Compressibility and Fractal Dimension Analysis in the Bituminous Coal Specimens

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Abstract: The fractal characteristics of pore structures in six coal samples with the same rank were investigated. Insights into the relationship between fractal dimension and pore structure parameter were provided. Compressibility effect on the mercury intrusion porosimetry (MIP) data was evaluated. N\textsubscript{2} adsorption (NA) and mercury intrusion porosimetry were applied to analyze the pore structure of coal. The mercury intrusion process was divided into three stages including interpore filling, large intrapore filling and small intrapore filling with compression. Three pore fractal dimensions corresponding to the three stages, \(D_{1C}\), \(D_{2C}\), and \(D_{3C}\) were calculated with the Brooks–Corey capillary pressure model. \(D_{f}\) calculated with Farin model is used to estimate an overall fractal dimension. The correlation between the pore fractal dimension and the pore size distribution (PSD) characteristics was further discussed.

Keywords: Pore Size Distribution, Fractal Dimension, Compressibility, Mercury Intrusion Porosimetry (MIP), Nitrogen Adsorption (NA)

1. Introduction

Fractal theory as a powerful analytical tool has been widely applied to depict the texture of porous materials [1]. The fractal properties of porous materials can be obtained from nitrogen adsorption (NA) [2], small-angle scattering of X-rays (SAXS) or neutrons (SANS) [3], scanning electronic microscopy (SEM) [4], and mercury intrusion porosimetry (MIP) [5]. The pore size ranges from these measurements are different: SAXS, SANS and NA cover the range of 1 nm–100 nm, SEM images can capture pore geometry in range of >10\textmu m, MIP can cover a large range of 1 nm–1 mm. The MIP and NA measurements were chosen for fractal analysis in this study. Previous studies have demonstrated that the pore structure of coal has obvious fractal characteristics, and many fractal models have usually been applied to coal for dimension calculation [6, 7]. The coal structures are too complex to describe, with pore structures have strong heterogeneities, consisting of porous network; these structures contain cracks as well as open and closed pores that vary with the rank and type of coal [8]. Coal samples differ in pore structure because coal is created under varied and complex environmental conditions. Porosity, pore size distribution and pore surface area are still regarded as fundamental parameters for pore structure. Due to the heterogeneous coal, effective methods of characterizing and evaluating the structural properties of coal samples are required.

Numerous studies have been done to investigate the pore structure of coal. Pan et al. [9] distinguished closed pores from open pores using small-angle X-ray scattering and gave a result that the volume of a closed pore have a little change under the tectonic stress. Li et al. [10] evaluated the fractal dimension by MIP data and proposed the data corrected by coal compressibility should be used since effects of the interparticle voids. Gregory et al. [11] discussed and compared the surface area, porosity and other structural properties of coals from different coal fields using different standard methods. Zeng et al. [12] evaluated the pore surface fractal properties of porous materials using Neimark’s model and
Zhang’s model and investigated the relationship between the pore surface fractal dimensions and the porous material composition. Liu et al. [13] obtained the pore fractal dimensions and surface fractal dimensions of pulverized coal particles by N$_2$ adsorption and small-angle X-ray scattering, and the results showed that the surface fractal dimension increases with the increasing size of pulverized coal particle and the pore fractal dimension increases with the decreasing average pore diameter.

This paper intends to evaluate the pore fractal properties and to understand the correlation between the pore fractal dimension and the pore structure properties including pore compressibility, pore size distribution and pore volume. For pore structure measurement, the Mercury Intrusion Porosimetry (MIP) method is chosen for its easy realization and large coverage of pore range. Mercury intrusion phase divided into three regions due to pressure effect was analyzed in order to acquire corrected MIP data. The compressibility of coal is also a remarkable property for pore size distribution. Followed by the MIP measurements using six coal samples, a comprehensive fractal analysis was carried out base on the mercury intrusion date and N$_2$ adsorption data.

2. Experiments and Methods

2.1. Samples Preparation

The samples tested were prepared from a large block obtained from the No.9 coal seam and at depth of 1084 m below the ground surface, in Zhaogezhuang Mine, Kaiping Basin, China, and was cut along with directions of cleats approximately. Six small samples were chosen with the same coal rank and type in this study. They are defined as No.1, No.2, No.3 and No.4.

2.2. N$_2$ Adsorption and Mercury Intrusion Experiments

Nitrogen adsorption (NA) were performed using an ASAP 2020 automatic gas adsorption apparatus at a temperature of 77 K to obtain pore volume in the pore size range of 1.0 nm to 289.3 nm. The super-micropore (<1nm) volume was interpreted from the Density Functional Theory (DFT) equation on the basis of the CO$_2$ sorption at a temperature of 273 K. Prior to the measurement, the samples were heated at 105°C overnight in the oven and then 12h of the vacuum. Pore size distribution was determined by DFT method according to the adsorption/desorption branch of the isotherm.

Mercury injection porosimetry (MIP) analysis was carried out using an AutoPore IV 9500 Instrument from Tsinghua University. The sample size for MIP experiment was about 1cm. Applying the Washburn equation, the pore diameter could be evaluated by using the contact angle between mercury and the pore surface of 130° and the surface tension of 0.485 N/cm. The intrusion pressure was from 0.0030 MPa to 413.47 MPa corresponding to the pore diameter from 1.0 nm to 289.3 nm. The intrusion pressure was from 0.0030 MPa to 413.47 MPa corresponding to the pore diameter from 1.0 nm to 289.3 nm. The super-micropore (<1nm) volume was interpreted from the Density Functional Theory (DFT) equation on the basis of the CO$_2$ sorption at a temperature of 273 K. Prior to the measurement, the samples were heated at 105°C overnight in the oven and then 12h of the vacuum. Pore size distribution was determined by DFT method according to the adsorption/desorption branch of the isotherm.

2.3. Mercury Porosimetry Analysis

Due to the fact that coals are compressible materials, it is necessary to consider the compressibility and correct the MIP data prior to their analysis. If not, abnormal values of the fractal dimensions could be obtained [14]. Some previous studies also found that coal compressibility has an obvious effect on MIP results. In this study, in combination with MIP data and gas (N$_2$ and CO$_2$) adsorption data, the MIP data was adjusted using Li method [10].

2.4. Calculation of the Pore Fractal Dimension

The Brooks–Corey capillary pressure model was applied theoretically in this fractal analysis. The self-affinity is an important feature with an integer fractal dimension for a fractal object, and the feature can be represented mathematically by a power-law function [15]:

$$ N(r) \propto r^{-D} $$ (1)

where $r$ is the radius (or characteristic length) as a unit used to fill the fractal object, $N(r)$ is the number of units with a characteristic linear dimension greater than $r$, and $D$ is the fractal dimension.

According to the previous description and the capillary tube model, $N(r)$ can be expressed by the following:

$$ N(r) = \frac{V_{Hg}}{\pi r^2 l} $$ (2)

where $V_{Hg}$ is the cumulative volume of mercury at a certain capillary pressure [16], $l$ is the length of a capillary tube. Pore radius $r$ which is filled by mercury at an applied pressure can be expressed as follows [17]:

$$ P_c = \frac{2\sigma \cos \theta}{r} $$ (3)

where $P_c$ is the capillary pressure, $\sigma$ is the surface tension, and $\theta$ is the contact angle.

Using Eqs. (1), (2) and (3), the Eq. (4) is derived:

$$ N(r) \propto P_c^{-(2-D)} $$ (4)

Then the mercury saturation can be expressed as follows [15]:

$$ V_s = \frac{V_{Hg}}{V_p} $$ (5)

where $V_s$ is the mercury saturation (%), $V_p$ is the pore volume of the core samples [15].

Eq. (6) can be obtained by substituting Eq. (5) into Eq. (4):

$$ V_s = aP_c^{-(2-D)} $$ (6)

where $a$ is a constant. Eq. (6) generally proves that the relationship between the mercury saturation ($V_s$) and the capillary pressure ($P_c$) is linear in a double-logarithm coordination ($\log(V_s)$-$\log(P_c)$). The fractal dimension can then be derived from the slope of the straight lines [15].

$$ D = S + 2 $$ (7)
where S is the straight line slope of $\log(V_s)$-$\log(P_c)$ plots.

3. Results and Discussion

3.1. Analysis for Coal Pore Experiment Data

Based on Hodot’s pore classification method, there are micropore (<10 nm), transition pore (10-100 nm), mesopore (100-1000 nm) and macropore (>1000 nm) in coal. In this paper, the classification method was applied to analyze the pore structure and the small pore including micropore, transition pore and mesopore. Pittman [18] proposed the plotting of mercury saturation versus the mercury saturation divided by capillary pressure. The plots of the mercury saturation/injection pressure versus mercury saturation are showed in Figure 1. The Pittman's plot produces a sharp and easily defined apex and a turning point. The maximum points in these curves indicate the transition from the broad and well-connected to small and poorly-connected pores. The turning points indicate that the progress of pores connecting needs more external force and the compressibility as a remarkable factor should be reckoned with in analyzing pore structure.
Figure 1. Mercury saturation /injection pressure (MPa) versus mercury saturation (V, %).

According to Figure 1, the interpore is directly connected to mercury. During MIP measurement, the process of interparticle filling continued until the applied pressure is increased to \( P_1 \). The intrapore is filled with mercury when the applied pressure is increased to \( P_2 \). The pressure of \( P_1 \) is not big enough to force mercury to intrude into the intrapores. However, the additional pressure has a very limited influence on large pore. At the pressure of \( P_{1,2} \), it is believed that the intruded mercury is attributed to the large intrapores filling. The applied pressure between \( P_1 \) and \( P_2 \) had almost no compressibility on coal samples. Only when the applied pressure is increased to \( P_2 \) or even greater than \( P_{1,2} \), mercury intrudes into small intrapores. In this phase, the capillary tube or new passageway due to intrusion pressure is a promotion factor for pore connection, and the volume of capillary tube or new passageway was also classified as smaller intrapores including mesopore, transition pore and micropore. As a result, the MIP measurement gives an overestimation of the volume of the small pore [19].

Li method [10] is used to calculate the compressibility of the studied samples with pressure over 1.2001 MPa, 1.1808 MPa, 1.1531 MPa and 1.1824 MPa (corresponding to the diameter about 1000nm, respectively) in this paper (Table 1). Porosimetric analysis of different coal samples indicates that there is much mercury intrusion and apparent compressibility for pressures above 1.15–1.20 MPa. So the diameter of the pores under 1000nm could be corrected based on the \( N_2 \) adsorption date. Figure 2 shows that the mercury intrusion volume sharply increased at lower pressure and the incremental intrusion volume of mercury apparently reduced in the range of higher pressure. A linear relationship fitting the MIP data well at higher pressures was found, and the slopes of these fitted lines have apparent difference range of 0.000029 to 0.000117 (Figure 2). Coal samples exhibit different
increments in mercury intrusion volume as the intrusion pressure increases under the higher pressures.

**Table 1.** Pressure classification corresponding to different mercury intrusion stages.

| Sample | Mercury intrusion pressure (MPa) |
|--------|----------------------------------|
|        | $P_1$     | $P_{1-2}$       | $P_2$         |
| No.1   | <0.0068   | 0.0068–1.2001  | >1.2001       |
| No.2   | <0.0085   | 0.0085–1.1808  | >1.1808       |
| No.3   | <0.0069   | 0.0069–1.1531  | >1.1531       |
| No.4   | <0.0068   | 0.0068–1.1824  | >1.1824       |

$P_1$ – interpore filling; $P_{1-2}$ – large intrapore filling; $P_2$ – small intrapore filling and coal compression

The original observed MIP data and the MIP data with compressibility correction were presented in Figure 3. It can be seen that the differences between original and corrected data are clearly. The results also indicate that coal compressibility as an important factor effect on MIP date a lot especially for pressure exceeding 20 MPa, which is compatible with the previous studies. Table 2 listed the corrected pore volume in the pore size range of 3–100 nm. The final pore size distributions were shown in Figure 4.
Figure 4. Pore volume percentage with different pore sizes.

Table 2. Comparison of pore volume in the pore size range of 3–100 nm estimated by mercury intrusion and N\textsubscript{2} adsorption.

| Sample ID | MIP a (ml/g) | NA b (ml/g) | c (ml/g) | \(K_{pc} \times 10^{-3}\) (MPa\textsuperscript{-1}) |
|-----------|--------------|-------------|----------|----------------------|
| No.1      | 0.0436       | 0.0083      | 0.0078   | 2.2117               |
| No.2      | 0.0236       | 0.0073      | 0.0011   | 1.1740               |
| No.3      | 0.0105       | 0.0023      | 0.0011   | 1.6022               |
| No.4      | 0.0394       | 0.0082      | 0.0055   | 1.7750               |

where a is the pore volume in the pore size range of 3–100 nm; b is the pore volume with correction in the pore size range of 3–100 nm; c is the pore volume in the pore size range of 3–100 nm from NA; \(K_{pc}\) is the pore compressibility of coal.

3.2. Pore Fractal Analysis

Log (Vs)-log (Pc) plots were constructed for all the four samples in Figure 5. The No. 1C (i = 1, 2, 3, 4) presents that the Log (Vs)-log (Pc) curves were plotted by using the corrected coal pore data. The plots pressures corresponding to these three processes, divided by \(P_1\) and \(P_2\), were as a reference for fractal dimension analyses, respectively, as shown in Table 2. The results show that all of the plots show good fits, indicating that these coal samples are general fractal and can be characterized by using the theories of fractal geometry. As can be seen in Figure 5, the curves of log (Vs) versus log (Pc) were divided into three segments at apex points and turning points. With the MIP data corrected, small pores (corresponding to high pressure) tend to have a slope of 0.2682, 0.0752, 0.0758 and 0.0985 while large pores were more likely to have two kinds of slope; the large pores at \(P_1,2\) have a slope of 0.1366, 0.0895, 0.1282 and 0.1158; the other large pores have a slope of 1.5260, 1.4928, 2.6164 and 3.4222. The ideal power-law for Vs against Pc hardly exists, and the corresponding slope varies with the scale Pc and the pore radius. In fact, all the log(Vs)-log(Pc) plots have obvious inflection points at certain capillary pressures (Figure 5).
Figure 5. Plots of log ($V_s$) versus log ($P_c$) for dimensions analysis.
For the small pores (<1000 nm), the calculated fractal dimensions are generally less than 2.3 (Table 3), which indicates that the pore systems consisting of small pores are less heterogeneous. Previous studies confirmed that micropores (<10 nm) have a greater impact on fractal dimensions than mesopores and macropores [20]. They used the MIP date without correction, so the number of micropore increased apparently during mercury intrusion. Although the results of fractal dimension have great differences, an undoubtedly point is that the micropores play an important role on fractal dimensions. $D_{1C}$, $D_{2C}$, $D_{3C}$ and $D_1$ are near to the truly original pore fractal of coal samples. Large pores have larger fractal dimension values than 3.0, the result is contrary to previous studies. The reason is that we calculated fractal dimension applying the capillary tube model. The process of mercury intrusion is also the process of pore connection which depends on capillary tube, so it is exact to use capillary tube model for fractal dimension calculation. Combination with the other experiment results [21-23], under the increasing intrusion pressure the values of $D_1$ become smaller; $D_2$

| Sample | Li and Gao, H model | Farin model |
|--------|-------------------|-------------|
|        | $D_1$ | $D_{1C}$ | $D_2$ | $D_{2C}$ | $D_3$ | $D_{3C}$ | $D_f$ |
| No.1   | 4.6813 | 3.5260 | 2.2567 | 2.1366 | 2.3387 | 2.2682 | 3.0266 |
| No.2   | 3.4969 | 3.4928 | 2.0891 | 2.0895 | 2.1439 | 2.0752 | 2.8762 |
| No.3   | 4.6164 | 4.6164 | 2.1282 | 2.1282 | 2.1879 | 2.0758 | 2.9060 |
| No.4   | 5.4458 | 5.4222 | 2.1162 | 2.1158 | 2.2311 | 2.0985 | 2.9681 |

Table 3. Fractal dimensions of coal samples with different methods.

Figure 6. Plots of $\ln(\text{dV/dP})$ versus $\ln(P)$ for dimensions analysis.
becomes greater but the values are still ranging from 2.0 to 3.0. The values of $D_3$ become greater than 3.

Applying Farin’s method, $D_f$ represents an overall fractal dimension for all pore sizes ranges. The values of $D_f$ have almost no change in the process of mercury intrusion which are about 3.0. $D_f$ is used to evaluated the pore fractal dimension for the whole coal sample, and the value of $D_f$ is affected by many properties of coal. In this study, the $D_f$ has an negative correlation with the percentage of macropore volume (Figure 4 and Figure 6). The value of $D_f$ decreases with the increasing percentage of macropore volume. The result also proves that small pore (<1000nm) has a remarkable effect on fractal dimension for coal.
As showed in Figure 7, although there are no clear correlations with coal samples for $D_1$ and $D_{1c}$, $D_2$ and $D_{2c}$, all the $D_3$ values of corrected are less than that of uncorrected ones which means that the small pores could be compressed easily compared to the other pores. $D_{2c}$ is more likely used to describe the interparticle pore, and $D_{1c}$ is used to describe the large intraparticle pores. The $D_{3c}$ can be represented the compression of coal samples, it also indicates small intraparticle pores manipulate the compressibility. The small pores consist of mesopore (100-1000nm), transpore (10-100nm) and micropore (<10nm). Since the $D_3$ can be used as an index to measure the coal compression, the variability of the coal can be described by the difference between the fractal dimensions $D_3$ both with and without correction, as shown in Figure 7c, the bigger difference values of $D_3$ means coal samples can be easily influenced by increasing intrusion pressure. Different values of $D_3$ for the coal samples studied in this paper are as follows: $\Delta D_3$ is 0.0705, 0.0687, 0.1121 and 0.1326 corresponding to No.1, No.2, No.3 and No.4 coal sample, respectively.
Figure 8. The influence of pore volume for fractal dimension.
The differences of $D$ before and after MIP data correction are shown in Figure 8. The variations of volume percentage were calculated with the origin date and corrected data at different pore size ranges. It is clear that $D$ varied with pore volume. However, the pore size corresponding to changed pore volume also has a remarkable effect on the fractal dimension. Volume percentage variation of micropores is larger than the ones of transpores and mecropores (Figure 8).

So the volume variation of micropores could lead to the $D_3$ changed but there is no apparently linear relationship between the value of $D_3$ and $\Delta D_3$. It indicates that pore volume and pore size of macropore volume. It also proves that small pore (<1000 nm) has a remarkable effect on fractal dimension of coal. The volume changes of macropore size range of 1000 nm-100000 nm almost have no effects on $\Delta D_2$ and $\Delta D_1$. It indicates that pore volume and pore size of macropore structure are not the dominant factors for fractal dimension.

4. Conclusion

Pore compressibility and pore types including interpore, large intrapore and small intrapore should be taken into consideration for MIP data correction. Pore fractal dimension not only correlate with micropore or transition pore size and its volume, but also is controlled by the connectivity with the increasing external pressure, which lead to the apparent change of volume percentage. The difference between $D_3$ and $D_{3c}$ has an apparent correlation with the micropore properties. The value of $D_3$ decreases with the increasing percentage of macropore volume. It also proves that small pore (<1000nm) has a remarkable effect on fractal dimension of coal.

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