Experimental Research on the Influence of Microwave Radiation on Coal Permeability and Microstructure

Zhijun Wang,*, Xuelong Li,*, Xin Gao, Deyou Chen, and Zhiguan Zhu

ABSTRACT: At present, there is limited information available about the effects of microwave radiation on desorption characteristics, microstructures, and functional groups of coal. This research focuses on the influence of microwave radiation on coal sample desorption and examines the changes in pore structures and oxygenic groups of different coal samples using liquid nitrogen adsorption, nuclear magnetic resonance, and X-ray photoelectron spectroscopy. Results prove that the methane desorption capacity and desorption rate are proportional to the increase in microwave energy; the initial dynamic diffusion coefficient is also proportional to microwave energy but negatively proportional to the attenuation coefficient. As a result of microwave radiation, the Brunauer–Emmett–Teller (BET) surface area, pore size, and Barret–Joyner–Halenda (BJH) pore volume decreased. The specific surface area of BET decreased and then increased as microwave energy increased, while the average pore size increased and then decreased. However, the change in the BJH cumulative adsorption pore volume was complicated. The microwave radiation decreases the volume and number of micropores while increasing the volume and number of medium pores. With the increase in microwave energy, the number and volume of micropores continue to decrease, while the number and volume of medium pores continue to increase. An increase in microwave energy increased the surface area of oxygenic groups with the increasing relative content of COO\(^-\), C\(=\)O, and C\(\equiv\)O bonds; however, the relative content of C\(=\)C/C\(=\)H bonds decreased. These findings deepen the understanding of the antireflection effects of microwaves on coal.

1. INTRODUCTION

Coalbed methane (CBM) is a gas that coexists with coal in most deposits. It is a clean and valuable energy source. It can be used as an alternative energy source to coal, oil, and natural gas. Unfortunately, it poses a hazard to coal mine safety. It can cause disasters such as gas explosions and coal and gas outbursts during coal mining and seriously affect the safety of coal mines.\(^1\) In addition, methane is also a potent greenhouse gas if it is emitted into the atmosphere during mining. This not only wastes energy resources but also pollutes the atmospheric environment. Therefore, CBM extraction and utilization before mining can have a dual benefit by transforming a mine safety hazard into a source of clean energy. In China, most coal seams that are likely to be explored for CBM have strong adsorption and low permeability, which makes efficient gas extraction difficult before mining.\(^2\) Coal is porous, with many pores and cracks, which accounts for its high adsorption capacity. In the coal seam, adsorbed gases account for approximately 80–90% of the gas content, and it is very hard to desorb gas during coal seam drainage.\(^3\) Therefore, enhancing CBM extraction provides possible solutions to solve the gas problem in underground coal mines.

Previous studies have made remarkable achievements in stimulation technologies for gas extraction. The prominent technologies include deep hole presplitting and blasting,\(^4\) hydraulic punching,\(^5\) cavitation and fracture-adding,\(^6\) hydraulic fracturing, hydraulic cutting,\(^7\) physicochemical method,\(^8\) gas injection displacement,\(^9\) heat injection,\(^10\) and physical field excitation.\(^11\) Due to the limitation of technology and the difference in coal seam conditions, these technologies still have some defects and cannot be popularized comprehensively. In recent years, studies have found that external fields, such as sound fields and electromagnetic fields,\(^12\)–\(^14\) can affect gas adsorption and desorption to increase gas production. According to Jiang et al., sound waves significantly increase production and methane desorption is increased as well.\(^12\) Lu

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et al. found that coal seams’ desorption and seepage are enhanced by cavitation water jet acoustic shock, which can effectively increase the desorption speed and desorption volume. He and Zhang found that low-frequency electromagnetic fields reduce the amount of methane adsorption and promote methane desorption.

A microwave is a special electromagnetic wave with high frequency and high permeability. It is less affected by the geological condition and is capable of distributing heat through a large volume. Compared with conductive heating, the microwave has many advantages such as the following: (1) it uses volumetric heating (it does not rely on conduction to heat the material); (2) it is noncontact, rapid, and efficient; (3) it heats selectively; (4) it is easy to operate and starts and stops the heating cycle faster than any other heating system; and (5) it provides a safer method than conventional heating in part because microwave heating can be automated. Further, the microwave offers good environmental protection by utilizing electrical energy that does not pollute the environment. Microwaves are usually generated and transmitted in enclosed spaces and waveguides without emitting harmful gases. A microwave heating is not detrimental to the material being processed and waveguides without emitting harmful gases. A microwave heating is not detrimental to the material being heated without requiring a special heating body. Therefore, microwave heating is a healthy, environmentally friendly, safe, and reliable advanced technology. With these unique properties, the microwave has been applied in coal processing operations such as cleaning, drying/dewatering, coking, floating, grinding, and desulfurization.

The use of microwaves for recovering CBM has recently drawn some attention. Scholars studied the microwave’s effect on coal by two means, numerical simulations or experimental studies. For numerical simulations, Huang et al.,15,16 Li et al.,17,18 Hong et al.,19,20 Lin et al.,21 Su et al.,22 Xue et al.,23 and Lan et al.24,25 carried out simulations of the heating behavior of coal under microwave using COMSOL Multiphysics. Multiscale physics and reported that microwaves can rapidly heat coal. For experimental studies, the effect of microwave on coal microstructures was investigated by comparing the pore structure or fracture before and after microwave using one or more experimental methods. The methods in determining pore structure in coal include nuclear magnetic resonance (NMR), mercury intrusion porosimetry (MIP), and N2/CO2 adsorption experiments. Among the methods, NMR is the most widely used method. Yao et al.,26 Hong et al.,27,28 Xue et al.,29 and Lan et al.30 analyzed the pore structure of coal before and after microwave by NMR. Hong et al.31 studied the pore structure of coal before and after microwave by MIP.32 The methods in investigating the fracture in coal include X-ray computed tomography (CT), scanning electron microscopy (SEM), X-ray diffraction (XRD), and ultrasonic methods. Zhang et al.,33 and Kumar et al.34 analyzed the fracture change in coal before and after microwave by CT. Li et al.35 studied the fracture in coal before and after microwave by SEM and the ultrasonic method.36 Hong et al.37 evaluated the crack propagation in coal subjected to microwave energy using camera pictures and the ultrasonic method.38 Some scholars studied the pore structure and fracture in coal before and after microwave by two or more methods, for example, Huang et al.39 used NMR and SEM;40 Li et al. used NMR, CT, and P-wave;41,42 Liu et al.,43 Hu et al.,44 and Wang et al. used N2 adsorption experiments and SEM;45 Hu et al. used N2 adsorption experiments and MIP.46 Fu et al. studied the influences of microwave field irradiation on the physiochemical property and methane adsorption and desorption capability of coals by CO2 adsorption, N2 adsorption, XPS, and high-pressure CH4 adsorption.47 Lu et al.48 analyzed microwave-induced microstructure evolution of coal and its effects on the methane adsorption characteristic by Fourier transform infrared (FTIR) spectroscopy, N2 adsorption, SEM, and high-pressure CH4 adsorption.49 Furthermore, Xu et al.,50 Hong et al., and Lan et al. investigated the temperature-rising characteristics of coal by experimental studies.51,52,53

The changes of the pore structure, fracture, and temperature of coal after microwave were well studied. Pores and fractures play controlling roles on CBM migration, while the temperature is an important factor affecting the change of pores and fractures. Almost all the previous studies focused on the microwave’s effect on coal’s physical and petrographic characteristics, while few experiments have been conducted to investigate the direct effect of microwave on methane migration in coal. As 80–90% of methane in original coal seams is absorbed on the surface of micropores and internality of micrograins, the desorption process of methane from the adsorption state to the free state plays a decisive role in the CBM recovery. Therefore, the investigation of desorption characteristic variation before and after microwave can more intuitively show microwave’s effect on CBM recovery. Additionally, the surface chemical properties of coal affect its adsorption and desorption capability. There are many oxygenic groups in the surface structure of coal. In laboratory studies, oxygenic groups have been shown to have a negative effect on the adsorption of CH4 on coal.54 Coal with more oxygenic groups will have a weaker ability to adsorb methane.55 Few studies focused on the microwave’s effect on oxygenic groups in coal. Most of the scholars studied the pore structure and fracture in coal subjected to microwave by only one method, while few scholars used two or more methods. As each experimental method has limitations, the lack of methods makes it difficult to figure out the accuracy of experimental results.

To fill the research gap, this paper comprehensively studied the effects of microwaves on adsorption and desorption characteristics, desorption kinetics, microstructures, and surface oxygenic groups of anthracite with high adsorption and low permeability. To avoid the shortcomings of using a single method, three experimental methods are used in this work, liquid nitrogen adsorption (LNA), NMR, and XPS. LNA is a common way to test a coal’s pore structure, but its testing range is limited and it can only evaluate the connectivity of micropores and mesopores. NMR has the most extensive range. While the original pore structure of the coal sample is retained with the NMR test, the NMR test does not reveal the pore morphology distribution of the sample, which is precisely what the LNA does. LNA and NMR can complement each other. XPS is an important method for surface composition analysis. The combination of the three experimental methods can make up for each other’s shortcomings and verify each other’s experimental results and improve the accuracy of the experimental results.

2. EXPERIMENTAL RESULTS

2.1. Desorption Characteristics of Coal Samples Treated by Microwave. Figure 1 shows a plot of the desorption amount as a function of time. It is evident from Figure 1 that a greater cumulative amount of methane has been desorbed under the action of microwaves. The cumulative
The desorption amount increases with the increase in microwave energy. In the absence of microwaves, the final desorption amount was 288.60 mL. A microwave power of 180 W brought a final desorption amount of 342 mL, and the methane desorption amount was increased by 18.5% compared to those without the microwave. After the application of 360 W of microwave, the final desorption amount was found to be 374 mL, and the corresponding methane desorption amount was increased by 29.60%. Increasing after the microwave power to 540 W yielded a final desorption amount of 386 mL and a 33.75% rise in methane desorption. The desorption of methane from coal appeared to be significantly influenced by methane. The methane desorption capacity of coal samples continued to increase with the use of microwave energy.

### 2.2. Kinetics Analysis of Methane Desorption from Coal Treated by Microwave

In order to further understand the effects of microwave radiation on the desorption characteristics of coal samples, the dynamic diffusion coefficient model was used to assess the kinetics of methane desorption from coal.

#### 2.2.1. Dynamic Diffusion Model

The dynamic diffusion coefficient model is used to analyze the kinetics of methane desorption from coal after application of microwave radiation:

$$\frac{Q_t}{Q_\infty} = 1 - \sum_{n=1}^{\infty} \frac{1}{(n^2 \pi^2 D_0 r_0^2 \beta)} e^{-n^2 \pi^2 D_0 r_0^2 (1 - e^{-\beta})}$$

where $r_0$ is 0.0375 cm, $D_0$ is the initial diffusion coefficient at $t = 0$, $\beta$ is the attenuation coefficient of the dynamic diffusion coefficient, and the limit desorption quantity $Q_\infty$ is equal to the difference between the adsorption quantity $Q_p$ at the adsorption equilibrium pressure of 0.9 MPa and the adsorption quantity $Q_a$ at an atmospheric pressure of 0.09923 MPa as given below:

![Figure 1. Accumulative desorption amount of methane from Jiulishan coal sample.](image1)

![Figure 2. Regression curves based on the dynamic diffusion models for experimental data under different microwave radiation powers: (a) raw coal (without microwave), (b) 180 W microwave, (c) 360 W microwave, and (d) 540 W microwave.](image2)
\[ Q_{\infty} = Q_f - Q_a \]  
\[ Q_f \text{ and } Q_a \text{ can be calculated by} \]
\[ Q = \frac{abp}{1 + bp} \frac{100 - A_{ad} - M_{ad}}{100(1 + 0.31M_{ad})} + \frac{10bp273}{\rho(273 + t_w)} \]  
where \( Q \) is the methane adsorption quantity at a specific temperature and pressure, \( \text{cm}^3/\text{g} \); \( a \) is the Langmuir volume, \( \text{cm}^3/\text{g} \); \( b \) is the reciprocal of Langmuir pressure, \( \text{MPa}^{-1} \); \( p \) is the methane adsorption equilibrium pressure, \( \text{MPa} \); \( A_{ad} \) is the ash content of coal, \( \% \); \( M_{ad} \) is the moisture content, \( \% \); \( \rho \) is the porosity, \( \% \); \( \phi \) is the apparent relative density, \( \text{g}/\text{cm}^3 \); and \( t_w \) is the saturated saltwater temperature, \( ^\circ\text{C} \).

2.2.2. Kinetics of Methane Desorption after Microwave Radiation. Combined with the experimental results pertaining to the desorption amount, desorption parameters in the dynamic diffusion models are obtained using nonlinear regression. The curves generated by these regressions and the corresponding experimental data are shown in Figure 2. The diffusion coefficients and the correlation coefficients for these curves are provided in Table 1.

| coal samples  | \( D_0 \) (10^{-6}\text{cm}^2s^{-1}) | \( \beta \) (s^{-1}) | correlation coefficient |
|-------------|----------------|----------------|-----------------------|
| raw coal     | 1.96823248     | 0.01405391     | 0.993858              |
| 180 W microwave | 2.51339957 | 0.01019804 | 0.998975              |
| 360 W microwave | 3.07754357 | 0.00732768 | 0.999524              |
| 540 W microwave | 4.85897013 | 0.00545334 | 0.998908              |

Figure 2 shows the variation of \( Q_f/Q_{\infty} \), which is the percentage of total methane adsorbed to microwave power levels that was desorbed. In Figure 2, it is shown that the total amount of adsorbed methane after microwave radiation application is higher than that of raw coal at all times and gradually increases with the amount of microwave power used. The percentage of total adsorbed methane of raw coal after 2 h reached 22.22%, while the percentages of total adsorbed methane after 180, 360, and 540 W microwave reached 24.22, 30.43, and 39.41%, respectively. It shows that methane in the coal sample is easily desorbed by microwave radiation, and thereafter, methane in the coal sample will further be released. Using the data in Table 1, the diffusion coefficient—time curves of the desorbed methane are drawn, as shown in Figure 3.

It can be found from Table 1 and Figure 3 that \( D_0 \) approaches higher values after the application of microwave radiation compared to raw coal. A microwave power leads to a greater value of \( D_0 \). The \( D_0 \) of raw coal is 1.96823248 \( \times 10^{-7} \text{cm}^2\text{s}^{-1} \), while the \( D_0 \) for microwave powers of 180, 360, and 540 W is increased by 1.27, 1.56, and 2.47 times, respectively. \( \beta \) describes the degree of attenuation of a dynamic diffusion coefficient. A greater value of \( \beta \) indicates a larger amount of attenuation and vice versa. In this study, \( \beta \) has positive values, reflecting the fact that the diffusion rate of coal samples decreased with time. It was found that microwave radiation reduced the value of \( \beta \), which continues to decline with an increasing microwave power, resulting in a larger dynamic diffusion coefficient.

In summary, the \( D_0 \) of the coal sample increases and the \( \beta \) decreases after the application of microwave radiation. With the increase in microwave power, the \( D_0 \) gradually increases, and the \( \beta \) gradually decreases. It shows that microwave radiation can promote the desorption of methane in coal, and the final desorption amount of methane increases with the increase in microwave power.

3. DISCUSSION

3.1. Pore Structure Characteristics of Coal Samples Treated by Microwave. The BET surface area has a significant effect on the macroscopic properties of coal. A fitting calculation is performed on coal samples with different microwave power levels, and its results are presented in Table 2.

| coal samples  | BET surface area (s/m^2g^-1) | linear fitting degree |
|-------------|----------------|-----------------------|
| raw coal     | 0.306954      | 0.999999              |
| 180 W microwave | 0.173491     | 0.999124              |
| 360 W microwave | 0.096194     | 0.999734              |
| 540 W microwave | 0.145494     | 0.999893              |

It can be seen that the linear fitting degree of the BET specific surface area of coal samples is higher than 0.99, indicating that the surface area could be reasonably predicted from experiments. The BET surface area of coal samples after microwave treatment was lower than that of raw coal, and as the microwave energy was increased, the BET surface area of coal samples first decreased and then increased. The BET surface area of raw coal is 0.306954 s/m^2g^-1; however, it decreased by 43.48, 69.64, and 52.6% after 180, 360, and 540 W microwave treatments, respectively. It indicates that microwave radiation can change the pore structure of the coal sample surface. A decrease in the BET specific area leads to a decrease in methane adsorption capacity. In addition, in the LNA tests, the BJH (Barrett–Joyner–Halenda) cumulative adsorption volume and average pore size effectively represent the macroscopic properties of coal samples, as shown in Table 3.

Table 3 shows that the BJH cumulative adsorption pore volume and average pore diameter of coal samples after microwave treatment are lower than those of raw coal. It appears that coal samples’ adsorption decreases after the microwave treatment and methane in coal is easier to diffuse and be desorbed. With the increase in microwave power, the average pore size of coal samples first increases and then decreases.
than 100 nm. Since coal samples have a significant impact on a large pore test, granular coal samples were used in the experiments. Table 4 shows the $T_2$ distribution of coal samples.

The $T_2$ distribution mainly reflects the pore size distribution and the pore volume of coal samples. Analyzing changes in $T_2$ distribution can reveal the pore size distribution of coal samples. The transverse relaxation time $T_2$ depends on the size of the pores. Generally, the longer transverse relaxation time $T_2$ of coal indicates larger pores; therefore, different pore sizes correspond to different transverse relaxation times. The number of pores and the corresponding volume are represented by the peak and area distribution of the $T_2$ spectrum, respectively. The main source of gas in coal and rock mass lies in coal holes and cracks. By using the $T_2$ spectrum area, the actual gas adsorption amount in coal can be determined, and the gas adsorption law in coal can be quantified.

It can be seen from Figure 5 and Table 4 that the peak value and area of the $T_2$ spectrum of micropores gradually increase from raw coal to 180 W microwave, 360 W microwave, and 540 W microwave. The peak and area of the $T_2$ spectrum of the mesopores gradually increase by lowering the microwave power from 540 W microwave to 360 W, 360 to 180 W, and 180 W to raw coal. It shows that microwave radiation can change the pore structure of the coal surface. In coal samples, microwave radiation decreases the number of micropores and the volume of the pore but increases the number of mesopores and the volume of the pore. The number and volume of micropores continue to decrease as the microwave power increases, while the number and volume of mesopores continue to increase. It indicates that the influence of microwave radiation on the pore structure of coal samples increases with the increase in microwave power. Further, the $T_2$ spectrum of coal samples gradually shifted to the right as the microwave energy increased, indicating that the minimum pore size of micropores gradually increased. It is believed that micropores are the main sites for methane adsorption. The results show that the methane adsorption capacity of coal samples after microwave treatment is reduced in comparison to raw coal. Coal samples continue to lose their ability to absorb methane as the microwave energy increases. As methane diffuses through mesopores, the increased number and area of mesopores after microwave radiation indicate the opening of methane diffusion channels. Hence, the methane diffusion and desorption capacity of coal samples increased after microwave radiation and will continue to increase with the increase in microwave energy.

### 3.3. XPS Characteristics of Coal Treated by Microwave

Figure 6 shows the XPS spectrum and peak fitting diagram of the C element in coal samples treated by microwave. Table 5 lists the peak area and relative content of C element functional groups in coal samples.

Figure 6 shows that the types of oxygenic groups on the surface of coal samples are different, as is the binding energy. The binding energies of the COO− bond, C−O bond, C==O

### Table 3. Cumulative Adsorption Pore Volume and Average Pore Size of BJH in Coal Samples

| coal samples   | BJH cumulative adsorption pore volume (cm$^3$) | average pore size (nm) |
|---------------|-----------------------------------------------|------------------------|
| raw coal      | 0.001306                                      | 14.420976              |
| 180 W microwave| 0.002266                                      | 39.654191              |
| 360 W microwave| 0.000783                                      | 18.024499              |
| 540 W microwave| 0.001043                                      | 16.450812              |

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### Table 4. Area of $T_2$ Distribution of Coal Samples

| coal samples   | area of first peak | area of second peak | area of third peak | gross area     |
|---------------|--------------------|--------------------|--------------------|---------------|
| raw coal      | 4278.871229        | 210.719588         | 8262.949207        | 12752.54002   |
| 180 W microwave| 3852.866163        | 236.366507         | 7915.649465        | 12004.88214   |
| 360 W microwave| 3686.898606        | 247.175019         | 3982.458393        | 7916.532018   |
| 540 W microwave| 3852.866163        | 236.366507         | 7915.649465        | 12004.88214   |

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The peak areas of the C−C/C−H bond are found to be 290.0, 285.9, 287.3, and 284.6 eV, respectively.

It can be observed from Table 5 that the surface oxygenic groups of coal samples are changed after the application of microwave radiation. With the increase in microwave energy, the total area of oxygenic groups of element C continues to increase as well as the relative content of oxygenic groups of the COO− bond, C−O bond, and C═O bonds, while the relative content of oxygenic groups of C−C/C−H bond decreases. By applying a 180 W microwave energy, the peak areas of oxygenic groups of the COO− bond, C−O bond, C═O bond, and C−C/C−H bond were changed from 910.26, 4146.77, 668.54, and 3640.17 to 2009.12, 5520.41, 1563.61, and 3924.5, respectively, compared with raw coal. Meanwhile, the total area was changed from 4212.94 to 4834.14 by the application of 180 W microwave compared to raw coal. The corresponding relative contents changed from 2.16, 9.84, 1.59, and 86.41% to 4.16, 11.42, 3.23, and 861.19%, respectively.

![Figure 6](https://doi.org/10.1021/acsomega.1c04291)  
Figure 6. XPS spectrograms of the C element in Jiulishan coal samples. (a) Raw coal, (b) 180 W microwave, (c) 360 W microwave, and (d) 540 W microwave.

Table 5. Peak Area and Relative Content of C Element Functional Groups in Coal Samples

| functional groups | raw coal (CPS. eV) | 180 W microwave (CPS. eV) | 360 W microwave (CPS. eV) | 540 W microwave (CPS. eV) |
|-------------------|--------------------|--------------------------|--------------------------|--------------------------|
| C−C/C−H peak area | 36407.17 ± 124.2   | 39248.5 ± 94.6           | 45345.25 ± 113.4         | 53776.75 ± 131.4         |
| C−C/C−H relative content (%) | 86.41 ± 4.2 | 81.19 ± 2.6 | 76.69 ± 1.8 | 73.21 ± 2.4 |
| C−O peak area (CPS.eV) | 4146.77 ± 32.5     | 5520.41 ± 42.6           | 7484.24 ± 59.5           | 9870.62 ± 62.8           |
| C−O relative content (%) | 9.84 ± 0.87       | 11.42 ± 1.5              | 12.66 ± 2.1              | 13.44 ± 1.9              |
| C═O peak area (CPS. eV) | 668.54 ± 17.6      | 1563.61 ± 36.4           | 2816.69 ± 39.5           | 4410.15 ± 45.8           |
| C═O relative content (%) | 1.59 ± 0.06       | 3.23 ± 0.14              | 4.76 ± 0.18              | 6.01 ± 0.21              |
| COO− peak area (CPS. eV) | 910.26 ± 19.6     | 2009.12 ± 53.6           | 3478.61 ± 69.3           | 5397.17 ± 74.8           |
| COO− relative content (%) | 2.16 ± 0.17       | 4.16 ± 0.25              | 5.89 ± 0.38              | 7.34 ± 0.87              |
| Total peak area (CPS.eV) | 42129.74 ± 278.6  | 48341.64 ± 318.4         | 59124.79 ± 539.6         | 73454.7 ± 683.9          |
indicates that microwave radiation breaks the C–H bond on the coal surface, and oxygen atoms replace hydrogen atoms to generate the COO− bond, C–O bond, and C=O bond.

The literature reveals that the amount of methane adsorbed on coal gradually decreases with increasing stacked layers of aromatic units. It was found that the adsorption characteristics of coal were different due to the different surface C−C/ C−H bonds. With the increase in relative content of C−C/C− H bonds on the coal surface, the saturated adsorption capacity and the adsorption constant of coal decrease. It can be seen that microwave radiation can inhibit the methane adsorption capacity of coal samples by changing the oxygenic groups on the coal surface. The methane adsorption capacity of coal samples decreases with an increase in microwave energy.

3.4. Modification of Coal Samples by Microwave. In a previous study, it was reported that microwave radiation could change the pore structure of the coal sample surface. After microwave treatment, the number and volume of micropores on the surface of coal samples decreased, the number and volume of mesopores increased, the total BET surface area decreased, the BJH adsorption pore volume decreased, and the average pore size increased. The results indicate that microwave exposure reduces the methane adsorption capacity of coal while increasing its diffusion desorption capacity. The porosity and permeability of the coal sample increase. As a result of microwave radiation, coal samples showed a change in oxygenic groups on their surface. The C−H bond on the surface of the coal sample was broken, resulting in COO−, C−O, and C=O bonds on the surface, leading to lower levels of the C−H bond on the surface and increased levels of the other three oxygenic groups. Consequently, coal samples lose methane adsorption ability after microwave radiation.

A microwave is a type of electromagnetic energy that creates oscillations in polar molecules to heat coal (such as water, inorganic polar molecules, and polar groups in macromolecular structures). This volumetric heating will produce a sharp thermal effect. In coal, selective microwave heating leads to thermal stress and detrimental effects. Under the action of a high-energy microwave, water vapors are formed by the vaporization of free water or bound water in the microstructure of coal. Vapor pressure opens and connects closed pores, which results in pores opening. A pore-dredging effect is caused by microwave-induced thermal decomposition of organic macromolecules and mineral removal from coal pores. In addition, the water adsorbed on the surface of the coal body is removed, resulting in shrinkage and deformation of the coal matrix. It leads to the collapse of pore channels and the formation of blockages. The effects of pore opening and pore dredging are of particular importance. The damaging effects of microwaves on coal do not disappear with the removal of the microwave field. It causes permanent changes in the pore structure of the coal, which affect the adsorption ability of coal as well as its permeability.

In summary, microwave radiation not only reduces the adsorption ability of coal but also improves the ability of methane diffusion and desorption. Moreover, it improves the connectivity of coal pores and increases the permeability of coal samples. Microwave technology can therefore be used to stimulate gas extraction in coal seams.

3.5. Potential Applications of the Microwave for Coalbed Gas Extraction. A conceptual design of a gas drainage system in a coal seam assisted by microwave radiation is proposed, as shown in Figure 7. The microwave is imported into the coal seam through the drilling antenna so that the microwave directly acts on the coal body. The microwave system used to stimulate coal seam gas extraction typically consists of the following components:

(1) Microwave heating system. It includes a microwave generator, microwave control system, microwave transmission line, drilling antenna, etc. Its main function is to produce microwave radiations and control the power and frequency of the output radiations.

(2) Temperature monitoring system. The system includes a temperature sensor, temperature alarm, and high-temperature automatic cutting system. The temperature monitoring system is designed to continuously monitor the coal seam temperature around the drilling antenna in real time. As soon as the coal seam temperature exceeds the preset limit, it will alarm and cut off the power supply so the microwave loading will cease.

(3) Gas monitoring system. The system mainly includes a gas concentration sensor and an alarm. This device monitors coal seam gas concentration in real time, triggering an alarm when levels exceed a specified limit.

(4) Gas drainage system. It mainly consists of a pump, pipe, borehole, and sealing device for gas extraction.

Coal seam gas extraction with a microwave radiation antireflection system ensures the microwave radiation antireflection in the real-time gas extraction process. This can greatly improve gas extraction efficiency and significantly reduce time spent on the process. Thus, coal mining enterprises can enjoy significant economic benefits from it.
The microwave system injects heat into the coal seam during the gas extraction. Under the action of microwave, the temperature of the coal body is increased, methane diffusion and desorption are greatly improved, and the internal gas seepage channel becomes smoother, thereby aiding in the extraction of natural gas. Microwave radiation from one borehole can cover two adjacent boreholes on either side, thus accelerating gas extraction from three boreholes. The use of a microwave system not only increases the gas extraction rate and reduces the gas extraction time significantly. Therefore, microwave systems have the potential to stimulate gas extraction.

4. CONCLUSIONS

As a result of microwave treatment, the desorption amount of methane in coal samples increased by 18.5, 29.60, and 33.75%, respectively, and the initial dynamic diffusion coefficient $D_0$ increased by 0.08, 0.59, and 1.47 times, respectively.

Coal samples treated with microwave radiation had a lower BET specific surface area, average pore size, and BJH cumulative adsorption pore volume than raw coal samples. In response to the increase in microwave energy, the BET specific surface area of coal samples progressively decreased and then increased. Also, the average pore size first increased and then decreased. As microwave energy increases, the peak value and peak area of micropore $T_2$ of coal samples continue to decrease, while the peak value and peak area of the mesopore $T_2$ spectrum continue to increase. The total area of oxygenic groups of the C element in coal samples increases continuously after the application of microwave irradiation. The relative content of C–C/C–H bonds on the coal surface decreases, while the relative content of COO–, C–O, and C=C–O bonds increases continuously. It indicates that microwave irradiation is capable of changing the oxygenic groups on the coal surface and inhibiting its ability to absorb methane. The three methods reveal that microwaves can inhibit methane adsorption and promote methane desorption from different aspects.

This paper presents a conceptual design for a gas drainage system assisted by microwave radiation in coal seams and examines the microwave system and its working principles.

5. EXPERIMENTAL SECTION

5.1. Sample Preparation. The coal sample used in this experiment was anthracite from Jiulishan Coal Mine in Henan Province, China. Fresh coal was collected underground and stored in the laboratory for the experimental investigation. After grinding, the coal samples with a particle size of 0.5–1 mm were screened out. The screened coal samples were put into a drying oven and processed at 105 ºC for 8 h, then taken out, and put in a dryer. The coal samples were then cooled to room temperature and sealed for use.

5.2. Experimental Procedures. Experimental procedures included degassing, adsorption, microwave radiation, desorption, and microstructure testing, as shown in Figure 8.

1. Degassing. Fifty grams of coal sample was weighed and placed in the adsorption and desorption tank on the experimental platform followed by degassing for 48 h using a vacuum pump.

2. Adsorption. Methane adsorption amount was measured by the manometric method. A defined amount of methane was successively transferred from the buffer tank into the adsorption and desorption tank containing the coal sample before obtaining the adsorption equilibrium. Prior to the adsorption experiment, the void volume ($V_0$) of the adsorption and desorption tank was determined by helium inflation. The adsorption amount was calculated using eq 4,

$$m_{\text{gas}} = \frac{VM}{ZRT}(P_{\text{equilibrium}} - P)$$

(4)

where $m$ is the mass of gas, $P$ is the pressure, $T$ is the temperature, $M$ is the molar mass of methane, $Z$ is the compressibility coefficient of methane, which is calculated by the Redlich–Kwong equation (when pressure is less than 9 MPa), and $R$ is the universal gas constant.

3. Microwave radiation. The adsorption equilibrium of coal samples was maintained for 5 min with microwave powers of 180, 360, and 540 W. After the treatment, the coal sample is kept for 48 h to bring the temperature of the coal sample to room temperature.

4. Desorption. To measure the amount of desorption gas, the exhaust valve was opened and let to run for 5 s. The data were recorded at a certain time interval to obtain the variation of desorption amount with time.

5. Microstructure testing. The microstructure test was then performed, including adsorption tests with liquid nitrogen, NMR tests, and XPS tests.

5.2.1. LNA Tests. A V-Sorb2800s pore structure analyzer was used for liquid nitrogen adsorption tests using the static capacity method. As an integrated and assembled system with a high vacuum, it is capable of automatic and intelligent...
measuring of specific surface areas. The instrument has the following specifications:

Vacuum limit: $4 \times 10^{-2}$ Pa (3 × 10$^{-4}$ Torr). Partial pressure range: $P/P_0$, the accurate controllable range of $5 \times 10^{-6}$–0.995. Measurement accuracy: 0.35–400 nm (average pore size), 0.01 (m$^2$/g)–no upper limit (specific surface area), measurement error: less than 1.5%. The number of samples tested simultaneously: two.

5.2.2. NMR Tests. The low-field NMR instrument model used in this experiment is the Meso MR23-060H-I, which is manufactured by the Shanghai Newmai Company. The parameters pertaining to the NMR test are as follows:

- RF pulse frequency (resonant frequency): 21.67568 MHz;
- Magnet temperature: 31 °C ± 0.1 °C;
- Magnetic field intensity: 0.5 ± 0.08 T.

The instrument is equipped with nuclear magnetic resonance imaging application software. The pulse sequence library is comprehensive, and the operation is simple and convenient. It can be used for the NMR transverse relaxation $T_2$ spectrum of coal samples, permeability, movable fluid saturation, porosity, pore size distribution, and other parameters.

5.2.3. XPS Tests. In the experiment, a Thermo Fisher Scientific ESCALAB Xi+ was used as an XPS device. The instrument includes a crystal cleaner, sample heating/cooling device, a high-pressure reaction chamber, a sample product stage, a sample scraper, and sputtering clean separation to satisfy analytical requirements for XPS and imaging. A macrofocus monochromator has the following analytical size: 20–900 μm, with a target working point of 20. The spatial resolution was higher than 1 μm, and the XPI imaging resolution was 1 μm.

5.3. Experimental Apparatus. 5.3.1. Microwave Devices.

The microwave generator is a G90F23CN3PV-BM1 (G1) microwave oven that operates at a frequency of 2450 MHz and can be controlled according to the user’s needs. Microwave has a power rating of 900 W, which can be set to 20, 40, 60, 80, and 100% of the maximum power, i.e., 180, 360, 540, 720, and 900 W, respectively. The coal samples can be treated with different microwave powers by adjusting the setting parameters. In the experiment, the first three stages of the microwave oven were used to treat coal samples.

5.3.2. Experimental Apparatus for Adsorption and Desorption. According to the requirements of the experimental scheme, the experimental platform of methane adsorption and desorption was designed, as shown in Figure 9. The device consisted of a gas supply device, a vacuum degassing device, adsorption and desorption tank, and a measurement device.

The gas supply device consisted of high-pressure cylinders, gas buffer tanks, pressure relief valves, and precision pressure gauges. The purity of methane gas in a high-pressure cylinder was 99.99%. The precision pressure gauge range was 6 MPa, the unit scale value was 0.02 MPa, and the precision was 0.25 MPa. Vacuum degassing devices include vacuum gauges and vacuum pump units. The vacuum pump used was a 2XZ-4 rotary vane vacuum pump, which generates a vacuum of up to $6 \times 10^{-2}$ Pa. The adsorption and desorption tank was made of Teflon to ensure that microwaves can penetrate the interior of the coal sample. The gas flow meter and gas chromatograph were used to measure the flow rate and constituents of desorbed gas, respectively.

Figure 9. Experimental device of gas desorption.

AUTHOR INFORMATION

Corresponding Authors
Zhijun Wang — State Key Laboratory Cultivation Base for Gas Geology and Gas Control, Henan Polytechnic University, Jiaozuo, Henan 454000, China; Collaborative Innovation Center of Central Plains Economic Region for Coalbed/Shale Gas, Henan Province, Jiaozuo, Henan 454000, China; orcid.org/0000-0002-5465-6932; Email: wzhj0537@163.com

Xuelong Li — College of Energy and Mining Engineering, Shandong University of Science and Technology, Qingdao, Shandong 266590, China; Mine Disaster Prevention and Control-Ministry of State Key Laboratory Breeding Base, Shandong University of Science and Technology, Qingdao, Shandong 266590, China; orcid.org/0000-0003-2037-2525; Email: lixlcumt@126.com

Authors
Xin Gao — College of Energy and Mining Engineering, Shandong University of Science and Technology, Qingdao, Shandong 266590, China
Deyou Chen — College of Energy and Mining Engineering, Shandong University of Science and Technology, Qingdao, Shandong 266590, China
Zhiguan Zhu — China Construction Fifth Engineering Division Henan Branch, Zhengzhou, Henan 450000, China

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.1c04291

Notes
The authors declare no competing financial interest.

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