Experimental study on the effect of intermittent microwave on coal adsorption performance

Abstract: Most of the coal seams in China belong to low permeability coal seams, and microwave permeability enhancement method can effectively improve the mining effect of coalbed methane. In order to study the effect of microwave irradiation on the adsorption performance and pore structure of coal, the lignite samples of Hulun Buir Linglu Mine in Inner Mongolia were irradiated by intermittent microwave. The LPGA-N_2 experiment and LPGA-CO_2 adsorption experiment were carried out on the irradiated coal samples, and the NLDFT model was used to calculate the effect of microwave irradiation on the pore volume, specific surface area and proportion of coal samples. The results show that with the increase of irradiation time, the adsorption amount of CO_2 and N_2 on coal decreases linearly. After 10 min of microwave irradiation, the adsorption amount of CO_2 decreases by 14.13 %, and the adsorption amount of N_2 decreases by 34.34 %. The total pore volume and total specific surface area of coal samples decreased with the increase of irradiation time due to microwave action. Therefore, it was confirmed that microwave action would reduce the adsorption performance of coal samples. The experimental results will help to improve the gas extraction rate and provide reference for reducing the risk of coal seam outburst.

Key words: Coalbed methane, microwave irradiation, lignite, adsorption performance, pore structure.

Coalbed methane, also known as ' gas ', is a kind of gas accompanied by coal formation and metamorphism, with methane as the main component. China is a country with abundant coalbed methane reserves. China 's proven coalbed methane reserves below 2000 m are 3.68 × 1013 m^3, which is one of the most representative clean energy sources in China[1]. At present, the permeability of coal seam in China is generally 0.1×10^{-5}-$0.1\times10^{-3}m^2$, which is a typical low permeability coal seam. In the process of mining, free gas is difficult to release quickly, and the extraction effect is poor, which seriously restricts the efficient utilization of coalbed methane resources in China, and also brings serious environmental pollution. Therefore, improving the permeability of coal is the key to improve the efficiency of gas extraction.

At present, a variety of anti-reflection technologies are used at home and abroad. Among them, hydraulic fracturing[2], hydraulic slotting[3], liquid nitrogen fracturing[4], supercritical CO_2 anti-reflection[5], high-energy gas fracturing[6] and other technical measures have achieved good results, but these methods have technical limitations and complex geological conditions. It is difficult to implement and other issues. In order to make up for the shortcomings of the above methods, in recent years, domestic and foreign scholars have carried out research on the influence of acoustic field[7], electric field[8], alternating electromagnetic field[9] and other methods on the adsorption/desorption behavior of coal. Among them, the electromagnetic wave with a frequency of 300MHz-300GHz is called microwave. It has the advantages of controllable heating, selective heating, rapid heating, strong penetration ability, small heating inertia and high transmittance. It is a hot spot for scholars at home and abroad. In recent years, microwave technology has been widely used in coal heating dehumidification, coal desulfurization, coal pyrolysis, coal assisted rock breaking and oil recovery.

Wen et al.[10] conducted gas desorption experiments on bituminous coal. The results showed that when the microwave radiation was 3 minutes and 5 minutes, the instantaneous desorption amount, cumulative desorption amount, micropore specific surface area and pore volume of bituminous coal all showed a trend of increasing first and then decreasing, and then increasing.

Wang et al.[11] compared the columnar coal by two methods of no microwave and intermittent loading, and found that microwave can effectively promote the desorption of methane in coal. Yu et al[12] carried out continuous-discontinuous irradiation and gas adsorption and desorption experiments on granular coal. The results show that the thermal effect generated by microwave has a significant effect on gas desorption. Song et al.[13] studied the conventional pyrolysis and microwave pyrolysis of three low rank coals. It was found that microwave pyrolysis can reduce the methane content of coal, and the gas yield is 3%-5% higher than that of conventional pyrolysis. Hu et al.[14] carried out low temperature nitrogen adsorption and mercury intrusion tests on coal samples under microwave field. The results showed that the specific surface area and pore volume of coal samples under microwave field decreased significantly, and this change did not disappear with the disappearance of microwave field.

At the same time, in order to reveal the influence of microwave energy on the pore-crack structure of coal, Li et al.[15] studied the evolution characteristics of pore-crack structure of coal under microwave irradiation by means of infrared thermal imaging, ultrasonic testing, nuclear magnetic resonance and high-precision X-CT.Through the cyclic microwave irradiation test of coal and rock, the structural characteristics and evolution law of the surface crack of coal and rock under microwave irradiation were studied. Microwave irradiation has a promoting effect on the pore structure of coal.Lin et al.[16] through the cyclic microwave irradiation test of coal and rock, the structural characteristics and evolution law of the surface crack of coal and rock under microwave irradiation were studied. Microwave irradiation has a promoting effect on the pore structure of coal. Zhang et al.[17] carried out microwave experiments on anthracite with different cycles, and confirmed that microwave radiation made the pores of coal samples connect and expand, resulting in an increase in permeability. Dong et al.[18] measured the porosity, adsorption volume and volume of coal. The results showed that with the extension of microwave treatment time, the porosity and permeability of coal increased first and then decreased, while the adsorption volume of coal decreased first and then increased. Kumar et al.[19] found that new cracks were produced while microwave action increased coal pores during the study of coal pores under the action of energetic microwave.

Some scholars have found that microwave energy will also affect the functional group structure of coal. Xu et al.[20] found that the quality of coal decreased after radiation treatment of coal samples at different time periods. The reason is mainly due to the selective removal of water and some other polar small molecules under heating conditions. Ge et al.[21] found that water molecules and active molecules precipitated under microwave heating conditions through the microwave drying test of lignite. Ren et al.[22] studied the oxygen-containing functional groups in lignite by chemical titration method, and concluded that 600-800W microwave power had the highest efficiency in removing oxygen-containing functional groups in lignite. He et al.[23] showed that under the condition of microwave heating, the oxygen-containing groups and aliphatic hydrocarbons in bituminous coal were cracked, which increased the hydrophobicity of coal. Zhou et al.[24] and Tahmasebi et al.[25] showed that under the condition of microwave radiation, the oxygen-containing functional groups in lignite decreased, the content of aliphatic hydrogen decreased, and the aromaticity remained basically unchanged or even increased. Wang et al.[26] studied the microwave pyrolysis reaction of Indonesian lignite. The results showed that microwave radiation could increase the yield of aromatic hydrocarbons and was proportional to the radiation power.

The results show that the heating effect of microwave can not only accelerate the desorption of coalbed methane, but also have a certain influence on the pore structure and chemical structure of coal. At present, the high power excitation mode is widely used in the study of the mechanism of microwave enhanced gas desorption. However, due to the high power continuous microwave, the coal will be rapidly heated and pyrolyzed in a short time, and the pyrolysis products will have an impact on the pores of the coal body, which is not conducive to gas extraction. Therefore, this paper studies the effect of intermittent microwave on the adsorption characteristics of coal by means of intermittent microwave action and carrying out the adsorption experiments of N_2 and CO_2 under low temperature conditions, which provides a certain reference for microwave antireflection technology.

1 Experimental method

1.1 Coal sample preparation

In this paper, the experimental samples were selected from the lignite raw coal sample of Hulunbeier Linglu in Inner Mongolia. According to the national standard ' preparation method of coal sample ' (GB/T474-2008), the coal sample was pre-crushing first, and then the coal sample sieve was used to separate the coal sample into 0.18-0.25mm (60-80 mesh) and less than 0.18mm (<80 mesh) according to the particle size. According to the national standard " Industrial analysis method for coal " (GB/T212-2008), the moisture (Mad), ash (Aad), volatile matter (Vad) and fixed carbon content Fad) of coal samples with less than 80 mesh were measured. Specific parameters are shown in Table 1.

Table 1 Coal sample industrial analysis

Coal samples	Industrial analysis/%			
	Mad/%	Aad/%	Vad/%	Fad/%
Brown coal	1.69	6.15	52.96	39.2

1.2 Microwave radiation experiment

Compared with the traditional heating method, the working frequency of microwave heating is usually higher than 300MHz, and its radiation mode is divided into two types, one is irradiation in the resonant cavity, and the other is based on the radiation field of the microwave antenna. In this paper, the former microwave irradiation method is selected, and the Midea M1-L213B microwave oven is selected. The output frequency of the microwave generator is 2450MHz, and the power is adjustable in five gears. In this paper, the output power is 700W, and the intermittent microwave irradiation experiment is carried out on the coal sample, that is, the radiation is stopped for 300s after 2min of each microwave, and then the irradiation is continued. Taking the irradiation time as a variable, the total irradiation time of coal samples is often 2min, 4min, 6min, 8min, and 10min. The coal samples without irradiation experiments are used as the control group (hereinafter referred to as '0 min' coal samples).

1.3 Low temperature adsorption experiment

The American Micromeritics ASAP 2020 Plus physical adsorption instrument was used in the test. The adsorption capacity and pore structure of coal samples were characterized by carbon dioxide adsorption experiment at 273K and nitrogen adsorption experiment at 77K. The prepared coal sample is loaded into the test tube and installed in the degassing station. The purpose of this step is to remove the impurities in the coal sample. After the degassing is completed, it is installed in the analysis station to perform CO_2 and N_2 adsorption/desorption experiments.

A large number of research results show that CO_2 is suitable for the analysis of micropore stage in coal, especially ultra-micropore (≤1nm), and N_2 is suitable for the analysis of non-micropore stage. Therefore, this paper will use LPGA-N_2 and LPGA-CO_2 adsorption methods to more comprehensively analyze the effect of microwave irradiation on the adsorption capacity and pore results of coal. The overall process of the experiment is shown in Figure 1.

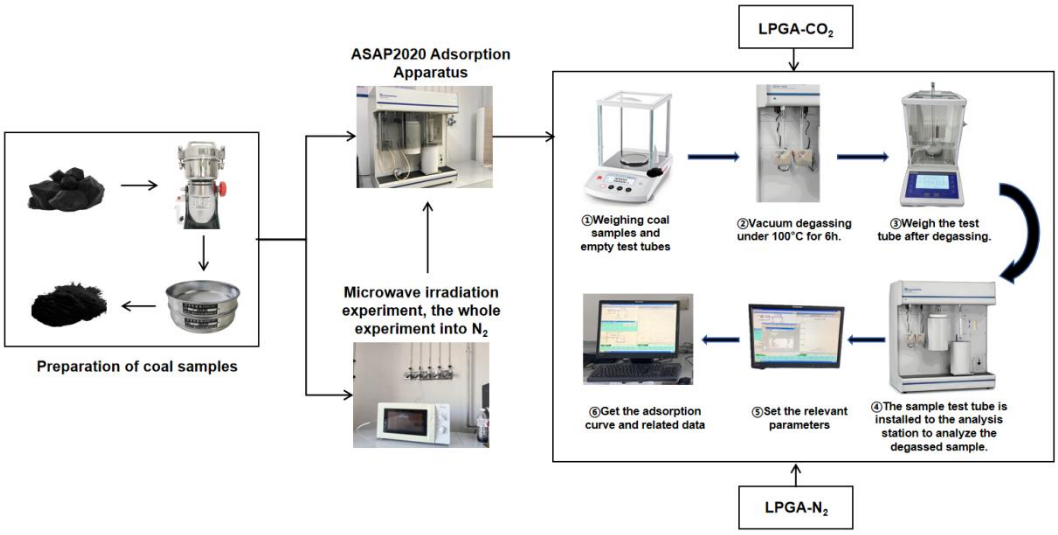


Fig.1 The overall flow chart of the experiment

1.4 Pore characterization method

At present, there are a variety of adsorption theoretical models used to study the pore structure of coal, including Brunauer-Emmett-Teller (BET), Barret-Joyner-Halenda (BJH) and Density Functional Theory (DFT).

Non-Local Density Functional Theory (NLDFT) is an extension of the traditional density functional theory (DFT). It has been widely used in the study of pore structure of materials, especially in the adsorption and desorption of pores, pore size distribution and specific surface area. By introducing nonlocal modification, NLDFT takes into account the change of the interaction force between the pore wall and the surface adsorption molecules with space, which can better reflect the adsorption characteristics in the real porous system. For microporous and mesoporous materials, especially the fitting of pore size distribution, pore volume and adsorption isotherms at high pressure adsorption, more accurate results can be provided. This paper will combine the NLDFT model to analyze the adsorption isotherm and quantitatively analyze the pore structure of coal samples.

1.5 Principle of microwave action

Microwave heating is a body heating from inside to outside. Due to the different dielectric material properties inside the object, if the medium is placed between two metal plates and direct current is applied, the dipoles in the medium will rearrange and form polar molecules with certain orientation and regular arrangement. If the direct current is replaced by alternating current, the electric field between the poles will change alternately at the same frequency, and the dipoles in the medium will also swing rapidly accordingly. For example, in the electric field of 2450 MHz, the dipoles swing rapidly at a speed of 2.45 × 109 times / s ; due to the thermal motion of molecular oscillation and the interaction of adjacent molecules, the moving molecules obtain energy and form multiple internal heat sources, resulting in the overall temperature change of the material[27]. The schematic diagram is shown in Fig.2.

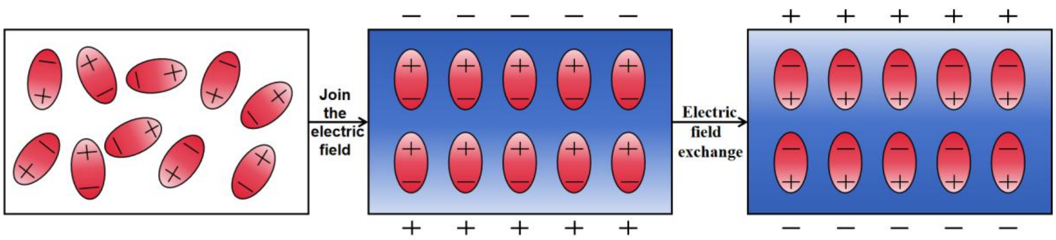


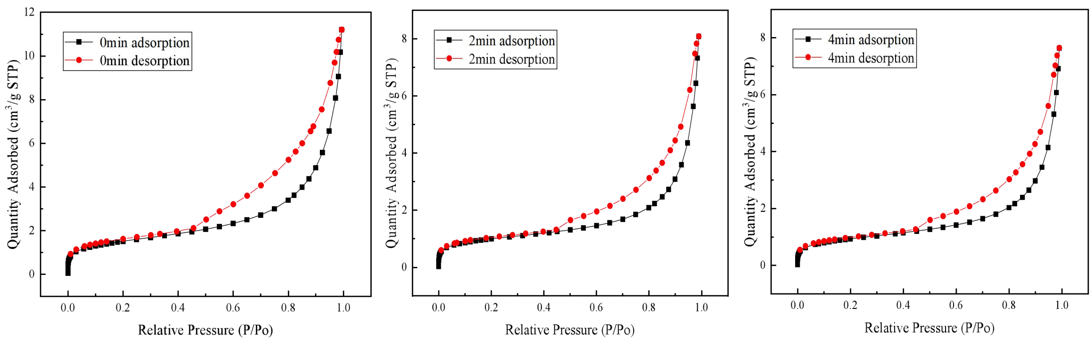
Fig.2 Principle of microwave heating

2 Results and analysis

2.1 Effect of microwave irradiation on adsorption isotherms and adsorption capacity

The isothermal adsorption curve of coal samples is of great significance for studying the adsorption characteristics of coal samples. The complexity of the pore structure of coal samples makes the isothermal absorption curve of coal samples not only follow an adsorption model, but also generally show a complex morphology, that is, a combination curve composed of various standard isotherms.

The N_2 adsorption isotherm curves of coal samples under different microwave irradiation time are shown in figure 3. According to the IUPAC isotherm classification method[28], the nitrogen adsorption isotherm curves of Liuzu coal samples all show the characteristics of type I(a) and type II isotherms, and the type I(a) isotherm is the main type, indicating that in the lower pressure range, with the occurrence of micropore filling, the adsorption capacity increases rapidly and forms an obvious adsorption platform. However, when in the higher pressure range, the growth rate of adsorption capacity began to slow down and remained at a stable growth rate. When the saturated vapor pressure is quickly reached, due to the gap between the particles, the large pores in the coal sample will adsorb, so that the adsorption capacity increases rapidly. The experimental results show that under the action of microwave, the pore size, shape and distribution of coal change.



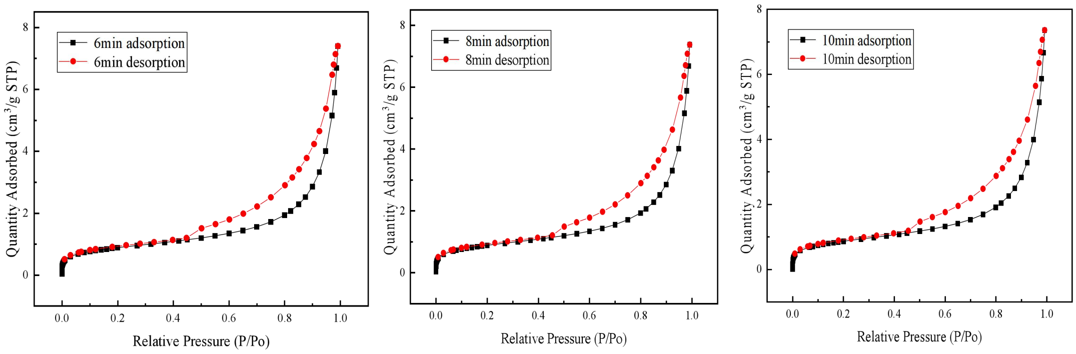
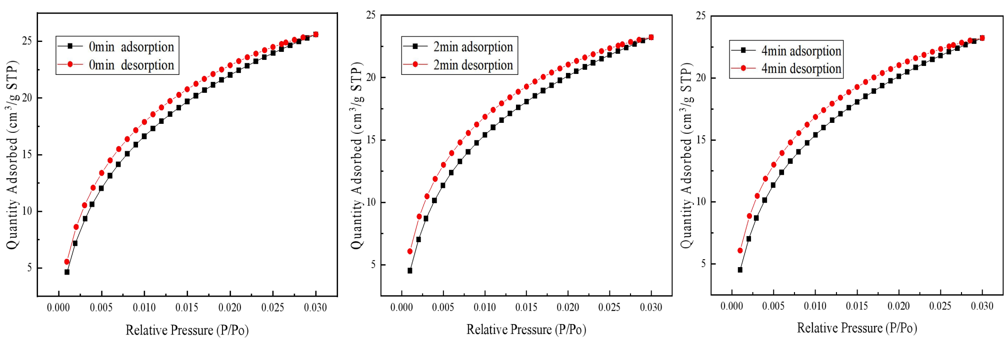


Fig.3 Grlots of low temperature N_2 adsorption / desorption of lignite at different microwave irradiation times

Fig.4 is the CO_2 adsorption isotherm curve of coal samples under different microwave irradiation time. It can be seen from the adsorption isotherm that the adsorption capacity of coal samples is positively correlated with the relative pressure of adsorption. Especially when the relative pressure is in the range of 10^{-4}-0.025, the adsorption capacity increases most obviously, which indicates that the pressure range is the best adsorption range of CO_2, corresponding to the micropore filling stage of coal samples.

After comparing and analyzing the 10 groups of adsorption isotherm curves, the results show that the coal samples treated by microwave irradiation and the untreated coal samples show the same trend on the adsorption isotherm curve, and the overall shape of the adsorption line is not significantly different. This shows that microwave irradiation does not change the primary pore type of coal samples. However, with the extension of microwave irradiation time, the adsorption capacity of coal samples changed significantly when reaching the maximum equilibrium pressure. This phenomenon shows that microwave irradiation mainly affects the pore structure of coal samples, which is manifested in the change of pore connectivity and pore size distribution. Microwave irradiation may change the connectivity between pores, making some pore channels more unobstructed or more closed, and the pore size distribution may also be adjusted, such as the decrease in the number of micropores and the increase in the number of mesopores and macropores. These structural changes eventually lead to the difference in the adsorption capacity of coal samples. Under the same equilibrium pressure, the adsorption capacity of coal samples treated by microwave irradiation is lower than that of 0 min coal samples.



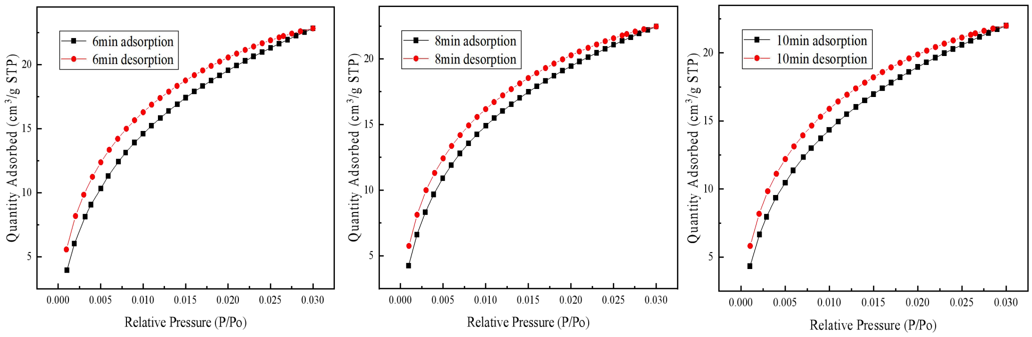


Fig.4 Grlots of low temperature CO_2 adsorption / desorption of lignite at different microwave irradiation times

In the process of microwave irradiation, due to the microwave energy, the molecular oscillation in the coal body is enhanced, the heat energy is generated, and the structure of the coal body is changed, which affects the pore distribution of the coal body, and then affects its adsorption capacity. Fig.5 is based on the LPGA-N_2 and LPGA-CO_2 experiments, the maximum adsorption capacity after microwave irradiation changes with the irradiation time curve：

(1) he experimental results show that in the different time range of microwave field action, compared with 0 min coal sample, the decreasing trend of the adsorption capacity of CO_2 and N_2 by coal sample after microwave field action is basically the same. The adsorption capacity of CO_2 and N_2 by coal sample after microwave field action is smaller than that of coal sample before microwave field action, which means that microwave field can effectively inhibit the adsorption performance of coal sample.

(2) The adsorption capacity of coal to CO_2 and N_2 decreases linearly with the increase of microwave field action time. However, due to the complexity of pore structure of coal samples, there are significant differences in the ability to adsorb N_2 and CO_2. The specific performance is that the adsorption capacity of coal samples to N_2 and CO_2 varies greatly with the same irradiation time, and the adsorption capacity of CO_2 is 1.9-4 times that of N_2. The effect of microwave field on the adsorption capacity of the two gases was the most significant in 10 min. At this time, the reduction of CO_2 adsorption decreased by 3.62cm^3/g (STP), a decrease of 14.13%. At this time, the reduction of N_2 adsorption decreased by 3.85cm^3/g(STP), a decrease of 34.34%, it can be seen that the adsorption capacity of coal samples also decreases with the increase of microwave action time, which further proves that the microwave field will inhibit the adsorption of coal samples.

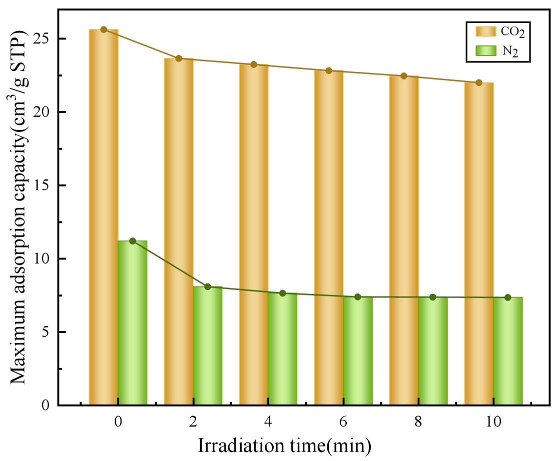


Fig.5 Change curve of the maximum adsorption volume at different times of microwave irradiation

2.2 Effect of microwave irradiation on pore volume and specific surface area

The pore classification method proposed by IUPAC[28] : micropores (50nm). It has been found that pore size can reflect the pore volume distribution characteristics of coal samples, including micropores, mesopores and macropores. The nitrogen adsorption experiment can not accurately determine the pore size less than 2nm, and the CO_2 molecule can penetrate into the micropores of coal samples because of its small size. Therefore, the CO2 adsorption experiment mainly reflects the transformation of micropores, which can accurately describe the micropores less than 2nm compared with the N_2 adsorption experiment. Therefore, this paper uses LPGA-N2 experimental data to characterize meso / macropores, and uses LPGA-CO_2 experimental data to characterize micropores. Combined with nonlinear density functional theory (NLDFT model) calculation, the total pore volume and total specific surface area of meso / macropores and micropores of coal with irradiation time are obtained.

According to Figure 6 and Figure 7, in the N_2 adsorption experiment results, with the increase of microwave irradiation time, the pore volume of the meso/macropores of the coal sample increases linearly, while the specific surface area decreases in a power function trend, and the change rate slows down with the increase of time ; in the results of CO_2 adsorption experiments, the pore volume and specific surface area of coal samples decreased linearly with the increase of irradiation time. The R^2 values of the pore volume and specific surface area fitting curves of micropores, mesopores and macropores under the results of the two adsorption experiments were all above 0.95, with a high degree of fitting.

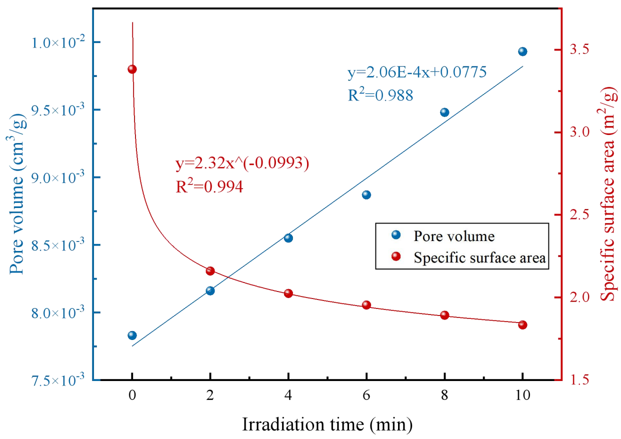


Fig.6 The specific surface area and pore volume of LPGA-N_2 changed with irradiation time.

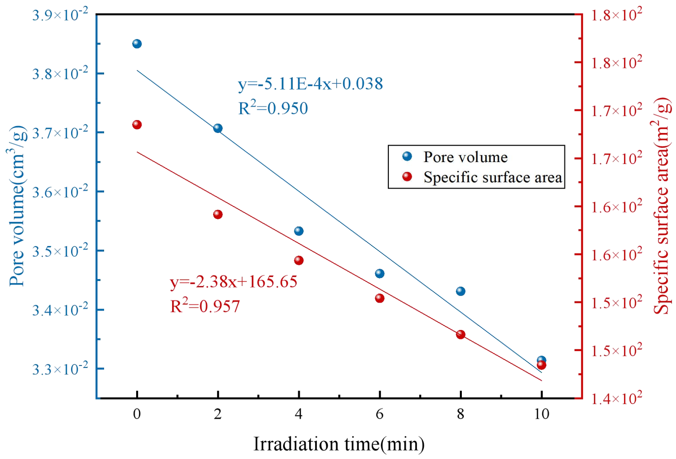


Fig.7 The specific surface area and pore volume of LPGA-CO_2 changed with irradiation time.

The specific surface area is one of the important indexes to measure the adsorption performance of coal samples, which is usually expressed by the pore area per unit area. Because the active sites adsorbed by coal samples are mainly concentrated on the surface, the larger the specific surface area of coal samples, the more active sites, the better the adsorption effect, the stronger the adsorption capacity, and the weaker the adsorption capacity. From Figure 6 and Figure 7, it can be seen that with the increase of microwave irradiation time, the pore specific surface area of coal samples in each stage decreases, indicating that the adsorption capacity of coal samples gradually decreases under the action of microwave field. Due to the large diameter of coal meso / macropores, it can be used as a free gas seepage channel. The increase of meso / macropore volume makes gas easier to diffuse, which further confirms that microwave has an inhibitory effect on the adsorption performance of coal samples and promotes their desorption.

2.3 LPGA-CO_2-N_2 combined characterization of pore parameters in the whole pore section

Combining the results of low-temperature CO_2 adsorption experiment with the results of low-temperature N_2 adsorption experiment, as shown in Table 2, the comprehensive characterization of the pore volume and pore specific surface area of the whole pore section of the coal sample is realized.

Table2 Joint characterization of whole-well pore parameters of LPGA-CO_2-N_2

Irradiation timemin	Total pore volume (cm^3/g)	Stage pore volume(cm^3/g)		Proportion(%)		Total specific surface area(m^2/g)	Stage specific surface area(m^2/g)		Proportion(%)	
		V_1	V_2	n_1	n_2		S_1	S_2	n_1	n_2
0	0.04633	0.0385	0.00783	83.10	16.90	171.873	168.492	3.381	98.03	1.97
2	0.04523	0.03707	0.00816	81.96	18.04	161.305	159.145	2.16	98.66	1.34
4	0.04388	0.03533	0.00855	80.52	19.48	156.381	154.357	2.024	98.71	1.29
6	0.04348	0.03461	0.00887	79.60	20.40	152.37	150.416	1.954	98.72	1.28
8	0.04379	0.03431	0.00948	78.35	21.65	148.529	146.637	1.892	98.73	1.27
10	0.04307	0.03314	0.00993	76.94	23.06	145.283	143.449	1.834	98.74	1.26

(Note : V_1 and S_1 are the pore volume and specific surface area measured by LPGA-CO_2, V_2 and S_2 are the pore volume and specific surface area measured by LPGA-N_2, n_1 and n_2 are the proportion of pore volume and specific surface area in different stages.)

It can be seen from Table 2 that with the increase of microwave irradiation time, the total pore volume and total specific surface area of coal samples show a downward trend. Among them, the proportion of micropore pore volume gradually decreased from 83.1 % to 76.94 %, and the proportion of meso / macropore pore volume gradually increased from 16.9 % to 23.06 %. With the increase of microwave irradiation time, the proportion of micropore specific surface area of coal samples increased from 98.03 % to 98.74 %, and the proportion of meso / macropore specific surface area decreased from 1.97 % to 1.26 %.

This is because under the action of microwave field irradiation, the polar molecules inside the coal sample move violently, causing a thermal effect, which promotes the gas produced by the decomposition of volatiles and functional groups in the coal matrix to escape from the pores. This process makes the pore surface of the coal sample tend to be smooth, reducing the complexity and number of micropores. At the same time, the microwave energy causes the collapse of the micropore structure, and the cementing material generated inside the coal body returns, which in turn seals or blocks the pores, resulting in a decrease in the proportion of micropore volume in the coal body. However, with the extension of microwave irradiation time, the temperature of the coal sample continues to rise, which promotes the removal of water inside the coal body, because water usually occupies part of the pore volume. During the removal process, the pores will be eroded and the pore connectivity will be improved, resulting in an increase in the proportion of mesopore / macropore volume. In addition, microwave energy may cause new micro-cracks in coal samples, resulting in an increase in the proportion of micropore specific surface area.

According to the data of table 2, the micropore volume of coal sample accounts for more than 76 %, and the specific surface area accounts for more than 98 %, which indicates that micropores dominate the adsorption process of coal sample. With the extension of microwave irradiation time, the pore volume of micropores decreases, which leads to the gradual decrease of adsorption capacity of coal samples, thus further verifying the negative effect of microwave irradiation on the adsorption performance of coal samples.

3 Conclusion

In this paper, through nitrogen adsorption and carbon dioxide adsorption experiments, the adsorption properties and pore structure characteristics of coal samples before and after microwave irradiation experiments were studied, and the effects of microwave action on the adsorption capacity, pore volume and specific surface area of coal samples were analyzed. The main conclusions are as follows :

(1) There was no significant difference in the shape of nitrogen adsorption / desorption isotherms and carbon dioxide adsorption / desorption isotherms of coal samples before and after microwave irradiation. The main effect of microwave action on the adsorption performance of coal was that the adsorption capacity decreased with the increase of action time. The adsorption capacity of coal to CO_2 and N_2 decreased linearly with the increase of microwave field action time. However, due to the complexity of the pore structure of coal samples, there was a significant difference in the adsorption capacity of coal samples when adsorbing N_2 and CO_2. The specific performance was that after 10 min of microwave irradiation, the adsorption capacity of CO_2 decreased by 14.13 %, while the adsorption capacity of N_2 decreased by 34.34 %.

(2) Based on the LPGA-N_2 and LPGA-CO_2 experiments, combined with the NLDFT model, it was found that due to the thermal effect of microwave energy and microwave irradiation, the pore structure of the coal sample collapsed and the pores expanded, so that the micropore volume and specific surface area of the coal sample decreased linearly. The pore volume of the meso / macropores of the coal sample increased linearly, while the specific surface area decreased in a power function trend and slowed down with time.

(3) Under the action of microwave field, the decomposition of volatiles and some functional groups in coal makes the coal sample show a tendency to be smooth, and the microwave energy causes the micropore structure to collapse, resulting in a gradual decrease in the proportion of micropore volume to 76.94 %. In the process of removing water from coal by microwave thermal effect, water vapor erosion holes promote the increase of connectivity of coal samples, so that the proportion of meso / macropore volume of coal samples gradually increases to 23.06 %. In addition, microwave energy leads to the formation of new microcracks in coal samples, which leads to an increase in the proportion of micropore specific surface area to 98.74 % and a decrease in the proportion of meso / macropore specific surface area to 1.26 %.

Data availability

All data supporting the findings of this study are available from the corresponding author Yuxing Zhang upon request.

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Author contributions

The data were analyzed by Z.D.and H.L., G.D., Z.Y. analyzed the images and wrote the manuscript.

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Competing interests

The authors declare no competing interests.

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