Low-Field NMR Experimental Study on the Effect of Confining Pressure on the Porous Structure and Connectivity of High-Rank Coal

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ABSTRACT: To study the influence of different confining pressures on the pore structure and connectivity of high-rank coal, the high-rank raw coal of the Shanxi Xinjing Mine No. 9 coal seam was studied. A low-field nuclear magnetic resonance (LNMR) test system and a vacuum pressurized water saturation system were used to analyze the samples. The $T_2$ spectra of samples, saturated with water under different confining pressures and containing residual water after centrifugation, were tested. The coal sample pore size distributions, permeabilities, free fluid values, bound fluid values, and other parameters were obtained, and a calculation model of the coal pore connectivity ratio was established. The results were as follows. When the saturated pressures were 5, 10, 15, 20, 25, and 30 MPa, the pore diameters of the coal samples were mainly concentrated in the ranges of $0.00023 - 0.069$ and $1.29 - 24.09 \mu m$. Among them, micropores ($<10 \text{ nm}$) and small pores ($10 < 100 \text{ nm}$) account for the main part, mesopores ($100 < 1000 \text{ nm}$) were underdeveloped, and relatively few macropores ($>1000 \text{ nm}$) and fissures developed. As the confining pressure increased, the coal porosity and connectivity showed a trend of decreasing, then increasing, and finally remaining basically unchanged. The total pore connectivity rates of the coal samples were $37.0 - 62.6\%$. The interconnection rates of the micropores, small holes, mesopores, and macropores are $2.90 - 34.55$, $89.09 - 99.03$, $97.09 - 100$, and $100\%$, respectively. The total pore connectivity followed an exponential functional relationship with permeability, and the critical confining pressure of high-rank coal was $25 \text{ MPa}$. These results provide a scientific basis for the high-pressure water injection of high-rank coal seams.

1. INTRODUCTION

Coal has an extremely complex structure and is a flammable porous medium. The pore and fissure structure characteristics of coal determine the physical and chemical properties of coal seams to a certain extent.\textsuperscript{1–3} Coalbed methane (CBM) is mainly stored in coal reservoirs in an adsorbed form or a free state, which is high-quality clean energy.\textsuperscript{4–6} China is rich in CBM resources, ranking third in the world.\textsuperscript{7,8} According to the national CBM resource evaluation results in 2015, the total reserves of CBM resources within 2.0 km of coal mines in China were $30.05 \times 10^{12} \text{ m}^3$, and the recoverable resource reserves were $12.50 \times 10^{12} \text{ m}^3$. Of these, high-rank CBM reserves are $3.56 \times 10^{12} \text{ m}^3$ and the total reserves and recoverable reserves are $35$ and $25.2\%$, respectively.\textsuperscript{7} As a high-rank coal seam enters a deep mine, the air permeability of the coal seam is low, and the mining of the coal seam becomes more difficult, which significantly restricts the development of CBM and gas control.\textsuperscript{10,11} The cleats and pore connectivity of high-rank coals are poor,\textsuperscript{12–15} and the contribution of exogenous fissures to the coal permeability is high, micropores and cracks have a significant effect on the connectivity.\textsuperscript{16–20} Therefore, understanding the pore and fissure structure characteristics of high-rank coals under different confining pressures and conducting in-depth research on their influence...
on the connectivity are of great significance for the efficient and safe development of coalbed methane.

At present, the research on the micropores and fissures of coal seams is mainly conducted via optical microscopy (OM), scanning electron microscopy (SEM), mercury intrusion porosimetry (MIP), N2 and CO2 adsorption experiments, and other traditional methods, but these methods are difficult to effectively solve the problems of the development characteristics of nanoscale micropores and cracks and understand the connection relationship between pores and cracks. In recent years, nuclear magnetic resonance (NMR), X-ray computed tomography (CT), focused ion beam scanning electron microscopy (FIB-SEM), helium ion microscopy (HIM), and other new technologies and methods have been applied to the study of coal pores and cracks, which can be carried out on test samples nondestructively, these techniques can be used to characterize nanoscale pores and cracks. The experimental research on the development of the pore and fissure structures of high-rank coals has mainly been from the perspectives of structural systems, spatial distributions, and seepage and adsorption characteristics. However, little attention has been paid to the influence of the development and connectivity of high-rank coal pores and fractures on the coal permeability under different confining pressures.

Based on this, fast and nondestructive low-field NMR testing technology was used in this study. Experiments were carried out on the changes in the pore and fissure structures of high-rank coal under different confining pressures. The pore size distribution, permeability, free fluid value, bound fluid value, and other parameters of high-rank coal under different confining pressures were studied and the variation characteristics of the coal pore structure were analyzed. A calculation model of the coal pore connectivity rate was established, and the changes in pore size distribution, permeability, and connectivity rate of high-rank coal under different confining pressures were analyzed. The relationship between the pore connectivity and permeability of high-rank coal under different confining pressures was explored. This study provides a scientific basis for high-rank coalbed methane development and high-pressure water injection.

2. EXPERIMENT RESULTS AND DISCUSSION

2.1. T2 Analysis. Nuclear magnetic resonance testing technology is mainly a time-domain technique that obtains the signal relaxation time distribution to obtain molecular interactions (physical information). This experimental device was used to determine the transverse relaxation times (T2) distribution of water in the pores of water-saturated coal samples. To characterize the sizes and distribution of pores in the corresponding coal sample, the relationship between the transverse relaxation time and the pore radius of the coal sample was as follows

$$\frac{1}{T_2} = \rho \left( \frac{S}{V} \right) = \frac{F_5 \rho}{r}$$

(1)

where T2 is the transverse relaxation time (ms), \(\rho\) is the transverse surface relaxation strength (\(\mu\)m/ms), S is the pore surface area (\(\mu\)m²), V is the pore volume (\(\mu\)m³), F5 is the pore shape factor (spherical pores, F5 = 3; columnar pores, F5 = 2; and fissure F5 = 1), and r is the aperture (nm).

Then, the relationship between the pore size of the coal sample and the transverse relaxation time is as follows

$$r = \alpha T_2$$

(2)

where \(\alpha\) is the conversion coefficient, \(\alpha = \rho \times F_5\).

The area enclosed by the T2 map distribution curve and the horizontal axis, the number of peaks, and the peak widths can well characterize the distribution of the internal pore structure of a coal sample and the degree of pore connectivity. The length of the transverse relaxation time (T2) is proportional to the pore size of the internal pore structure of the coal; the longer T2 is, the larger the corresponding coal hole diameter is. The T2 distributions measured after the coal samples were saturated with water under different confining pressures are shown in Figure 1.

![Figure 1. T2 patterns of water-saturated coal samples under different confining pressures.](https://doi.org/10.1021/acsomega.2c01154)

Under different confining pressures, the T2 distributions of the experimental coal samples showed bimodal structures, and the relaxation times of the spectrum peaks were 0.05–3.5 and 75–9000 ms. The short relaxation time of the T2 curve was the largest peak, which showed that the pores of the coal samples were mainly micropores. The mesopores were basically not developed, relatively few large pores and fissures developed, and there was no continuity between the two peaks. This also indicated that the coal samples had poor connectivity.

2.2. Pore Size Distribution. The pore structure of high-rank coal is dominated by micropores and poor connectivity, which directly affect the mining of CBM. According to XoAoT’s research, the pore structure of coal can be divided into micropores (<10 nm), small pores (10 < 100 nm), mesopores (100 < 1000 nm), and macropores (>1000 nm). Of these, the gas in the coal was mainly stored in the micropores in an adsorbed form, and the diffusion space for the gas was mainly small holes. Gas mostly existed in the coal and macroporous structure in flowing form. The pore size distribution curves of the coal samples under different confining pressures measured after being saturated with water are shown in Figure 2.
The pores of the coal samples were mainly micropores. Mesopores did not develop significantly, and a few macropores were formed. The pore diameters of the coal samples were mainly concentrated in the ranges of 0.00023–0.069 and 1.29–24.09 μm. Under different confining pressures, when the confining pressure of the coal sample was 5–15 MPa, the tiny hole peaks in the coal sample shifted left. The peak value (pore size distribution) dropped from 2.09 to 1.88%, and its peak area decreased. When the confining pressure of the coal sample was 15–20 MPa, the peak of the coal sample suddenly increased to 2.01%, and the peak area also suddenly increased. When the confining pressure of the coal sample was 20–30 MPa, the spectrum peak exhibited shifted left again. The peak value (pore size distribution) was reduced from 2.01 to 1.86%, and its peak area decreased. At the same time, with the increase in the confining pressure, for the large pores of the coal sample, the peak of the spectrum shifted left. The center of the peak was reduced from 14.78 to 2.47 μm, then increased to 9.07 μm, and finally, decreased to 2.10 μm.

2.3. Porosity. Coal porosity is an important indicator for the development process of the coal pore structure, and it has a direct effect on the coal permeability. According to the principle of NMR, the relationships between the total porosity, effective porosity, and residual porosity are as follows:

\[ \varphi_E = \varphi \times \frac{\text{FFI}}{\text{BVI} + \text{FFI}} \]  
\[ \varphi_B = \varphi \times \frac{\text{BVI}}{\text{BVI} + \text{FFI}} \]

where \( \varphi \) is the total porosity of the coal, \( \varphi_E \) is the effective porosity of the coal, \( \varphi_B \) is the residual porosity of the coal, FFI is the free fluid index, and BVI is the bound fluid index.

The porosity standard sample of the Niumai LNMR test system was used. A five-point method for calibration was applied. The calibration relationship between the nuclear magnetic signal intensity and porosity had a correlation coefficient \( R^2 = 1 \), which showed that the instrument could accurately measure the porosity parameters of the experimental coal samples, as shown in Figure 3.

The changes in the coal porosity are shown in Figure 4. As the confining pressure increased from 5 to 10, 15, 20, 25, and 30 MPa, the porosities of the coal samples were 2.41, 2.38, 2.24, 2.26, 2.39, and 2.38%, respectively. The porosity showed a trend of decreasing, then increasing, and finally remaining basically constant. The value was minimum at 15 MPa and maximum at 25 MPa.

2.4. Pore Connectivity Analysis. The effective porosity of coal represents the percentage of the connected pore volume of coal to the total pore volume, but it cannot characterize the connectivity of each pore. This paper proposes the use of the pore connectivity \( C_R \) to characterize the connectivity of the pores. The specific calculation process was as follows:

1. The \( \varphi_E \) and \( \varphi_B \) values of the coal samples under different confining pressures were measured according to the principle of nuclear magnetic resonance. The total pore volume of the coal sample was equal to the sum of the volume of free and bound water, and the volume of the residual pores in the coal sample was equal to the volume of bound water in the coal sample. The \( T_2 \) test curves of the saturated and centrifuged coal samples were converted into cumulative pore volume curves, as shown in Figure 5.

2. The pore sizes were \([r_1, r_2]\). Correspondingly, vertical lines were drawn along the horizontal axis in Figure 5. The closed pore volume \( V_{c}\) was the point at which the vertical line crossed the cumulative pore curve of residual water, and the total pore volume \( V_{t}\) was the point at which the vertical line crossed the saturated pore cumulative curve. Then, the connectivity rate \( C_{r_1\sim r_2}\) for aperture \([r_1, r_2]\) was calculated as follows:

\[ C_{r_1\sim r_2} = \frac{(V_{c} - V_{1})}{V_{t}} \]  

The pore connectivity rates of all of the pore sections of the coal samples under different confining pressures are shown in Table 1. As shown in Table 1, the total pore connectivity rates of the coal samples were 37.0–62.6% and the interconnection rates of the micropores, small holes, and mesopores of the experimental coal samples were 2.90–34.55, 89.09–99.03, and

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Figure 2. Pore size distributions of water-saturated coal samples under different confining pressures.

Figure 3. Calibration of low-field nuclear magnetic resonance (LNMR) signal intensity and porosity.
The connectivity rates were high, and large pores had the highest connectivity of 100%. As the confining pressure increased, the total connectivity rate of the experimental coal sample showed a trend of first decreasing, then increasing, and finally remaining basically constant, and the micropores changed most significantly with the confining pressure. This showed that the large and medium pores in the coal sample were basically connected, and the pore

**Figure 4.** Changes in porosities of water-saturated coal samples under different confining pressures.
connectivity was high; the tiny holes were mostly closed holes, and the connectivity was poor. The relationship between the total pore connectivity and the permeability of the coal samples is shown in Figure 6.

The total pore connectivity of the coal samples followed an exponential relationship with the permeability, and the functional relationship was

\[ y = 0.00516 \times \exp \left( \frac{x}{21.484} \right) + 0.0567, \]

with a correlation coefficient \( R^2 = 0.999 \). As the total connectivity rate increased, the permeability of the coal samples increased. The permeability of a coal sample was directly affected by the pore connectivity of the coal sample. The more the pores developed, the better the connectivity between the pores was, and the higher the permeability of the coal sample became.

### 3. CONCLUSIONS

In summary, in this study, the influences of different confining pressures on the pore structures, connectivities, and permeabilities of high-rank coals were examined. The LNMR testing of high-rank coal samples saturated with water under different confining pressures and containing residual water after centrifugation was performed. A calculation model of the coal pore connectivity rate was established, and the relationship between the pore connectivity and the permeability for high-rank coal under different confining pressures was explored. The main conclusions were as follows:

1. When the saturated pressures were 5, 10, 15, 20, 25, and 30 MPa, the \( T_2 \) curves of the experimental coal samples had bimodal shapes. The pores of the coal samples were mainly micropores, and mesopores had basically not developed. There were relatively few large pores and fissures that developed, and there was no continuity between the two pore size peaks. Thus, the coal samples had poor connectivities. As the confining pressure increased, the coal sample porosity showed a trend of first decreasing, then increasing, and finally remaining basically constant. The minimum value of porosity was at 15 MPa and the maximum value was at 25 MPa.

2. With the increase in the experimental confining pressure, the change rule of the coal pore system and connectivity is as follows. In the first stage, when the coal sample confining pressure increased from 5 to 15 MPa, the surface moisture entered the coal body through the large pores and cracks on the surface of the coal body under pressure. The water and minerals in the macropores and fissures of the coal were squeezed and migrated into the coal. The pore volume of the coal body became smaller or blocked. At the same time, local expansion and deformation of the coal matrix occurred due to water soaking, and the pore volume and specific surface area of the coal body were further reduced. As a result, the connectivity of the coal body decreased, and the minimum connectivity was achieved when the confining pressure was 15 MPa, as shown in Figure 7b. In the second stage, when the coal sample confining pressure increased from 15 to 25 MPa, as the confining pressure continued to increase, the coal body softened due to prolonged soaking in water. The partially closed pores of the coal body were squeezed and fractured, the coal matrix swelled and deformed overall, and the originally

| coal sample number | permeability \( \times 10^{-15} \text{ m}^2 \) | connectivity rate (%) |
|--------------------|---------------------------------|----------------------|
|                    | <10 nm  | 10−100 nm | 100−1000 nm | >1000 nm | total connectivity rate |
| M1                 | 0.1112  | 15.61     | 96.45      | 100      | 100       | 50.8     |
| M2                 | 0.1013  | 6.79      | 94.33      | 100      | 100       | 45.9     |
| M3                 | 0.0854  | 2.90      | 89.09      | 97.09    | 100       | 37.0     |
| M4                 | 0.1049  | 13.22     | 99.00      | 98.48    | 100       | 48.1     |
| M5                 | 0.1516  | 34.55     | 99.03      | 100      | 100       | 62.6     |
| M6                 | 0.1433  | 31.76     | 99.01      | 100      | 100       | 62.1     |

Figure 5. Cumulative pore distributions of experimental coal samples obtained by nuclear magnetic resonance (NMR).

Figure 6. Relationship between total connectivity of coal sample pores and permeability.

Table 1. Pore Connectivity Rate of Each Pore Section of Coal Body under Different Confining Pressures

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closed pores of the coal body interpenetrated and connected to each other, leading to an increase in the overall connectivity rate of the coal body. The connectivity reached a maximum when the confining pressure was 25 MPa, as shown in Figure 7c. In the third stage, when the coal sample confining pressure increased from 25 to 30 MPa, as the confining pressure continued to increase, the total connectivity of the coal pores was basically unchanged, as shown in Table 1. Therefore, the confining pressure of 25 MPa is the critical pressure for water injection in high-rank coal seams.

(3) The total pore connectivity rate of the experimental coal sample was 37.0−62.6%. The interconnection rates of the micropores, small holes, mesopores, and macropores of the experimental coal samples were 2.90−34.55, 89.09−99.03, 97.09−100, and 100%, respectively. The total pore connectivity followed an exponential functional relationship with the permeability: y = 0.00516 × exp (x/21.484) + 0.0567, and the correlation coefficient was R^2 = 0.999. The more developed the pore structure of coal was, the better the connectivity between the pores was, and the higher permeability of the coal sample was. The permeability of high-rank coal was very small, which is extremely unfavorable for coalbed methane development and gas control. When injecting water into a low-permeability coal seam to suppress gas gushing, considering the influence of the confining pressure on the pores and the connectivity of the coal, the on-site coal seam water injection pressure should be controlled at about 25 MPa.

4. Materials and Methods. 4.1. Experimental Materials and Sample Preparation. The experimental coal sample was high-rank raw coal from the Shanxi Xinjing Mine No. 9 coal seam. The experimental coal sample was high-rank anthracite with a medium ash content, low sulfur content, and low phosphorus content. The proximate analysis of the coal samples was performed according to the GB/T212-2008 standard “Industrial Analysis Method of Coal”, and the results are shown in Table 2, where M (ad) is the moisture content of the coal sample, A (ad) is the ash content of the coal sample, V (ad) is the volatile content of the coal sample, and FC (ad) is the fixed carbon content of the coal sample. A large piece of fresh coal was wrapped with plastic. After returning to the laboratory, the surface of the coal sample was peeled off. A multifunctional jade carving machine was used for cutting and polishing. Specimens with dimensions of 20 mm × 20 mm × 20 mm were cut from fresh coal, and regular-shaped coal samples with no cracks on the surface were selected for the experiment.

4.2. Experimental Methods. 4.2.1. Experimental Facility. The experimental device was mainly composed of a Niumai low-field nuclear magnetic resonance test system, constant-
temperature drying system, centrifugal treatment system, and vacuum pressurized saturated water system. The experimental setup is shown in Figure 8.

4.2.2. Experimental Procedure. The experimental procedure was as follows:

(1) The coal samples were numbered (M1–M6), and the mass of the coal sample in its initial state was determined by weighing. The coal sample was placed in an electric heating blast drying oven at 100 °C and dried for 12 h, after which the mass of the dried coal sample was measured by weighing.
(2) The dry coal sample was placed in a vacuum pressure saturation device. A vacuum pressure of −0.08 MPa was applied, and the duration was 12 h.
(3) After the vacuum process was completed, the coal samples were pressurized and saturated in the device. The saturated pressures were 5, 10, 15, 20, 25, and 30 MPa (corresponding to the coal sample numbers of M1–M6, respectively), and the duration was 10 h.
(4) The moisture on the surface of the coal sample after being saturated with water was wiped away. NMR testing was performed, and the initial T2 spectrum, saturated porosity, permeability, and other data of the coal samples under different saturation pressures were measured.
(5) The water-saturated coal samples were centrifuged at 3000 rpm for 90 min.
(6) NMR testing was performed on the centrifuged coal sample, and data such as the T2 spectrum and porosity parameters were obtained.

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