



Article

Study on the Influence of Characteristics of Pore Structure on Adsorption Capacity of Tectonic Coals in Guizhou Province

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Abstract: The occurrence and migration of coalbed methane (CBM) is inseparably associated with the pore structure within the coal seams. Three Permian Longtan Formation tectonic coal samples (QL, XL, XT) from Guizhou Province were studied to determine pore size distribution and characteristics, as well as factors that influence adsorption. Adsorption test results show that all samples generally have "ink bottle"-type pores, with large pore capacity but poor connectivity. Furthermore, the fractal dimension D_f , the tortuosity " τ ", and tortuous fractal dimension D_T of samples were calculated. Among the studied tectonic coals, moisture, ash, tortuosity, and volatile fraction have a positive effect on the maximum adsorption capacity (V_L), whereas intact coals' tortuosity volatile has a negative correlation with the maximum adsorption capacity (V_L).

Keywords: tectonic coal; pore structure; adsorption capacity; fractal dimension



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1. Introduction

Coal is a porous and heterogeneous mixture composed of organic macerates and small amounts of inorganic minerals which exhibit complex microscopic porous structures [1–3]. Coalbed methane is stored in the pores of the matrix, and gas migrates along the channel of those cracks. The micro-pore system of coal plays a key role in the adsorption, desorption, diffusion, and migration of CBM [4–6]. Adsorption capacity of coal is an important indicator to characterize the production of coalbed methane and gas migration, and is also an important parameter to prevent or control gas disasters [7]. Li et al. [8] showed the intact coal seams have fractal characteristics, and adsorption capacity of the coal seam is closely related to the distribution of microscopic pores and fractures. Therefore, it is of great significance to use fractal theory to investigate how the microscopic pore structure of coal impacts the adsorption capacity of CBM.

In recent years, the porosity characteristics of tectonic coals have been investigated by several researchers [9–11]. Moreover, the porosity characteristics of Chinese coals have been studied on the basis of fractal theory by researchers [12,13]. The fractal scale selection, fractal dimension calculation [14], and physical characterization of fractal geometric parameters of coal reservoir pores have been studied extensively, but the conclusions are quite different or completely opposite [15,16], which means that the pore systems of coal reservoirs in different areas varies due to the influence of metamorphism degree and reservoir physical properties.

Tectonic coal has good adsorption capacity because it is crumpled and destroyed by geological tectonics and has more systems of pores and fractures compared to primary coal. Although the fractal theory has been used to describe the adsorption capacity of coal, the influence of pore structure and capillary tortuosity on adsorption capacity has not been considered yet. Tectonic coals from Qinglong, Xiaotun, and Xinglong coal mines, which are representative and sampled from Guizhou province, China, have been selected for this study. The influence of fractal dimension of capillary tortuosity $(D_{\rm T})$ on the adsorption

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characteristics of CBM was analyzed by calculating the fractal $D_{\rm T}$ of coal samples with different structures. The results of this study will provide insight into how to prevent natural gas disasters and improve CBM production in the region. In this study, pore characteristics were studied, and adsorption characteristics were analyzed and combined with fractal dimension theory.

2. Geological Setting and Experimental Methods

The location of Guizhou province was shown in Figure 1, experimental samples were taken from three coal mines located in Guizhou province. The studied Qinglong coal mine is located about 14 km east of Qianxi city, Guizhou Province, whereas Xinglong coal mine is located in the southeast of Xishui county, Guizhou province. The coal seam sampled in this study is located in the fault zone in the 1802 return air passage of Xinglong coal mine, with a burial depth of 258 m. Xiaotun coal mine is located in the south of Dafang County, Guizhou Province, and the coal seam sampled in this study is 100 m deep at the No.6 coal working surface.



Figure 1. Location of study area; Drawing approval number: GS[2019]1652; Producer: Ministry of Natural Resources of China [17]).

The basic information of each coal sample is shown in Table 1.

Table 1. Basic information of tectonic coal samples.

Coal Samples	Rank	Depth(m)	Coalfield
QL Coal	anthracite	264	Qianbei
XL Coal	anthracite	258	Qianbei
XT Coal	anthracite	382	Qianbei

2.1. Sample Overview

Coal samples were prepared with a particle size range of 0.18–0.25 mm (60–80 mesh). The test-air-dry-based moisture (M_{ad}), dry base ash (A_{ad}), and dry ash-free base volatile matter (V_{daf}) were obtained according to Chinese standards (GB/T212-2008). The measured results are shown in Table 2.

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Table	2.	Standard	coal	characteristic.

Coal Samples	Moisture (M _{ad})%	Ash (A _{ad})%	Volatile (V _{daf})%	True Density (g/cm³)	Apparent Density (g/cm³)	Porosity ф/%
QL Coal	3.43	22.96	8.75	1.61	1.54	4.35
XL Coal	3.14	10.41	8.10	1.56	1.49	4.49
XT Coal	1.67	19.99	7.86	1.57	1.51	3.85

2.2. Methane Adsorption and Desorption Test

In each group of CH₄ adsorption/desorption experiments, 0.25–0.3 mm (50–60 mesh) coal samples were selected, and samples were placed in a drying box for 8 h at 368 K to prevent moisture. Initial speed of methane diffusion (ΔP) and gas adsorption constants (a, b) were test with analyzer(WT-1, HCA) at Guizhou University Safety Engineering Lab. The results are listed in Table 3.

Table 3. Diffusion property constants and adsorption constants.

Coal Samples	ΔP (kPa)	a (cm ³ /g)	b (MPa ⁻¹)
QL Coal	15.209	33.5761	0.6338
XL Coal	15.904	38.5189	0.5384
XT Coal	19.779	41.1743	0.6590

2.3. Nitrogen (N_2) Adsorption and Desorption Test

In each group of N_2 adsorption/desorption experiments, 0.25 mm-0.3 mm (50–60 mesh) coal samples were selected to avoid analyzing the microscopic pore characteristics with different sample sizes. Before starting the experiment, the coal samples were placed in a drying box for 8 h at 373 K to prevent excessive moisture and impurities in the samples from damaging the turbo molecular pump. Subsequently, 2 g of sample was weighed and placed in the sample tube and installed on the degassing station of the analyzer (Beishide 3H-2000PS1/2). The sample tube was then installed in the analysis station for the LT- N_2A experiment to determine the adsorption/desorption isotherm of the coal samples. The low-temperature nitrogen adsorption method was used to test the nano-scale pore size of coal in the range of 1.5–100 nm.

3. Experimental Result and Analysis

3.1. Experiment Result and Analysis

The N_2 adsorption and desorption isotherms were acquired in the relative pressure (P/P_0) range between 0.01 and 0.99 [18]. Based on the adsorption branch of isotherms, the specific surface areas analyzed by using BET theory. Then, pore volumes and pore size distribution were analyzed by using the BJH theory reported by Barrett et al. [19]. The parameters of the coal samples measured in the test are shown in Table 4.

Table 4. Pore structural parameters of coal specimens from the LT-N₂A measurement.

Coal Samples	BET Surface Area (m²/g)	Pore Volume (mL/g)	Average Pore Radius (nm)	Most Probable Pore Radius (nm)
QL Coal	0.9762	0.0047	19.26	2.98
XL Coal	1.4713	0.0054	14.68	2.98
XT Coal	4.3790	0.0106	9.68	1.76

The total volume of a pore of tectonic coal has a good positive correlation with total pore-specific surfaces, and the pore-specific surface area is negatively correlated with mean pore size, as shown in Figure 2.

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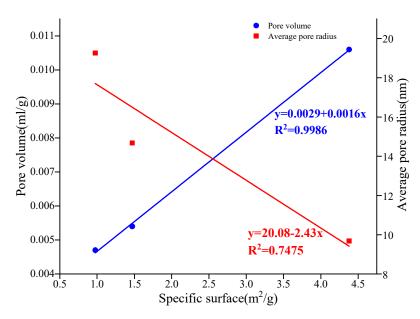


Figure 2. Correlation of specific surface with pore volume and average pore radius.

Figure 3 demonstrates that there is a negative correlation between specific surfaces with moisture and ash for all three samples, whereas volatile matter has a higher effect on specific surfaces than moisture.

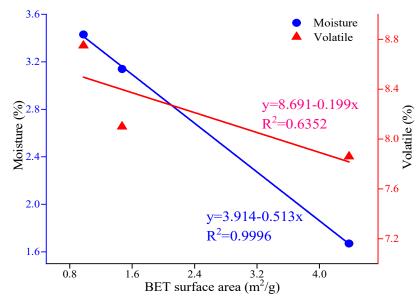


Figure 3. Correlation of specific surface with moisture and volatile.

There is a certain amount of gas adsorption on the surface of the solid under constant temperature which corresponds to a certain adsorbent pressure. The adsorption isotherms are obtained by measuring the amount of adsorption at a given pressure. The interaction between adsorbent and adsorbate is reflected by the morphological changes of adsorption isotherms, and then the properties of surface and characteristics of pore distribution of adsorbents can be analyzed. The N_2 adsorption isotherm curves according to Thommes et al. [20] of three tectonic coal samples are shown in Figure 4.

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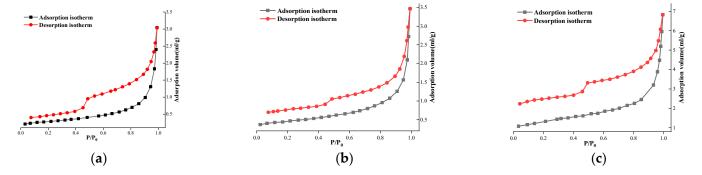


Figure 4. Nitrogen adsorption/desorption of isotherm curves. (a) QL, (b) XL, (c) XT.

It can be seen from Figure 4 that the hysteresis phenomenon was shown in the three tectonic coal samples, and a large hysteresis range of rings belonged to tectonic coal samples QL and XT. Capillary cohesion played a major role in the interaction between N₂ molecules and coal samples as the relative pressure gradually increases. When the relative pressure is about 0.5, more obvious "skip points" appeared from the QL and the XT samples on the desorption isotherm curve, more so than that of XL. Ampoule-shaped pores (also called "ink bottle-shaped" pores) existed in the pore systems of the three structural coals. When P/P_0 was large, a loop was generated due to the different shapes of the interface of gasliquid during the condensation and evaporation of the open hole, so the desorption curve declined slowly before plunging. The liquid in the open hole with the smallest diameter evaporated when P/P_0 fell to the value corresponding to the inflection point. The internal condensed liquid was released rapidly as the pressure continued to decrease, indicating a trend towards a steep decline in the desorption curve. At this time, the liquid in the open pores had vaporized, thus the pores turned to semi-closed status and the desorption curves appeared to overlap the adsorption curves. The quantity of adsorption where P/P_0 was large declined slowly before the desorption curve dropped sharply due to the existence of the "ampoule-shape neck" of the hole. The P/P_0 was much lower than the relative pressure of decondensation required by the radius of the "bottleneck" after the condensed liquid at the "ampoule-shape neck" had evaporated. Consequently, the liquid in the "bottleneck" was released at this time [8].

The shape of the gas-liquid interface during condensation and evaporation was different at the initial stage of desorption because of the existence of open pores. The reduction of adsorption capacity and the decrease of the desorption curve resulted from the corresponding larger pores beginning to evaporate when the relative pressure declined to reach a certain n value. However, it was unable to return to the starting point of the adsorption curve at the end, indicating that part of the gas still remained in the coal during the desorption process and was not discharged due to the existence of various levels of pores.

3.2. Distribution of Pore Size and Pore Volume for Coals

The structure of the pore for the tectonic coal was investigated deeply by the calculation of pore size distribution with the adsorption–desorption curve. From Figure 5, the number of micropores of the same coal was calculated from the two curves of adsorption and desorption separately.

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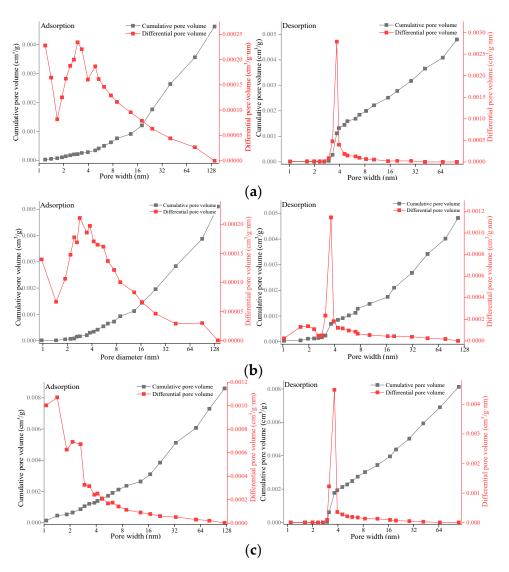


Figure 5. Cumulative pore volume and differential pore volume diagram of coals (adsorption/desorption). (a) QL coal, (b) XL coal, (c) XT coal.

The result shows that there are many micropores calculated from the adsorption curves, whereas that of the desorption curve was hardly calculated. This is because most of the micropores in tectonic coal are "ink bottle" type pores with faint connectivity, and it is difficult to completely release the nitrogen in the pores when desorption occurs.

4. Analysis of Pore Fractal Characteristics and the Main Control Factors on Gas Adsorption Capacity of Tectonic Coals

The gas adsorption method is a common method to calculate fractal dimension. Pfeifer et al. [21] proposed that fractal dimension was calculated by the adsorption experiment of N_2 with the FHH model. Both surface information of coal pores and dimension of surface roughness were obtained accurately by the model [22], and the calculation formula is:

$$ln V = D ln \left[ln \left(\frac{p_0}{p} \right) \right] + c$$
(1)

where, V (mL/g) is the volume of adsorption at balance pressure, P_0 (MPa) is the saturation vapor pressure of gas adsorption, P (MPa) is balance pressure, D (dimensionless) is the fitting slope, and c is constant.

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According to Song et al.'s [12] research results, the linear relationship was presented for fitting slope (D) and fractal dimension (D_f), and fractal dimension was calculated by two formulas as follow:

$$D_f = D + 3$$
$$D_f = 3D + 3$$

The results of the fractal dimension calculated by the two methods are shown in Table 5. Generally, the fractal dimension of the coal pore structure surface is $2\sim3$, where a fractal dimension close to 2 indicates a smooth coal pore surface, and close to 3 indicates a rough pore surface. According to the value range of the fractal dimension ($2 < D_f < 3$), it can be seen that the result calculated by the front formula has a better fitting degree with the actual situation. Therefore, the formula selected in this study to calculate the fractal dimension is:

$$D_f = D + 3 \tag{2}$$

Table 5. Fractal dimension calculation result.

Coal Samples	D	$D_f = D + 3$	$D_f = 3D + 3$
QL Coal	-0.44	2.56	1.65
XL Coal	-0.36	2.64	1.92
XT Coal	-0.32	2.68	2.04

4.1. Fractal Dimension of Capillary Tortuosity

Coal is a typical porous medium, and complex curved channels are formed due to the considerable pores inside [1]. The circuitous degree of gas flow in the channel is expressed by tortuosity. Xu and Yu [23] proposed the calculation formula of tortuosity as follows:

$$\tau = \frac{1}{2} \left[1 + \frac{1}{2} \sqrt{1 - \varphi} + \sqrt{1 - \varphi} \frac{\sqrt{\left(\frac{1}{\sqrt{1 - \varphi}} - 1\right)^2 + \frac{1}{4}}}{1 - \sqrt{1 - \varphi}} \right]$$
(3)

Yu and Li [24] proposed the correlation function to reflect the microstructure characteristics of capillary tortuosity as follows:

$$D_{\rm T} = 1 + \frac{L_{\rm n}\tau}{L_{\rm n}\left(\frac{L_{\rm m}}{2r_{\rm a}}\right)} \tag{4}$$

where r_a (um) is the mean radius of capillary, $r_a = \frac{D_f \cdot r_{min}}{D_f - 1}$, r_{min} (um) is the minimum pore radius, L_m (um) is the characteristic length of the capillary.

The characteristic length of the capillary ($L_{\rm m}$) in two-dimensional space can be expressed as follows by Wu and Yu [25]:

$$L_m = \left[\frac{1 - \varphi}{\varphi} \cdot \frac{\pi D_f r_{\text{max}}^2}{\left(2 - D_f\right)} \right]^{\frac{1}{2}}$$
 (5)

where D_f (dimensionless) is the fractal dimension of pores, and r_{max} (um) is the maximum pore radius.

The above three Formulas (3)–(5) were combined to calculate the fractal dimension (D_T) The results are shown in Table 6.

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	Coal Samples	D_{T}	c
	QL Coal	1.33	11.86
	XL Coal	1.34	12.41
	XT Coal	1.35	14.02

Table 6. Capillary mean tortuosity fractal dimension calculation result.

4.2. Influence of Tortuosity and Fractal Dimension of Tortuosity on Gas Adsorption

The adsorption capacity of coal is closely related to its physical properties. In the Langmuir equation, V_L represents the maximum monolayer adsorption capacity. Therefore, D_T and τ are closely related to V_L . The effects of D_T , τ on V_L are shown in Figure 6.

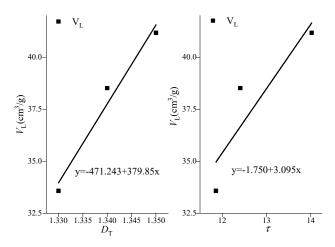


Figure 6. Effect of D_T and τ on gas adsorption capacity.

As the $D_{\rm T}$ increased, the $V_{\rm L}$ rose in Figure 6. The main reason is that the specific surface area increases with the roughness of the pore surface of the coal body increases, and the sites available for methane adsorption increase, and the adsorption capacity of the coal body is strengthened. $V_{\rm L}$ also shows an upward trend with the increasing of τ , indicating that the path of gas migration is complicated; therefore, it is difficult for methane to desorb from reservoirs of tectonic coal.

4.3. Analysis of the Effect of Structural Parameters on Adsorption Capacity for Coals

Figure 7 also indicates the factors influencing adsorption capacity. Figure 7a,b show that the adsorption capacity of the samples has a negative correlation with moisture and ash. In Figure 7c, there is an opposite trend for tectonic and intact coals.

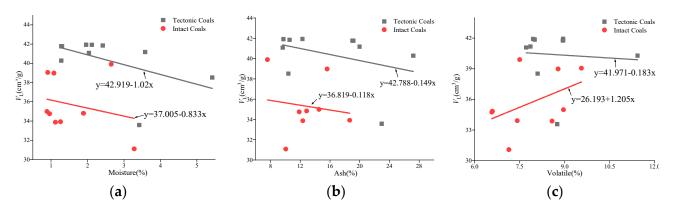


Figure 7. The effect of parameters on gas adsorption. (a) Moisture, (b) Ash, (c) Volatile. Note: The data in Figure 7 are derived from the references and have been reprocessed by the authors [26–28].

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5. Discussion

The coalfields in the Guizhou region have undergone multiple phases of tectonics, resulting in complex formations. This has led to coal deformation and dramatic changes in coal properties, forming tectonic coal [29]. Based on the research work of Wang et al. [30] and Zhang [31], it can be concluded that tectonic coal is formed when the primary structure of intact coal is impacted. Long-term intense extrusion and shearing lead to the pore structure characteristics of tectonic coals being dramatically different from intact coals [32].

Li et al. [8] summarized the reason for the appearance of such curves and concluded that there are some open pores in coal, but there are more pores in the "ink bottle" type, which has a large volume of pores and negative connectivity. As seen in Figure 4, some liquid nitrogen cannot be discharged normally during the process of desorption. Therefore, the three tectonic coal samples have large number of pores which are "ink bottle" in shape. Meanwhile, the pore-size-distribution curves of tectonic coal from N_2 adsorption measurement are exhibited in Figure 4. For the coal specimens, the proportions of mesopores and micropores all decreased with the increasing of the pore diameters.

The D_f , τ , and D_T of the coal samples were calculated. As Figure 6 shows, τ and D_T were found to be linearly and positively correlated with the maximum adsorption capacity. Figure 7a,b indicate that the high content of moisture and ash are not favorable for gas adsorption [1–3], because water molecules are more easily adsorbed on the surface of coal pores than gas. When the moisture content in the coal body is low, water molecules are adsorbed in some microporous pores and there are more adsorption sites for gas adsorption, and the pores of the coal body have a strong adsorption capacity for methane molecules. As the moisture content increases, water molecules gradually fill the microporous pores and provide fewer adsorption sites for methane molecules, and the adsorption capacity of the coal gradually decreases. Similarly, the increase of mineral composition of coal also reduces the adsorption sites for methane molecules [1,33,34], leading to the decrease of adsorption capacity of coals. The results of the sample tests indicate that the maximum adsorption capacity of tectonic coals is higher than that of intact coals. In addition, Cheng and Pan [7] pointed out some characteristics of tectonic coal which are also the key to the outburst. Among them, low strength characteristics make coal more prone to failure; therefore, tectonic coal has low mechanical requirements for outburst. Tectonic coal is easily broken into granular coal, which has a very fast initial gas desorption rate and provides sufficient gas supply during the outburst development stage. Hou et al. [35] found that the structure deterioration coefficient of the outburst prone coal seam was three times as high as that of the non-outburst prone coal seam, and there was a large amount of tectonic coal in outburst prone coal seam. In order to prevent the occurrence of such disasters as outbursts, the reservoir should be analyzed and tested before mining, and the gas content of the coal seam, especially the tectonic coal reservoir, should be reduced through gas extraction and other processes.

6. Conclusions

- a. According to the experiment of liquid nitrogen adsorption, coal samples have different adsorption characteristics when the relative pressure is 0~0.5 and 0.5~1.0. respectively. The "hysteresis" phenomenon was presented in the desorption process of the three kinds of tectonic coal samples, and it is difficult to reach the starting point of adsorption when the tectonic coal is desorbed. Therefore, tectonic coals in Guizhou have a large volume of pores and negative connectivity.
- b. The distribution of pore size was calculated from the curves of liquid nitrogen adsorption and desorption. It is found that the pore size distribution in the three tectonic coals, which mainly contributed to the specific surface area of the pores, were distributed in the range of 1 to 4 nm. Among the three coals, the pores with a diameter of less than 4 nm accounted for the largest proportion in all levels.
- c. The fractal dimensions of porosity and tortuosity in the three tectonic coals were calculated, and it was found that tortuosity and fractal dimension have a linear

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correlation with adsorption capacity. The maximum adsorption capacity (V_L) of the three tectonic coals increased linearly with the increase of D_T and τ , indicating that the adsorption capacity of the tectonic coals was enhanced with the increase of D_T and τ .

d. The potential relationship between the relevant parameters and adsorption capacity of tectonic coals is discussed, and it can be seen from the Figure 6 that the maximum adsorption capacity is negatively correlated with moisture and ash and volatile fraction. In contrast to the positive correlation of volatile fraction of tectonic coals on the adsorption capacity, the intact coals exhibit the opposite trend.

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Informed Consent Statement: All authors have been informed and agreed to publish.

Data Availability Statement: The data presented in this study are openly available in reference number [26–28].

Conflicts of Interest: The authors declare no conflict of interest.

Nomenclature

 D_f fractal dimension

*D*_T fractal dimension of tortuosity

D fitting slope

V volume of adsorption at balance pressure, mL/g

 P_0 saturation vapor pressure of gas adsorption, MPa

P balance pressure, MPa

 $V_{\rm L}$ Langmuir volume, cm³/g

 $L_{\rm m}$ characteristic length of the capillary, um

 r_{min} minimum pore radius, um r_{max} maximum pore radius, um r_{a} mean radius of capillary, um

 φ porosity: %

τ tortuosity

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