STUDY OF FRACTAL BASED OXYGEN ADSORPTION EXPERIMENT OF POROUS COAL

by

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Coal is China's main energy source and fuel. Coal spontaneous combustion is one of the most prominent issues that threaten the production safety of coal mining, storage, and transportation. In order to explore the factors affecting coal spontaneous combustion, we investigated the pore structure characteristics of the coal based on the fractal theory, through the low temperature liquid nitrogen adsorption experiment of coal. The fractal dimension of the coal sample was calculated, and the oxygen adsorption quantity of the same coal sample was obtained by using the physical adsorption experiment of coal. Experimental and fitting results showed that coal sample has obvious surface fractal dimension features and pore structure fractal features. Fractal dimension expressed coal oxygen adsorption well. In the meantime, the coal samples with lower fractal dimension, higher temperature, smaller porosity usually have less oxygen adsorption quantity. This research cannot only enrich the study of oxygen adsorption in porous media such as coal, but also help to understand its spontaneous combustion mechanism in depth, thereby reducing the occurrence of spontaneous combustion disasters.

Key words: *coal oxygen adsorption, physical adsorption, porous medium, coal spontaneous combustion, fractal*

Introduction

Coal is China's main energy source and fuel. The spontaneous combustion of coal is one of the most prominent issues that threaten the production safety of coal mining, storage, and transportation. Fires resulted from coal spontaneous combustion cause serious environmental pollution problems and produce a resource of waste, reduce coal quality, and seriously threaten the safety of coal mine production. It is of great practical significance to study the spontaneous combustion law of coal and preventive measures [1].

Because the physical structure of porous media impacts the physicochemical properties [2], many scholars have studied the structure influencing factors of coal spontaneous combustion, such as the influence of particle size [3], pore size distribution [4], and the contribution of each size of pores [5]. As the application of fractal geometry theory can accurately locate the pore structure information of coal porous media [6], to study the mechanism of adsorption, transportation, and penetration in porous media based on fractal geometry theory receives special attention [7-9]. Li *et al*. [10] confirmed the classification and fractal characteristics of

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coal and rock debris under uniaxial cyclic loading conditions, this provided a basis for further research on coal fractal. Liu *et al*. [11] investigated the effect of fractal dimensions on methane adsorption capacity by using SEM and low pressure nitrogen adsorption (LP-N2GA), found that fractal dimensions comprehensively reflected the differences in the physical properties of coal and can be used to evaluate the adsorption capacity of coal for methane. Xu *et al*. [12] studied the effect of surface roughness on the diffusion and adsorption of coalbed methane through the 3D-Laser profiler testing and fractal-based lattice Boltzmann simulation, which can better evaluate the effect of the microscopic mechanism of coalbed methane migration/adsorption on the overall recovery factor of the reservoir.

Studies have shown that the oxygen adsorption of coal during spontaneous combustion is the main factor for coal spontaneous combustion, thus the primary study of coal spontaneous combustion prevention is to explore the mechanism of coal oxygen adsorption [13-15]. The research on the fractal characteristics of coal has made significant progress, but the research on the influence of fractal dimension on coal oxygen adsorption has been seldom reported [16]. In view of the excellent performance of fractal theory in the field of gas adsorption [17], we employ the fractal dimension to represent the pore structure of coal. To find the relationship between coal's fractal dimension and coal oxygen adsorption can help us to have a better understanding of the structural mechanism of coal spontaneous combustion. This article will carry out relevant experimental research and analysis to provide new ideas for studying the influencing factors of the coal oxygen adsorption process.

Materials and method

This study conducted liquid nitrogen adsorption and coal oxygen physical adsorption experiments to explore the effect of fractal dimension on coal oxygen physical adsorption. The low temperature liquid nitrogen adsorption experiment of coal was employed to determine the fractal dimension and pore structure characteristics of the coal sample, and the physical oxygen adsorption experiment was carried out on the same batch of coal samples to determine the oxygen adsorption of the coal.

The coal samples used in this study were taken from six coal-producing areas in China, Sunjiawan, Hongyang (Northeast of China), Yulin, Baiyangling, Qujiagou (North of China), and Guizhou (South of China). They were selected for experimental analysis. Before the adsorption experiment for testing pore structure and oxygen adsorption, we did the industrial analysis to quantify the ingredient of coal samples, tab. 1. The industrial analysis was carried out using the standard automatic industrial analyzer, and there were three replications of each coal sample.

Table 1 Proximate analysis of coal samples

Sample number	Moisture, M_{ad} [%]	Ash, A_{ad} [%]	Volatile matter, V_{ad} [%]	Fixed carbon, FC_{ad} [%]	Species
1#	3.21	10.86	19.42	66.51	Coking coal
2#	3.52	12.72	18.27	65.49	Coking coal
3#	3.88	10.15	17.35	68.62	Lean coal
4#	2.17	12.37	20.98	64.48	Coking coal
5#	3.89	11.55	16.28	68.28	Lean coal
6#	4.25	10.47	20.38	64.90	Coking coal

Low temperature liquid nitrogen adsorption experiment of coal

The low temperature adsorption method determines the specific surface area and pore size distribution of solids according to the law of gas adsorption on the solid surface. At a certain pressure, the adsorption rate is equal to the desorption rate. In this equilibrium state, the pressure corresponds to the adsorption amount of liquid nitrogen, and pressure is changed to obtain the corresponding gas adsorption amount [18]. The curve corresponding to the equilibrium adsorption volume and pressure change is the adsorption isotherm. The pore structure, distribution, and calculation of fractal dimension can be obtained by studying the adsorption isotherm. This experiment uses the ASAP2020 physical adsorption instrument to determine, the specific adsorption equation:

$$V = \frac{V_m CP}{\left(P_0 - P\right)\left[1 + \left(C - 1\right)\left(\frac{P}{P_0}\right)\right]} \tag{1}$$

After the transformation, we can get:

$$\frac{P}{V\left(P_0 - P\right)} = \frac{1}{CV_m} + \frac{C-1}{CV_m}\frac{P}{P_0} \tag{2}$$

where V [mlg^{-1}] is the amount of gas adsorbed, V_m [mlg^{-1}] – the amount of adsorbed monolayer, P [MPa] – the adsorbate pressure, P_0 [MPa] – the saturated vapor pressure of adsorbate, and C – a constant.

From the pressure data and coal liquid nitrogen adsorption data obtained from the experiment, a function-line of P/P_0 about $P/V(P_0/P)$ can be drawn. The value can obtain the value of V_m. The size of S_g (specific surface area) can be obtained:

$$S_g = 4.36V_m \tag{3}$$

The calculation of the fractal dimension of the adsorption pores uses the Frenkel-Halsey-Hill equation [19]:

$$\ln V = A\left\{\ln\left[\ln\left(\frac{P_0}{P}\right)\right]\right\} + B \tag{4}$$

where V is the amount of gas adsorption at equilibrium pressure P, P_0 – the saturated vapor pressure of the gas, P – the equilibrium pressure of gas adsorption, A – the slope of the fitted straight line, which is linearly related to the fractal dimension D, and B – a constant.

The fractal dimension D can be calculated from A. There are two different formulas derived under different assumptions: $A = D - 3$ and $A = (D - 3)/3$. Choose appropriate expressions through calculation results.

Coal oxygen physical adsorption experiment

Based on the principle of the Langmuir adsorption equation, at a low temperature state, the experimental pressure is controlled to atmospheric pressure to carry out the coal oxygen physical adsorption experiment [20], which is a single molecule physical adsorption state experiment. According to the chromatographic oxygen adsorption identification method [21, 22], the chromatographic peak is measured to obtain the oxygen adsorption of the coal sample, and the chromatographic peak area can be detected by the thermal conductivity cell. In this study, the ZRJ-1 coal spontaneous combustion tester is used to measure coal samples from six different coal-producing areas, the measurement is based on the chromatography oxygen identification method. The basic measurement process is that after the coal sample weighing and pretreatment, we put the coal sample into a sample tube, accessing to purge, to make sure that the original adsorbed gas was purged out. Secondly, start the oxygen adsorption procedure, oxygen is blown into the sample tube, and they are adsorbed by the coal sample, then fill the sample tube with nitrogen to quickly replace the adsorbed oxygen, and the thermal conductivity detector of gas chromatography will measure the amount of oxygen in nitrogen flow, thereby we can determine the physical adsorption value of coal sample for oxygen.

The adsorption amount of the coal sample is calculated according to the adsorption equation:

$$Q = KR_{c1}\left\{S_1 - \left[\frac{\alpha_1 R_{c1}}{\alpha_2 R_{c2}} \times S_2\left(1 - \frac{G}{D_{\mathrm{TRD}} V_s}\right)\right]\right\} \times \frac{1}{\left(1 - M_{ad}\right)G} \tag{5}$$

where Q is the oxygen adsorption, K – the instrument constant, R_{c1} and R_{c2} are the carrier gas-flow rate of the solid tube and the empty tube, α_1 and α_2 are the ratio of the partial pressure of oxygen and atmospheric pressure when the sample tube is non-empty and empty, S_1 and S_2 are the peak areas of the solid and empty tubes, G – the weight of the coal sample, D_{TRD} – the true specific gravity of the coal sample, V_{S} – the sample tube volume, and M_{ad} – the moisture of the coal sample.

Experimental results

Liquid nitrogen adsorption and desorption curve

After the coal sample undergoes the two steps of liquid nitrogen adsorption and desorption, the pore structure of the coal sample can be described according to the data it generates. The different textures of the coal samples lead to different degrees of metamorphism, which affects the pore adsorption and desorption curves of the coal samples, as shown in fig. 1.

The adsorption hysteresis phenomenon exists in each coal sample, that is, the adsorption curve and the desorption curve do not coincide. The adsorption hysteresis phenomenon is caused by the capillary condensation phenomenon between the mesopores and macropores of the coal sample and the adsorbent surface. The adsorption lag of the No. $6^{\#}$ coal sample is the most obvious in six samples. No. $1{\sim}5^{\#}$ coal samples have good overall coincidence, but when the relative pressure is large ($P/P_0 > 0.5$), a slight adsorption hysteresis phenomenon can still be observed. However, at the low pressure section ($P/P_0 < 0.5$), there is almost no adsorption hysteresis, due to the capillary condensation phenomenon occurring less. From the behavior of liquid nitrogen adsorption/desorption curve, we can find that the proportion of micropores in coal samples $1{\sim}5^{\#}$ is larger, while the proportion of medium pores and macropores in coal sample $6^{\#}$ is larger.

Research on fractal characteristics of coal sample

Through comparative analysis, the adsorption curve and desorption curve of coal sample can be seen that the curve of coal sample in the desorption state is more stable than in the adsorption state, thus the desorption curve is used when calculating the fractal dimension of coal sample. Observing the liquid nitrogen adsorption curve and desorption curve of coal samples, it can be seen that the $P/P_0 > 0.5$ and $P/P_0 < 0.5$ curves in the low pressure section have different coincidence states.

For the low pressure section, the curve coincides well, while the high pressure section has different degrees of adsorption lag. The reason for this phenomenon is mainly due to the

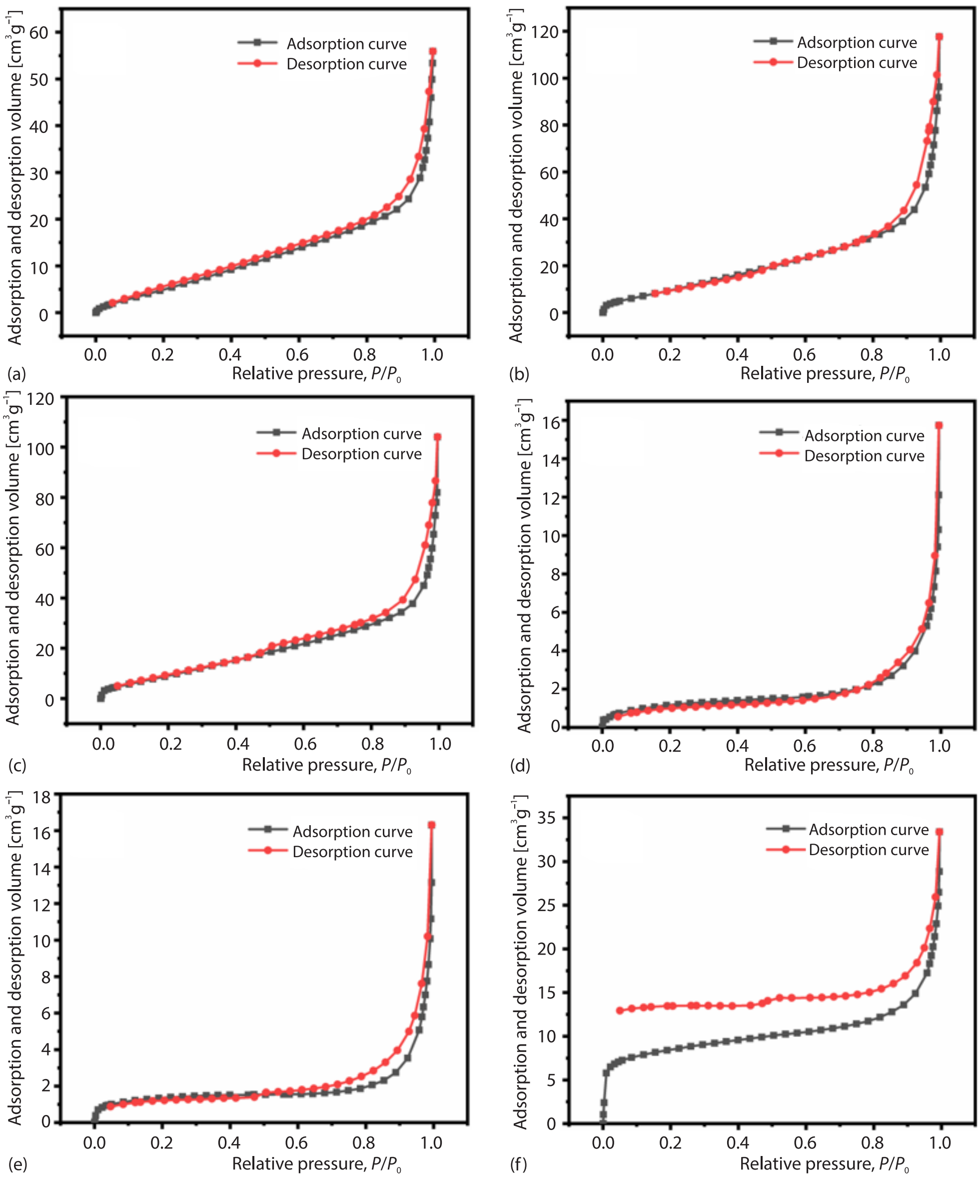

Figure 1. Liquid nitrogen adsorption/desorption curve; (a) 1#, (b) 2#, (c) 3#, (d) 4#, (e) 5#, and (f) 6#

different adsorption force of coal-like structure on molecules under different pressures. In the low pressure section, the van der Waals force plays a major role, while in the high pressure section, the capillary cohesion between the pore structure and the molecules is the force. This phenomenon leads to a two stage structure that requires a second calculation. Different pressure stages correspond to different fractal dimensions to characterize the different characteristics of coal samples.

Coal samples in different pressure sections show different fractal characteristics, which reflects that the adsorption pores of coal samples have different adsorption capacities under different pressure from the side. That is to say, at low pressure, the micropores are dominant, the adsorption capacity is highly correlated with the surface roughness of coal samples. While at relatively high pressure, with the influence of the condensation effect of capillary, the adsorption capacity is related to the pore structure of coal sample. Therefore, the surface fractal dimension of the coal sample is expressed as the fractal dimension D_1 ($P/P_0 < 0.5$) of the coal sample, while the fractal dimension of the pore structure of the coal sample is expressed as D_2 ($P/P_0 > 0.5$) in this study.

It can be seen from fig. 2 that the desorption curve of liquid nitrogen shows a clear fractal scaling law in both low pressure and high pressure parts. In the low pressure area, van der Waals force is the main adsorption force. The gas adsorption capacity is related to the surface roughness of the coal sample. Therefore, the fractal dimension D_1 of the low pressure portion represents the surface fractal dimension of the porous material. The gas adsorption in the high pressure section mainly depends on the condensation of the capillary, and the adsorption capacity is related to the pore structure of the coal sample. That is to say, the fractal dimension D_2 of the high pressure cross section represents the fractal dimension of the pore structure. According to the concept of fractal, the value of fractal dimension in 3-D space is usually in the range of 2.0-3.0.

It can be found in tab. 2 that the fractal dimension of coal surface D_1 varies from 1.80-2.96, which indicates that the surface roughness of coal is relatively large. In tab. 2, A is the slope of the fitted straight line, which is linearly related to the fractal dimension D. The A_1 and A_2 are determined by $A = D - 3$ and $A = (D - 3)/3$. As the degree of metamorphism increases, the surface roughness of the coal sample first decreases and then increases. This is why the fractal dimension D_1 decreases first and then increases as the coal metamorphism increases. The fractal dimension of coal pore structure D_2 varies from 2.45-2.82.

Table 2. Fractal dimension results of coal samples

Sample number	A_1	$D_1 = 3 + A_1$	$D'_1 = 3 + 3A_1$	Fit, R^2	A_2	$D_2 = 3 + A_2$	$D'_2 = 3 + 3A_2$	Fit, R^2
1#	–1.2	1.8	–0.6	0.98	–0.32	2.68	2.04	0.98
2#	–0.86	2.14	0.42	0.99	–0.38	2.62	1.86	0.97
3#	–0.92	2.08	0.24	0.99	–0.33	2.67	2.01	0.99
4#	–0.48	2.52	1.56	0.90	–0.55	2.45	1.35	0.99
5#	–0.30	2.70	2.10	0.90	–0.51	2.49	1.47	0.99
6#	–0.04	2.96	2.88	0.76	–0.18	2.82	2.46	0.95

According to the previous analysis, the pore structure fractal dimension and surface fractal dimension of the experimental coal samples are 2.45~2.82 and 1.8~2.96. According to the principle of coal surface roughness, the degree of surface roughness is negatively correlated with the degree of metamorphism of coal samples. The calculation method of surface roughness changes when the degree of metamorphism reaches a higher level. Regarding the numerical distribution of pore structure, it can be seen that the pore structure of coal samples varies in size and is very complicated.

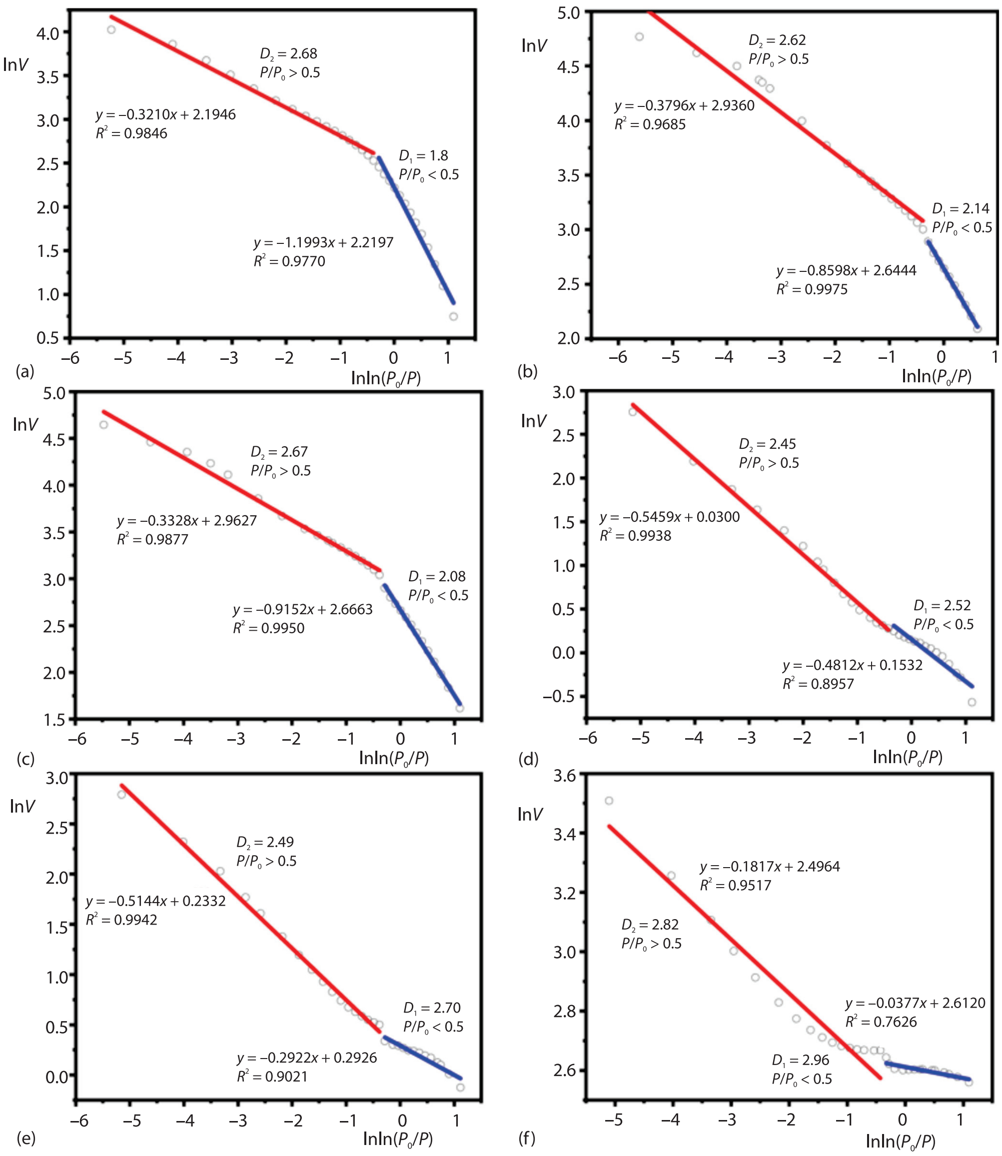

Figure 2. Fractal calculation results of liquid nitrogen desorption curve (original experimental data was represented by hollow symbols in plotted figures): (a) 1#, (b) 2#, (c) 3#, (d) 4#, (e) 5#, and (f) 6#

Results and discussion

Effect of porosity on oxygen adsorption

The coal sample related data obtained by the liquid nitrogen adsorption experiment, the porosity size of the coal sample obtained by calculation, and the size of the fractal dimension of the coal sample of the same batch are summarized in tab. 3.

It can be seen from fig. 3 that the coal sample with larger porosity has a larger value of oxygen adsorption. The oxygen adsorption of coal samples shows an increasing trend with

Table 3. Structural information of coal samples

Sample number	Bulk density, $\rho_{p,d}$ [gcm^{-3}]	Skeletal density,* ρ_d [gcm^{-3}]	Porosity [%]	Fractal dimension, D_f
1#	1.105972	1.28545	13.96228	1.8
2#	1.112062	1.29825	14.34144	2.14
3#	1.072638	1.2512	14.27128	2.08
4#	1.274886	1.491237	14.50814	2.52
5#	1.113322	1.30562	14.72844	2.7
6#	1.142731	1.35058	15.38963	2.96

* Definition of Skeletal density: the particle skeletal density is the density of the material that constitutes the particle, without porosity.

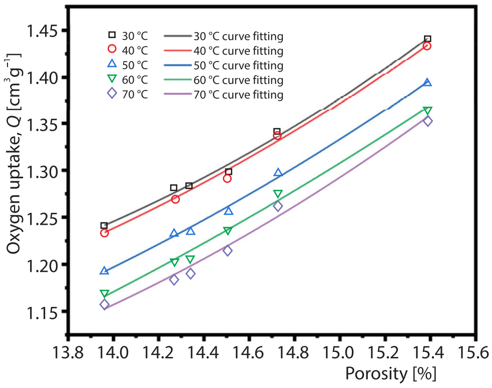

Figure 3. Relationship between porosity and oxygen uptake of coal samples (original experimental data was represented by hollow symbols in plotted figures)

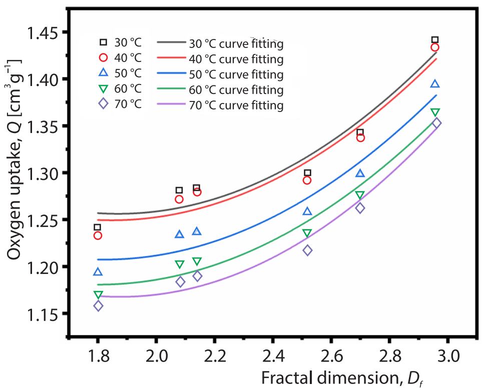

Figure 4. Relationship between fractal dimension and oxygen uptake (original experimental data was represented by hollow symbols in plotted figures)

the increase of porosity. This may be due to the relatively large porosity of coal samples with large porosity, and the large gas storage space. The adsorption capacity is strong as well, and the coal with smaller pores tends to be denser, leading to a decrease in adsorption capacity. According to the numerical simulation results of Yu and Liang [23], the porosity and oxygen adsorption are also positively correlated, which is consistent with what this study has done.

Effect of fractal dimension on oxygen adsorption

Fractal objects are unique in that they do not depend on units of measurement and the following formulae follow the rules of scale [24]:

$$M(L) \sim L^{D_f} \tag{6}$$

where M can be the length of a straight line, L – the area of a surface or the volume of an object and D_f – the fractal dimension.

The relationship between the fractal dimension of the coal sample measured by the liquid nitrogen adsorption experiment and the physical oxygen adsorption of the coal sample is shown in fig. 4.

This result tells us that in the condition that the experimental temperature is constant, the fractal dimension of coal samples and oxygen adsorption shows a positive correlation trend. The fractal dimension characterizes the pore structure and pore surface area of coal samples. When the fractal dimension increases, the pore surface area also increases accordingly. The stronger the compressibility, the stronger the adsorption capacity of coal. Experiments have fully proved that the fractal dimension is closely related to the oxygen adsorption of coal.

Effect of temperature on oxygen adsorption

With a certain fractal dimension, five temperature points of 30 °C, 40 °C, 50 °C, 60 °C, and 70 °C are taken to conduct the oxygen adsorption measurement experiment.

Experimental data show that under the same fractal dimension, as the experimental temperature rises, the oxygen adsorption values of the three coal samples all show a downward trend. This is because when the temperature increases, the random thermal movement of oxygen molecules will strengthen, and oxygen molecules will easily break away from the pore surface; as the temperature increases, the internal structure of the molecules will change, and the adsorption sites on the coal surface decrease. The fluctuation of oxygen adsorption in certain temperature ranges is caused by the development of pores inside the coal in this temperature range, which results in an increase in the surface area of the pores that adsorb oxygen, and the resulting impact exceeds the reduction of surface adsorption sites and oxygen molecules. The effect caused by detachment leads to an increase in oxygen adsorption [25].

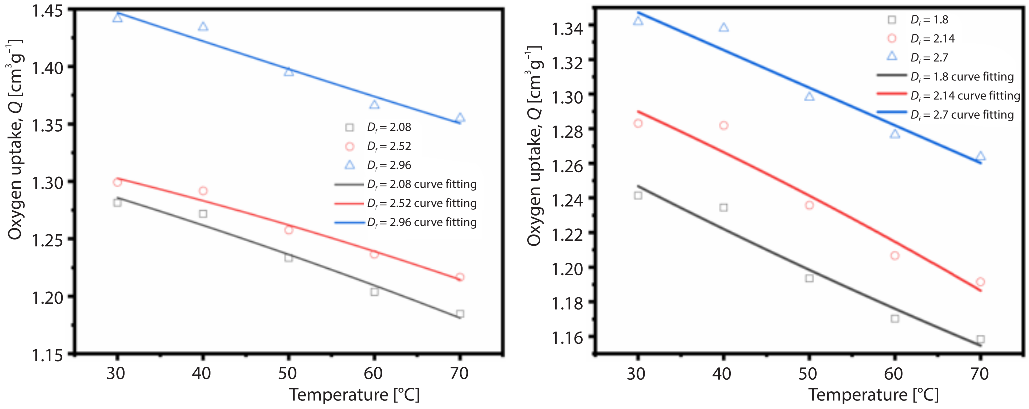

Figure 5. Relationship between temperature and oxygen uptake (original experimental data was represented by hollow symbols in plotted figures)

Conclusions

- Through the low temperature liquid nitrogen adsorption experiment of coal, the adsorption and desorption curves of 6 coal samples were obtained. In addition, the 6# coal sample, there were some adsorption lags in the adsorption and desorption process, and other coal samples showed no obvious lag.
- We calculated the fractal dimension of the desorption curve. The six coal samples all have good fractal dimensions and dual characteristics. The D_1 is used to describe the surface fractal characteristics of the coal sample, and D_2 can be used to describe the pore structure fractal of the coal sample. The fractal dimension D_1 is distributed between 1.8~3.0, while the D_2 distribution is 2.4~2.82.
- The effect of porosity, temperature, and fractal dimension on oxygen adsorption was analyzed by the coal oxygen physical adsorption experiment. It was found that at the same temperature, fractal dimension and coal oxygen adsorption are positively correlated, while porosity and oxygen adsorption are in a positive correlation as well. However, temperature

and oxygen adsorption are negatively correlated. This work helps to understand the mechanism of coal oxygen adsorption and can provide a useful reference for strategies to reduce the risk of coal spontaneous combustion.

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