



Study on Adsorption Characteristics of Coal Under Different Crushing Degrees-Taking Chengzhuang No.3 Coal As an Example

Dongshan Li*

School of Resources and Environment, Henan Polytechnic University, Jiaozuo 454003, China

*Corresponding Author: Dongshan Li

ABSTRACT

This study examines the adsorption characteristics of coal under varying degrees of fragmentation using Chengzhuang No.3 coal as a case study. The research explores how fragmentation impacts the adsorption capacity of gases like methane (CH₄) and carbon dioxide (CO₂), which is critical for optimizing gas extraction and predicting coal and gas outburst risks. Coalbed methane (CBM) is a significant energy resource but poses safety risks in mines due to potential explosions. Previous studies have focused on the effects of coal metamorphism on adsorption capacity, often neglecting the impact of fragmentation on pore structure. This study collected 500 grams of coal samples from Chengzhuang No.3 seam, crushing them into 5-60 mesh and 60-80 mesh sizes for analysis. Experimental methods included scanning electron microscopy (SEM) to analyze pore characteristics, low-temperature nitrogen adsorption to determine pore size and type, and high-pressure CH₄ and CO₂ adsorption experiments to measure adsorption capacity. SEM revealed metamorphic pores, dissolution pores, and flat slit pores in the coal. Low-temperature nitrogen adsorption showed a significant hysteresis loop, indicating complex pore structures. High-pressure adsorption experiments demonstrated that increased fragmentation enhances adsorption capacity, with finer coal samples showing higher adsorption rates. CO₂ exhibited greater adsorption capacity than CH₄ due to its stronger interaction with coal. The study concludes that fragmentation increases adsorption capacity by expanding pore volume and specific surface area. This finding is essential for improving gas extraction processes and mitigating coal and gas outburst risks.

KEYWORDS

Coal fragmentation; Adsorption capacity; Methane; Carbon dioxide; Coal bed methane.

1. INTRODUCTION

Fossil fuels, mainly coal, have a significant impact on global warming. Deep coal seam is rich in a large number of coal bed gas, also known as gas (the main component is CH₄), is of great significance to the energy structure transformation of our country, the fifth national coal bed gas evaluation results show that 2000m to shallow coal bed gas resources of 30.05 trillion, ranking third in the world. From the perspective of safety field, gas is a harmful factor in coal mine production. It not only pollutes the air, but also when the gas content in the air is 5% ~ 16%, it will cause an explosion in case of fire, resulting in accidents. From the perspective of geological energy, coal bed methane can be regarded as a clean energy source to replace coal.

The exploitation of coalbed methane often needs to go through the four processes of "depression-desorption-diffusion-percolation", and the desorption and diffusion of methane are the most important in the exploitation. In the process of methane extraction, coal fragmentation is often accompanied.



The adsorption-desorption process of coal seam gas (the main component is methane) is the dynamic basis of coal mine gas disaster. As a porous medium, the adsorption capacity of coal is regulated by many factors such as pore structure, specific surface area and surface chemical properties. The results show that the increase of coal crushing degree will significantly change the pore volume distribution: with the aggravation of the failure type, the volume of micropores, mesopores and macropores in the coal body increases, thus expanding the physical space for gas adsorption. This phenomenon is particularly prominent in deep high-stress mines, where the high adsorption capacity of broken coal may lead to abnormal gas emission and increase the risk of explosion. Therefore, exploring the adsorption characteristics of coal under different crushing degrees has important engineering value for optimizing the gas extraction process and predicting the risk of coal and gas outburst.

Existing studies mostly focus on the influence of coal metamorphism degree on adsorption capacity. For example, Lu Shuoqing et al.[1] found through Langmuir equation and thermodynamic model that there were significant differences in the change value of surface free energy and equivalent adsorption heat of coals with different metasormalization degrees: the change value of surface free energy of coals was the largest, while the initial equivalent adsorption heat of anthracite was the highest. However, such studies usually assume that the coal structure is complete, and do not consider the pore reconstruction effect caused by coal fragmentation in actual mining. Recent studies based on the adsorption potential theory show that the increase of coal failure types will increase the adsorption capacity of methane, and the adsorption potential energy decays nonlinearly with the expansion of adsorption space[2]. However, there is still a lack of quantitative models for the correlation mechanism between the degree of fragmentation and the thermodynamic parameters of adsorption (such as isoadsorption heat and surface free energy), especially the dynamic adsorption law under the coupling of temperature and pressure.

Many researchers have done a lot of research on CBM since 1983. On the basis of predecessors, Huo Xiaoyan et al.[3] systematically summarized the latest progress of coal bed methane reservoir geology research in recent years, comprehensively combed the latest progress of coal bed methane reservoir geology research, and emphasized the importance of coal bed methane reservoir geology research for coal bed methane exploration and development. Zheng Yongwang et al.[4] found in their study of Jinzhong area of Qinshui Basin that the adsorption capacity of methane from coal seam increased with the increase of equilibrium pressure, and influenced by the characteristics of coal seam pore fracture development and CO₂ characteristics, the adsorption capacity of CO₂ from coal seam increased continuously at first, then decreased sharply near the critical pressure and then increased sharply. In addition, after adsorbed in coal seam, CO₂ shows obvious characteristics of adsorption and desorption lag, and a large proportion of CO₂ is retained in coal seam in the form of adsorption and residual sequestration, which can not be desorbed, becoming a favorable condition for large-scale CO₂ sequestration and CH₄ replacement. In order to reflect the influence of temperature and pressure in the process of gas adsorption, Wu Shouya et al.[5] constructed a non-uniform distribution model of gas density near the wall, and then obtained the calculation equations of adsorption thickness and adsorption layer number, deduced the calculation model of adsorbed gas density, and then established the calculation model of adsorption capacity. Through molecular dynamics verification, the adsorption gas density is affected by the gas-solid interaction strength, temperature and pressure: the stronger the gas adsorption capacity of coal, the greater the gas-solid interaction strength, the greater the gas density near the wall, the greater the adsorption gas density, and the greater the number of adsorption layers. The adsorption region is divided into strong adsorption region and weak adsorption region according to one molecular unit distance of the wall, and the main controlling factor of adsorption behavior in the strong adsorption region is external pressure. In addition, it is also considered that the gas adsorption and desorption within 0.2nm of the wall is only affected by the gas-solid interaction by taking 0.2nm as the limit.

Many experimental methods have been developed since 1983. The common ones include low temperature nitrogen adsorption experiment, rock fracture see-through experiment, high and low

pressure gas adsorption experiment, etc.[6] However, limited by the experimental conditions, a large number of numerical simulation methods have also been developed, which can be divided into finite element simulation method and molecular simulation method according to the research scale. In general, gas seepage and displacement are often realized by finite element method. The commonly used software platforms include Comsol Multiphysics and Ansys Fluent platform, and the research scale is generally from micron to millimeter.[7] At the nanoscale, coalbed methane adsorption and desorption processes occur. Studies at this scale are often carried out with a range of molecular dynamics software, including Materials Studio (MS), Large-scale Atomic/Molecular Massively Parallel Simulator (Lammps), Gromacs and so on, can simulate and describe the adsorption and desorption process of gas between coal seams.

Before the molecular simulation method is used to calculate the adsorption of gas molecules between coal seams, it is often necessary to describe and model coal macromolecules, which is the first problem in front of all researchers. In order to solve this problem, many researchers often need to collect coal samples first, then calculate the photon energy spectrum by X-Ray photoelectron spectroscopy (XPS), and then determine the material composition by comparing the PDF of Jade software[8]. The specific gravity was determined by coal industry analysis and elemental analysis, and the functional group structure peak and proportion of each group of organic matter were clear by FTIR spectroscopy[9]. After that, the chemical shift distribution of various carbon structures and the relative percentage of coal ^{13}C NMR spectrum were determined by ^{13}C NMR technology. After that, the optimal proportion model of coal molecules was modeled and verified by reasonable calculation [10]. Up to now, many classical models have been developed to describe various possible situations of coal macromolecules, including Fuchs model, Wisser model, Shinn model, etc. In addition, since coal macromolecules are composed of a series of aromatic compounds, some researchers have directly used graphene to simulate coal macromolecules. The advantage of this is that it is easier to study the influence of a certain characteristic of coal on gas adsorption.

In essence, the adsorption characteristics of broken coal mass are the comprehensive embodiment of pore structure evolution and surface energy change. On the one hand, the increase of fracture degree will enlarge the pore volume, but excessive fracture may lead to the closure of some pores or the decrease of connectivity[11]. On the other hand, changes in the surface free energy of coal may alter its interaction strength with methane molecules. In this study, the method of experimental analysis was used to explore the adsorption characteristics of coal under different fragmentation degrees, and the relationship between adsorption strength and coal fragmentation degree was analyzed. This study provides guidance for the engineering practice of gas mining, and also has reference significance for the efficient development of coalbed methane and the optimization of the adsorption behavior of porous media in CO_2 geological storage.

2. EXPERIMENTAL METHODS AND SAMPLE COLLECTION

2.1. Sample collection and mining area selection

No. 3 coal seam in Chengzhuang Mine field has good conditions of "generation", "storage" and "cap" accumulation. In the process of coalization, a large number of organic microcomponents and thermal metamorphism provide good hydrocarbon generation conditions for coalbed methane accumulation, which is the basis of coalbed methane accumulation. A large number of microscopic pores are developed in the coal, which provides a lot of space for coalbed methane adsorption and storage[12]. The adsorption energy is enhanced, and the amount of adsorbed coalbed methane is increased, which provides a guarantee for coalbed methane accumulation. In the isothermal adsorption experiment, the Langmuir volume of air dry base reached $45.46\text{m}^3\cdot\text{t}^{-1}$, and the adsorption volume was large.

500 grams of original coal samples were collected from No. 3 coal seam in Chengzhuang mining area, and after crushing them, 5-60 mesh and 60-80 mesh coal samples were selected as experimental samples.

2.2. Experimental instruments and methods

Pore characteristics were experimentally analyzed by Scanning electron microscope(SEM).The pore size and pore type characteristics were measured by low temperature liquid nitrogen adsorption experiments, and the adsorption capacity differences under different crushing degrees were determined by high pressure CH₄ adsorption experiments and high pressure CO₂ adsorption experiments.

The models of field emission scanning electron microscopy used in the experiment were Quanta FEG 250 and ZEISS Sigma 300, produced by FEI Company of the United States and Carl Zeiss Joint-stock Company of Germany respectively. The instrument consists of electronic optical system, signal detection system, vacuum system and power supply and control system. The images obtained by SEM were processed by ImageJ software.

The cryogenic liquid nitrogen experiment was selected to be carried out in the Autosorb-iQ automatic specific surface and pore size distribution analyzer from Kona Instruments and Equipment. In this test, BJH method was used to process the adsorption and desorption data, and the processed data started from 2nm[13]. After data processing, we would get the nitrogen adsorption-desorption isotherm and BJH pore size distribution. High pressure CH₄ adsorption experiment by Qingdao Ruian Xinda production of intelligent briquette methane adsorption capacity tester WX-II to carry out the high pressure methane adsorption test. The instrument consists of test environmental parameter measurement system, pulverized coal sample preparation system, coal sample drying system and degassing system, coal sample moisture based preparation system, coal sample weighing system, coal sample test constant temperature system, gas tight inspection and free volume measurement system, adsorption aeration system, touch screen test parameter acquisition and display system, stage adsorption pressure change curve system. It consists of high pressures compression index test system, adsorption pressure self-calibration system, adsorption state indicator system, computer analysis and calculation system. Exploring the high pressures CO₂ adsorption can identify the difference in the adsorption capacity of coal samples for the two gases. GAI-100 gas isothermal adsorption apparatus produced by Temco Company of the United States was used in the experiment, as shown in Figure 25. The device includes an adsorption capacity test system, a gas supply system, a temperature control system, and a pore volume measurement system. Adsorption capacity test system, the adsorption capacity test system includes three independent balance chambers, each chamber can be loaded into 80~100g of powdered coal sample at a time[14]. In order to ensure the comparative significance of the experiment, the same coal mass used in the high-pressure methane adsorption experiment was selected, and two coal samples with different particle sizes of 5~60 mesh and 60~80 mesh were obtained after crushing. After placing them into the GAI-100 isothermal adsorption instrument, the temperature was set at 25°C. Because the CO₂ gas begins to gradually transition to the supercritical state after the pressure exceeds 6MPa. Therefore, the adsorption pressure range was set from 0 to 6MPa.

3. RESULTS AND DISCUSSION

3.1. SEM analysis

In order to facilitate subsequent experiments and simulations, we took the lead in scanning electron microscopy observation of the original coal sample. In order to enhance the electrical conductivity of the original coal sample, the coal sample was treated with gold injection. After that, it was numbered

and put into the scanning electron microscope chamber, and then vacuumed. The pore morphology of coal samples was imaged by computer software.

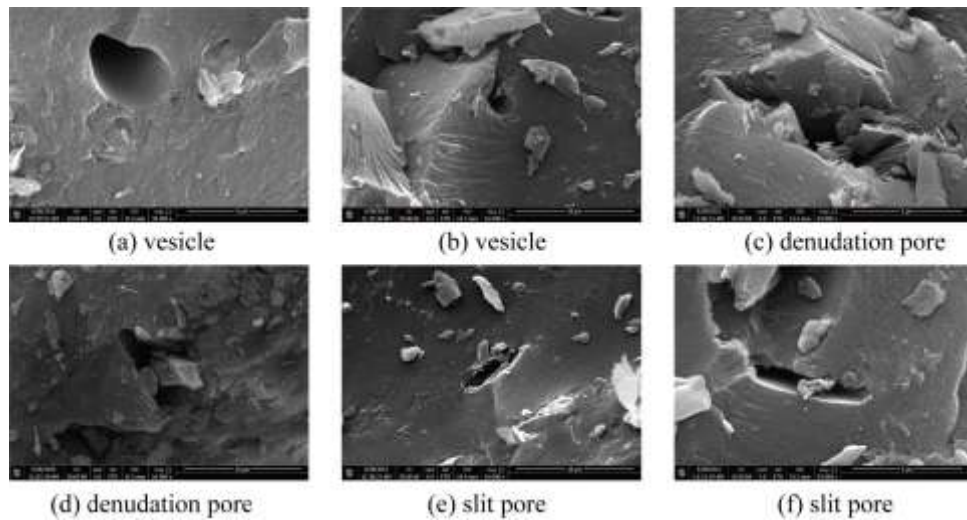


Figure. 1 Pore morphology characteristics of No.3 coal in Chengzhuang Mining area

Figure 1 shows the basic pore types of the original coal sample, which provides a basis for the construction of the model below[15]. It can be found that according to the test results, the coal samples in the study area mainly develop metamorphic pores, dissolution pores and flat slit pores. Stomata are mainly developed in metamorphic pores. In addition, No. 3 coal seam also has a variety of pore shapes, such as circular holes, wedge-shaped holes, triangular holes, etc.

3.2. Nitrogen adsorption characteristics at low temperature

Since adsorption often occurs in nano-scale pores, IUPAC proposed the pore classification standard for coal (micropore $d < 2$ nm, mesopore 2-50 nm and macropore $d > 50$ nm) and the standard adsorption curve type. The pore size measured by low-temperature liquid nitrogen adsorption (LP-N₂GA) was 2-300 nm, which was basically within the range of adsorption. Therefore, the low temperature nitrogen adsorption experiment can be carried out to determine the pore type. Affected by the pore characteristics, the adsorption and desorption of gas have hysteresis. Therefore, the desorption curve of nitrogen often does not completely coincide with the adsorption curve, resulting in the formation of a "hysteresis loop". At present, according to the adsorption and desorption curve, researchers divide the pore types into three categories: open-shaped pores, one-end closed pores, and pores with special structures[16]. According to the adsorption isotherm, with the analysis of the shape and width of the hysteresis loop, the main information of the pore structure and texture characteristics of the adsorbent can be obtained. There is a saturation adsorption platform and uniform decline in the hysteresis ring, generally one end of the closed pore, such as cylindrical pore; There is no obvious saturation adsorption platform, indicating that the pore structure is very irregular, generally flat slit structure, crack and wedge structure. There are saturated adsorption platform and cliff fall, the hole shape is generally more complex, such as ink bottle shaped hole.

The data of raw coal samples after low temperature N₂ adsorption test were plotted, as shown in Figure 2.

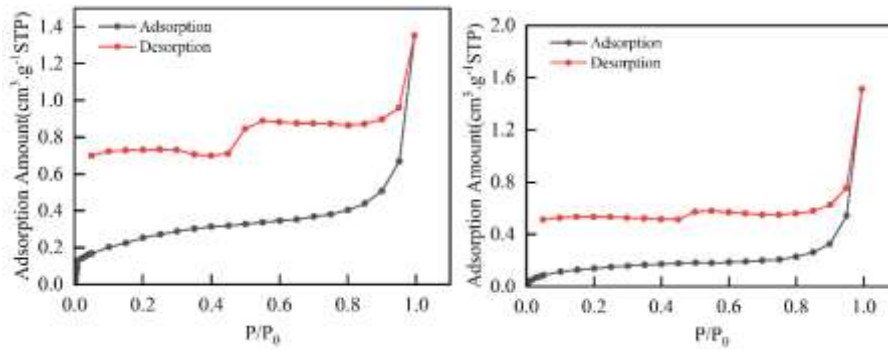


Figure. 2 Low temperature liquid nitrogen adsorption and desorption curves of No. 3 coal from Chengzhuang Mine

According to the above analysis of N_2 adsorption and desorption curve, and combined with the adsorption results of No.3 coal in Chengzhuang Mine, we can find that the N_2 adsorption and desorption curve is not closed under the standard condition of 77K, and it is assumed that the desorption is incomplete due to the influence of the functional groups of the coal body itself. No. 3 coal in Chengzhuang Mine does not have obvious saturation adsorption platform, and it is speculated that No. 3 anthracite in Chengzhuang mine is mainly composed of flat slit holes.

3.3. CH₄ adsorption characteristics of different particle sizes

Through the methane adsorption experiment, the adsorption data of coal samples at 25 ° C were obtained. Langmuir equation was used to fit the above data, and methane isothermal adsorption curves at different particle sizes were drawn, as shown in Figure 3.

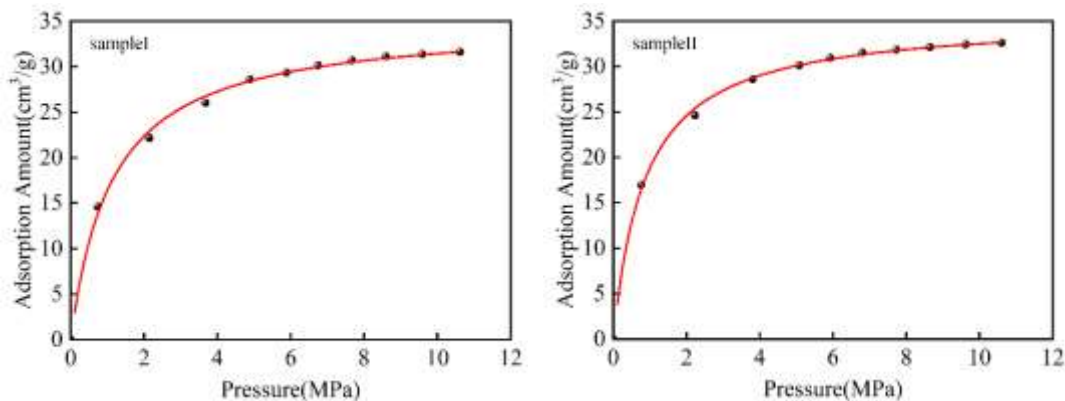


Fig. 3 Results of methane isothermal adsorption at 25 ° C

By analyzing the adsorption data of methane with different particle sizes at 25°C, we find that the adsorption amounts of coarse and fine coal samples are relatively close to each other on the whole. This is consistent with the conclusion of previous studies that adsorbent particle size has basically no effect on the measured value of adsorption capacity. With the increase of coal fragmentation, the adsorption of methane increased slightly. The equilibrium adsorption capacity of fine-grained coal samples is slightly larger than that of coarse-grained coal samples. This is because the degree of coal fragmentation exposes more pores, which can participate in the adsorption process. The fine coal sample has a larger specific surface area than the coarse coal sample, which provides adsorption space for methane adsorption[17]. In the pressure range of 0~6MPa, the adsorption amount and pressure of methane show a nonlinear growth trend. This is because under the low pressure condition, there is a lot of adsorption space in the original coal sample, and the coal surface is in a high-energy state. When methane gas is adsorbed, energy is released, and the two are in a relatively stable state. The adsorption rate of coal under different pressures is different. With the increase of pressure, the

adsorption sites on the surface of coal are gradually occupied completely, and the coal is in a relatively stable state. At this time, the adsorption rate of methane decreases.

After fitting the adsorption capacity of methane by Langmuir equation, the limiting adsorption capacity of methane under this condition can be obtained, and the adsorption performance of coal mass can be judged by the limiting adsorption capacity. See Table 1 for the Langmuir fitting parameters of coal at two particle sizes.

Table 1 Langmuir fitting coefficient of adsorption capacity of No. 3 coal in Chengzhuang Mining area at 25°C

sample	a	b	R2
sampleI	34.5222	0.8619	0.9997
sampleII	34.7063	1.1287	0.9999

It can be found that the values of a and b increase with the increase of the degree of coal fragmentation, in contrast, the increase of b value is larger. With the increase of coal fragmentation degree, the number of methane adsorption channels increases significantly, and the methane adsorption rate accelerates.

In addition, the free space volume of methane was affected by the sample mass at 25°C. The experimental dosage of sample I was 36.9816g, and the measured free space volume was 29.7499 cm³. The experimental mass of sample II was 32.1728g, and the measured free space volume was 31.9963 cm³. After conversion, the mean free space volume of the coarse sample is 0.8045 cm³/g, and the mean free space volume of the fine sample is 0.9945 cm³/g. The increased fragmentation of the sample allows more pores to be entered by methane molecules, resulting in an increase in the measured mean free space volume. Macros.

3.4. CO₂ adsorption characteristics under different particle sizes

Through meticulous CO₂ adsorption experiments, comprehensive adsorption data of coal samples at 25°C were meticulously obtained. These experiments were designed to explore how varying particle sizes of coal samples influence the adsorption capacity of CO₂, a critical parameter for understanding the behavior of coal as a porous medium under different fragmentation conditions. The Langmuir equation, a well-established model in adsorption studies, was employed to fit the experimental data and draw the isothermal adsorption curves of CO₂ under different particle sizes, as vividly illustrated in Figure 4.

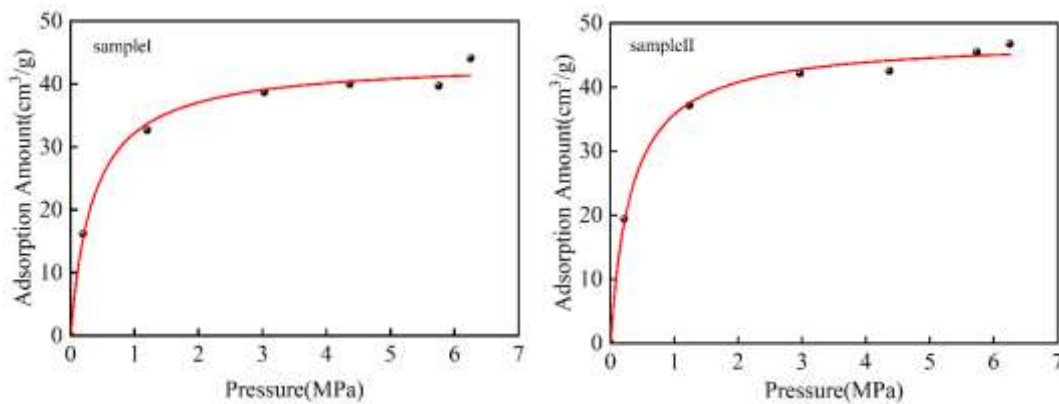


Fig. 4 Results of isothermal adsorption of CO₂ at 25 ° C

The Langmuir equation, given by

$$v = \frac{v_L P}{P + p_L} \quad (1)$$

Where p is the pressure, Pa; V_L is the Langmuir volume, m^3/t ; p_L is the Langmuir volume, Pa. The Langmuir isothermal adsorption equation is most commonly used in gas-solid adsorption analysis and can also be derived by statistical thermodynamics and Gibbs equations.

The resulting isothermal adsorption curves, depicted in Figure 4, provide a clear visualization of how CO_2 adsorption varies with pressure for different coal particle sizes.

The Langmuir fitting parameters, summarized in Table II, offer quantitative insights into the adsorption behavior of CO_2 on coal samples with varying degrees of fragmentation. These parameters are crucial for understanding the underlying mechanisms and for comparing the adsorption capacities of different samples.

Table 2 Langmuir fitting coefficient of CO_2 adsorption capacity of No. 3 coal in Chengzhuang mining area at $25^\circ C$

sample	a	b	R2
sampleI	43.7335	2.7938	0.9925
sampleII	47.4822	3.0435	0.9958

According to Figure 4 and the fitting results in Table II, it is evident that the Langmuir equation effectively describes the adsorption behavior of CO_2 on the coal samples, as indicated by the high R^2 values (0.9925 for Sample I and 0.9958 for Sample II). This high degree of fit confirms the validity and reliability of the experimental results, reinforcing the applicability of the Langmuir model in this context.

A notable observation is that the adsorption capacity of the 60-80 mesh coal sample (Sample II) is higher than that of the 5-60 mesh coal sample (Sample I). This finding is consistent with the results obtained from high-pressure methane adsorption experiments, further validating the conclusion that increased fragmentation of coal provides more adsorption space. As the coal is crushed into finer particles, more pores are exposed, offering additional adsorption sites for gas molecules. This phenomenon underscores the importance of considering coal fragmentation in the design of gas extraction processes and in predicting the risk of coal and gas outbursts.

In addition to adsorption capacity, the free space volume of CO_2 adsorption was also measured in this experiment. For Sample I, with an experimental dosage of 79.3028 grams, the measured free space volume was 50.419 cm^3 . For Sample II, with an experimental mass of 66.6285 grams, the measured free space volume was 55.0155 cm^3 . After normalization by sample mass, the average free space volume of the coarse sample (Sample I) was calculated to be 0.6358 cm^3/g , while that of the fine sample (Sample II) was 0.8407 cm^3/g . The increased fragmentation of the sample allows more pores to be accessible to CO_2 molecules, resulting in a higher measured mean free space volume.

It is also worth noting that the free space volume measured by CO_2 is smaller than that measured by methane. This difference can be attributed to the molecular characteristics of the gases involved. The molecular radius of methane is 0.2 nm, and under standard conditions ($0^\circ C$, 1 atm), its average molecular free path is slightly less than 6.9×10^{-8} meters. In contrast, the average radius of CO_2 molecules is 0.25 nm, and its average molecular free path is 6.29×10^{-8} meters under the same conditions. The larger molecular size and higher thermal motion intensity of CO_2 molecules result in stronger interactions with the coal matrix, enabling CO_2 to penetrate into smaller pores compared to methane. This enhanced adsorption effect of CO_2 on coal is a significant factor in the observed differences in free space volume and adsorption capacity.[18]

These findings highlight the complex interplay between coal fragmentation, pore structure, and gas adsorption behavior. Understanding these relationships is essential for optimizing gas extraction

processes, enhancing the efficiency of coal bed methane recovery, and mitigating the risks associated with coal and gas outbursts in mining operations. Future research should continue to explore the detailed mechanisms underlying these phenomena, potentially leveraging advanced imaging and simulation techniques to further elucidate the behavior of gases within the intricate pore networks of fragmented coal.

4. SUMMARY

In this paper, through low temperature liquid nitrogen experiment, CH₄ adsorption experiment and CO₂ adsorption experiment, the adsorption characteristics of coal under different crushing degrees are explored, and the following conclusions are finally obtained:

- (1) SEM experiments show that anthracite coal of No.3 coal seam in Chengzhuang mining area mainly develops pores of different shapes, dissolution pores and plate-like slit pores.
- (2) The low temperature nitrogen adsorption experiment shows that there is an obvious "hysteresis loop" in the low temperature nitrogen adsorption and desorption curve. According to the classification of low temperature liquid nitrogen adsorption curve proposed by IUPAC, the adsorption and desorption curve of coal samples from No. 3 coal seam in Chengzhuang mining area belongs to class III, and there is no obvious saturated adsorption platform. It is presumed that No. 3 anthracis in Chengzhuang Mine is mainly dominated by flat type slot holes.
- (3) High-pressure CH₄ adsorption experiments show that the degree of coal fragmentation is positively correlated with the adsorption capacity of CH₄. In the pressure range of 0-6mpa, the adsorption capacity of methane increases sharply with the pressure. This is because under low pressure conditions, there is a large amount of adsorption space in the original coal sample, and the coal surface is in a high-energy state, and energy is released when methane gas is adsorbed. The two are in a relatively stable state. With the increase of pressure, the adsorption sites on the coal surface are gradually occupied completely.
- (4) The high pressure CO₂ adsorption experiment shows that the adsorption capacity of CO₂ is greater than that of CH₄ under the same particle size, temperature and pressure conditions, because CO₂ is more easily adsorbed by the coal sample itself. Under the same conditions, the free space volume measured by CO₂ is slightly smaller than that measured by CH₄.

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