



Experimental analysis of pore structure and fractal characteristics of soft and hard coals with same coalification

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Abstract

Accurate and quantitative investigation of the physical structure and fractal geometry of coal has important theoretical and practical significance for coal bed methane (CBM) development and the prevention of dynamic disasters such as coal and gas outbursts. This study investigates the pore structure and fractal characteristics of soft and hard coals using nitrogen and carbon dioxide (N₂/CO₂) adsorption. Coal samples from Pingdingshan Mine in Henan province of China were collected and pulverized to the required size (0.20–0.25 mm). N₂/CO₂ adsorption tests were performed to evaluate the specific surface area (SSA), pore size distribution (PSD), and pore volume (PV) using Brauner-Emmett-Teller (BET), Barrett-Joyner-Halenda (BJH), and Density Functional Theory (DFT). The pore structure was characterized based on the theory of fractal dimensions. The results unveiled that the strength of coal has a significant influence on pore structure and fractal dimensions. There are significant differences in SSA and PV between both coals. The BJH-PV and BET-SSA obtained by N₂-adsorption for soft coal are 0.029–0.032 cm³/g and 3.523–4.783 m²/g. While the values of PV and SSA obtained by CO₂-adsorption are 0.037–0.039 cm³/g and 106.016–111.870 m²/g. Soft coal shows greater SSA and PV than hard coal, which is consistent with the adsorption capacity (V_L). The fractal dimensions of soft and hard coal are respectively different. The Ding coal exhibits larger D_1 and smaller D_2 , and the reverse for the Wu coal seam is observed. The greater the value of D_1 (complexity of pore surface) of soft coal is, the larger the pore surface roughness and gas adsorption capacity is. The results enable us to conclude that the characterization of pores and fractal dimensions of soft and hard coals is different, tending to different adsorption/desorption characteristics. In this regard, the results provide a reference for formulating corresponding coal and gas outburst prevention and control measures.

Keywords Methane adsorption · Soft and hard coal · Pore structure · Fractal characteristics · N₂/CO₂ adsorption

1 Introduction

Coal and gas outburst is a complex dynamic phenomenon associated with coal, rock, and gas in coal mines, which is one of the most serious natural disasters in the process of coal mining (Li et al. 2017; Wang et al. 2017a; Yue et al. 2019a, b; Zhao et al. 2020). Coalbed methane (CBM) is considered a driving force of coal and gas outbursts. It occurs in

multilayer deposition in coal seam; adsorbed on the pore surface of coal, as a part of coal molecular structure, in cracks and large pores, the adsorbed gas controls the occurrence of coal and gas outburst (Zarebska and Ceglarska-Stefańska 2008; Sun et al. 2020; Liu et al. 2021). It is generally believed that coal and gas outbursts result from gas pressure, in-situ stress, and the mechanical properties of coal. Gas pressure and in-situ stress are positively correlated with coal and outburst strength. In contrast, coal's mechanical properties are negatively correlated with the intensity of coal and gas outbursts (Chen 2011; Wang et al. 2013, 2017b; Xue et al. 2014). According to the literature, the influence of stress and gas in the process of coal and gas outburst has been comprehensively studied. However, the relationship between coal's mechanical properties and coal and gas outbursts still needs to be discussed.

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Coal with different destruction degrees formed by tectonic movement is often called tectonic coal, which is considered an essential condition for coal and gas outbursts. Previous research has revealed it as an abundant factor in coal and gas outbursts (Yue et al. 2019b). In addition, coal with different destruction degrees has different pore structures and adsorption properties. Therefore, it is worth investigating the pore structure characteristics and adsorption characteristics of tectonic coals to enhance CBM recovery and prevent coal and gas outbursts (Yue et al. 2019b). The original structure of coal or slightly damaged coal is designated as hard coal, while seriously damaged coal is specified as soft coal in the production practice (Tang et al. 2005). The adsorption and desorption characteristics of coal show significant differences with coal strength. If the research based on coal strength is not differentiated, it will lead to inaccurate prediction and inadequate performance tests (Sun et al. 2020). Moreover, the behavior of gas adsorption, flow, and transport is affected by pore morphology (i.e., coal seam pore system), which is the key to understanding the adsorption/desorption mechanism in the long-term storage process (Clarkson and Bustin 1999b). Generally, pore morphology includes PV, SSA, PSD, pore shape, connectivity, and fractal dimension (Zhang et al. 2013). Based on the complexity of coal pore structure, including the universality of PSD, diversity of pore geometry, and network structure, coal pores are classified into different types that have different effects on gas adsorption and seepage in the coal matrix (Yin et al. 2018). The latest and widely used pore classification standard defined by the International Union of Pure and Applied Chemistry (IUPAC) classifies pores as macropore (> 50 nm), mesopore (2–50 nm), and micropore (< 2 nm), while micropore is divided into ultra-micropore (< 0.7 nm) and super-micropore (0.7–2.0 nm) (Yao et al. 2009; Wu and Wang 2013; Li et al. 2014; Thommes et al. 2015). Coal pores are also divided into seepage pores (> 100 nm) and adsorption pores (< 100 nm). The pores smaller than 100 nm are usually defined as nanopores (Barsotti et al. 2016; Fu et al. 2017; Zhang et al. 2017; Ren et al. 2019). Various methodologies have been developed and used to evaluate the PSD and porosity of the porous materials. These methods can be categorized into liquid invasion method and radiation method (Clarkson et al. 2013; Zhao et al. 2014b). Fluid intrusion methods include high-pressure mercury intrusion (MICP) and low-pressure adsorption (LPA), using N₂ and CO₂. Radiation methods contain optical microscope, scanning electron microscope (SEM), transmission electron microscope (TEM), synchrotron small-angle X-ray scattering (SAXS), small-angle neutron scattering (SANS), nuclear magnetic resonance (NMR), and X-ray micro-computed tomography (micro-CT) (Zhao et al. 2014c).

Some recent studies have revealed the role of pore morphology of coal based on its strength in outburst initiation

and CBM development. For instance, Zhao et al. (2016b) clarified that the rapid desorption capacity of pulverized coal provides a driven force to throw out broken coal that plays an important role in coal and gas outburst initiation. The effect of soft and hard coal pore structure on gas adsorption was studied. The results showed that the PV and SSA of soft coal were comparatively higher than that of hard coal (Zhao et al. 2021b). Though coal is used as the main energy source, pulverized coal is also the form of coal that can be used significantly for production (Shi et al. 2021a, b). Sun et al. (2020) studied the effect of coal strength on coal and gas outburst occurrence. The obtained results revealed that coal strength pointedly influences adsorption, desorption, and outburst occurrence. Wang et al. (2020) analyzed the pore characteristics and gas desorption properties of pulverized tectonic and intact coal. In addition, the effect of gas expansion energy of pulverized tectonic coal on gas outbursts in an underground mine and its influence on the environment were discussed. Wang et al. (2019) studied the pore structure and fractal characteristics of pulverized intact coal and tectonic coal. The results showed that micropore and mesopore volumes and specific surface areas (SSAs) positively correlated with decreasing particle size. According to Yu et al. (2017), the SSA and PV of mesopores increased with the enhancement of tectonic deformation. The study also indicated that the gas desorption and diffusion properties of pulverized tectonic coal exhibited large variations (Guo et al. 2016). Jin et al. (2016) studied the effect of pulverization on coal's pore structure, and it was concluded that the pulverization process significantly increases the SSA and PV. Yue et al. (2019c) investigated the pore structure characteristics and adsorption characteristics of coals with different destruction types. The PV and SSA were increased with destruction type. The results showed that the SSA was mainly provided by micropores, and the PV was provided by macropores. Liu et al. (2021) studied the variation of the pore structure and adsorption capacity of deformed and undeformed coal (soft and hard) with the particle size. The structural characteristics of pores and fractures in soft coal masses were explored and compared with those of hard coal from the same mining area (Wei et al. 2019). Guo et al. (2017) studied the effect of structural characteristics of deformed coal with different degrees of deformation on methane adsorption. Fourier transform infrared (FTIR) spectroscopy and low-temperature N₂ adsorption experiment were applied to analyze the macromolecular structure and pore structure, respectively. The results indicated that coal structures with different deformation mechanisms have different evolution rules. Soft coal shows dynamic phenomenon or outburst risk because of its poor permeability, low strength, rapid gas desorption speed, and high gas content. The pore structure connectivity is influenced by the strength of coal, which may lead to a difference in adsorption/desorption properties (Cheng et al.

2011; Wei et al. 2019) and coal and gas outburst mechanism. So far, available literature has unveiled that the coal destruction degree and pulverization have a significant role in pore structure evolution, adsorption/desorption characteristics, and coal and gas outburst development. Although several analyses have been performed, the influence of coal with different destruction degrees (soft and hard) and the same coalification on pore morphology, adsorption and outburst development are rarely reported. Therefore, the pore structure and fractal characteristics of soft and hard coal from same site will provide a substantial understanding in CBM development and coal and gas outburst mechanism.

This study aims to investigate the pore structure and fractal characteristics of soft and hard coals of same rank. The coal strength is discriminated by means of firmness coefficient f : $f > 0.5$ designates hard coal, while $f < 0.5$ designates deformed or soft coal. The study was undertaken on samples collected from Ding and Wu coal seams of the Pingdingshan No. 6 Coal Mine in Henan province, China. The mining region demonstrates a complex geological structure, and abundant deformed and undeformed coal containing high gas pressure, and high gas content. As a result, it is prone to high frequency outburst disasters, with a total of 12 mines vulnerable to severe coal and gas outbursts (Zhai et al. 2016). Moreover, the tendency of coal and gas outburst accident often occurs in the coal seams where the soft coal is more developed. Based on this status, this paper chooses the coal samples from soft and hard seams with same rank to differentiate the pore morphology and their role on methane adsorption which play a key role in coal and gas outburst initiation. For this purpose, a couple of N_2/CO_2 adsorption tests were performed to evaluate the PSD, SSA, and PV of micropores and mesopores. Furthermore, the correlation of fractal characteristics and V_L was also discussed. The results of this study will provide a reference for CBM development and prevention of dynamic disasters like coal and gas outbursts in the studied coal mine.

2 Material and methods

2.1 Sample preparation and geological overview

Coal samples were collected from two coal seams (Ding 5–6 and Wu 9–10) of Pingdingshan No. 6 Coal Mine, China. The geographical coordinates of the studied mine are $33^\circ 40' 15''$ – $33^\circ 48' 45''$ N and $113^\circ 11' 45''$ – $113^\circ 22' 30''$ E. The minable coal seams in mine include Bing 3, Ding 5–6, Wu 8, Wu 9–10, Ji 15, Ji 16–17, and G 20. Among them, the Ding and Wu seams are taken as the focus of research in this study. Each seam is divided into two layers: Ding 5 and Ding 6 and Wu 9 and Wu 10. The coal-bearing area of the Ding seam is 24.3 km^2 , and the burial depth is 138–1050 m. The

thickness of the coal seam is 0.65 m to 12.65 m. The total coal-bearing area of the Wu seam is larger than that of Ding, i.e., 31.3 km^2 with a burial depth of 150–1100 m. The coal seam thickness is 0.40–5.39 m. In this study, Ding 5–6 and Wu 9–10 are referred to as DN56 and WU910, respectively.

Before performing N_2/CO_2 adsorption tests, the samples were wrapped in an isolated container to avoid the influence of external moisture and brought to the laboratory to determine the physical parameters. The moisture content, ash content, volatile matter, and fixed carbon in coal according to the China National Standard GB/T 212–2008 using a 5E-MAG6600 automatic proximate analyzer (Changsha Kaiyuan Instruments, China) (Yi et al. 2020). The size and weight of the coal sample for proximate analysis were 0.074–0.200 mm and 1 g, respectively.

The samples were pulverized to the required size (0.20–0.25 mm) to conduct N_2/CO_2 adsorption and CH_4 adsorption tests. The samples from the DN seam were labeled as DN56-1 and DN56-2, and those from WU coal seams were named as WU910-1 and WU910-2, respectively. The type and densities of coal were determined according to the Chinese Classification of Coals (GB/T5751–2009) and China National Standard for Determining True Density of Coal and Rocks (GB/T 23,561.2–2009).

2.2 Firmness coefficient

The coal hardness was determined using China National Standard for Determining Coal Hardness Coefficient (GB/T 23,561.12–2010). The coal firmness coefficient f can reflect the outburst danger of coal face to a certain extent. The coal strength coefficient was calculated by adopting the drop-weight method in which the power consumption is directly proportional to the increased surface area of broken material. When the power consumption and average diameter of the material before broken are of a certain value, the firmness coefficient f of material is proportional to the crushing ratio. If the crushing ratio gets larger, the f gets smaller. The firmness coefficient of a coal sample is calculated by the following formula Eq. (1):

$$f_{20-30} = 20n/h \quad (1)$$

where f_{20-30} is the measured firmness coefficient of coal samples with granularity between 20 and 30 mm, n is the number times of drop-weight; h is the height of measured powder by a cylinder. The coal samples (1–3 mm) obtained from Pingdingshan No. 6 Coal mine were used to determine the firmness coefficient. Then, according to article 30 of “Rules of coal and gas outburst prevention”, if the granularities of soft coal samples are less than 10–15 mm, the f can be converted by the f_{1-3} (when $f_{1-3} > 0.25$, $f_{20-30} = 1.57 \times f_{1-3} - 0.14$; when $f_{1-3} \leq 0.25$, $f_{20-30} = f_{1-3}$) (Ze-gong 2010). Coal

with a firmness coefficient f more than 0.5 is called hard coal, and coal with a hardness coefficient less than 0.5 is called soft coal (Yue et al. 2019b; Sun et al. 2020).

2.3 LPGA-N₂/CO₂ adsorption experiments

The mesoporous/micropores morphology was characterized by the physical adsorption method (N₂ and CO₂ as probe molecules) with an automatic Autosorb IQ2 gas adsorption analyzer (Quantachrome instrument, manufactured in the USA), as shown in Fig. 1. Before the test, the coal samples were dried in a vacuum oven at 50 °C for 48 h, and degassed by a molecular pump for 12 h. After degassing and back-filling with nitrogen, the final analysis of the samples was carried out. For N₂ adsorption at 77 K, the measurement range of mesopore is 2–50 nm, and the N₂ adsorption isotherms can give better results at a relative pressure (P/P_0) from 0.001 to 0.995. The saturation pressure (P_0) of the nitrogen at 77 K was measured by the instrument using a P_0 cell. Nevertheless, due to the large size of the N₂ molecule, this method may not be suitable for pores with a width of less than 2 nm (Zhao et al. 2021a). N₂ can form a well-defined monolayer on the surface of the adsorbent because of its eternal quadrupole moment (Vishal et al. 2019). For micropores characterization, CO₂ adsorption isotherms (at 273 K) were obtained. Later those isotherms were analyzed by DFT to obtain PV and SSA of micropores. In the analysis of micropores morphology, CO₂ adsorption at 273 K can overcome the shortcoming of N₂ adsorption to determine micropores smaller than 2 nm. Due to the smaller molecular dynamic diameter and shorter adsorption equilibrium time, it is more accurate to obtain a small pore size in the

microporous area (Clarkson and Bustin 1999b). The low temperature liquid N₂ adsorption and CO₂ adsorption experimental data were attained by ASiQwin software analysis.

2.4 Gas adsorption characteristics

Methane adsorption potential provides a theoretical basis for carbon dioxide–methane displacement in coal seams, which is of great significance for forecasting gas emissions, as well as coal and gas outbursts (Cheng et al. 2017). Gas adsorption is usually described by isotherms under constant temperature, and there is a functional relationship between the amount of adsorbed gas and pressure. The methane adsorption test was performed using the MT/T752–1997 method for determining the methane adsorption capacity in coal (China Department of Coal Industry, 1997). First, the coal samples were placed into coal adsorption tanks. The tank was connected to a vacuum pump for vacuumization (8 h) under 60 °C. The adsorption tank was filled with methane to obtain the experimental equilibrium pressure of 5 MPa at 30 °C. The particle size of 0.20–0.25 mm was used to determine the adsorption quantity. To study the adsorption of methane in coal, the Langmuir equation, given in Eq. (2), was used to calculate the Langmuir parameters (V_L and P_L) (Zou and Rezaee 2016).

$$V_{\text{ad}} = \frac{P}{P + P_L} \quad (2)$$

The following rearranged Langmuir equation was used to determine the Langmuir gas pressure and volume.

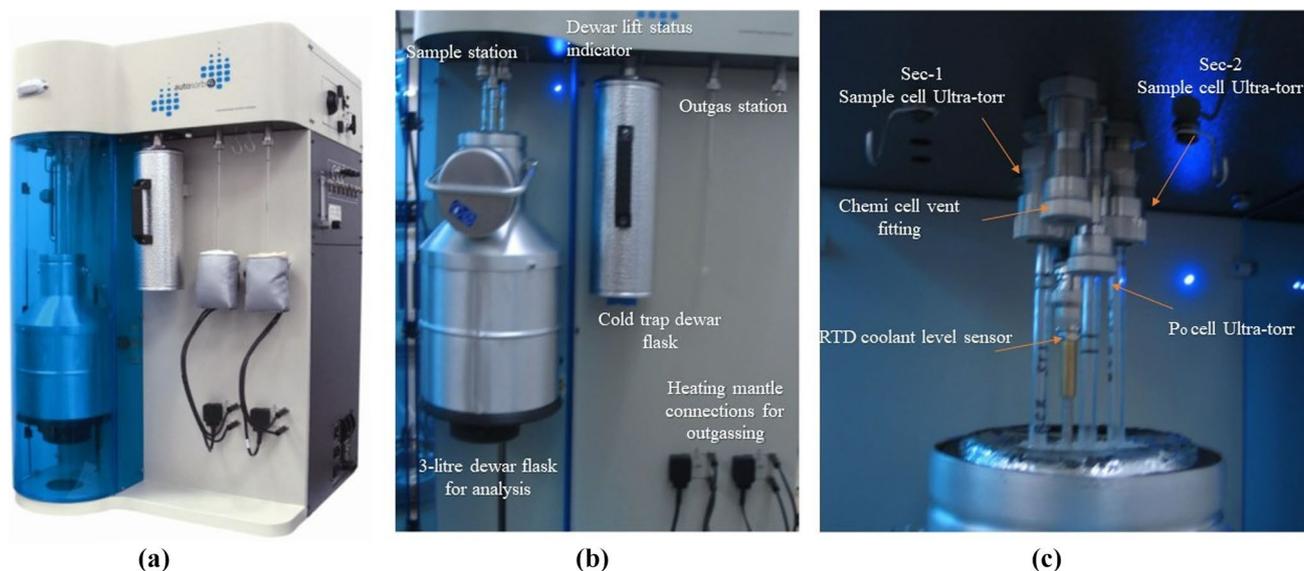


Fig. 1 a Quanta chrome instrument main body; b Front panel; c Analysis Stations

$$\frac{P}{V_{ad}} = \frac{P}{V_L} + \frac{P_L}{V_L} \tag{3}$$

where: P is the reservoir gas pressure, V_{ad} is the adsorbed volume, V_L represents the Langmuir volume per gram of coal particles without moisture and ash, P_L is the adsorption constant describing the adsorption energy intensity,

From Eq. (3), a fitting line between $\frac{P}{V_{ad}}$ and P was drawn, and the obtained slope and intercept were used to determine the adsorption volume and pressure as mentioned in Table 1.

3 Results and discussion

3.1 Physical characteristics of coal

The proximate analysis results, density (true and apparent), and porosity of coal samples are shown in Table 1. The values of physical parameters are consistent. While the f values of coal particles are 0.16, 0.18, 0.71, and 0.67, respectively, which reveals that the Wu seam has high strength than Ding. The value of V_{daf} falls in the range of ($10 < V_{daf} < 37$), categorizing the DN and WU coals as high volatile bituminous coal.

3.2 N₂-adsorption isotherms

The adsorption isotherms obtained from N₂ adsorption (at 77 K) were analyzed by BET, BJH, and DFT methods to obtain the PSDs, PV, and SSA. However, based on the BJH model, the PSD, PV, and SSA of mesopores and a small number of macropores can be calculated, while the DFT model is favorable to determine PSD, PV, and SSA of mesopores and low range of partial micropores. Therefore, the BJH model was better for accurately determining the PSD, PV, and SSA of mesopores. On the other hand, the BET model is widely used to obtain the SSA of mesopores (Sun et al. 2015; Jiang et al. 2019; Wang et al. 2019).

Figure 2 shows the adsorption isotherms of coal samples DN56-1, DN56-2, WU910-1, and WU910-2, respectively.

According to the IUPAC classifications of isotherms, the adsorption isotherms obtained from low-temperature N₂-adsorption experiments are classified as type IV (A) and type II. The shape of this kind of adsorption branch of the hysteresis loop is caused by the adsorption of a monolayer on the surface. Then, multilayer and capillary condensation of gas molecules occur (Thommes et al. 2015; Wei et al. 2019). The significant rise of adsorption isotherms at low pressure (P/P_0) region corresponds to micropores filling, while at the beginning of isotherm, indicating the completion of monolayer adsorption and starting of multilayer adsorption (Thommes et al. 2015; Jin et al. 2016).

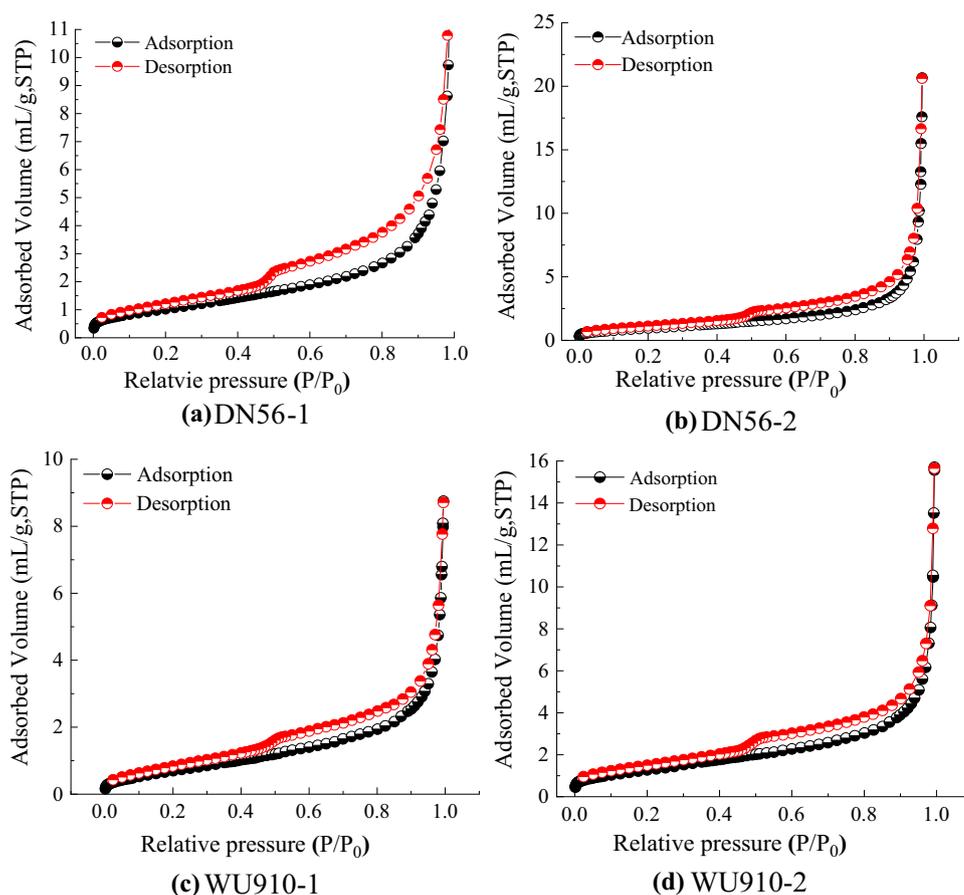
Capillary condensation occurs in mesopores with pore size larger than a certain critical size, which is mainly caused by adsorption metastable and/or pore network effect, and the width of adsorption hysteresis loop increases with the increase of pore size (Nie et al. 2015; Barsotti et al. 2016). With a gradual increase of relative pressure P/P_0 , the rise in the curve happens again, which results in capillary condensation during mesopores adsorption (Barsotti et al. 2016; Jiang et al. 2019). The presence of the hysteresis loop indicates that the evaporation in the pore is significantly different from the condensation in the pore and that capillary condensation occurs under mesopores (Clarkson and Bustin, 1999a, b; Wang et al., 2019).

The desorption branch of the isotherm of coal depends on the influence of the pore network and various forms of pore blockage, thus reflecting the network structure characteristics of the pores (Jiang et al. 2019). From Fig. 2, it can be seen that at high relative pressure $P/P_0=0.98-0.99$, the desorption branch decreases sharply and coincides with the adsorption branch at the equilibrium desorption zone. With the decrease of relative pressure ($P/P_0=0.5$), the adsorption and desorption branches detach from each other, which indicates evaporation and desorption of condensed phase in pores. Finally, the desorption branch drops at the extremely lower pressure phase, showing the detachment of monolayers adsorbed molecules. The shape of pores corresponds to hysteresis loops, and the adsorption/desorption isotherm of coal samples from DN and Wu belongs to type B, indicating the presence of numerous slit-shaped pores. Adsorption/

Table 1 Values of physical parameters

Sample	Proximate analysis				Langmuir constant			True density (t/m ³)	Apparent density (t/m ³)	Porosity (%)	f	
	M_{ad}	A_d	V_{daf}	FC_{ad}	V_L (mL/g)	P_L (1/MPa)	R^2					
Soft coal	DN56-1	1.52	17.85	33.8	53.3	27.02	0.85	0.96	1.42	1.33	6.37	0.16
	DN56-2	1.53	19.97	34.4	51.4	28.8	0.54	0.97	1.45	1.36	6.25	0.18
Hard coal	WU910-1	1.47	22.86	34.6	49.44	26.28	0.77	0.96	1.46	1.37	6.11	0.71
	WU910-2	1.39	29.69	34.8	44.9	26	0.65	0.97	1.48	1.39	5.79	0.67

Fig. 2 Adsorption isotherms obtained by N_2 -adsorption



desorption branches at low pressure regions did not depart from each other, giving evidence of less cylindrical type pores (Nie et al. 2015). The width of adsorption isotherms explains the PSD and pores shape. Adsorption isotherms obtained in this study are obvious, which indicates the micro-mesoporous nature of coal.

3.3 Measurement of pore size distribution, pore volume, and specific surface by N_2 -adsorption

Various studies confirmed that PV and SSA of mesopores by N_2 -Adsorption test could be quantitatively determined by BET and BJH models (Yao et al. 2009). The results of PSDs, PV, and SSA from low temperature N_2 - adsorption experiment were obtained with the help of ASiQwin software from Quantachrome (United States). Figure 3 explains PSD results determined by the BJH and DFT model based on the N_2 -adsorption experiment. PSD results show multimodal mode. According to the BJH result of PSDs, DN56-1 contains micropores and fewer amount of mesopores. The PSD of DN56-1 ranges from 0.9 to 4.0 nm. While DN56-2 exhibits PSD of mesopores and a small number of macropores,

ranging from 2 to 9 nm. Coal samples WU910-1 and WU910-2 are dominated by mesopores. The pore size distributions of WU910-1 and WU910-2 are concentrated around 2–8 nm. More precisely, the coal samples from the DN coal seam are more dominant in quantity by mesopores and micropores, evidenced by pore size distributions, BET SSA, and BJH PV (see Table 2).

N_2 adsorption using DFT (DFT- N_2) primarily shows the lower pore size range of mesopores and low range of partial micropores (<30 nm) (Zhang et al. 2017). Compared to non-local density functional theory (NLDFT), the quenched solid density functional theory (QSDFT) considers the heterogeneity of materials, which is highly effective in the analysis of pore size of micro-mesoporous materials (Neimark et al. 2009; Jin et al. 2016). Based on the QSDFT approach (kernel based on carbon, slit/cylinder pores), the PSD of mesopores and partial micropores is presented in Fig. 3. It can be seen that coal samples DN56-1, DN56-2, and WU910-2 are dominated by mesopores, and a reasonable concentration of micropores (<2 nm) is observed, while WU910-1 exhibits mesoporous concentration. According to the QSDFT model, the PSDs peaks are bimodal. One of the peaks for DN56 and WU910 samples is located between 1–2, and another is

Fig. 3 Pore size distribution of coal determined by BJH and DFT Model

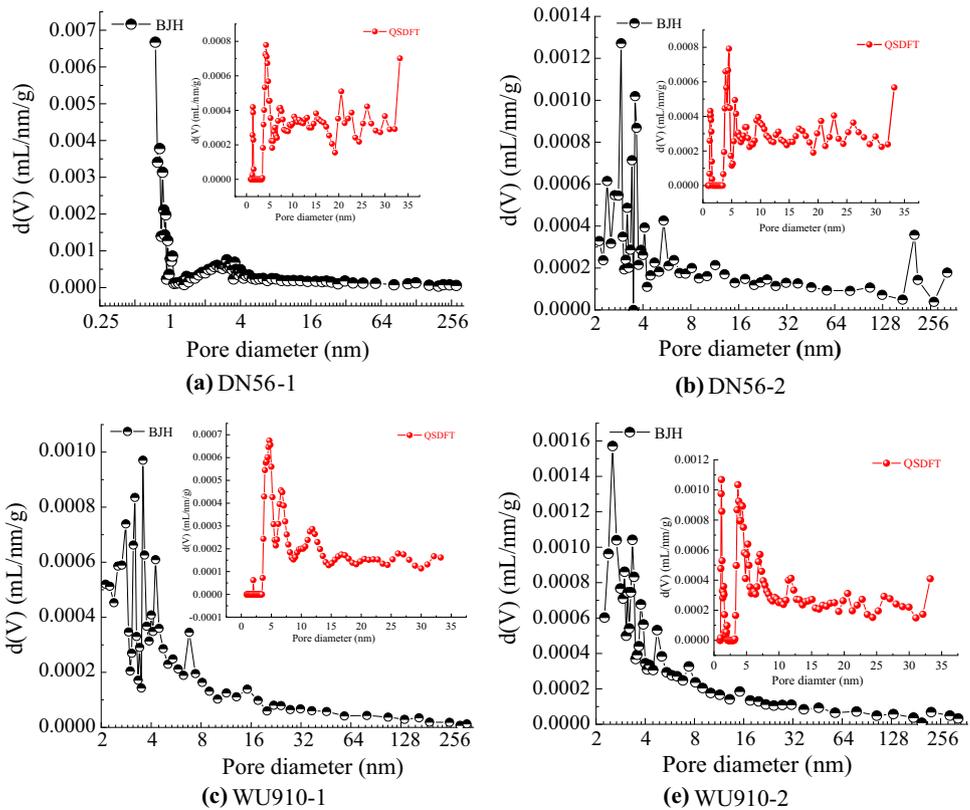


Table 2 PSDs, pore volume and SSA obtained by N₂-adsorption test

Coal sample	BJH pore size (nm)	BJH pore volume (cm ³ /g)	BET SSA (m ² /g)	STD	
				BJH (PV)	BET (SSA)
Soft coal	DN56-1	0.747	3.523	0.002	0.890
	DN56-2	2.922	0.032	4.783	
Hard coal	WU910-1	3.568	0.024	2.941	0.007
	WU910-2	2.522	0.014	2.819	0.086

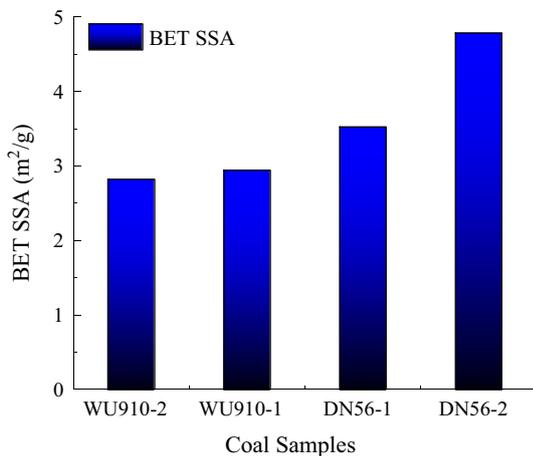


Fig. 4 BET-SSA comparison with samples from Ding5-6 and Wu9-10 coal seams

located in 4–5 nm. While Wu910-1 exhibits a different trend of PSDs peak located between 4–5 and 6–7 nm.

The SSA of the samples was determined by the BET method, and the results are displaced in Fig. 4. For DN seam (soft coal), the SSA increases with the increase of destruction type. The reason is that the SSA is closely related to the microporous structure of coal. The structural responses of destruction types of coal under periodic and anisotropic tectonic stress are different. The contact area of coal samples with low damage degrees can be increased by displacement, rotation, and rearrangement of coal skeleton particles. The internal stress of coal skeleton particles is balanced again. With the increase of the damage degree, the development of multiple groups of fractures gradually disappears in the macropore’s phase, and many nanopores are formed in the micropore’s aspect. Tectonic stress produces strong crushing

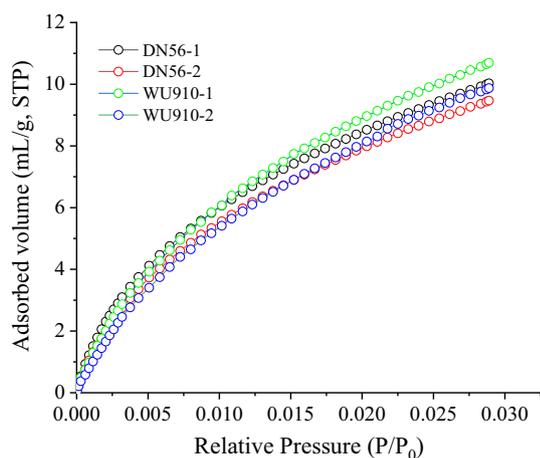


Fig. 5 CO₂ adsorption isotherms

and pulverization, which strongly influences mesopores and especially micropores evolution, which is evident by a study (Yue et al. 2019b). This also indicates that pulverized intact coal does indeed affect the coal's pore structure. An increase in PV and BET-SSA may be related to new pores that appear during the crushing/pulverization process (Hou et al. 2017). The SSAs of coal samples DN56-1 and DN56-2 (soft coals) are greater than WU910-1 and WU910-2 (hard coal). The result implies that DN seam has high adsorption capacity than WU. The PSD, PV, and SSA obtained by BJH and BET models and their corresponding standard deviation (STD) values are depicted in Table 2. Table 2 shows that the PSDs, TVP, and BET-SSA determined by N₂-adsorption ranging from 0.7 to 3.5 nm, 0.014–0.032 cm³/g, and 2.819–4.783 m²/g, respectively.

3.4 Analysis of micropores by CO₂-adsorption

Micropore analysis can be conducted under nitrogen adsorption at 77 K. However, due to the slow diffusion and large molecular diameter of nitrogen at 77 K, this method is not ideal in the quantitative evaluation of micro-porosity, especially in the range of ultra-micropores (pore diameter < 0.7 nm) (Jin et al. 2016). In addition, due to the larger specific interaction of CO₂ than N₂, the volume and SSA of coal micropores measured with these two gases are quite different. The SSAs of coal measured by CO₂ adsorption can give a value of several hundred square meters per gram (Amarasekera et al. 1995; Jin et al. 2016).

Figure 5 shows the CO₂-adsorption isotherms, showing that the adsorption quantity increases gradually with increasing relative pressure. Comparing coal samples DN56-1, DN56-2, WU910-1, and WU910-2, CO₂-adsorption isotherms showed dramatic changes. The adsorption curve of the DN56-1 coal sample shows an increasing trend in

the low-pressure region, and the DN56-1 curve is lagged by WU910-1 in the high-pressure region. The exact change can be seen between DN56-2 and WU910-2 curves.

Micropore is an important index to evaluate the enrichment and adsorption capacity of coalbed methane. However, at present, the super micropores with a diameter less than 2 nm have not apprehensive enough attention. The characteristics of super micropores have a decisive impact on the adsorption performance of methane (Zhao et al. 2016a; Chen et al. 2017). Based on the CO₂ adsorption experiment, PSD, PV, and SSA of DN and WU coal seams are determined. CO₂ Isotherms analyzed by AsiQwin software and DFT model give a favorable approach to obtain PSDs, TPV, and SSA of micropores (super and ultra-micropores). DFT model is also favorable to determine the PSD of the micropores with pore size in the range of 0.35–1.50 nm, as depicted in Fig. 6 (Jiang et al. 2019; Clarkson and Bustin 1999b). PSDs of coal samples are mainly concentrated by ultra-micropores, and a notable amount of super-micropores is also revealed. The obtained PSD peaks have multimodal mode, and all of them are located between 0.30–0.39 and 0.4–0.7 nm. Among the four samples, the PSDs peak in the region of ultra-micropores, especially in the range of 0.30–0.39 nm, the peaks of samples taken from DN seam are higher than WU seam. It can be evident that the DN coal seam is enriched by ultra-micropores, and the adsorption quantity is attributed maximum in the ultra-microporous region. PSDs, TPV, and SSA of DN56-1, DN56-2, WU910-1, and WU910-2 are displayed in Table 3, ranging from 0.36 to 0.58 nm, 0.033–0.039 cm³/g, and 101.01–111.87 m²/g, respectively. It can be noticed that the SSA of micropores of soft coal is greater than hard coal, which is attributed to the high adsorption capacity of soft coal.

3.5 Fractal dimensions of adsorption pores through N₂-adsorption

The Frenkel-Halsey-Hill (FHH) model is the most effective method for calculating the fractal dimensions of coal adsorption pore based on gas adsorption isotherm (Fu et al. 2017; Wang et al. 2018; Hazra et al. 2018; Jiang et al. 2019). Therefore, the fractal features of coal samples were determined by the FHH model, expressed in Eq. (4).

$$\ln\left(\frac{V}{V_0}\right) = A + (D_n - 3) \ln\left(\frac{P_0}{P}\right) \quad (4)$$

where: V represent the adsorption volume, V_0 monolayer absorption volume, A is a constant value that depends on the fractal dimension (D).

Fractal dimension is widely used to describe the anisotropy and complexity of pore structure. For coal, fractal features represent the complexity of pores and the roughness

Fig. 6 Pore size distributions by CO₂-adsorption

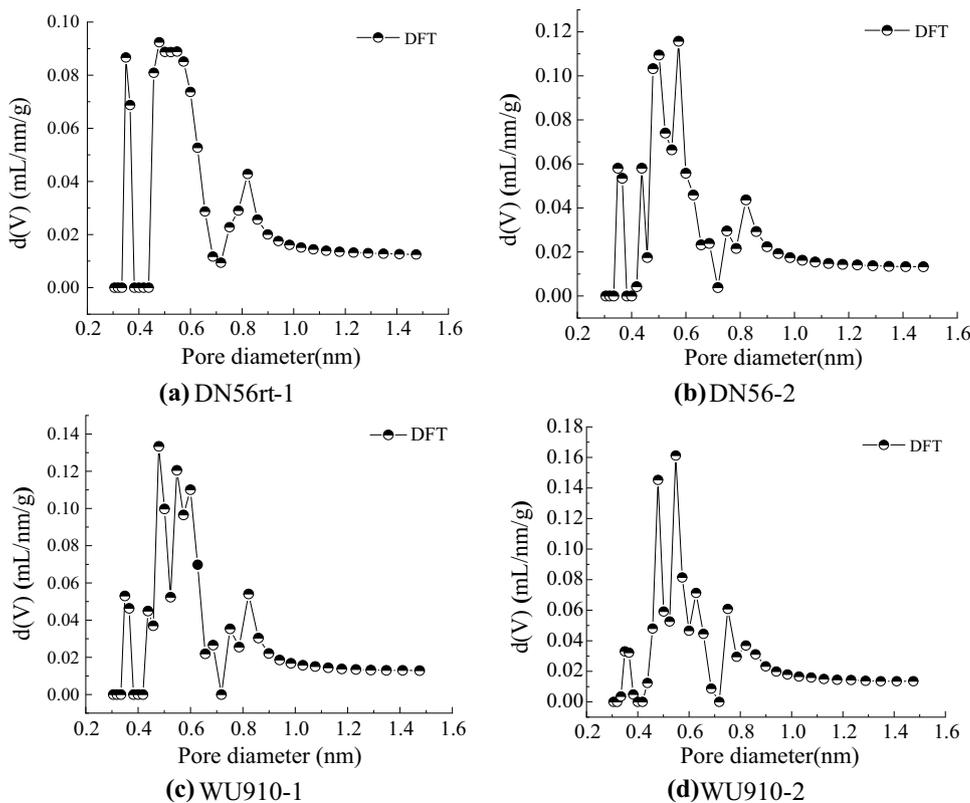


Table 3 Pore size distribution, pore volume, and SSA obtained by CO₂-adsorption test

Coal sample	DFT micropores size (nm)	DFT micropore volume (cm ³ /g)	DFT micropore SSA (m ² /g)
Soft coal	DN56-1	0.548	106.016
	DN56-2	0.573	111.872
Hard coal	WU910-1	0.479	103.822
	WU910-2	0.365	101.181

of the pore surface, which can be determined by N₂ (77 K) adsorption data. The FHH model has been verified to be the most efficacious method to obtain fractal dimensions. Many studies showed that the dividing point of the FHH fractal curve “(P₀/P) = -0.5” (corresponding aperture about 5 nm) divides the curve into two stages: D₁ and D₂. Generally, D₁ and D₂ are used to characterize the complexity of pore surface and pore structure, respectively (Wang et al. 2018).

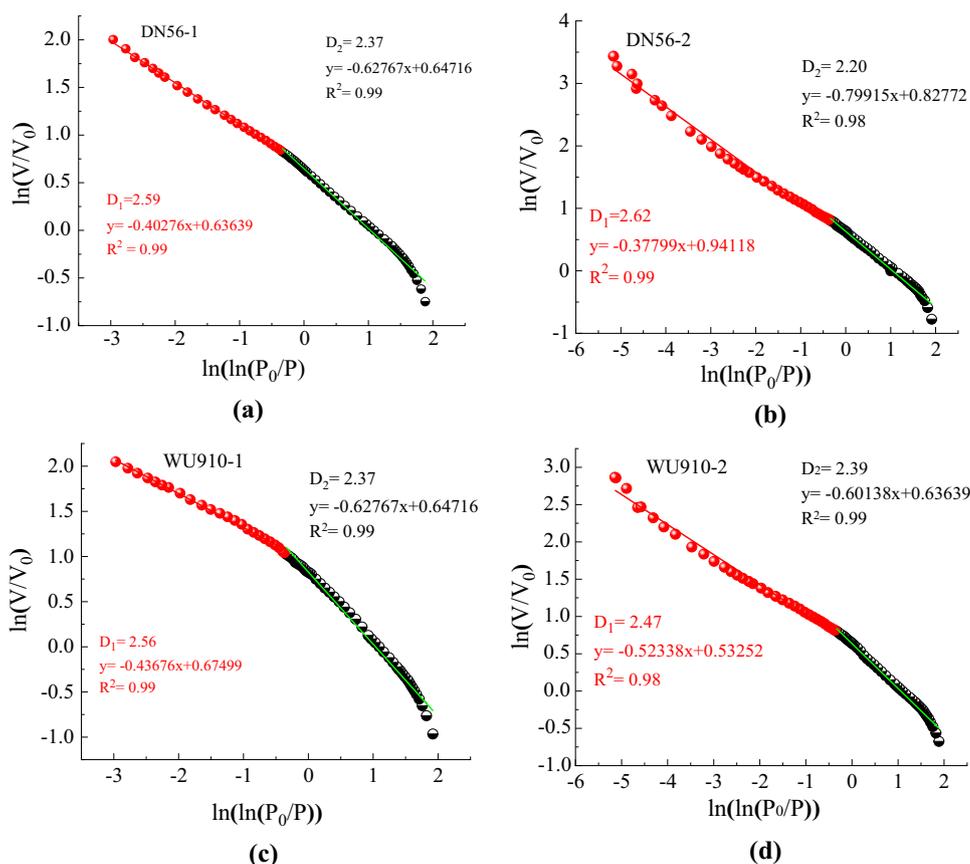
FHH fractal results, shown in Fig. 7, indicate an obvious segmentation, and both intervals have a different outcome, which resembles previous works. According to Eq. (4) the straight line between ln V versus ln(P₀/P) was plotted and the slope S was obtained (Hazra et al. 2018). Later the fractal dimensions D₁ (P₀/P < 0.5) and D₂ (P₀/P > 0.5) were estimated using Eq. (5), and all values are lying between 2 and

3, which yield to the fractal dimensions range (Yao et al. 2008; Fu et al. 2017).

$$D_n = 3 + S \tag{5}$$

Previous studies illustrate that D₁ and D₂ represent surface fractal dimensions and pore structure complexity, respectively (Yao et al. 2008; Fu et al. 2017; Wang et al. 2018). The higher the value of D₁, the rougher the surface, corresponding to more adsorption sites and higher gas adsorption capacity in coal. On the contrary, the higher the D₂, the more complex the pore structure of coal, indicating more vigorous capillary condensation and lower methane adsorption capacity of gas (Yao et al. 2008; Ren et al. 2019). The values of D₁ and D₂ for samples are displaced in Fig. 7 and Table 4. It can be noticed that the value of D₁ for DN coal increased from 2.59 to 2.62 which while D₁ for WU coal is reduced from 2.56 to 2.42. Considering the above observation, it can be concluded that the D₁ value is verily different in soft and hard coal. D_n seam showed a greater value of D₁ confirms the high adsorption capacity. On the contrary, the value D₂ of coal Ding reduced from 2.37 to 2.20 between the samples, while WU evidenced the same value of 2.39. According to the meaning of D₂, the smaller its value, the smaller the pore structure anisotropy leads to

Fig. 7 Fractal characterization determined by N_2 -adsorption experiment



greater gas adsorption capacities are (Wang et al. 2019). It is confirmed from the above results that, DN coal seam has a high adsorption capacity with a lower D_2 value. It is confirmed from the above analysis that the deterioration in the roughness of coal surface and reduction in anisotropy of the pore structure is mainly occurred by geological tectonic moments (Wang et al. 2019).

Correlation plots of PV and SSA of mesopores and micropores versus fractal dimensions are depicted in Fig. 8. It can be seen in Figs. 8a and b that the BJH-PV and BET-SSA of coals exhibit a positive correlation with D_1 . The correlation coefficient (R^2) of BET-SSA and BJH-PV are 0.65 and 0.99, respectively. The results indicate that BJH-PV has a good fitting relation than BET-SSA. However, there

is negative relation with D_2 , indicating that BET-SSA and BJH-PV decreased gradually with the increase of D_2 value, as confirmed by a previous study (Wang et al. 2019). On the contrary, the DFT-SSA of micropores also showed a positive linear correlation with D_1 and a negative relation with D_2 (Fig. 8c). On the other hand, DFT-PV has a polynomial correlation with D_1 and revealed a U-shaped trend with D_2 (see Fig. 8d). The obtained curves revealed that the rougher the pore surface of the particles is, the larger the PV and SSA are. The rough pore surface provides more adsorption sites for gas adsorption, resulting in stronger gas adsorption capacities (Yao et al. 2008). It indicates that although the samples are taken from the same coal seam, their different strengths show different fractal characteristics and adsorption properties. At the same time, D_1 of soft coal is more significant than that of hard coal, which is one of the reasons that the former's adsorption capacity (V_L) is greater than that of the latter (Wang et al. 2019).

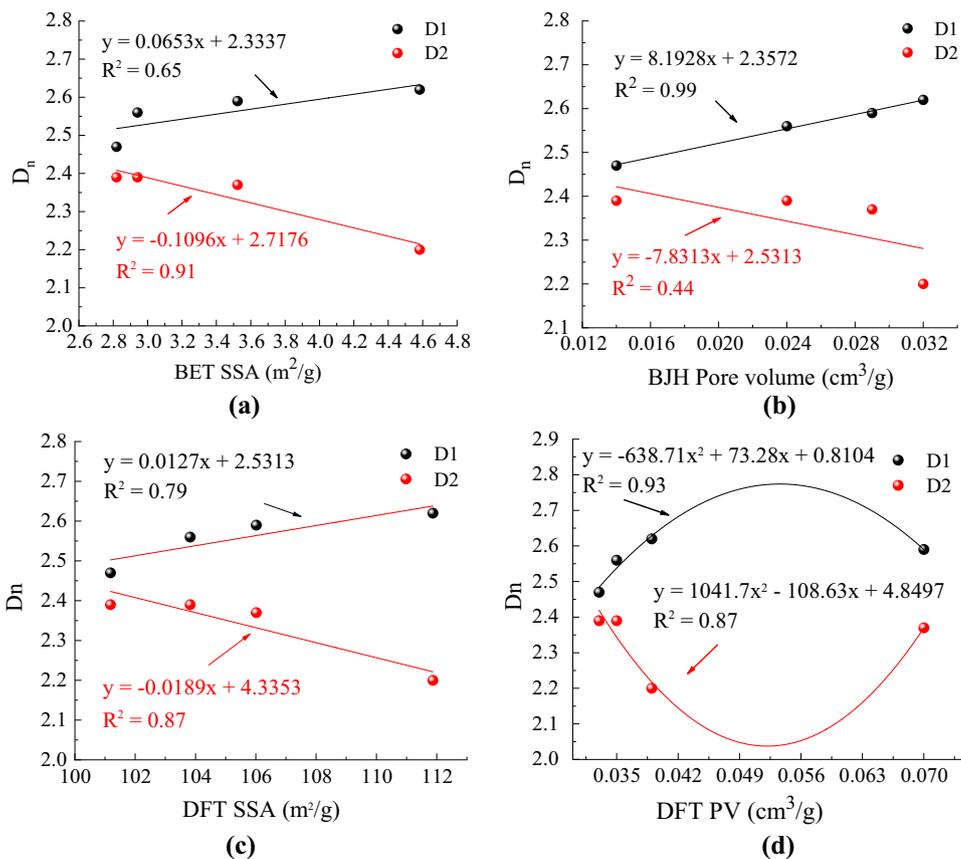
Table 4 Pore fractal dimensions of coal samples

Coal sample		Fractal dimensions			
		D_1	R^2	D_2	R^2
Soft coal	DN56-1	2.59	0.99	2.37	0.99
	DN56-2	2.62	0.99	2.20	0.98
Hard coal	WU910-1	2.56	0.99	2.39	0.98
	WU910-2	2.47	0.98	2.39	0.99

3.6 Influence of pore SSA and pore volume on gas adorability

Gas adsorption in coal occurs in three steps: (1) gas molecules diffuse through the surface layer of coal, (2) gas molecules travel through internal pores, and (3) gas molecules

Fig. 8 **a** Correlation of fractal dimensions with BET-SSA; **b** Correlation of fractal dimensions with BJH pore volume; **c** Correlation of fractal dimensions with DFT-SSA; and **d** Correlation of fractal dimensions with DFT pore volume



are assembled by pore walls (Zhao et al. 2014a). Therefore, the pore structure of the coal matrix has a significant influence on the adsorption of gas (Lu and Cheng 2015). In the adsorption state, CBM is retained on the inner surface of coal micropores and mesopores, so the SSA and PV of micropores and mesopores are of great significance to the adsorption capacity and gas accumulation. According to the basic physical adsorption theory on a solid surface, the

adsorption capability of the object is in direct proportion to the SSA (Laxminarayana and Crosdale 1999; Chen et al. 2017).

The BET surface area result of coals determined by the N_2 adsorption experiment is presented in Fig. 4 and Table 2. The BET-SSA decrease between four coal samples in the following order: DN56-2 > DN56-1 > WU910-1, > WU910-2, showing consistency with the V_L of samples evaluated.

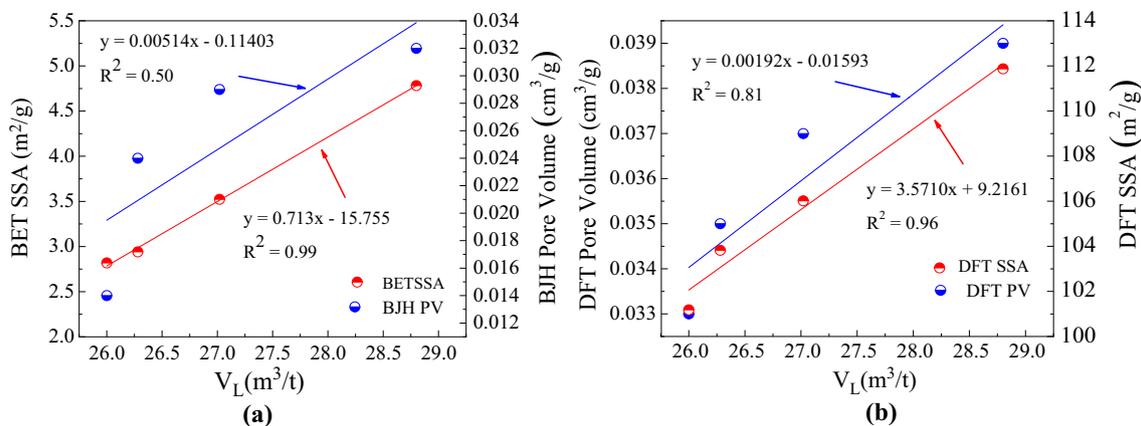


Fig. 9 **a** Correlation of V_L with BET-SSA and BJH-PV; **b** Correlation of V_L with DFT-SSA and DFT-PV

The BET-SSA of the DN56-2 has an extremely high surface area compared with the other samples, which implies to higher adsorption capacity, based on the results of a previous study (Qi et al. 2017).

Figure 9a shows that BJH-PV and BET-SSA are linearly correlated to adsorption volume. The increase of SSA and PV of micropores and mesopores has an increasing influence on gas adsorption in the coal body. Therefore, SSA and PV of micropores and mesopores contribute to the gas adsorption capacity of the coal. By comparing BJH-PV and BET-SSA, BET-SSA has a good linear fitting with the adsorption volume, which indicates its influence on methane adsorption in coal is greater than BJH-PV. The BET-SSA of soft coal is higher than hard coal lead to high V_L mentioned in Table 1. This may be due to the more developed pore structure of soft coal, resulting in greater gas adsorption capacity. At the same time, under the influence of tectonic action, the fragmentation degree of soft coal increases, which may lead to the increase of adsorption sites. Therefore, the gas adsorption capacity of soft coal is greater than that of hard coal. The adsorption capacity of both coals increases with the increase in SSA of the pores, i.e., the increase of SSA promotes gas adsorption. Simultaneously, the influence of micropores on V_L is greater than that of transition and mesopores, as evident by a study (Meng et al. 2015). SSA and PV of micropores show the same trend with adsorption volume. Figure 9b depicts the relationship of SSA and PV of micropores obtained by the CO₂ adsorption experiment. The linear increase of V_L with the micropore surface area and micropore volume among the coal samples indicates that the micropores in the coal are the main factor affecting V_L . The increase of micropore SSA and micropore volume increased the maximum adsorption capacity of soft coal, which is actually denoted by V_L in Table 1. The results presented in Fig. 9a and b conclude that the adsorption capacity of coal seams is greatly influenced by specific surface areas of micro and mesopores. The influence is followed by DFT-PV and BJH-PV. It is also evident from the results that the different coal strengths evaluated different pore structure and fractal characteristics that ensure the difference in V_L of Ding and Wu coal seams. Therefore, the obtained results are useful to be considered while investigating CBM development and the mechanism and sensitivity of coal and gas outburst in Pingdingshan No.6 coal mine.

4 Conclusions

This study evaluated the pore structure and fractal characteristics of soft and hard coals with the same coalification. In order to clearly understand the influence of coal strength

on pore structure and fractal characteristics and their role on adsorption capacity and outburst development, the LPGA-N₂/CO₂-adsorption tests were performed, and the following conclusions were drawn.

- (1) N₂-adsorption isotherms at low temperature experiments are classified as type IV (A) and type II. The shape of hysteresis loops indicates numerous slit-shaped and a small number of cylindrical pores. The BJH-PV and BET-SSA of mesopores are significantly increased with the decrease of coal hardness. This increase in PV and SSA in soft coal may be related to new pores that appear during the crushing/pulverization process. The SSAs of coal samples DN56-1 and DN56-2 (soft coals) are greater than WU910-1 and WU910-2 (hard coal). The result implies that the DN seam has high adsorption capacity than WU, indicating consistency with the V_L .
- (2) CO₂ isotherms were analyzed by the DFT model to obtain PSDs, PV, and SSA of micropores (super and ultra-micropores), and it is concluded that the PSDs peak in the region of ultra-micropores for DN seam is higher than WU seam, especially in the range of 0.30–0.39 nm. It can be evident that the DN coal seam is enriched by ultra-micropores, and the adsorption quantity is attributed maximum in the ultra-microporous region. The SSA of micropores of soft coal is greater than hard coal, which is attributed to the high adsorption capacity of soft coal.
- (3) The fractal dimensions values (D_1 and D_2) vary with coal strength. The D_1 of soft coal increased from 2.59 to 2.62, while that for hard coal decreased from 2.56 to 2.42. It leads to the conclusion that D_1 is different in both coals, and soft coal shows greater value, which confirms the high adsorption capacity. The value of D_2 between soft coal samples decreased from 2.37 to 2.20, while that for hard coal remained the same, i.e., 2.39. Therefore, the smaller the D_2 value is, the smaller the pore structure anisotropy leads to greater gas adsorption capacities.
- (4) Detailed analysis of pore structure and fractal characteristics performed based on coal strength found that low strength coal (soft coal) dramatically influences the evolution of pore structure and adsorption capacity, which can be traced to the strong sensitivity of coal gas outbursts in the particular coal seam. Therefore, the current study can be extended to evaluate the effect of geological structure on coal's pore structure and fractal dimensions. Additionally, the relationship between coal seam thickness and coal strength is also worth investigating.

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Declarations

Conflict of interest The authors declare no potential conflicts of interest.

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