of the three core samples, mainly distributing at 0–1 ms, illustrating that there is residual crude oil in the micropore, which has not been extracted, so that the $T_2$ amplitude weakly exists due to the hydrogen atoms from crude oil. For the above reasons, the real total porosity should
Table 4. \( T_2 \) Spectrum Distributions Obtained from NMR for the Three Core Samples

| core no. | experimental condition | NMR \( T_2 \) distribution, % |
|----------|------------------------|---------------------------------|
|          | 0−1 ms | 1−10 ms | 10−100 ms | 100−10,000 ms |
| 1        | dried  | 81.66    | 18.34     | 0           | 0         |
|          | saturated with brine  | 32.45    | 54.88     | 12.36       | 0.31      |
|          | DI water flood       | 28.94    | 56.87     | 14.19       | 0         |
| 2        | dried  | 76.55    | 23.45     | 0           | 0         |
|          | saturated with brine  | 30.72    | 53.71     | 15.07       | 0.50      |
|          | fluorinated oil flood | 51.33    | 46.06     | 2.61        | 0         |
|          | brine flood          | 36.44    | 53.41     | 10.06       | 0.08      |
|          | fluorinated oil flood | 43.29    | 51.94     | 4.76        | 0         |
| 3        | dried  | 87.92    | 12.08     | 0           | 0         |
|          | saturated with brine  | 33.73    | 51.75     | 12.54       | 1.99      |
|          | fluorinated oil flood | 52.32    | 44.98     | 2.70        | 0         |
|          | DI water flood       | 32.94    | 55.83     | 10.55       | 0.68      |
|          | fluorinated oil flood | 38.13    | 57.21     | 4.66        | 0         |

To see the changes at the pore level, scanning electron microscopy (SEM) is conducted. The SU8010 cold field emission scanning electron microscope of Hitachi, Japan, used in this experiment is a micro-imaging equipment with high precision. When the acceleration voltage is 15 kV, the resolution of the equipment can reach 1.0 nm, and the magnification is 100−300,000x in high power mode and 20−2000x in low power mode. The experiment is carried out according to the industry standard.43 The core samples before and after treatment by the same displacement experiment method were dried and vacuumed to prepare small samples, and the changes of pore structure and clay on the fresh surface of the core samples were observed under a scanning electron microscope. As shown in Figure 8a−d, the SEM images of the untreated core samples (Figure 8a1−a3), the core samples after water sensitivity damage only (Figure 8b1−b3), the core samples after water locking damage only (Figure 8c1−c3), and the core samples after water lock and water sensitivity damages simultaneously (Figure 8d1−d3) were respectively represented.

It can be seen from Figure 8a that the clay mineral content of the core is high, and the micro- and nanopore throats of the rock are developed. There are certain cracks between the minerals due to the difference in mechanical properties. Figure 9 shows the experimental results of energy dispersive X-ray spectroscopy (EDS). It can be found that the clay minerals are mainly silicate aluminates.

It can be found from Figure 8b that many “new” clay particles are distributed in the originally relatively “clean” pores or fractures, which is due to the fact that the minerals tend to get expanded after the water sensitivity damage on the core. Also, when these minerals interact with foreign fluids, their stability is reduced and they are apt to come off from the rock surface and migrate with the fluids in the pore throat. Figure 8c shows the microscopic results of pores in cores after water locking damage, and it can be found that there is no particularly significant change in pores and fractures relative to undamaged cores, as well as the minerals. Interestingly, nanoscale fractures have been added in some areas of the rock (cf. Figure 8c3), which is not found in scanning electron microscopy experiments of undamaged cores, and these fractures may be occasionally generated by pressure during displacement. The performance of pores, cracks, and clay in Figure 8d is mainly similar to that in Figure 8b. Clay increases, expands, and adheres to the crack wall, indicating that the
water sensitivity damage has a significant impact on the original pore structure of the rock, and this effect is very weak for the water locking damage. At the same time, it can be found that there are many very large fractures in rocks and clays. After water sensitivity damage and water locking damage, the experimental sandstone core is very unstable.

According to previous studies, it is defined that the pore throats corresponding to $T_2 < 10$ ms are small pores with a radius of less than 0.0787 mil, the pore throats corresponding to $10 < T_2 < 100$ ms are medium pores with a radius range of $0.0787 - 0.7874$ mil, and the pore throats corresponding to $T_2 > 100$ ms are large pores with a radius of more than 0.7874 mil. With this method, the $T_2$ distribution of the three core samples in Table 4 is approximately converted to the pore size distribution, as shown in Table 5.

It can be seen from Table 5 that for the three core samples, the average pore radius size is less than 0.0787 mil. Few pores have the radius in the range of 0.0787–0.7874 mil, and very few pores have a radius greater than 0.7874 mil. With the flooding, the distribution of pore size together with its average value decreases as a whole for core no. 1 and no. 3 due to the water sensitivity damage, while the large pore and extra-small pore reduce and the small-medium pore increases relatively. It can be attributed to the fact that the swelling damage on clay in an extra-small pore would lead to the blockage, which will lead to the disappearance or smaller size of extra-small pores. Also, the measured permeability also decreases, indicating that the permeability is closely related to the core pore size. Therefore, the water sensitivity damage has a serious impact on low permeability damage. The pore size for core no. 2 shown in Table 4 also decreases, but rarely, correspondingly, the permeability decreases, too. However, the main reason for the $T_2$ spectrum decline is the small pores occupied by residual
Table 5. Pore Size Distribution of the Three Core Samples

| core no. | experimental condition | core pore size distribution, $T_2$ amplitude |
|----------|------------------------|---------------------------------------------|
|          |                       | <0.0787 mil | 0.0787–0.7874 mil | >0.7874 mil |
| 1        | dried                  | 92.13      | 0               | 0           |
|          | saturated with brine   | 934.17     | 132.21          | 3.36        |
|          | DI water flood         | 718.51     | 118.84          | 0           |
| 2        | dried                  | 124.84     | 0               | 0           |
|          | saturated with brine   | 981.55     | 175.14          | 5.79        |
|          | fluorinated oil flood  | 471.07     | 12.64           | 0           |
|          | brine flood            | 871.86     | 97.64           | 0.82        |
|          | fluorinated oil flood  | 755.18     | 37.76           | 0           |
| 3        | dried                  | 112.90     | 0               | 0           |
|          | saturated with brine   | 898.65     | 131.79          | 20.93       |
|          | fluorinated oil flood  | 416.95     | 11.56           | 0           |
|          | DI water flood         | 583.92     | 69.40           | 4.47        |
|          | fluorinated oil flood  | 525.17     | 25.68           | 0           |

Fluorinated oil because of the water locking/Jamin effect, not the finding that the pore size has really reduced.

3.5. Quantitative Analysis for Porosity Damage. As analyzed in Section 3.3, the real total porosity of cores should be calculated as the sum of irreducible liquid volume (using NMR) and brine-filled volume (using the vacuum saturation method). The $T_2$ coverage of cores saturated with brine, $S_1$ (red area in Figure 10), can approximately represent the whole fluorinated oil because of the water locking/Jamin effect, not the finding that the pore size has really reduced.

The actual volume of brine $V_b$ in saturated core samples measured by the vacuum saturation method is known, and the real pore volume $V_p$ can be calculated by the following formula,

$$V_p = S_1 \times \frac{V_b}{S_1 - S_0}$$

The results of the real pore volume and porosity of the three core samples calculated after conversion from eq 8 are shown in Table 6. The helium porosity is also tested for each core, and results are shown in Table 6 for comparison.

However, it is worth mentioning that even if the converted real porosity is larger than that measured by the vacuum saturation method, the effective porosity during core flooding is smaller than the real porosity due to the existence of residual and immovable crude oil in the core.

From the analysis of Sections 3.3 and 3.4, we know that the pore size distribution of all the core samples decreases and the $T_2$ spectrum amplitude has changed a lot after damage, which is obvious for core no. 1 and no. 3. It is not difficult to find that the $T_2$ coverage of core no. 1 after DI water flooding, $S_2$ (blue area in Figure 10), is smaller than before, indicating that the pore volume decreases because of the water sensitivity damage, so does the porosity. The reduction multiple of the $T_2$ coverage is the multiple of porosity damage. As for core no. 2 and no. 3, the $T_2$ coverage after brine/DI water flooding could not fully reflect the current pore volume, and it needs to add part of area, $S_2$, which is converted by the volume of residual fluorinated oil using eq 8. Then, the porosity damage degree could be calculated by eq 9,

$$I_p = 1 - \frac{S_1 + S_2}{S_1}$$

The porosity damage degree results of the three core samples after damages are calculated and shown in Table 7. It can be found from Table 7 that the porosity of core no. 1 and no. 3 has been reduced due to water sensitivity damage, and the decrease in porosity is around 20%, which is the main reason for causing a significant decrease in permeability. The porosity of core no. 2 hardly changes, and the main reason for the decrease in its permeability is the capillary resistance in two-phase flow.

4. CONCLUSIONS

Understanding the mechanism and evaluating the degree of formation damage in advance are very important for the development of specific reservoirs, followed by taking effective mitigation of formation damage. This study provides a reliable basis, core flooding experiments coupled with the real-time NMR tests, for understanding the effect and mechanism of both water sensitivity and water locking damages on a low-permeability sandstone reservoir during drilling, completing, or fracturing.

Based on the experiment results of this work, the key findings are summarized as follows:
1. For the Jilantai low-permeability sandstone reservoir, we find from the assessment experiment results that the water sensitivity damage degree of the reservoir is “strong” with the permeability damage degree of 61.33% and the water locking damage degree of the reservoir is “strong” with the permeability damage degree of 71.79%. The two formation damages have become one of the most important problems affecting the production in Jilantai oilfield.

2. Water sensitivity damage can reduce porosity, decrease the large and extra-small pore radii, and increase the small pore radius.

3. Water locking damage has hardly any effect on the pore radius and porosity of the reservoir, but the capillary resistance in two-phase flow and irreducible water will result in seriously lower flooding efficiency.

4. The forward and backward flooding process of cores could more truly simulate the actual production operation. The real-time NMR tests designed can reflect more information about fluid saturation and accumulation of fluid in core pores, and structure characteristics can be obtained to explore the mechanisms behind the formation of water sensitivity and water locking damages.

Therefore, as for a specific reservoir, an effective layer production method should be considered in advance. The following recommendations are made for further studies: (1) research and development (R&D) of an agent that can coat the borehole wall with a film so as to prevent the working fluid from injecting into the formation and then mitigate the water sensitivity damage; (2) R&D of an agent that is able to turn the rock surface from being hydrophilic to hydrophobic, making the rock surface hydrophobic and oleophobic, so as to mitigate the water locking damage.

5. MATERIALS AND METHODS

5.1. Regional Overview. Quite a few efforts have been expended in the target Jilantai sandstone oil reservoir in the southwest of Linhe Depression, Hetao Basin, Bayannaoer City, in Central Inner Mongolia Autonomous Region, China, in an attempt to exploit oil reservoirs. The Hetao area is a fast-deposited supercompensated basin, and reservoirs in this area that are buried in less than 6562 ft have notable characteristics of “three lows and one high”, that is, low maturity, low permeability, low strength, and high shale content. The reservoir rock type is mainly clastic lithic feldspar sandstone, followed by arkoses and feldspar lithic sandstone. The pore type consists of mainly intergranular pores. The total amount of clay minerals is about 10%, in which montmorillonite is the dominant clay mineral, followed by illite and kaolinite. The relative content of clay minerals is 37%, 38%, and 25%, respectively. The interstratified ratio is 85%.

Table 7. Porosity Damage on Three Cores Caused by Water Sensitivity/Water Locking

| core no. | $S_1$ | $S_2$ | $S_2'$ | $I_\phi$ | primary real porosity, % | porosity after damage, % |
|----------|-------|-------|--------|----------|--------------------------|--------------------------|
| no. 1    | 1069.74 | 837.35 | 0      | 21.72    | 13.81                    | 10.81                    |
| no. 2    | 1162.48 | 970.32 | 193.95 | -0.15    | 14.67                    | 14.69                    |
| no. 3    | 1051.17 | 657.79 | 200.42 | 18.37    | 14.61                    | 11.93                    |

Table 8. Mineral Content of Cores in the JH2X Well

| mineral content (%) | quartz | potassium feldspar | plagioclase | calcite | dolomite | magnesite | total clay minerals |
|---------------------|--------|--------------------|-------------|---------|----------|-----------|---------------------|
|                     | 53.8   | 16.4               | 12.3        | 5.5     | 1.2      | 1.1       | 9.7                 |

Figure 11. (a) Cores of the JH2X well before washing with oils. (b) Same core broken after washing with oils.

Figure 12. XRD result of cores in the JH2X well.

Figure 13. XRD results of three different core slices (N, EG, and T).

Table 9. Clay Mineral Content of Cores in the JH2X Well

| relative content of clay minerals (%) | interstratified ratio (%) |
|-------------------------------------|---------------------------|
| 1/S                                 | 1/S                       |
| 73                                  | 85                         |
| It                                  | 14                         |
| Kao                                 | 7                          |
| C                                   | 6                          |
most abundant content, accounting for 60–90%, for which it can be estimated that the reservoir rocks have strong water sensitivity. Poor cementation of the rocks makes it difficult to core and prepare core samples that are easy to break when washed with oils (cf. Figure 11). Poor rock cementation also seriously affects the production rate of crude oil.46

A reservoir in the depth range of 7081.56–7104.82 ft (K1g1) is mainly categorized by mid-porosity and mid-low permeability, an average porosity of 14.8%, and an average permeability of 30.2 mD, which is a sandstone oil reservoir with a normal pressure and temperature system and severe reservoir heterogeneity. The saline type of formation water is with a normal pressure and temperature system and severe permeability of 30.2 mD, which is a sandstone oil reservoir is mainly categorized by mid-porosity and mid-low permeability, an average porosity of 14.8%, and an average permeability of 30.2 mD, which is a sandstone oil reservoir with a normal pressure and temperature system and severe reservoir heterogeneity. The saline type of formation water is with a normal pressure and temperature system and severe permeability of 30.2 mD, which is a sandstone oil reservoir

Table 11. Ion Content in Simulated Formation Water

| water type | Na\(^+\) + K\(^+\) | Ca\(^{2+}\) | Mg\(^{2+}\) | SO\(_4\)\(^{2-}\) | HCO\(_3\)\(^-\) | Cl\(^-\) | I\(^-\) | B |
|------------|-----------------|-------------|-------------|--------------|-------------|--------|--------|---|
| CaCl\(_2\)  | 19219.6         | 3306.6      | 486         | 720.5        | 61.0        | 36336.3| 5.9    | 11.9|

5.2. Materials and Instruments. 5.2.1. Core Samples. Three rock core plugs were obtained from the JH2X well, Jilantai oilfield (cf. Figure 14). The lengths of the cores are in, the average porosity is approximately 13%, the permeability is approximately 2 mD, and the cores are water-wet. Detailed information of each core is shown in Table 10. The samples are representative of typical sandstone reservoirs not only in the region but also around the world.

5.2.2. Flooding Fluid. 5.2.2.1. Fluorinated Oil. Fluorinated oil (no. 4891), whose API gravity is 55.4°, used in the experiment was produced by Sinopec Great Wall Lubricant Oil
with a density of 1.86 g/cc and a viscosity of 52 cst at atmospheric pressure and 68 °F. It is a per-fluorinated solvent, which contains no hydrogen atom. Due to its chemical inertness and excellent thermal and oxidation stability, fluorinated oil does not interact with either the pore or simulated formation water.

5.2.2.2. Simulated Formation Water. The simulated formation water was prepared according to the properties of formation water with the salinity of 60,130 mg/L, which is compatible with the core samples. The ion content in simulated formation water is shown in Table 11.

5.2.2.3. DI Water. DI water is nearly pure water obtained by removing ionic impurities such as Ca²⁺/Mg²⁺ from water by ion exchange resin. DI water was used to enable the core to form water sensitivity damage. This is because the core salinity after DI water flooding will be reduced to a very low level, while the salinity of simulated formation water is high. This concentration difference can cause water sensitivity damage, so the water sensitivity-related problems can be studied in the following.

5.2.3. Experimental Setup. 5.2.3.1. Core Flooding Setup. The core flooding setup (cf. Figure 15) includes a core holder, a “constant flow rate and pressure syringe pump” (HUA AN Technology, model HAS-200HSB) for introducing flooding fluid to the core sample at a constant pressure, a “confining pressure pump” (SYRINGE PUMP, model 250D) for supplying the confining pressure for the core holder, cylinders for containing flooding fluid, and other connecting lines.

5.2.3.2. NMR Core Analysis Apparatus. The NMR test was carried out with an NMR Core Analyzer (Beijing SPEC) (cf. Figure 16). The magnetic field intensity of the NMR instrument is 0.3 ± 0.05 T, and the main frequency is 12.8 MHz. The echo spacing of 300 μs was chosen to capture fast relaxation components in the core samples (including fluids in small pores and heavy hydrocarbon components). The magnetic pole is a permanent magnet, whose diameter is 14.72 in. and the gap is 11.81 in.

5.2.3.3. Other Setups. A precision electronic balance was used to measure the changes of dried and saturated weights of cores.

A vacuum pump (412722, WELCH) was used to extract vacuum to allow brine to enter rock voids more fully. An incubator (DGG-9053A) was used to dry the cores at a hot temperature.

5.3. Experimental Methods. The most direct assessment for water sensitivity and water locking damage degree is to compare the oil phase permeability of core samples before and after. In this experiment, simulation of core damage and measurement of core permeability reduction were realized by the core flooding experiment, and the water saturation distribution of core samples in each state was measured by NMR. The procedure schematic of the experiment is shown in Figure 17.

One purpose of core flooding and NMR experimental design is to assess the water sensitivity/water locking damage degree of the core rock from the Jilantai low-permeability sandstone reservoir. The other purpose is to visually observe the changes of moveable fluid in the core to show the porosity variation before and after the two damages. Three groups of core flooding and NMR experiments were used to simulate water sensitivity damage, water locking damage, and comprehensive damage, respectively. During water locking damage, the
wettability of rock changes from water wetting to mixed wetting (water-wet and oil-wet) and then to oil wetting. The specific experimental procedures of the three cores are as follows and shown in Figure 18.

5.3.1. Preparation before Core Flooding and NMR Experiment. Fluid in cores was extracted by the solvent extraction method, and then all the cores were put in an incubator under 176 °F for 24 h until the weight stabilized. After drying, cores were measured by NMR to get the first group of $T_2$ spectra of dried cores. Then, the cores were put in vacuum to be saturated with brine (simulated formation water) at 7.25 psi for 24 h and subsequently were measured by NMR to get the second group of $T_2$ spectra of saturated cores. The changes of dried and saturated weights of cores were measured by an electronic precision balance. The total pore volume of brine-filled volume was measured using the vacuum saturation method.

Before core flooding experiments, to avoid the disintegration of the core when being flooded, a heat-shrinkable film was used to wrap the cores in advance.

5.3.2. Core Flooding and NMR Experiment. After the above preparations were adopted, brine-saturated sandstone cores were placed in the core holder, applying a confining pressure of 435 psi, brine flooding with the constant rate of 0.1 cc/min (lower than critical flow rate) started, and the stable water phase permeability was measured, as shown in Table 1. Then, the cores were forward-flooded with the first fluid (the first, second, and third fluids are shown in Table 2) in the same way for 10 PV injection, and the initial oil phase permeability $K_1$ was measured. Flooding was stopped and the cores were kept still in the core holder for 24 h to keep the same time with the saturation process, and then the cores were measured by NMR to get the third group of $T_2$ spectra. Flooding was stopped and the cores were backward-flooded with the second fluid in the same way for 5 PV injection, kept them still for 24 h, and also measured by NMR to get the fourth group of $T_2$ spectra. Finally, the cores were forward-flooded with the third fluid in the same way for 5 PV injection, the damaged oil phase permeability $K_2$ was measured, and the fifth group of $T_2$ spectra was obtained in the same way for 24 h.

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