Study on the Quantitative Characterization and Seepage Evolution Characteristics of Pores of Loaded Coal Based on NMR

Zehua Wang, Hongqing Cui,* Guoying Wei, Tianrang Jia, Jingyuan Guo, and Xin He

ABSTRACT: Quantitative characterization of the pore structure and gas seepage characteristics of loaded coal is of great significance to the study of high-efficiency gas drainage in coal seams. Aiming at the problem of imperfect characterizations of coal seepage characteristics based on nuclear magnetic resonance (NMR), a calculation method for the pore permeability of coal with different pore diameters is proposed. The pore structure and seepage characteristics of coal have been studied using a nuclear magnetic resonance (NMR) system. The results show that with increasing external load, the proportion of the pore volume of the coal sample in the range of 0.01–0.52 μm gradually decreases, while that in the range of 5.11–352.97 μm increases. In this process, the porosity increases from 0.9967% to 1.0103%, the connectivity increases from 0.1718 to 0.2391, and the permeability increases from 2.64 × 10⁻⁶ to 8.20 × 10⁻⁵ μm². The calculation of the coal sample connectivity and permeability using the improved NMR permeability component proves that 94.37–352.97 μm pores are the main channel of fluid flow. When the axial pressure increases, the coal body permeability in the aperture range of 94.37–352.97 μm rapidly increases. The improved permeability component calculation model can better reflect the variation law of pore permeability of the loaded coal body.

1. INTRODUCTION

Coal is a type of porous medium with many pore/fracture structures of different scales. Coal seam gas is mainly adsorbed in coal pores,4,8 which is an important factor that causes disasters in coal mines,3,6 and is a high-quality clean energy source.7,9 The development of coal seam gas can ensure safe and efficient mining of coal resources and promote the utilization of unconventional natural gas. Coal seam gas drainage refers to the process by which the desorbed gas in the coal seam is transported to the drainage pipeline through the seepage channel composed of a pore-fracture network under negative pressure.10 Fractures are the main migration pathways for coalbed methane, which dominate the permeability of coal reservoirs.11,12 During the process of coal seam gas drainage, internal fractures in the coal body develop, which cause dynamic changes in the coal body permeability (Figure 1). Accurate characterization of the pore structure and gas seepage characteristics is the key to coalbed methane exploitation.13–15

At present, there are many methods to study the coal structure, such as mercury porosimetry (MIP),16 gas adsorption (N₂/CO₂),17 scanning electron microscopy (SEM),18 and μ-CT scanning.19 These methods play an important role in detecting coal pores and fracture structures. However, it is limited by the sample size, measurable pore size, testing time, damage, cost, and other factors. The low-field nuclear magnetic resonance (NMR) method has been increasingly widely used in the evaluation of the pore structure of coal because of its high speed, nondestructive nature, large sample size, wide measurement range, etc.20,21 By measuring the relaxation characteristics of the ¹H fluid (water) in pores by low-field NMR, the pore structure and flow characteristics of coal samples can be obtained. By analyzing the T₂ spectra of saturated and centrifuged samples, one can obtain information about the porosity, permeability, pore size distribution, and fluid saturation.3,5,12–15 For example, Lucas-Oliveira et al.26 compared experimental and simulated NMR data with gas permeability through the combination of experiments and simulations and proved that the pore size distribution could be obtained using the T₂ relaxation spectrum. Zheng20 and Liang et al.27 obtained accurate pore size distributions from NMR T₂ distributions by combining NMR and centrifugation experiments. According to the results of the T₂ distribution of saturated samples, the pores in coal can be divided into three types: adsorption pores (T₂ < 2.5 ms), percolation pores (2.5 ms < T₂ < 50 ms), and fracture pores (T₂ > 100 ms). In addition, the connectivity characteristics of the samples can be

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estimated according to the shape of the $T_2$ spectrum. Continuous $T_2$ spectra usually reflect good connectivity among different pores, while discontinuous $T_2$ spectra show poor connectivity.\textsuperscript{28} These scholars have quantitatively characterized the pore structure parameters and permeability of coal based on NMR technology. However, there are a few studies on the quantitative characterization of the pore structure complexity and permeability components of loaded coal based on NMR. The porosity, connectivity, and permeability of coal are determined by the complexity of the coal pore structure. The permeability component reflects the real seepage of gas in pores. Therefore, it is very important to quantitatively characterize the complexity and permeability evolution characteristics of the pore structure of loaded coal and analyze its influence on gas migration characteristics.

To date, the existing methods are not perfect for the comprehensive characterization of the structure and seepage characteristics of creep-loaded coal, so it is difficult to effectively and quantitatively evaluate different pore permeabilities of creep-loaded coal. In this paper, a low-field NMR technique is used to quantitatively characterize the pore structure and seepage characteristics of coal and study the effect of creep load on the development degree of the pore-fracture structure and seepage characteristics of coal. By comparing and analyzing different NMR permeability models, a suitable NMR permeability calculation model is selected, and an NMR permeability component calculation model is proposed. The relationship between the pore size distribution and the permeability component of loaded coal is also discussed. Based on this relationship, the pore structure and fluid distribution characteristics of coal samples are obtained, which can provide references for technical engineering operations related to coaled methane extraction.

2. RESULTS AND DISCUSSION

2.1. Pore Size Distribution by NMR. The NMR signal results from the superposition of water signals in different pores, and the NMR $T_2$ spectrum is obtained by a multi-exponential fitting. The transverse relaxation time $T_2$ of coal is estimated according to the shape of the $T_2$ spectrum. Continuous $T_2$ spectra usually reflect good connectivity among different pores, while discontinuous $T_2$ spectra show poor connectivity.\textsuperscript{28} These scholars have quantitatively characterized the pore structure parameters and permeability of coal based on NMR technology. However, there are a few studies on the quantitative characterization of the pore structure complexity and permeability components of loaded coal based on NMR. The porosity, connectivity, and permeability of coal are determined by the complexity of the coal pore structure. The permeability component reflects the real seepage of gas in pores. Therefore, it is very important to quantitatively characterize the complexity and permeability evolution characteristics of the pore structure of loaded coal and analyze its influence on gas migration characteristics.

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The $T_2$ distribution can reflect the signals generated by the pore structures over a wide range, and the signal peak corresponding to its peak is consistent with the relative volume percentage of the corresponding pores in the total volume of pores for each coal sample. When the core is saturated with a single fluid, the $T_2$ distribution is proportional to the pore size. Therefore, the distribution of the pore structures of coal can be determined based on the changes in the $T_2$ spectrum of the coal samples in the water-saturated state. According to the research results of some scholars, coal pores can be divided into micropores ($0.01−0.1 \mu m$), small pores ($0.1−1 \mu m$), medium pores ($1−11 \mu m$), macropores ($11−1000 \mu m$), and fissures ($>100 \mu m$) under different relaxation times. Based on the "$T_2C$ method" previously proposed by other researchers, the pore size distribution of coal samples can be obtained. The principle is as follows: the pore radius $r$ corresponding to the $T_2$ cutoff value, which is determined based on the saturated water and bound water, is a constant. Therefore, the aperture $r_d$, corresponding to the $i$th relaxation time $T_2i$, is

$$r_d = r \frac{T_{2i}}{T_{2C}}$$

In this study, the magnitude of $r$ corresponding to the $T_{2C}$ value is $0.1 \mu m$ according to the centrifugal experiment. Formula 3 can be used to obtain the $r_d$ values at different time periods and build the pore size distribution characteristics based on the NMR $T_2$ spectrum analysis. Figure 3 shows the pore size distribution and pore volume ratio of the A1−A4 coal samples.

The $T_2$ relaxation spectrum of the A1 coal sample in Figure 3 has three peaks, among which the porosity component at the relaxation time ($r_1$) of $0.1−4.8 \text{ ms}$ is the largest and accounts for $91.2\%$ of the total pore volume. After being subjected to an external load, coal samples A2−A4 show four peaks in the $T_2$ relaxation spectrum. With increasing axial pressure, the porosity component in the range of $r_1$ gradually decreases, and the percentage of porosity in the total pore volume gradually decreases. With the gradual increase in the external load, the porosity component, and pore volume ratio in the relaxation time ($r_2$) of $47.7−3293 \text{ ms}$ for A2−A4 coal samples gradually increase, and the overall porosity is larger than A1.

These characteristics prove that the external load promotes the development of pores/cracks in the coal sample. The increase in the pore size promoted by the external load
decreases the number of pores in the corresponding pore size range and results in a gradual decrease in the first peak. However, the development and expansion of primary cracks in coal samples under external loading cause the gradual increase of porosity components from the second peak to the fourth peak in the relaxation spectrum of coal samples.

2.2. Verification of Connectivity by NMR. Connectivity is an important component in the evaluation of coal bodies to study their pore structure characteristics, and its numerical value can directly affect the difficulty of gas seepage in coal bodies. Through NMR experiments of coal samples, the porosity components of the total pores and residual pore-volume fraction of the coal sample can be obtained. The connectivity of coal can be calculated by the following formula

$$C = 1 - \frac{\theta'}{\theta}$$

where $\theta'$ is the residual pore volume fraction and $\theta$ is the total pore volume fraction.

Through water saturation and centrifugal NMR experiments of coal samples, the porosity components of the total pores and residual pores of coal are measured. According to formula 1, the connectivity of coal samples is calculated, as shown in Table 1.

| coal sample number | total pore volume fraction (%) | residual pore volume fraction (%) | connected pore volume fraction (%) | connectivity  |
|--------------------|-------------------------------|-----------------------------------|-----------------------------------|--------------|
| $A_1$              | 0.9967                        | 0.8265                            | 0.1702                            | 0.1708       |
| $A_2$              | 0.9989                        | 0.8100                            | 0.1889                            | 0.1891       |
| $A_3$              | 1.0056                        | 0.7935                            | 0.2121                            | 0.2110       |
| $A_4$              | 1.0103                        | 0.7687                            | 0.2416                            | 0.2391       |

2.3. Quantitative Characterization of Coal Permeability. The permeability of the coal seam can be deduced and calculated according to the pore size distribution and signal intensity of the $T_2$ relaxation spectrum. There are three types of NMR permeability calculation models: the traditional SDR model, Coates model, and improved model based on the former two. Han et al. proposed an improved permeability calculation model based on the combination of nuclear magnetic resonance multicomponent pore components. The calculation formulas of the three models are shown in Table 2.

### Table 2. NMR Permeability Calculation Model

| the model     | calculation formula                                                                 |
|---------------|--------------------------------------------------------------------------------------|
| Coates model  | $K_{\text{Coates}} = \left( \frac{\phi}{C_1} \right)^{\frac{1}{2}} \left( \frac{\text{FEI}}{\text{BVI}} \right) \cdot \Omega$ |
| SDR model     | $K_{\text{SDR}} = C_2 \phi^2 T_{2gm} \cdot \Omega$                                    |
| improved model| $K_{\text{im}} = \phi^3 \frac{S_{4b}^d}{S_{1b}^d} \cdot \Omega$                      |

In the formulas in Table 2, $K_{\text{Coates}}$, $K_{\text{SDR}}$, and $K_{\text{im}}$ are the permeability of the Coates model, SDR model, and improved model (10^{-3} \, \mu m^2), respectively; $\phi$ is the porosity of the coal sample; and $C_1$ and $C_2$ are constant coefficients. According to laboratory correction experience, $C_1 = 5.1$ and $C_2 = 0.00045$; BVI is the irreducible fluid saturation; $T_{2gm}$ is the geometric average of nuclear magnetic resonance $T_2$; and $S_1 - S_4$ are the pore composition ratios of micropores, small pores, medium pores, and macropores. According to previous research, the classification criteria of the NMR $T_2$ relaxation time for different sizes of pores can be classified according to Table 3.

### Table 3. NMR $T_2$ Classification Criteria of Different Apertures

| pore type          | $T_2$ (ms) |
|--------------------|------------|
| large holes        | >200       |
| medium holes       | 90–200     |
| small holes        | 30–90      |

Through saturated and centrifugal NMR experiments of loaded coal samples, the permeability of coal samples is calculated using three permeability models. The permeability of coal samples was also measured in the laboratory, as shown in Figure 4.

The laboratory uses the TCQT-III-type experimental system for gas-phase displacement and stimulation in low-permeability coal seams to measure the coal samples. The test gas was nitrogen, the inlet pressure was 1 MPa, and the outlet pressure was atmospheric pressure. All axial pressure and confining pressure were 1 MPa.

As shown in the figure, the permeability calculation results of the Coates model are 1 order of magnitude different from those of the other two models. With increasing axial pressure, $K_{\text{SDR}}$ slowly increases, while the permeability of the improved model greatly changes. Because the improved model fully considers the influence of cracks of different pore sizes on the permeability, which is closer than the measured gas permeability, it better reflects the influence of pore-size change on permeability. Therefore, for testing coal samples, the
The Coates model and the SDR model are commonly used to calculate the permeability in NMR logging but they also have limitations in application. For conventional reservoir cores with simple pore structures, the two methods show satisfactory permeability characterization results. However, when the pore space of rock samples becomes more diverse and the pore size distribution becomes increasingly complex, the calculation results are not accurate. The improved model is more suitable for calculating the permeability of coal rock with cross-scale characteristics and wide distribution of pore throats.

2.4. Seepage Characteristics. The influence of different pore sizes on coal permeability is different and mainly depends on the size and proportion of throats. If the diameter of the connected pore throat is large and the proportion is relatively high, the permeability is higher; in contrast, if the throat diameter is small and the proportion is high, the permeability is lower. Therefore, it is necessary to calculate the porosity components of throats of different sizes.

According to scholars,35 the contribution of different throat sizes to the permeability can be calculated by formula 10. However, formula 10 is suitable for comparing the contribution of throat permeability in a single coal sample. For different coal samples, it is of little significance to compare with one another because of different permeabilities. As shown in Figure 5, the contribution of throat permeability of the coal samples with diameters ranging from 100 to 378 μm is obviously larger than throat diameters ranging from 5.8 to 100 μm. However, there is no obvious difference among the A1−A4 coal samples.

\[
\Delta K_j = \frac{d_j^3 \alpha_j}{\sum d_j^3 \alpha_j}
\]

where \(\Delta K_j\) is the contribution to the permeability (dimensionless) of interval \(j\), \(d_j\) is the throat radius (μm) of interval \(j\), and \(\alpha_j\) is the throat frequency (%) of interval \(j\).

According to the above NMR permeability calculation model and permeability contribution calculation formula, an NMR permeability component calculation model is proposed as follows:

\[
K_j = K_{\text{mr}} \Delta K_j
\]

where \(K_j\) is the NMR permeability component of the \(j\)th interval throat (10\(^{-3}\) μm\(^2\)).

Understanding the pore size distribution of coal cracks is of great significance for predicting the coal gas distribution and guiding mine gas drainage. Figure 3 shows that most of the pores of the coal samples are concentrated in the range of 0.01−0.52 μm. With increasing axial pressure, the proportion of pore volume for the range of 0.01−0.52 μm decreases, while that for the range of 5.11−352.97 μm increases. The results show that under the action of pressure, the pores and primary cracks of coal samples expand along the direction of stress concentration, which increases the pore size. As a result, the proportion of pore volume of small pore diameter decreases and that of large pore diameter increases.

Through formula 11, the permeability components of different throat diameters of A1−A4 coal samples are calculated, and the results are shown in Figure 6. There is a significant difference between Figure 6 and Figure 5. If the throat diameter is less than 5 μm, \(K_j\) of the throat is close to 0, which can be ignored. When the throat diameter is 5.8−100 μm, the permeability component is close to 1, indicating that the permeability of the throat is mainly determined by the diameter of the throat.

![Figure 4. Comparison of permeability results calculated by laboratory measurements and using Coates, improved, and SDR models.](https://example.com/figure4)

![Figure 5. Contribution value of permeability of coal samples under load.](https://example.com/figure5)

![Figure 6. Permeability component of the coal sample under load.](https://example.com/figure6)
μm, $K_1$ is lower than that in the range of 100−378 μm. With increasing axial pressure, the pore size in the range of 5−100 μm slowly increases, and the wave crest of the curve tends to slightly increase and move backward. The throat of 100−378 μm rapidly increases with increasing axial pressure, and the peak of the curve greatly increases. This result shows that the micropores and small pores in the coal body have little influence on the coal permeability, and cracks with large pore diameters are the main channel of gas seepage in the coal body. With increasing axial pressure, the primary fractures of coal samples are developed and connected with one another, which results in an increase in the pore size. Figure 3 shows that the pore volume increases the most when the throat diameter is 100−378 μm. With the increase in the volume ratio of large throats, the permeability component is greatly increased. Obviously, comparing Figure 5 with Figure 6, the permeability component of coal samples in Figure 6 is closer to the real situation of pore permeability changes in the loaded coal body (in Figure 4).

3. CONCLUSIONS

Using an NMR test system, the porosity, connectivity, and permeability of loaded coal samples are quantitatively characterized. Based on the quantitative description of the pore size distribution using the proposed permeability component model, the relationship among porosity, connectivity, and permeability is analyzed. According to the analysis, the following conclusions are drawn:

(1) Pores of 0−11 000 μm were quantitatively characterized by NMR. Under stresses of 0, 6, 9, and 12 MPa, the porosities of the four coal samples are 0.9967, 0.9989, 1.0056, and 1.0103%, respectively. The pore volume proportion of coal samples is in the range of 0.01−0.52 μm gradually decreases, while that of 5.11−352.97 μm increases.

(2) The pore structure parameters are calculated based on the pore size distribution and volume ratio of saturated and centrifuged coal samples. The 4 coal samples with adsorption pores smaller than 0.1 μm have the highest signal strength. The water signals of $A_1$ and $A_2$ are mainly distributed in micropores, while $A_3$ and $A_4$ have many water signals in larger pores and fissures. It is known that the micropore structure of $A_1$ and $A_2$ are more than those of $A_3$ and $A_4$, which indicates that creep promotes the development of coal pores and fractures.

(3) The permeability of the loaded coal sample is quantitatively characterized using the NMR permeability optimization model. With increasing axial pressure, the permeability of the coal sample increases from $0.00264 \times 10^{-3}$ to $0.00820 \times 10^{-3}$ μm$^2$, and the increase rate increases. The improved model permeability calculation model better reflects the influence of pore size changes on the permeability.

(4) The improved NMR permeability component calculation model shows that pores with a diameters in the range of 94.37−352.97 μm are the main channels for gas seepage of the test coal sample. With increasing axial pressure, the permeability component of the pore size range of 94.37−352.97 μm rapidly increases. Compared with the permeability contribution model, the improved permeability component calculation model can better reflect the change law of the pore permeability of the loaded coal.

4. EXPERIMENTS AND METHODS

4.1. Samples and Creep Experiment. The experimental coal samples in this paper are anthracite from Zhaogu No. 2. coal mine, Jiaozuo City, Henan Province. The bulk coal samples obtained underground are made into standard cylindrical coal samples with a diameter of 50 mm along the vertical bedding direction using a core sampler. The end face
of the coal sample was ground to make its surface smooth. The coal samples were numbered A₁, A₂, A₃, and A₄ (as shown in Figure 7). The prepared coal samples were placed into a constant-temperature drying box for 24 h. A 5E automatic industrial analyzer was used for the industrial analysis of coal samples, and the results are shown in Table 4.

| Table 4. Basic Parameters of Experimental Coal Samples<sup>a</sup> |
| --- |
| Parameter | Value |
| Mₐd (%) | 0.89 |
| Aₐd (%) | 15.70 |
| Vₐd (%) | 6.43 |
| real density (g/cm³) | 1.59 |
| apparent density (g/cm³) | 1.51 |
| firmness coefficient | 1.31 |

<sup>a</sup>Mₐd, moisture; Aₐd, ash yield; Vₐd, volatile matter.

We used the TCQT-III-type experimental system for gas-phase displacement and stimulation in low-permeability coal seams in the experiment. The axial pressure and confining pressure pump adopted a constant-speed and a constant-pressure pump, which can realize linear loading. Before the triaxial compression test, the triaxial compression failure test of coal samples was performed, and the triaxial compression failure strength of experimental coal was obtained. Because 17.5 coal is a viscoelastic-plastic material, when the stress of coal is less than the long-term strength of coal (σ < σₛ), the stress–strain curve of coal shows two stages: attenuating creep and stable creep. When the stress of coal is equal to or greater than the long-term strength of coal (σ ≥ σₛ), there are three complete creep stages: the attenuation creep stage, stable creep stage, and accelerated creep stage. To prevent the instability of coal samples from entering the accelerated creep stage, axial pressures of 6, 9, and 12 MPa were applied to coal samples A₂, A₃, and A₄, respectively, while maintaining the constant confining pressure of 3 MPa. When the loading stress reached the predetermined value and the loading pressure was kept constant for 8 h, the data of changes in stress with time of the coal sample were recorded by a computer. The time-dependent strain curves of coal samples obtained from the experiment are shown in Figure 7.

4.2. Fully Saturated and Centrifugation NMR Experiment. The nuclear magnetic signal strength is proportional to the number of movable 1H atoms in the sample. The transverse relaxation time (T₂) spectrum of the sample was measured by low-field nuclear magnetic resonance (NMR). The instrument used was a MesoMR23-060H-I nuclear magnetic resonance instrument made by Shanghai Newmai Company. The resonance frequency was 21.67568 MHz, the magnet temperature was 31.99–32.01 °C, and the magnetic field intensity was 0.5 ± 0.05 T.

To understand the change characteristics of the internal pore structure of coal under creep, NMR experiments were performed on the scanned coal samples. First, samples A₁–A₄ were vacuum-treated for 8 h, and the coal samples were saturated with water and at the same time treated with vacuum, as shown in Figure 8. The water-saturated coal samples were tested by NMR to obtain their T₂ spectrum distribution. According to Wang et al.,<sup>13</sup> coal samples used in the test is anthracite with a size of φ 50 × 100 mm. They can be centrifuged at high speed under 1.5 MPa centrifugal pressure for 2 h, and the ideal hydration state can be achieved. Therefore, at the end of the experiment, the coal samples were centrifuged. The centrifugal force was 1.5 MPa, and the centrifugation time was 2 h. Then, the porosity was measured by NMR to obtain the T₂ spectrum distribution of the centrifuged coal samples.

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Figure 8. NMR experiment process of the coal sample.
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Notes

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