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Study on multi-scale characterization and fractal characteristics of coal pore structure in Wuyang Coal Mine of Luan Mining Area

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Abstract: In order to explore the microscopic pore structure characteristics and fractal law of coal reservoir in Wuyang Coal Mine of Lu 'an mining area, the coal samples of No.3 coal seam in Wuyang Coal Mine were selected, and the microscopic morphology, pore structure and surface complexity were characterized by scanning electron microscopy (SEM), low temperature liquid nitrogen adsorption experiment and FHH fractal model. The results show that the matrix of coal sample is relatively dense, mainly develops pores and dissolution pores, and is accompanied by micro-fractures. The pore structure is mainly mesoporous with an average pore diameter of 8.255 nm. The pore volume is mainly concentrated in the range of 10-50 nm and > 50 nm. The nitrogen adsorption isotherm is type IV and has a H3 hysteresis loop, indicating that the pore morphology is mainly slit-like pores formed by irregular particle accumulation. FHH fractal analysis shows that the pore structure has obvious segmentation characteristics. The surface fractal dimension (D1) of the low pressure zone is higher (reflecting the roughness of the pore surface), and the fractal dimension (D2) of the high pressure zone structure is relatively low (reflecting the pore size distribution tends to be concentrated). The research shows that the coal reservoir in Wuyang Coal Mine has formed a multi-scale pore system dominated by mesopores, accompanied by macropores and fissures. The mesopores dominate the gas adsorption capacity, while the macropores and fissures provide channels for gas migration. The synergistic effect of the two determines the adsorption-diffusion-seepage characteristics of the coal reservoir.

Keywords: Wuyang Coal Mine, coal pore structure, low temperature liquid nitrogen adsorption, fractal characteristics, multi-scale characterization.

1. Introduction

Coalbed methane (CBM), as a typical unconventional natural gas resource, has become an important part of the global energy structure transformation due to its abundant reserves, wide distribution and great development potential [1-2]. Unlike conventional natural gas, coalbed methane mainly exists in the pores of coal matrix in the form of adsorption, and its occurrence state and migration behavior are highly dependent on the pore structure characteristics of coal reservoirs [3]. Therefore, the pore structure of coal not only determines the reservoir capacity of coalbed methane, but also controls its diffusion and seepage process, which is the key factor affecting the development efficiency and productivity prediction of coalbed methane [4].

A large number of studies have shown that the pore structure of coal reservoirs has significant multi-scale, heterogeneity and complexity, and its formation and evolution are controlled by multiple factors such as sedimentary environment, coalification and tectonic transformation [5-6]. On the micro scale, coal pores can be divided into micropores, mesopores and macropores. Different scales of pores play different roles in gas adsorption and migration: micropores mainly provide adsorption space, mesopores play a transitional role between adsorption and diffusion, while macropores and fractures constitute the main channels of gas seepage [7]. Therefore, the fine characterization of coal pore structure from a multi-scale perspective is an important prerequisite for in-depth understanding of coalbed methane reservoir mechanism.

In recent years, with the development of testing technology, low temperature liquid nitrogen adsorption (LTNA) and scanning electron microscopy (SEM) have become the core means of coal pore structure research. Among them, low-temperature nitrogen adsorption technology can effectively characterize the mesoporous structure in the range of 2-50 nm, and can be combined with BET and BJH models to achieve quantitative analysis of pore size distribution and specific surface area [8-9]. SEM technology can directly reveal the pore morphology and micro-crack development characteristics [10]. On this basis, fractal theory has been widely introduced into the research field of coal pore structure, which is used to quantitatively describe the surface roughness and spatial complexity of pores [11]. In particular, the Frenkel-Halsey-Hill (FHH) model can calculate the fractal dimension based on the adsorption isotherm data, thus reflecting the heterogeneous characteristics of pore structures at different scales [12]. Studies have shown that there is a significant correlation between fractal dimension and pore specific surface area, pore volume and gas adsorption capacity, which is an important parameter to evaluate the complexity of coal reservoirs [13-14].

Although some progress has been made in the characterization of coal pore structure at home and abroad, there are still some shortcomings. On the one hand, the research on specific mining areas mostly focuses on macroscopic geological characteristics and coalbed methane enrichment law, and the multi-scale quantitative research on microscopic pore structure is relatively insufficient. On the other hand, the internal relationship between pore structure parameters and adsorption-diffusion behavior of coalbed methane still needs to be further discussed in combination with fractal theory [15]. As an important area of coalbed methane development in Qinshui Basin, Lu 'an mining area has high development potential in its main coal seam (such as No.3 coal seam), but the research on pore structure and fractal characteristics of specific mines such as Wuyang coal mine is still limited.

Based on this, this paper takes No.3 coal seam of Wuyang Coal Mine in Lu 'an Mining Area as the research object, and comprehensively uses scanning electron microscopy (SEM), low-temperature liquid nitrogen adsorption experiment and FHH fractal model to characterize the microscopic morphology, pore structure parameters and fractal characteristics of coal samples at multiple scales. The purpose of this study is to reveal the pore types, pore size distribution and fractal characteristics of coal in the study area, to explore the control mechanism of pore structure on the adsorption and migration behavior of coalbed methane, and to provide a theoretical basis for the efficient development of coalbed methane resources in Lu 'an mining area.

2. Sample Collection and Test Methods

2.1. Sample and Pretreatment

The Luan mining area is located in the eastern part of the middle section of the Qinshui coalfield. The eastern boundary is the coal seam outcrop line, which is connected with the Qinyuan mining area and the Anze mining area in the west, the Yangquan mining area in the north, and the Jincheng mining area in the south. The strata in the study area belong to the Ningwu-Linfen area of Shanxi stratigraphic division in the North China stratigraphic area. The Ordovician, Carboniferous, Permian, Triassic and Quaternary are mainly developed in the mining area. Lu 'an mining area is based on the Ordovician limestone. The coal-bearing strata are mainly Taiyuan Formation and Shanxi Formation. The No.3 coal seam of Shanxi Formation and No.15 coal seam of Taiyuan Formation are the minable coal seams in the whole area. The No.3 coal seam is the main coal seam for the development of coalbed methane in Lu 'an mining area. The macroscopic coal rock type is semi-bright-bright briquette. The vitrinite group is developed, mainly matrix vitrinite, and the exinite group is not developed. The inorganic components are mainly clay and calcite, and the average mineral content is 5.95 %. The coal quality is low ash-extra low ash coal and extra low sulfur coal. The maximum vitrinite reflectance $R_{o,\,max}$ is 1.87 % -2.30 %, which belongs to lean coal-lean coal.

The basic physical parameters of coal samples in the study area are detailed in table 1. The experimental analysis results show that each sample shows high consistency in coal rock macerals, coal quality characteristics and metamorphic degree, which lays a reliable foundation for the subsequent comparative study. After the macroscopic morphology identification and microstructure observation of the sample were completed, it was crushed and ground to 40 ~ 60 mesh (0.250 ~ 0.425 mm), and the sampling amount of single test was controlled at 2 ~ 3 g. Under the environment of liquid nitrogen boiling point temperature (77.35 K, 101.3 kPa), the pore structure parameters with pore size greater than 2 nm were measured by ASAP 2020 automatic specific surface area and porosity analyzer with high purity nitrogen as adsorbate, covering core indicators such as specific surface area, pore volume and pore size distribution. In order to ensure the measurement accuracy, the sample should be continuously degassed for 24 h in a vacuum environment at 110 ° C before testing, so as to deeply remove the impurity gas and volatile matter adsorbed in the pores. Subsequently, according to the amount of nitrogen adsorption-desorption under different relative pressures, the specific surface area and pore volume of the samples were inverted by the classical BET and BJH models, respectively.

Table 1. Industrial analysis and elemental analysis results of coal samples from Wuyang Coal Mine

coal mine	industrial analysis /%				$S_{t,d}$/%	elementary analysis /%			
	M_{ad}	A_d	V_{daf}	FC_d		O_{daf}	C_{daf}	H_{daf}	N_{daf}
visceral yang	0.70	7.67	13.72	79.66	0.32	3.37	90.60	4.17	1.52

2.2. BET Specific Surface Area and BJH Pore Size Distribution Calculation Model

The adsorption-desorption data obtained from the low-temperature liquid nitrogen adsorption experiment can be used to quantitatively characterize the pore structure parameters of coal samples. In this paper, the specific surface area, pore volume and pore size distribution of coal samples were calculated and analyzed by Brunauer-Emmett-Teller (BET) model and Barrett-Joyner-Halenda (BJH) model.

The BET model is used to calculate the specific surface area. When the relative pressure P_0/ P is in the range of 0.05 ~ 0.30, the expression is:

$$\frac{1}{V\left(\dfrac{P_0}{P}-1\right)}=\frac{C-1}{V_m C}\cdot\frac{P}{P_0}+\frac{1}{V_m C} \tag{1}$$

In the equation: V is the adsorption capacity (cm ^{3} / g); the monolayer adsorption capacity (cm^3 / g); c is the BET constant.

The specific surface area calculation formula is:

$$S=\frac{V_m N_A}{m} \tag{2}$$

N_A is the Avogadro constant; V_m is the cross-sectional area of nitrogen molecules; m is the sample mass.

The BJH model is based on the Kelvin equation and capillary condensation theory, which is used to calculate the pore size distribution and pore volume in the range of 2-50 nm. This method inverts the pore size distribution by desorbing the branch line data, so as to obtain the pore volume and specific surface area in different pore size intervals.

2.3. Fractal Dimension Calculation Method (FHH Model)

Based on the Frenkel-Halsey-Hill (FHH) model, the fractal analysis of liquid nitrogen adsorption data is carried out. The expression is:

$$\ln V = A\ln\left[\ln\left(\frac{P_0}{P}\right)\right]+B\,) \tag{3}$$

In the equation: V is the amount of adsorption; p_0 is the equilibrium pressure; p is saturated vapor pressure; a and B are constants.

The calculation formula of fractal dimension is: D = A + 3 According to the characteristics of adsorption isotherm, the relative pressure is divided into low pressure zone ($P / P_0 < 0.5$), and the fractal dimension D_1 and D_2 are calculated respectively. Where: D_1 characterizes the surface roughness of the hole; D_2 characterizes the spatial complexity of pore structure. The fractal dimension ranges from 2 to 3, and the larger the value, the more complex the pore structure.

3. Experimental Results and Analysis

3.1. SEM Microscopic Pore Morphology and Cause Analysis

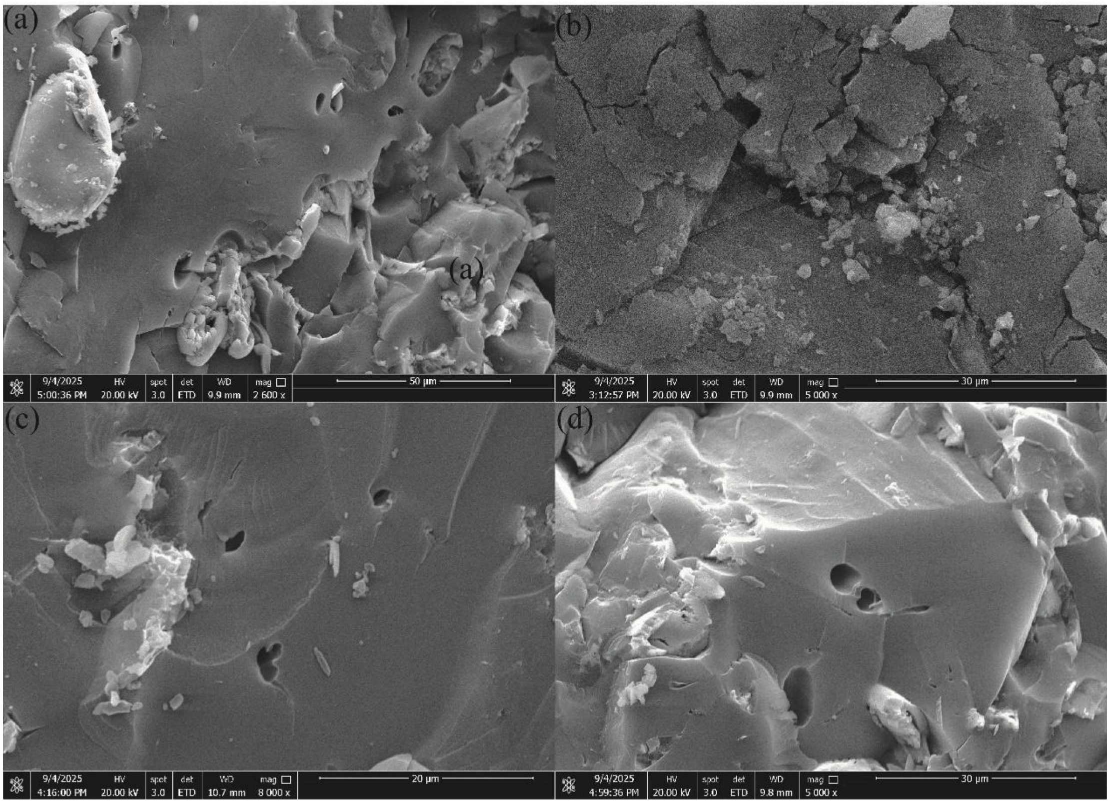

Figure 1. Scanning electron microscope (SEM) micro-morphology characteristics of coal samples from Wuyang Coal Mine

In order to intuitively reveal the microscopic occurrence state of coal samples, the surface morphology was characterized by scanning electron microscopy (SEM). Scanning electron microscopy (SEM) results can be used to visually characterize the surface morphology and pore-crack structure characteristics of the samples. It can be seen from Fig.1 (a-d) that the whole sample is dominated by relatively dense matrix, but a certain number of pores and fracture structures are developed on the surface, showing obvious heterogeneous characteristics. There are some differences in the degree of structural density and pore development in different regions, which together constitute the pore-fracture composite structure system.

From the perspective of pore type, the pores in the sample mainly include two types: pores and dissolution pores. The pores are mostly nearly round or oval, with smooth boundaries and relatively flat pore walls. It is speculated that they are mainly derived from the limited generation and dissipation of gas during the formation or treatment of materials. The gas is retained in the matrix and forms isolated or semi-closed pores. The dissolution pores are mostly irregular in shape and rough in boundary, often accompanied by local particle shedding or surface erosion, indicating that the sample may undergo fluid action or chemical erosion process in the later stage, resulting in dissolution and reconstruction of the local structure. Although the above qualitative observations reveal the existence of pores, it is necessary to rely on low-temperature liquid nitrogen adsorption experiments to accurately quantify pore structure parameters. Based on this, the pore structure of the sample was further quantitatively analyzed.

3.2. Analysis of Low Temperature Liquid Nitrogen Adsorption Characteristics

3.2.1. Characteristics of Specific Surface Area and Pore Structure Parameters

Based on 8 coal core samples collected from Wuyang Mine in Lu'an mining area of Qinshui Basin, low temperature liquid nitrogen adsorption experiments were carried out. The pore specific surface area of coal samples was calculated by BET method, and the parameters such as pore size and pore volume were calculated and analyzed by BJH method. The calculation results (Table 2) show that the BET total pore specific surface area of coal samples is 0.611 ~ 12.854 m^2 / g, with an average value of 2.638 m^2 / g ; the total pore specific surface area of BJH is 0.551 ~ 6.415 m^2 / g, with an average value of 1.885 m^2 / g. Compared with the BET calculation results, the specific surface area of BJH is slightly lower. The average pore size distribution of BJH is 5.775 ~ 17.842 nm, with an average of 8.255 nm. The total pore volume of BJH ranged from 0.0018 to 0.0112 cm^3 / g, with an average of 0.0037 cm^3 / g. In general, the pore size of high-rank coal in the study area is mainly concentrated in the range of medium pore scale, and the pore structure is mainly transition pore-medium pore, which indicates that the pore structure of coal reservoir has certain heterogeneity.

Table 2. Pore structure parameters of coal samples in Wuyang Coal Mine

specimen	BET Total pore specific surface area ($m^2 \cdot g^{-1}$)	BJH total pore specific surface area ($m^2 \cdot g^{-1}$)	Total pore volume of BJH (10^{-3} $cm^3 \cdot g^{-1}$)	BJH average pore size (nm)	N_2 adsorbed amount ($cm^3 \cdot g^{-1}$)
LA6801-9-1	15.62	14.10	20.4	5.21	6.82
LA6801-9-2	14.85	13.57	19.8	5.48	6.55
LA6801-9-3	16.03	15.02	21.3	4.07	7.02
LA6801-10-1	8.38	7.95	14.2	7.12	4.36
LA6801-10-2	7.48	7.01	13.6	7.56	4.01
LA6801-10-3	15.87	14.73	20.8	6.34	6.91
LA6801-12-1	3.52	3.21	10.5	5.08	2.64
LA6801-12-2	13.66	12.98	23.1	4.89	6.11

The total pore volume of BJH is distributed between 0.009 ~ 0.080 cm^3 / g, which shows that the development degree of pore structure of coal samples is obviously different. Through the analysis of the relationship between BET specific surface area, BJH specific surface area and BJH total pore volume (Figure 2a, b), it can be seen that there is a good positive correlation between the parameters, indicating that the pore volume has an important control effect on the specific surface area. Because the micropores are not developed, the BET specific surface area is basically the same as the BJH specific surface area, indicating that the specific surface area is mainly derived from the mesoporous structure. Figure 2c and d show that the adsorption amount of N_2 is positively correlated with the specific surface area and pore volume, and has a stronger correlation with the pore volume, indicating that the pore volume is the main controlling factor affecting the adsorption capacity.

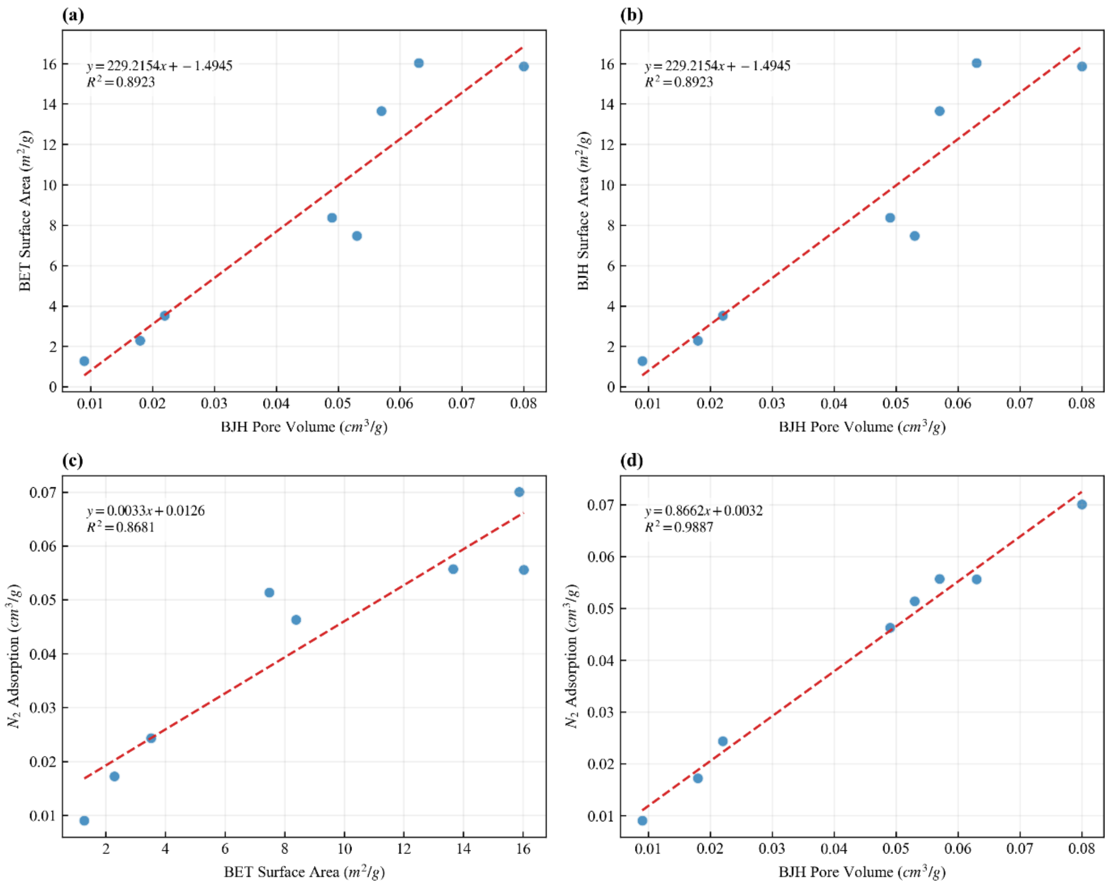

Figure 2. The relationship between specific surface area, pore volume and adsorption capacity of coal samples

3.2.2. Pore Distribution Characteristics

According to the experimental data of liquid nitrogen adsorption, the contribution of pore size and pore volume of different samples to pore specific surface area and pore volume was analyzed. As shown in figure 3 (a), the incremental pore volume distribution curves of each sample are responsive in the range of micropores to mesopores, showing obvious multi-peak characteristics as a whole, indicating that multi-scale pore structure is developed inside the sample. Among them, some samples have obvious peaks in the small pore size range, reflecting the development of micropores and small mesopores; at the same time, there is still a certain distribution in the larger pore size range, indicating that the system also contains a certain number of macroporous structures. It can be seen from Fig. 3 (b) that the BJH pore volume distribution curve shows an overall upward trend with the increase of pore size, and gradually tends to be gentle in the medium-macropore range, indicating that the main contribution of pore volume comes from mesoporous and macroporous structures.

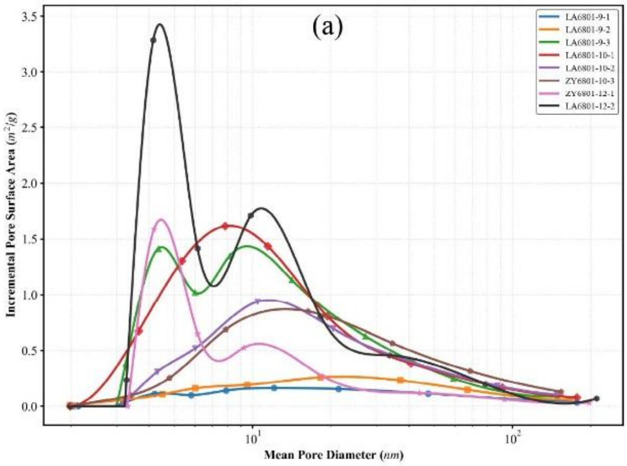

Figure 3. The relationship between the specific surface area of the BJH stage, the pore volume of the BJH stage and the average pore diameter of the BJH stage

There are differences in the curve shape between different samples, reflecting the heterogeneity of pore size distribution and pore volume development. Further combined with the statistical results of pore size classification (Fig.4), the pore volume of the sample is mainly concentrated in the range of 10-50 nm and > 50 nm, in which 10-50 nm mesopores dominate, while micropores (50 nm) contribute significantly, indicating that there are more developed pore or fracture structures inside. In general, the pore structure of the sample is dominated by mesopores, accompanied by a certain proportion of macropores, showing typical multi-scale pore system characteristics. This kind of multi-scale pore structure, which is dominated by mesopores and supplemented by macropores, is conducive to providing a good mass transfer channel while ensuring a certain specific surface area, thus having a positive impact on gas adsorption, diffusion and seepage behavior. On the basis of defining the pore size distribution, the shape of the nitrogen adsorption-desorption isotherm can further reflect the pore connectivity and pore shape.

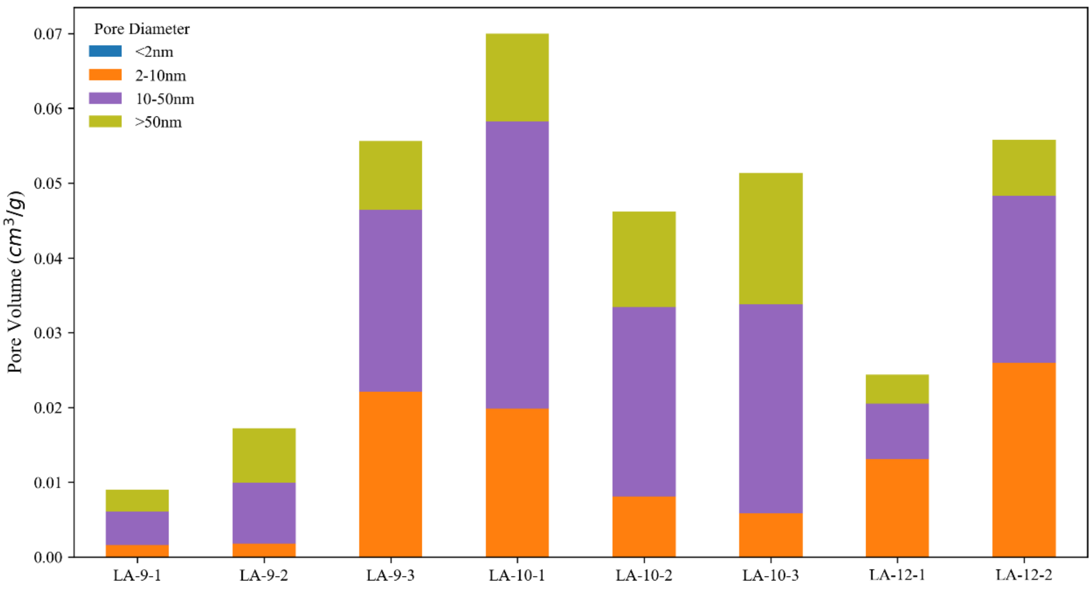

Figure 4. Pore size distribution and pore volume distribution characteristics of coal samples

3.2.3. Adsorption-desorption isotherm characteristics

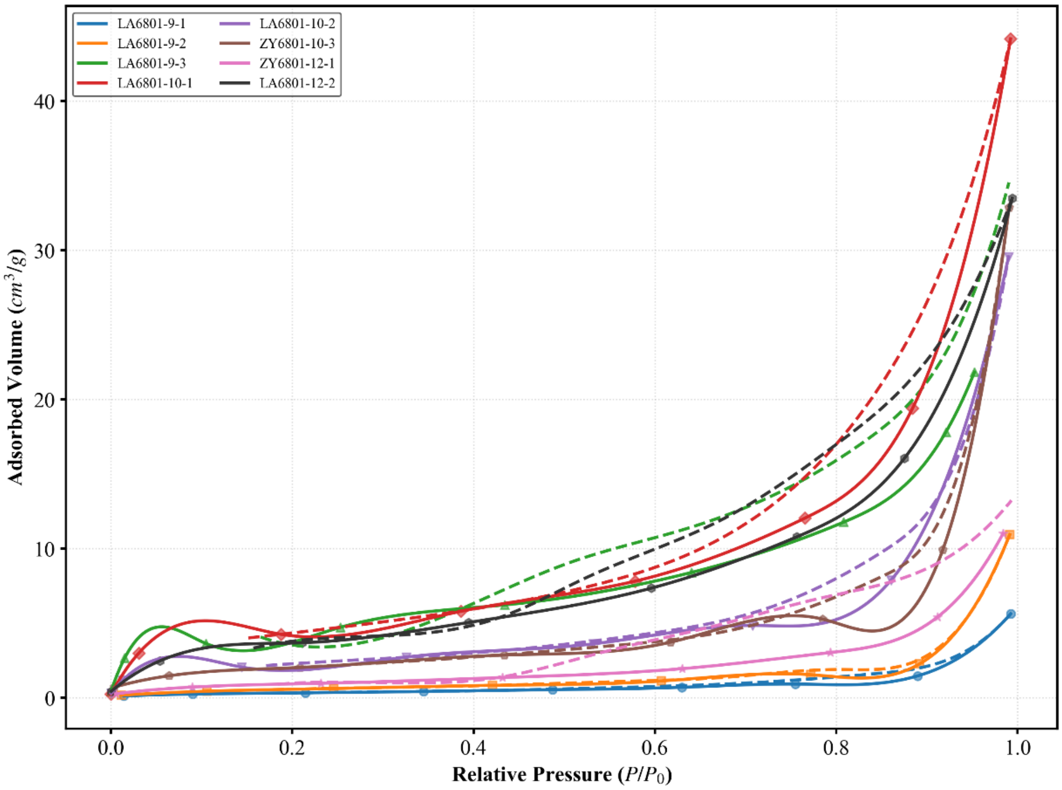

Figure 5. Nitrogen adsorption-desorption isotherm characteristics of coal samples

Fig. 5 shows the characteristics of nitrogen adsorption-desorption isotherms of the samples. The isotherms of this series of samples show typical IV-type characteristics, accompanied by obvious hysteresis loops, indicating that the pore structure is dominated by mesopores. In the low relative pressure region (P / P_0 < 0.2), the adsorption capacity increased slowly, indicating that the contribution of micropores was weak. In the middle and high relative pressure region, the adsorption capacity increased significantly, especially when P / P_0 was close to 1, which reflected the obvious capillary condensation behavior and indicated that there was a certain proportion of mesoporous and stacked pore structure in the system. From the perspective of hysteresis loop morphology, each sample is mainly characterized by H3 type, and there is no obvious adsorption platform in the high pressure zone, and the adsorption capacity continues to increase with P / P_0, indicating that the pore structure mainly comes from irregular particles or slit-like pores formed by the accumulation of flake units, rather than regular cylindrical pores. At the same time, the adsorption-desorption branch separation is relatively gentle, reflecting that the pore structure has certain non-rigid characteristics and the pore size distribution is wide. The differences between different samples are mainly reflected in the degree of mesoporous development and pore volume. LA6801-10-1, LA6801-9-3 and LA6801-12-2 have more obvious hysteresis loops and higher adsorption capacity in the high pressure region, indicating that their mesoporous structures are more developed and their pore connectivity is better. However, the adsorption capacity of LA6801-9-1 and LA6801-9-2 is low and the lag is weak, indicating that the pore structure development is relatively insufficient, which may only contain a small number of intergranular pores or discontinuous mesopores. In general, the series of samples are dominated by mesopores, accompanied by a certain contribution of macropores or stacked pores. The difference in performance is mainly due to the difference in mesoporous development and pore connectivity.

3.3. Fractal Characteristics of Pore Structure

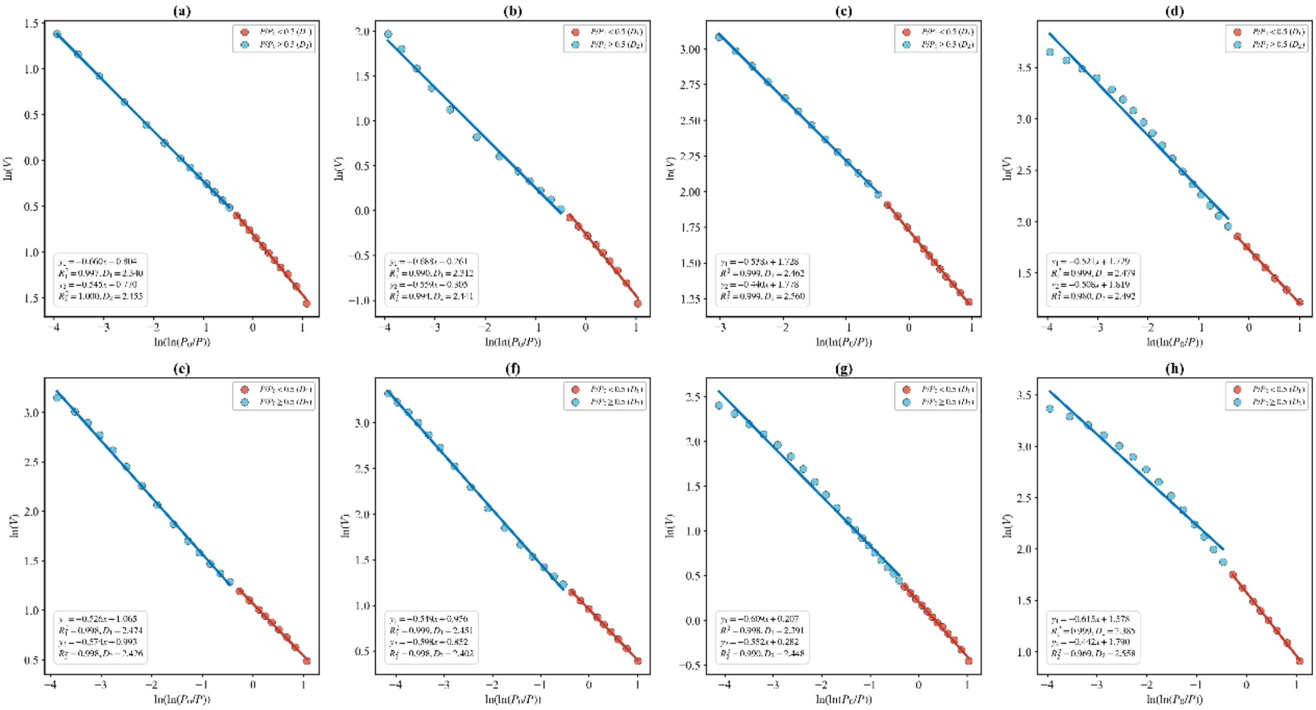

Figure 6. Fractal characteristic curve of coal sample based on FHH model

Figure 6 (a-h) is the fractal characteristic curve of each sample based on liquid nitrogen adsorption data and combined with FHH model. It can be seen from the figure that each sample has obvious piecewise linear characteristics near ln (ln (P_0 / P)) − 0.5, indicating that its pore structure has different fractal characteristics in different relative pressure intervals. By piecewise linear fitting, The corresponding fractal dimensions D_1 and D_2 for the low-pressure region ($P/P_0<0.5$) and the high-pressure region ($P/P_0>0.5$) can be obtained, respectively. Due to the difference in adsorption mechanism, D_1 and D_2 characterize the pore structure characteristics at different scales, respectively. Among them, the low pressure region mainly corresponds to the monolayer adsorption behavior of gas molecules on the pore surface, and D_1 reflects the roughness of the pore surface. The high pressure zone is controlled by multi-layer adsorption and capillary condensation, and D_2 characterizes the spatial complexity and homogeneity of pore structure. From the change of fractal dimension, the D value of each sample is higher, indicating that the surface of the hole has a certain roughness. At the same time, there is a certain difference in D_1 between different samples, indicating that the surface morphology complexity changes. In contrast, the overall D_2 value is slightly lower, and the variation range is relatively small, reflecting that the pore structure has a certain uniformity in spatial distribution. In general, the difference between D_1 and D_2 indicates that the pore structure of the sample shows obvious heterogeneity at different scales. Further analysis shows that the samples with a higher degree of mesoporous structure development usually correspond to a larger D value, indicating that the pore surface is more irregular and rough. The change of D_2 reflects the concentration of pore size distribution. When D_2 is relatively low, the pore structure tends to be simplified and the distribution is relatively concentrated. In general, the pore structure of the sample shows the characteristics of ' surface roughness enhancement-structure distribution tends to be uniform ' during the evolution process, that is, the pore surface complexity increases while the pore size distribution gradually concentrates. The results are in good agreement with the results of nitrogen adsorption-desorption analysis and SEM morphology observation.

4. Conclusion

(1) The results of scanning electron microscopy (SEM) show that the coal samples in the study area are mainly dense matrix, and there are two main pore types of pores and dissolution pores, accompanied by a certain number of micro-fracture structures. The pores are mostly round or oval and the pore wall is flat. The dissolution pores are irregular and the boundary is rough. The two together with the cracks constitute the pore-crack composite structure system, reflecting the obvious heterogeneity characteristics.

(2) The results of low temperature liquid nitrogen adsorption show that the pore size of coal samples is mainly concentrated in the range of 5.775 ~ 17.842 nm, and the whole is dominated by mesopores. BET and BJH specific surface area results have good consistency, indicating that the specific surface area is mainly derived from the mesoporous structure; the pore volume has a significant positive correlation with the specific surface area and adsorption capacity, and the pore volume has a more prominent control effect on the adsorption capacity. The pore size distribution shows multi-peak characteristics, and the pore volume is mainly concentrated in the range of 10-50 nm and > 50 nm, indicating that the coal sample has a typical multi-scale pore structure.

(3) The nitrogen adsorption-desorption isotherm is characterized by type IV, accompanied by H3-type hysteresis loop, indicating that the pore structure is dominated by mesopores, and the pore morphology is dominated by slit-like pores, mainly formed by irregular particles or sheet structure accumulation. The adsorption capacity in the high relative pressure area increased significantly, indicating that a certain number of macroporous or stacked pore structures were

developed in the sample. The differences between different samples are mainly controlled by the degree of mesoporous development and pore connectivity.

(4) The results of FHH fractal analysis show that the pore structure of coal samples has obvious segmentation characteristics in different pressure intervals. The low-pressure distinguishing dimension D_1 mainly reflects the roughness of the pore surface, and the high-pressure distinguishing dimension D_2 characterizes the spatial complexity and homogeneity of the pore structure. The overall performance is that D_1 is higher and D_2 is relatively lower, indicating that the surface roughness of the coal sample is higher, while the pore size distribution tends to be concentrated, and the pore structure shows the characteristics of' surface complexity-structure relative homogenization ' during the evolution process.

(5) Based on the results of SEM, pore size distribution and fractal analysis, the coal samples in the study area formed a multi-scale pore system dominated by mesopores, accompanied by certain macropores and fissures. Among them, mesopores mainly control the gas adsorption capacity, and macropores and fissures provide effective channels for gas migration. The two synergistically determine the adsorption-diffusion-seepage characteristics of coal reservoirs. Therefore, the degree of mesoporous development and pore connectivity are the key factors affecting the reservoir and migration capacity of coalbed methane in the study area.

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