Low Nuclear Magnetic Resonance Experimental Study on Gas Adsorption of High-Rank Coals with Different Beddings

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ABSTRACT: In order to deeply study the influence of the coal bedding structure on coal gas adsorption, low nuclear magnetic resonance (LNMR) and a confining pressure loading system were used to carry out the LNMR experiment of gas adsorption of high-rank coals with different beddings under different confining pressures. The results showed that the amount of gas adsorption of high-rank coals with different beddings increases with time and decreases with the increase of confining pressure. In the process from low confining pressure to high confining pressure, the coal sample with oblique bedding (bedding angles 30°, 45°, and 60°) has the largest average increment of gas adsorption, followed by the coal sample with vertical bedding (bedding angle 90°), and the coal sample with parallel bedding has the smallest increment of gas adsorption (bedding angle 0°). The linear function relation between the different-bedding high-rank coal gas adsorption state and the confining pressure is \( y = a - bx \). The relation between the free peak area and the confining pressure conforms to the exponential function \( y = a + b \exp(cx) \). Different-bedding high-rank coal adsorption peaks and the peak area decrease with the increase of confining pressure, and the free peak continues to move to the left; that is, the large pores gradually shrink. With the increase of angle and bedding, the area of the adsorption peak increases first and then decreases, presenting an “inverted V” shape on the whole. The area of the free peak decreases first and then increases, presenting a “V” shape on the whole.

1. INTRODUCTION

In recent years, a lot of coalbed methane development in China has been gradually carried out in high-rank coal seam. Coalbed methane (gas) is a kind of clean and efficient high quality energy, but it is also a dangerous source affecting coal mine safety. With the increase of coal mining depth in China, more and more coal seams show the characteristics of “three high and one low” (high ground stress, high gas pressure, high gas content, and low permeability). It is very important to accurately grasp the gas adsorption characteristics of high-rank coal to ensure the development effect of coalbed methane and improve the gas extraction rate underground.

Scholars at home and abroad have done a lot of research on the law of coal gas adsorption. Yang et al., Li et al., Yang et al., Yan et al., and He et al. have studied the relationship between gas adsorption and temperature. Zhang et al. and Wang et al. studied the influence of moisture on gas adsorption characteristics. Chattaraj et al. found that the adsorption capacity of gas was positively correlated with carbon content and vitrinite reflectance and negatively correlated with water content, ash content, and volatile substances. Li et al., Liu et al., Chen et al., and Xing et al. studied the influence of microstructure or fractal characteristics of coal on gas adsorption characteristics. Liu et al. found that coal gas adsorption behavior is not only affected by the pore structure but also closely related to the chemical structure. Guo et al. studied the dynamic damage caused by tectonic stress on aromatic lamella in coal and its influence on gas adsorption. Ji et al. studied the composition of the coal solvent extract and its influence on methane adsorption characteristics. Kang et al. found in their study that the maximum adsorption capacity of coal samples for methane decreased after electrochemical modification, and the decrease of methane adsorption capacity increased with the increase of the electric potential gradient. Qin et al. constructed a mathematical model of pressure swing adsorption for coal gas and compared the experimental results with theoretical calculations. Wei derived the multilayer adsorption theory of gas in coal and used this theory to fit and analyze the law of coal gas adsorption test. Ma established a double first-order function combination model to calculate and analyze the gas adsorption process. Zhao et al. found that the
adsorption capacity of CH4 increased with the increase of the coal rank. Zhou et al.21 studied the mesoscopic characteristics of coal adsorption of gas. The above scholars studied the theory of gas adsorption and established mathematical models of gas adsorption, and they mainly considered the influence of coal itself factors (such as coal fractal characteristics, pore structure, and chemical structure) and external factors (such as temperature, water, and stress) on gas adsorption.

Nuclear magnetic resonance (NMR) is an advanced testing method, which has the advantages of fast detection speed and high precision. At present, some scholars have used the NMR technology to study the law of coal gas adsorption. Liu et al.22 studied the gas adsorption characteristics of deep low-rank coal by using an NMR experimental system. Gao et al.23 studied the influence of moisture on the gas adsorption characteristics of anthracite by using the NMR technology. Xu et al.24 used the NMR technology to study the gas adsorption characteristics and pore structure change characteristics of coal under different pressure conditions. Yang et al.25,26 studied the influence of confining pressure on the gas adsorption law of high-rank coals by using the NMR technology.

At present, the study of the gas adsorption law of high-rank coals with different beddings by the low-field NMR technology has not been involved. The author collected the high-rank coal (vitrinite reflectance is 3.31%) from Zhongmacun Mine of Coking Coal Group studying the gas adsorption law of high-rank coals with different beddings (the included angles of bedding are 0°, 30°, 45°, 60°, and 90°, respectively). The research results provide a theoretical basis for the extraction and utilization of coalbed methane and the prevention and control of mine gas disaster in China.

2. PRINCIPLE OF LOW-FIELD NUCLEAR MAGNETIC RESONANCE

NMR refers to the response of atomic nuclei to radio frequency magnetized by a magnetic field. If one or both of the neutrons and protons in the nucleus are odd, the conditions for NMR signal generation are available, such as hydrogen 1H, carbon 13C, nitrogen 14N, and so on. Because hydrogen 1H is abundant in nature and easy to detect, almost all NMR techniques are based on the response of hydrogen nuclei. Low-field NMR is NMR with a lower magnetic field strength. Relaxation is the rapid change from a high energy state to a low energy state when nuclei are in resonance. For fluids in rock pores, there are three different relaxation mechanisms: free relaxation, surface relaxation, and diffusion relaxation, which can be expressed as follows:

$$\frac{1}{T_2} = \frac{1}{T_{2B}} + \frac{1}{T_{2S}} + \frac{1}{T_{2D}}$$  \hspace{1cm} (1)

where $T_2$ is the transverse relaxation time, ms; $T_{2B}$ is the free relaxation time, ms; $T_{2S}$ is the surface relaxation time, ms; and $T_{2D}$ is the diffusion relaxation time, ms.

Since a uniform magnetic field is used in the experiment environment, the principle formula 1 based on NMR can be modified as follows:

$$\frac{1}{T_2} = \rho_s \left( \frac{S}{V} \right)$$  \hspace{1cm} (2)

where $\rho_s$ is the transverse surface relaxation strength of the rock, $\mu$m/ms; $S$ is the surface area of the pores, $\mu$m$^2$; and $V$ is the pore volume, $\mu$m$^3$. According to eq 2, the relaxation time $T_2$ is proportional to the pore radius $r$.

$$r = CT_2$$  \hspace{1cm} (3)

where $r$ is the pore radius, nm; $C$ is the conversion coefficient; and $T_2$ is the transverse relaxation time, ms.

3. EXPERIMENTAL METHOD AND PROCESS

3.1. Experimental Equipment and Coal Sample Preparation. In this experiment, a MesoMR23-060H-I low-field NMR system (the resonance frequency of the device is 21.676 MHz, the magnetic field intensity is 0.5 T, and the magnet temperature is constant at 32 ± 0.01 °C) was used to conduct NMR experiments on coal samples. The coal samples were dried with a type 101 drying box produced by Beijing YongGuangming Company. The physical figure of the experimental test equipment is shown in Figure 1, and the gripper used in the low-field NMR system is shown in Figure 2.
The main parameter settings of the low-field NMR system are shown in Table 1, where SW is the signal frequency range received by the receiver during signal sampling, SF is the principal value of the signal frequency; RFD is the control parameters of the sampling starting point; PRG is the preamplification gain; TW is the interval between repeated samples; NS is the number of accumulations; TE is the echo time; and NECH is the number of echoes.

Fresh coal was collected from Zhongmacun Mine of Coking Coal Group, and cylindrical raw coal samples of Φ25 mm × 50 mm were drilled according to the included angles of coal stratigraphy of 0°, 30°, 45°, 60°, and 90°, respectively, and are marked as ZM1, ZM2, ZM3, ZM4, and ZM5, respectively. The prepared coal samples were sealed with a plastic film for reserve. The coal sample production process is shown in Figure 3.

![Figure 3. Production process drawing of coal samples.](image)

The prepared coal samples were reserved for experimental use, and the fresh small coals that could not be made into cylindrical coals were tested for maceral composition. The test results are shown in Table 2.

### 3.2. Establish the Transformation Relationship between Spectrum Area and Gas Quality.

1. First, put the pressure-bearing empty pipe into the gripper, then put it into the gasket, and finally install the plug of the holder.
2. Connect the equipment piping intact, use a pressurizing device to add 3 MPa confining pressure to the pressure-bearing empty pipe, open the valve to the atmosphere, and pass in 1.5 MPa helium gas, and close the valve to the atmosphere after 1 min (this step aims to remove gas that contains hydrogen atoms from the pipeline).
3. Continue ventilation to stabilize the gas in the pipeline at 1.5 MPa, close the gas valve at this time, and record the gas pressure value after stopping for 30 min. It is found that the gas pressure change is less than 0.01%, and it is considered that the gas tightness of the pipeline is good.
4. Use a vacuum pump to vacuum the pipeline for 60 min to remove the gas in the pipeline.
5. Open the valve of the gas cylinder, and fill the pipeline with gas of purity ≥99.9%. The gas pressure was successively set as 0.33, 0.74, 1.1, 1.47, and 1.98 MPa, and the T2 spectrum under each gas pressure was tested by low-field NMR. The measurement results are shown in Figure 4.

The ideal gas equation of state is corrected to obtain the actual gas equation of state:

\[ PV = \frac{m}{M} RTZ \]  

where \( P \) is the gas pressure, MPa; \( V \) is the measured volume of the test sample placed in the gripper, cm³, which is 16.85 cm³; \( m \) is the quality of the substance, g; \( M \) is the molar quality of the substance, g/mol; \( R \) is the gas constant, 8.314 J/(mol·K); \( T \) is the absolute temperature, K; and \( Z \) is the compression factor.

According to eq 4, the corresponding gas quality under different gas pressures can be obtained. The T2 spectral area and gas quality obtained in the experiment were fitted by a function, and the fitting results are shown in Figure 5.

According to Figure 5, the transformation relationship between T2 spectral area and gas quality is:

\[ y = 9604.59x + 450.766 \]  

### 3.3. Gas Adsorption Experiment Process.

1. Put ZM1, ZM2, ZM3, ZM4, and ZM5 coal samples into an electric blower drying oven and dry at 80 °C for 24 h.
2. Install gripper, and load coal sample ZM1. Use a pressurizing device to apply a confining pressure of 3 MPa and a vacuum pump to vacuum for 60 min. The gas pressure in the pipeline decreases by 0.1 MPa, ensuring the removal of the gas in the pipeline. After vacuumizing, close the valve to the atmosphere, and wait for 30 min, the gas pressure change in the pipeline is less than 0.01%, and it is considered that the gas tightness of the pipeline is good.
3. Use a low-field NMR system to test the semaphore of sample ZM1 as the base signal.
4. Open the valve of the gas cylinder, inject 1.47 MPa gas into the pipeline, and test the semaphore every half hour by using a low-field NMR system. When the semaphore

### Table 1. Main Parameters of the LNMR System

| magnet probe | sequence name | SW/KHz | SF/MHz | RFD/ms | PRG | TW/ms | NS | TE/ms | NECH |
|--------------|---------------|--------|--------|--------|-----|-------|----|-------|------|
| MesoMR-60 mm-jia | Q_CPMG | 250 | 21 | 0.08 | 1 | 5000 | 64 | 0.251 | 10,000 |

### Table 2. Microscopic Coal and Rock Component Detection Results

| result/% | vitrinite group | inert group | total organic | clay | sulfide | carbonate | total inorganic | vitrinite average maximum reflectivity |
|----------|----------------|-------------|---------------|------|---------|-----------|-----------------|-------------------------------------|
| value    | 61.7           | 25.3        | 87.0          | 10.9 | 0.1     | 2.0       | 13.0           | 3.31                                |

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does not increase, it indicates the gas adsorption balance, and stop the experiment.

(5) Set the confining pressure as 4, 5, 6, and 7 MPa in turn and repeat steps (2)~(4).

(6) Repeat steps (2)~(5) for ZM2, ZM3, ZM4 and ZM5 coal samples to conduct experiments successively.

4. RESULTS AND ANALYSIS

4.1. Micropore Structure Analysis of High-Rank Coals with Different Beddings. The different-bedding coal samples are put into a vacuum saturation device for saturation until the quality of the coal sample no longer increases. The fully saturated coal samples are tested with a low-field NMR system for the different-bedding high-rank coals. The $T_2$ spectrum test results of different-bedding coal samples are shown in Figure 6.

Analyzing the experimental results in Figure 6, we can see that the first peak areas (representing micropores) of the $T_2$ spectra of ZM1, ZM2, ZM3, ZM4, and ZM5 are 40,999.437, 29,781.694, 25,264.928, 22,693.186, and 27,466.478, respectively, accounting for 98.61, 99.664, 99.524, 97.363, and 99.657%, respectively. High-rank coals of different beddings all show the law of micropore development, while medium-large pores are not developed.

4.2. Analysis of the Law of Gas Adsorption in Different-Bedding High-Rank Coals. In order to deeply analyze the law of gas adsorption equilibrium of high-rank coals with different beddings under different confining pressures, the low-field NMR test was performed on dried ZM1, ZM2, ZM3, ZM4, and ZM5 coal samples after gas adsorption equilibrium under 3, 4, 5, 6, and 7 MPa confining pressures, respectively. The experimental results are shown in Figure 7.

Analyzing Figure 7 shows that the gas adsorption laws of different-bedding high-rank coals are significantly different. Under the confining pressures of 3, 4, 5, 6, and 7 MPa, ZM1 has the lowest NMR signal, ZM3 has the highest NMR signal, and the NMR signal of ZM5 is the second highest. Combining the transformation relationship between $T_2$ spectral area and gas quality to build the spectrum area and gas quality conversion relationship, the gas adsorption law of different-bedding high-rank coals under different confining pressures is obtained as shown in Figure 8.

Analyzing Figure 8 shows that the gas adsorption capacity of different-bedding high-rank coals has a certain similarity over time. With the increase of time, the gas adsorption capacity is higher, and the gas adsorption capacity first increases rapidly and then slowly as time increases.

Table 3. Basic Physical Property Parameters of Coal Samples

| coal sample number | high/mm | diameter/mm | volume/mL | quality in nature/g | quality after drying/g |
|--------------------|---------|-------------|-----------|--------------------|-----------------------|
| ZM1(0°)            | 49.46   | 25.46       | 25.17     | 39.78              | 38.97                 |
| ZM2(30°)           | 49.51   | 25.50       | 25.27     | 37.08              | 36.88                 |
| ZM3(45°)           | 49.55   | 25.48       | 25.25     | 35.80              | 35.64                 |
| ZM4(60°)           | 49.57   | 25.51       | 25.32     | 37.85              | 37.49                 |
| ZM5(90°)           | 49.61   | 25.48       | 25.28     | 36.45              | 36.02                 |

4.3. Analysis of the Law of Gas Adsorption...
The gas adsorption amount increases rapidly first and then slowly with the increase of time until the gas adsorption amount maintains a constant value at which time the gas adsorption is balanced; different-bedding high-rank coals have a law of decreasing gas adsorption volume with the increase of confining pressure, and a similar law is also present before the coal sample adsorption balance; that is, at 1, 2, 3, 5, 7, and 9 h,

the higher the confining pressure, the lower the gas adsorption capacity of different-bedding high-rank coals.

Considering the influence of coal heterogeneity, in order to further analyze the gas adsorption law of different-bedding
high-rank coals, the increase in adsorption equilibrium of different-bedding coal samples at 3–7 MPa is compared and analyzed. According to the experiment, we can get the results of the gas adsorption capacity of different-bedding high-rank coals at confining pressures of 3 and 7 MPa, and the increase of gas adsorption at confining pressures of 3–7 MPa is shown in Table 4 and Figure 9:

Analysis of Table 4 and Figure 9 shows that the bedding structure has a significant impact on the adsorption law of high-rank coals. When the confining pressure increases from 3 to 7 MPa, the bedding angle of the coal sample is different, and

| coal sample number | 3 MPa confining pressure gas adsorption capacity/g | 7 MPa confining pressure gas adsorption capacity/g | gas adsorption increment/g |
|--------------------|--------------------------------|--------------------------------|--------------------------|
| ZM1                | 0.797                           | 0.658                           | 0.139                    |
| ZM2                | 0.945                           | 0.614                           | 0.330                    |
| ZM3                | 0.999                           | 0.734                           | 0.264                    |
| ZM4                | 0.989                           | 0.788                           | 0.202                    |
| ZM5                | 0.968                           | 0.796                           | 0.172                    |

The increase in gas adsorption is also different. The difference is that the gas adsorption increase of coal samples with diagonal bedding (30° bedding angle) is the largest, and that of coal samples with parallel bedding (0° bedding angle) is the smallest. The reason is that for high-rank coals with bedding angles of 0° and 90°, gas enters the gripper from both ends of the gripper, when the confining pressure increases and the bedding is perpendicular to the confining pressure loading direction; the confining pressure has an obvious closing effect on the pores of the coal sample, which will cause the gas to flow into the coal sample to be blocked; as a result, the gas increment of the coal sample with an angle of 90° with bedding is larger than that of the coal sample with an angle of 0° with bedding. With the increase of confining pressure, some of the pores on the bedding structure surface become micropores due to extrusion and become gas adsorption sites. According to the Pythagorean theorem and calculus, it can be obtained that the integral area of the bedding plane of oblique bedding (with bedding angles of 30°, 45°, and 60°) coal samples is larger than that of vertical and parallel bedding coal samples. Therefore, the number of holes on the bedding plane of oblique bedding coal samples becomes gas adsorption sites because extrusion is also the largest. When the number of holes increases, the adsorption potential of gas in coal also increases, so the adsorption of gas also increases. Therefore, the average increment of gas adsorption in oblique bedding coal samples is the largest. In summary, in the process from low confining pressure to high confining pressure, the average increase of gas adsorption in the oblique bedding (bedding angles of 30°, 45°, and 60°) coal samples is the largest, followed by the vertical bedding (bedding angle of 90°) coal samples, and parallel bedding (bedding angle of 0°) coal samples have the smallest increase in gas adsorption.

In order to further analyze the influence of bedding structure on the adsorption and free peaks, the statistical results of the peak areas of the adsorption and free peaks of gas adsorption equilibrium of different-bedding high-rank coals under different confining pressures are shown in Figures 10 and 11.

Analyzing Figures 10 and 11, it can be seen that when the adsorption equilibrium of different-bedding high-rank coals is under different confining pressures, the adsorption peak area gradually decreases with the increase of confining pressure, and
the area of the adsorption peak first increases and then decreases with the increase of the bedding angle, presenting an “inverted V”. The free peak area and the adsorption peak area show different laws. The free peak area of different-bedding high-rank coals gradually decreases with the increase of confining pressure, and the free peak area first decreases and then increases with the increase of the bedding angle, presenting a "V" shape on the whole. In order to further study the gas adsorption law of different-bedding high-rank coals, the relationship between the adsorbed gas quality and the confining pressure is fitted, as shown in Figure 12.

Analyzing Figure 12, it can be seen that as the confining pressure increases, the quality of adsorbed gas gradually decreases. The reason is that the increase in confining pressure increases the extrusion of the coal wall, compresses the pore Figure 12. (a–e) Relationship between the adsorbed gas quality and confining pressure of high-rank coal with different beddings.

Figure 13. (a–e) Relation between the free peak area and confining pressure of high-rank coals in different beddings.
structure in the coal, and blocks or shrinks the gas adsorption space. There is a linear function relationship between the adsorbed gas quality and the confining pressure of different-bedding high-rank coals, that is, \( y = a - bx \) (where \( a \) and \( b \) are constants greater than 0), and the degree of fit is above 0.94, which indicates that there is a good correlation. Compared with the adsorption peak area, the free peak area is very small; that is, the quality of free gas is also extremely small. Therefore, when studying the law of free gas in different-bedding high-rank coals, it is necessary to further explore the relationship between the free peak area and the confining pressure. The relationship between the free peak area and the confining pressure is shown in Figure 13.

Analysis of Figure 13 shows that as the confining pressure increases, the free gas peak area gradually decreases, and the increase of the confining pressure leads to the blockage and narrowing of the gas migration channel and the compaction of the pores and cracks in the coal, which reduces the permeability. The free gas peak area and the confining pressure conform to the exponential function relationship, that is, \( y = a + b \exp(cx) \) (where \( a, b, \) and \( c \) are constants).

In order to further analyze the changes in the adsorption and free peaks of different-bedding high-rank coals under different confining pressures, the peak center positions of the adsorption and free peaks of different-bedding high-rank coals under different confining pressures are shown in Table 5.

Analyzing Table 5, it can be seen that the maximum center position of the adsorption peak is 1.390 ms, the minimum is 0.918 ms, and the difference between the maximum and minimum values is less than 0.5 ms. Therefore, it is considered that the position of the adsorption peak remains basically unchanged when the confining pressure increases. The maximum position of the center of the free peak is 129.172 ms, and the minimum is 37.192 ms. Obviously, the difference between the maximum and the minimum is large. The variation of the free peak center position of different-bedding high-rank coals under different confining pressures is shown in Figure 14.

Analyzing Figure 14 shows that as the confining pressure increases, the center position of the free peak gradually shifts to the left, which indicates that when the confining pressure increases, the free peak continues to move to the left. Combined with the physical meaning of the abscissa of the \( T_2 \) spectrum, it can be seen that the medium and large pores of high-rank coals will shrink as the confining pressure increases.

5. CONCLUSIONS

(1) The amount of gas adsorption of high-rank coals with different beddings increases with time and decreases with the increase of confining pressure. The amount of gas adsorbed at 1, 2, 3, 5, 7, and 9 h also shows a law of gradual decreases with the increase of confining pressure.

(2) In the process from low confining pressure to high confining pressure, the gas adsorption increments of different-bedding coal samples are obviously different, the average increase of gas adsorption in the oblique bedding coal samples is the largest, followed by the vertical bedding coal samples, and parallel bedding coal samples have the smallest increase in gas adsorption.

(3) The linear function relation between the different-bedding high-rank coal gas adsorption state and the confining pressure is \( y = a - bx \). The relation between the free peak area and the confining pressure conforms to the exponential function \( y = a + b \exp(cx) \); the area of the adsorption peak first increases and then decreases with the increase of the beding angle, presenting an “inverted V” shape on the whole. The area of the free peak first decreases and then increases with the increase of the beding angle, presenting an “V” shape on the whole. When the confining pressure increases, the free peak will continue to move to the left and the large and medium pores will shrink.

Table 5. Central Position of the Adsorption Front and Free Peak

| peak center position/ms | adsorption peak | free peak |
|-------------------------|-----------------|-----------|
|                         | ZM1  | ZM2  | ZM3  | ZM4  | ZM5  | ZM1  | ZM2  | ZM3  | ZM4  | ZM5  |
| 3 MPa                   | 1.297| 1.390| 1.390| 1.21 | 1.390| 91.225| 129.172| 92.087| 87.811| 106.268|
| 4 MPa                   | 1.054| 1.054| 1.054| 1.21 | 1.054| 84.814| 95.421| 86.783| 78.758| 74.226|
| 5 MPa                   | 0.984| 0.984| 1.054| 0.984| 0.918| 57.364| 93.908| 79.562| 78.677| 69.006|
| 6 MPa                   | 0.918| 0.984| 0.984| 1.054| 1.054| 54.805| 73.11 | 76.163| 77.850| 66.866|
| 7 MPa                   | 0.984| 0.984| 0.918| 0.984| 0.984| 37.192| 37.45 | 68.245| 66.286| 62.975|

Figure 14. Change law of the free peak center of high-rank coals with different beddings under different confining pressures.

\[ y = a - bx \]

\[ y = a + b \exp(cx) \]

\[ y = a - bx \]

\[ y = a + b \exp(cx) \]
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