Research Article

Effects of Thermal Treatment on Mineral Composition and Pore Structure of Coal

Bin Liu,1,2 Teng Teng,1,2 Zhenhua Jiao,2 and Shaobo Li2

1Key Laboratory of Safety and High-Efficiency Coal Mining, Ministry of Education (Anhui University of Science and Technology), Huainan 232001, China
2State Key Laboratory of Mining Response and Disaster Prevention and Control in Deep Coal Mines, Anhui University of Science and Technology, Huainan 232001, China

Correspondence should be addressed to Bin Liu; bqt1700101012@student.cumtb.edu.cn and Teng Teng; t.teng@cumtb.edu.cn

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With the increasing depth of coalbed methane (CBM) exploitation, temperature becomes the main factor affecting the efficiency of CBM exploitation. The change of temperature has significant influence on the mineral composition and pore structure of coal. To study the effects of thermal treatment on mineral composition and pore structure of coal, X-ray diffraction (XRD) test, scanning electron microscopy (SEM) test, and mercury intrusion test were carried out for three groups of coal. The mineral composition and pore structure of coal specimens after thermal treatment (25, 50, 75, and 100°C) were analyzed. The results show that the main mineral compositions of three groups of coal specimens after different temperatures are basically unchanged, and the maximum diffracted intensity after different temperature treatments decreases first and then increases with the increasing temperature. The count of fissures decreases first and then increases with temperature, and the count of pores increases first and then decreases with the increasing temperature. The velocity of mercury injection in high pressure (100–400 MPa) of coal specimens increases first and then decreases with temperature. The porosity, pore area, median pore diameter, and average pore diameter increase with the increasing temperatures. The volume of microfracture decreases, then increases, and finally decreases. The volume of macropore and mesopore increases slowly, and that of transition pore decreases slowly with the increasing temperature. Meanwhile, the volume of micropore increases first and then decreases during the process of thermal treatment. The fractal dimension of pore size ranges from 2.6 to 2.9 and increases linearly with the increasing temperature.

1. Introduction

With the depletion of shallow coal resources, more and more coal mines are entering the deep mining stages. The high ground stress, high temperature, and high water pressure restrict the safe and efficient mining of deep coal resources [1–3]. Coalbed methane (CBM) is a form of low-carbon clean energy, which is important for optimizing energy production and achieving carbon neutrality goals [4–6]. The temperature has significant influence on permeability and porosity of coal, which is important for the efficiency of CBM exploitation [7–9]. Therefore, it is urgent to study the evolution of mineral composition and microstructure of coal after different temperature treatments.

Previous studies have proved that rocks and rock-like materials have significant thermal effects, and the physical and mechanical properties change significantly after thermal treatment. The physical and mechanical properties of granite after temperature treatments have been a research hotspot for underground nuclear waste disposal, and many scholars have analyzed its physical and mechanical properties, such as fracture toughness [10], rockburst proneness [11], uniaxial compressive strength [12, 13], elastic modulus [13], longitudinal wave velocity [13], mechanical behavior [14, 15], tensile strength [12, 16, 17], acoustic emission characteristics [18, 19], mineral composition [20], pore structure [16, 20, 21], and permeability [22]. The physical and mechanical properties of sandstone after temperature treatments have also
attracted a lot of attention; the properties such as thermal cracking process [23], peak strength [24], mechanical behavior in unloading conditions [25], wave velocity [26, 27], porosity [27], triaxial mechanical behavior [28], permeability behavior [28], microstructure [26], pore characteristics [29, 30], elastic modulus [29], tensile strength [31], energy evolution [32], microstructure deterioration [33], and morphological properties [34] have been investigated in depth. The physical property and tensile strength of shale have been also analyzed [35, 36]. Peng et al. and Rong et al. analyzed the physical and mechanical behaviors of thermal-damaged marble [37, 38]. The microstructure characteristic, mechanical behaviors, pore distribution, and AE characteristic of limestone have been investigated [39–41]. Yavuz et al. [42] investigated the changes of physical properties of five carbonate rocks (two marbles and three limestones) after different heating temperatures. The unconstrained compressive strength and elastic moduli of gabbro after thermal loading have been studied by Keshavarz et al. [43]. Brotóns et al. [44] investigated the effect of thermal treatment on physical and mechanical properties of calcarenite. Ugur et al. [45] studied the changes in porosity features of natural stones after thermal treatment. The effect of thermal treatment on petrographic and mineralogical composition of coal mining wastes was analyzed by Nowak [46]. Tian et al. [47] investigated the changes of physical and mechanical behavior of claystone due to the thermal treatment, such as uniaxial compressive strength, triaxial compressive strength, and density. The grain size distribution and mineral composition of flux calcined porcelainites after thermal treatment have been studied by Saidi et al. [48]. Ersoy et al. [49] analyzed the mineralogical and geomechanical properties of volcanic rocks subjected to high temperatures. The effect of temperature on pore structure and mechanical properties of shotcrete was studied by Liu et al. [50]. Miao et al. investigated the evolution of coal pore-fracture during the thermal damage process [51]. The above studies mainly focus on physical and mechanical properties of dense hard rocks (granite, sandstone, marble, shale, and gabbro). The main reason is that these dense rock strata are often at high temperatures when storing nuclear waste. Meanwhile, the high temperature in those studies generally exceeds 500°C. However, the geothermal temperature increases by 25~30°C with an increase of 1000 m in mining depth, and the geothermal temperature is generally below 100°C in deep coal mining. Therefore, the temperature of thermal treatment ranges from 25 to 100°C in this study.

Coal, as an anisotropic medium, is highly sensitive to temperature. The structure and mechanical properties of coal will change significantly when the temperature changes, thus affecting the permeability and porosity of coal. To analyze the changes of physical and mechanical properties from a microscopic point of view, the X-ray diffraction (XRD) tests, mercury intrusion tests, and scanning electron microscopy (SEM) tests were carried out for three groups of coal after thermal treatment at different temperatures (25, 50, 75, and 100°C). The mineral composition and microstructure of coal specimens after thermal treatment were analyzed, and velocity of mercury injection, pore parameters, and distribution of pore size for three groups of coal were investigated based on the mercury intrusion tests. The result is significance for CBM exploitation and gas extraction.

2. Experimental Scheme
2.1. Specimen Preparation. As shown in Figure 1, the coal blocks were collected from Jingjia Coal Mine, Changzhi Coal Mine, and Pingdingshan Coal Mine, respectively. Jingjia Coal Mine is located in southwest of Guizhou Province, China. Changzhi Coal Mine is located in the southeast of Shanxi Province, China. Pingdingshan Coal Mine is located in the middle of Henan Province, China. The mineral composition and microstructure of coal specimens after thermal treatment were analyzed, and velocity of mercury injection, pore parameters, and distribution of pore size for three groups of coal were investigated based on the mercury intrusion tests. The result is significance for CBM exploitation and gas extraction.
Figure 2: Continued.
Figure 2: XRD patterns of three groups of coal specimens after different temperature treatments: (a) JJ coal specimen, (b) CZ coal specimen, and (c) PDS coal specimen.

Figure 3: The SEM images of JJ coal specimens after different temperature treatments.
lumps with smooth surface were selected for the mercury intrusion tests and SEM tests after thermal treatment. The coal lumps were milled to coal powder for XRD tests after thermal treatment. The coal specimens from Jingjia Coal Mine, Changzhi Coal Mine, and Pingdingshan Coal Mine were marked as JJ, CZ, and PDS, respectively.

2.2. Experimental Equipment and Process. Before the test, the three groups of coal lumps were heated to target temperature (25, 50, 75, and 100°C) in the drying oven and kept at a target temperature for 24 hours, and then, the coal lumps were sealed and cooled naturally to room temperature. The coal lumps after thermal treatments were prepared for the XRD, SEM, and mercury intrusion tests to analyze the variation of mineral composition, microstructure, and pore structure.

The XRD test was performed on D8 ADVANCE X-ray Diffractometer (BRUKER Corporation, Germany). The radius of goniometer is 250 mm, divergence slit is 0.6 mm, and antisscatter slit is 8 mm. The coal powder was milled and sieved to pass through a 325 mesh sieve, and the mass of each group of coal powder was not less than 0.5 g. The coal powder was poured into the center of a clean sample tray and then capped with a clean glass sheet to flatten the surface of the coal powder. Then, the mineral composition can be measured. The next coal powder is measured after the coal powder measurement completed.

The mercury intrusion test was conducted on Autopore IV 9510 Automatic Mercury Porosimeter. The working pressure ranges from 0 to 60000 psi (414 MPa), and the measurement range of pore size is 0.003~1000 μm. According to the previous research [52], the contact angle was set to 130°, and the mercury (Hg) surface tension was set to 0.485 N/m. The mercury intrusion tests were carried out on three groups of coal specimens, and the related parameters such as pore size distribution, total pore volume, and total pore area can be obtained after the test.

The specimens were scanned by Quanta 250™ SEM scanner (FEI, USA). The electron beam voltage ranges from 200 V to 30 kV, and the range of magnification is 6~100000. Before the test, the sample is first placed in the sample bin and then vacuumed. The region of interest (ROI) was magnified by 400, 1000, 3000, 10000, and 20000 times, respectively, and the SEM images were stored simultaneously.

3. Results and Discussions

3.1. Mineral Composition after Thermal Treatment. XRD tests are commonly used to investigate the mineral composition of coal and rock materials [20, 39, 46, 53, 54]. Therefore, XRD tests were carried out on coal powder after different temperature treatments (25, 50, 75, and 100°C). As shown in Figure 2, the main mineral composition of three groups of coal
specimens is basically unchanged; namely, no chemical changes occur after the thermal treatment. The result is consistent with that of granite [20]. As shown in Figure 2(a), the main mineral compositions of JJ coal specimens are kaolinite, calcium phosphate, and clinochlore. The maximum diffracted intensity is 1506 at 25°C, decreases at 50°C, and then increases to the peak of 1605 at 100°C. As shown in Figure 2(b), the mineral compositions are kaolinite, quartz, and clairite for CZ coal specimens. The maximum diffracted intensity is 1575 at 25°C, decreases to 1250 at 75°C, and then increases to 1323 at 100°C. As shown in Figure 2(c), the mineral compositions of PDS coal specimens are kaolinite, clairite, and montmorillonite. The maximum diffracted intensity is 1601 at 25°C, decreases to 1264 at 75°C, and then increases to 1492 at 100°C. Thus, the maximum diffracted intensity of three groups of coal specimens after different temperature treatments decreases first and then increases with the increasing temperature. The phenomena are mainly caused by thermal treatment and the anisotropy of coal specimens. In summary, there is no change in the main mineral compositions, and the maximum diffracted intensity changes a little after heat treatment. The maximum diffracted intensity after different temperature treatments decreases first and then increases with the increasing temperature.

3.2. Microstructure after Thermal Treatment. SEM tests are commonly used to investigate the microstructure of cross-sections of coal and rock materials [26, 33, 34, 39, 41, 51, 55]. Therefore, SEM tests were carried out on coal samples after different temperature treatments (25, 50, 75, and 100°C). The regions of interest (ROI) of coal specimens were magnified by 400, 1000, 3000, 10000, and 20000 times, respectively. The SEM images of three groups of coal specimens after different temperature treatments are shown in Figures 3–5, which were magnified by 1000 times. As shown in Figure 3, the fissure width is large at the temperature of 25°C, and there are no obvious fissures at the temperature of 100°C. Thus, the fissure width decreases with the increasing temperature. Meanwhile, there are a few pores generated after thermal treatment. Similarly, it can be seen from Figure 4 that the pores grow with the rise of temperature of thermal treatment, and the fissure width decreases with the increasing temperature. As shown in Figure 5, the fissure width decreases first and then increases with the rise of temperature of thermal treatment, and there are many pores generated after thermal treatment. Thus, thermal treatment has a significant effect on microstructure of coal specimens, which is related to the permeability. In summary, the count of fissures decreases first and then increases with the increasing temperature, which is mainly caused by thermal expansion and thermal cracking of coal matrix. Meanwhile, the count of pores increases first and then decreases with the increasing temperature of thermal treatment, which is mainly due to the thermal shrinkage and thermal expansion of coal matrix.
3.3. Pore and Fissure Distribution. Mercury intrusion tests are commonly used to investigate the pore and fissure characteristics of coal and rock [20, 50, 51, 56–58]. To obtain the pore and fissure characteristics of coal specimens after thermal treatment, mercury intrusion tests were carried out on coal samples after different temperature treatment (25, 50, 75, and 100°C). As shown in Figure 6, the mercury injection curve is S-shape, which can be divided into three stages, namely, initial injection stage, slow injection stage, and rapid injection stage. The cumulative pore volume increases fast at the initial injection stage, increases slowly during the slow injection stage, and increases rapidly at the rapid injection stage. There are significant differences in cumulative pore volume for different coal specimens. The cumulative pore volume is 0.03 mL/g in PDS coal specimen, and the cumulative pore volume is 0.12 mL/g in JJ coal specimen, which is 4 times higher than that in PDS coal specimen. The main reason is the differences in microstructure of coal specimens. Similarly, there also exist some differences in mercury injection curve of coal specimens after different temperature treatment. The cumulative pore volume increases first and then decreases with the increasing temperature for CZ coal specimen. For JJ coal specimen, the cumulative pore volume increases first, then decreases, and finally increases with the increasing temperature. The cumulative pore volume decreases first and then increases with the increasing temperature for PDS coal specimen. These phenomena indicate that the mineral composition and pore distribution have a significant effect on the mercury injection curve.

To analyze the velocity of mercury injection of coal specimens after different temperatures, the velocity in high pressure (100~400 MPa) is calculated using the least squares method. As shown in Figure 7, the fitted curves fit well for those data. The velocity of coal specimens after different temperatures.

Figure 6: Curves of mercury injection and ejection in three groups of coal specimens after different temperature treatments: (a) JJ coal specimen, (b) CZ coal specimen, and (c) PDS coal specimen.
the velocity treatment is shown in Figure 8. The velocity of JJ coal specimen is higher than $1.4 \times 10^{-4}$ mL/(g·MPa), and that of CZ and PDS coal samples ranges from $4 \times 10^{-5}$ to $6 \times 10^{-5}$ mL/(g·MPa), which is mainly caused by the differences in microstructure of coal specimens. Meanwhile, the velocity of JJ and CZ coal specimen increases first and then decreases with the increasing temperature. However, the velocity of PDS coal specimen decreases first and then increases with the rise of temperature. These phenomena are mainly caused by the mineral composition and pore structure.

The pore distribution of coal specimens can be measured by the mercury intrusion tests, and the relationship between the pore size and the applied pressure can be expressed as follows [52]:

$$p(r) = -\frac{2\gamma \cos \theta}{r},$$  \hspace{1cm} (1)

where $p(r)$ is the applied pressure, $r$ is the radius of pore, $\theta$ is the contact angle ($130^\circ$), and $\gamma$ is the Hg surface tension (0.485 N/m).

**Figure 7:** Relationship between cumulative volume and pressure in three groups of coal specimens after different temperature treatments: (a) JJ coal specimen, (b) CZ coal specimen, and (c) PDS coal specimen.

**Figure 8:** Relationship between velocity and temperatures in three groups of coal specimens after different temperature treatments.
### Table 1: Pore parameters of the three groups of coal specimens after different temperature treatments.

| Specimen source        | Temperature (°C) | Porosity (%) | Pore area (m²/g) | Median pore diameter (nm) | Bulk density (g/mL) | Average pore diameter (nm) |
|------------------------|------------------|--------------|------------------|---------------------------|---------------------|--------------------------|
| Jingjia Coal Mine      | 25               | 12.52        | 36.57            | 23.4                      | 1.12                | 12.2                     |
|                        | 50               | 13.70        | 41.05            | 21.0                      | 1.11                | 12.1                     |
|                        | 75               | 12.45        | 50.61            | 10.1                      | 1.10                | 8.9                      |
|                        | 100              | 13.92        | 41.11            | 23.5                      | 1.14                | 11.9                     |
| Changzhi Coal Mine     | 25               | 3.98         | 14.06            | 11.0                      | 1.27                | 8.9                      |
|                        | 50               | 4.35         | 15.51            | 11.6                      | 1.24                | 9.0                      |
|                        | 75               | 5.34         | 14.36            | 27.4                      | 1.29                | 11.6                     |
|                        | 100              | 4.71         | 11.59            | 63.7                      | 1.33                | 12.2                     |
| Pingdingshan Coal Mine | 25               | 3.80         | 13.98            | 10.9                      | 1.24                | 8.8                      |
|                        | 50               | 3.63         | 13.00            | 11.4                      | 1.24                | 9.0                      |
|                        | 75               | 3.61         | 12.63            | 11.2                      | 1.27                | 9.0                      |
|                        | 100              | 3.79         | 13.45            | 11.4                      | 1.25                | 9.0                      |

**Figure 9:** Relationship between pore volume and pore diameter in three groups of coal specimens after different temperature treatments: (a) JJ coal specimen, (b) CZ coal specimen, and (c) PDS coal specimen.
The pore parameters of the three groups of coal specimens after different temperature treatments are listed in Table 1. The porosity, pore area, median pore diameter, and average pore diameter for JJ coal specimens are higher than those for CZ and PDS coal specimens. However, the bulk density of JJ coal specimens is lower than that of CZ and PDS coal specimens. In general, the porosity, pore area, median pore diameter, and average pore diameter increase with the increasing temperatures. However, there are some exceptions. These phenomena are mainly caused by the mineral composition and pore structure.

Figure 9 shows the relationship between pore volume and pore diameter in three groups of coal specimens after different temperature treatments, and the pore diameter ranges from 3 nm to 150 μm. Previous studies show that the pore can be classified as microfracture (10~150 μm), macropore (1~10 μm), mesopore (0.1~1 μm), transition pore (10~100 nm), and micropore (3~10 nm) [52]. As shown in Figure 9, the pore volume of micropore and microfracture increases first and then decreases with the increasing temperature of thermal treatment, and the pore volume of macropore, mesopore, and transition pore changes a little with the increasing temperature. Those phenomena indicate that the micropore and microfracture are strongly influenced by the temperature. However, there exist significant differences among these three coal specimens. The pore diameter of JJ coal specimen is mainly concentrated on 3~100 nm, which mainly consisted of micropores and transition pores. On the contrary, the pore diameter is concentrated on 50~150 μm (microfracture) for CZ and PDS coal specimens.

To quantitatively analyze the pore distribution characteristics after different temperature treatments, the pore volume
with different diameters after the thermal treatments is calculated. As shown in Figure 10, the volume of microfracture decreases at 50℃, which is mainly caused by thermal expansion of coal matrix; it increases at 75℃ due to thermal shrinkage of coal matrix; it decreases at 100℃ due to thermal expansion. The volume of macropore and mesopore increases slowly, and that of transition pore decreases slowly with the increasing temperature. Meanwhile, the volume of micropore increases first and then decreases after the thermal treatment. These phenomena indicate that the micropore and microfracture are strongly influenced by the temperature. However, there are some exceptions due to the differences in mineral composition and pore structure.

Previous studies have demonstrated that the pore size distribution of coal and rock conforms to fractal characteristics [51, 59, 60]. Therefore, the effect of temperature on the pore structure of coal after different temperature treatments can be quantitatively analyzed based on fractal theory. According to the methods mentioned in previous studies [51, 59, 60],

\[ \lg \frac{dV}{dP} = k \lg P + b, \]  

where \( V \) is the pore volume, \( P \) is the applied pressure, \( k \) is the linear slope, and \( b \) is the constant.

And the fractal dimension (\( D \)) of pore size can be obtained:

\[ D = k + 4. \]  

As shown in Figure 11, the fractal dimension ranges from 2.6 to 2.9 and increases with the increasing temperature, which indicates that the pore surfaces is less and less smooth as the temperature rises. To analyze the evolution of fractal dimension with temperature quantitatively, the least squares method was used to fit the above data. As shown in Figure 11, the fractal dimension is nearly linearly related to the temperature. These phenomena indicate the pore surfaces of coal become rougher as the temperature rises.

In summary, thermal treatment has significant effects on mineral composition, microstructure, and pore structure of coal. The main mineral compositions after thermal treatment are basically unchanged, and the maximum diffracted intensity decreases first and then increases with the increasing temperature. The count of fissures decreases first and then increases, and the count of pores increases first and then decreases with the increasing temperature. The porosity, pore area, median pore diameter, and average pore diameter increases with the increasing temperatures. The fractal dimension of pore size ranges from 2.6 to 2.9 and increases linearly with the increasing temperature. The pore structure varies with mineral composition and microstructure due to the thermal shrinkage and thermal expansion of coal matrix. Meanwhile, the macroscopic mechanical properties are related to mineral composition, microstructure, and pore structure.

4. Conclusions

The X-ray Diffraction (XRD) test, scanning electron microscopy (SEM) test, and mercury intrusion test were carried out for three groups of coal, and the mineral composition and pore structure of coal specimens after thermal treatment (25, 50, 75, and 100℃) were analyzed. The main conclusions are as follows:

(1) The main mineral compositions of three groups of coal specimens after thermal treatment are basically unchanged, and the maximum diffracted intensity
changes a little after heat treatment. The maximum diffracted intensity after different temperature treatments decreases first and then increases with the increasing temperature

(2) The count of fissures decreases first and then increases with the increasing temperature, and the count of pores increases first and then decreases with the increasing temperature. Thermal treatment has a significant effect on microstructure of coal specimens

(3) The velocity of mercury injection in high pressure (100–400 MPa) of JJ and CZ coal specimens increases first and then decreases with the increasing temperature, but that of PDS coal specimen decreases first and then increases with the increasing temperature. The porosity, pore area, median pore diameter, and average pore diameter increase with the increasing temperatures

(4) The volume of microfracture decreases, then increases, and finally decreases. The volume of macropore and mesopore increases slowly, and that of transition pore decreases slowly with the increasing temperature. Meanwhile, the volume of micropore increases first and then decreases during the process of thermal treatment. The fractal dimension of pore size ranges from 2.6 to 2.9 and increases with the increasing temperature. The fractal dimension is nearly linearly related to the temperature

Data Availability

The experimental data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare no conflict of interest.

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