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Impact of Microbially Enhanced Coalbed Methane on the Pore Structure of Coal

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Microbially enhanced coalbed methane (MECBM) has important theoretical and practical significance for reforming coal reservoir structure, alleviating greenhouse effects and energy crises and developing new sources of clean energy. In this study, No. 3 coal seams in Qinshui Basin were taken as research objects to analyze the pore structure characteristics after microbial treatment by means of low-temperature nitrogen adsorption (LTNA), mercury porosimetry (MP), and isothermal adsorption/desorption experiments. The results showed that after bioconversion, the specific surface area and pore volume increased from 1.79 m^2/g and 0.0018 cm^3/g to 4.01 m^2/g and 0.0051 cm^3/g respectively under liquid nitrogen testing; however, the specific surface area was reduced from 5.398 m^2/g to 5.246 m^2/g and the pore volume was increased from 0.053 cm^3/g to 0.0626 cm^3/g under MP. The fractal dimension based on the LTNA data indicated that the fractal dimension of micropores and minipores was increased from 2.73 to 2.60 to 2.89 and 2.81, however the fractal dimension of meso-macropores was decreased from 2.90 to 2.85. The volatile matter and fixed carbon were both reduced from 6.68% to 78.63%–5.09% to 75.63%, and the Langmuir volume and Langmuir pressure were increased from 34.84 cm^3/g and 2.73 MPa to 36.34 cm^3/g and 3.28 MPa, respectively. This result indicated that microorganism participated in the degradation of coal reservoir and promoted the production of methane gas, the meso-macropores were more obviously modified by microorganism, so that the pore diameter stabilized, the pores became smoother, the specific surface area decreased, and the pore volume increased. These are more beneficial to the adsorption and production of coalbed methane (CBM) after microbial treatment.

Keywords: microbially enhanced coalbed methane (MECBM), pore structure, fractal theory, coalbed methane, qinshui basin

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INTRODUCTION

Coal, as an important energy resource, has been widely used since the industrial revolution. Approximately 71.4% of global fossil fuel reserves are in the form of coal (Faison, 1991; Park and Liang, 2016). However, a series of severe environmental problems have resulted, such as global climate change, extreme weather and land desertification. These problems have seriously affected and restricted the sustainable development of humankind with the development and utilization of

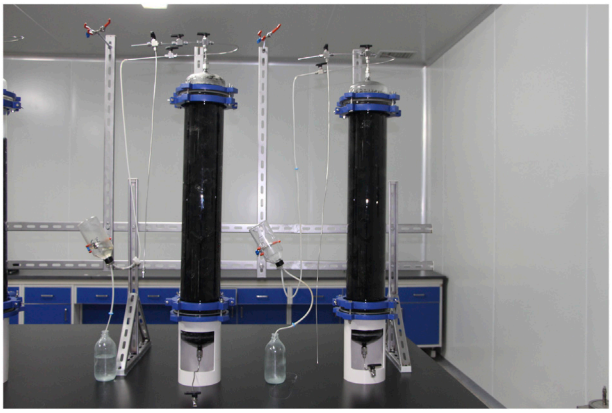

FIGURE 1 | Experimental devices used to test MECBM.

coal resources (Fakoussa and Hofrichter, 1999; Formolo et al., 2008; Byamba-Ochir et al., 2017; Su et al., 2021). Exploitation and utilization of coalbed methane (CBM) is of great significance to alleviate these problems. CBM is developed by way of two processes: thermogenic gasification and biogenetic gasification, so it is possible to obtain CBM in artificial conditions through microbial degradation (Fakoussa and Hofrichter, 1999; Park and Liang, 2016). Microbially enhanced coalbed methane (MECBM) has important theoretical and practical significance for reforming coal reservoir structure, alleviating greenhouse effect and energy crises and developing new sources of clean energy (Zhang et al., 2017; Lu et al., 2020; Hou et al., 2021). Meanwhile, the permeability of coal reservoir can be improved by using MECBM. This technique is believed to be related to low metamorphic coal, which is more conducive to microbial degradation (Harris et al., 2008; Robbins et al., 2016; Lu et al., 2020), but the bituminous-coal contents in some countries (such as the United States, Russia, and China) exceed 50% (Mcglade and Ekins, 2015; Lu et al., 2020). Especially in China, commercial CBM gas fields are mainly concentrated in anthracite coal mining areas. Therefore, MECBM research should not only focus on lignite, and but also highly metamorphosed bituminous coal, or even anthracite (Lu et al., 2020), many scholars have investigated this topic (Faiz and Hendry, 2006; Formolo et al., 2008; Boris et al., 2012; Bao et al., 2020; Zhao et al., 2020; Ashley et al., 2021; Guo et al., 2021). As a porous medium, the pores of coal are the main accumulation space and migration channels of CBM, and the pore structure not only affects the state of occurrence, adsorption/desorption, diffusion, seepage flow, gas content, but also restricts the ability to exploit CBM (Hudot, 1966; Clarkson et al., 2011; Moore, 2012; Gao et al., 2017; Chen et al., 2018), making it necessary to study the modification of coal pore structures by microorganisms.

MATERIALS AND METHODS

Coal Samples

The No. 3 coal seams of Early Permian Shanxi formation in Sihe mine, in the southern Qinshui basin of China were taken as research objects to study the pore structure characteristics after microbial treatment. These are typical primary mineable coal beds found in northern China. The study area lies in the southeast of Shanxi Province and forms a part to the western margin of North China Platform. The average thickness of No. 3 coal seam is about 6 m and $R_{o,\ Max.}$ is 2.54%. The research area is a focus of CBM research in China, and is the earliest and most successful commercialization area of the CBM industry in China. To maintain the contact between the coal and microorganisms as much as possible, while avoiding the channels becoming blocked by pulverized coal, the particle size of the granular coal specimens was about 20 × 20 mm and a coal sample of 18 kg was loaded into each experimental device. Proximate analysis, low-temperature nitrogen adsorption (LTNA), mercury porosimetry (MP) and isothermal adsorption measurements, and field emission scanning electron microscopy (FESEM) were applied to pre-test/post-test samples. Two sets of experimental devices were designed (**Figure 1**). The test data are summarized in **Table 1**.

Experimental Procedure

The MECBM treatments were performed using specially designed apparatus (**Figure 1**). The flora and experimental water were taken from CBM wells in the Sihe mine. The floras were cultured in the laboratory. To ensure the enrichment of methanogens in water samples, fluorescence observation and counting were performed before the experiment under a fluorescence microscope with 420 nm incident light. Preparation of 10 L of substrate was carried out, and the components mainly included glucose, peptone, sodium chloride, beef extract, L-cysteine hydrochloride, resazurin, sodium

TABLE 1 | Coal sample data.

Sample ID	M_{ad} (%)	A_d (%)	V_{daf} (%)	FC_{ad} (%)	Clay minerals	BET surface area m²/g, LTNA)	Pore volume cm³/g, LTNA)	Total pore area m²/g, MP)	Total intrusion volume cm³/g, MP)	Langmuir volume V_L/ (cm³/g)	Langmuirpressure P_L/MPa
Pretest	2.99	13.13	6.68	78.63	4.13	1.791	0.0018	5.398	0.0530	34.84	2.73
Posttest1	5.58	16.34	5.42	74.70	6.33	6.365	0.0050	5.434	0.0709	36.45	3.31
Posttest2	5.48	14.96	4.76	76.55	8.41	1.648	0.0051	5.058	0.0543	36.23	3.24

M_{ad}, moisture content, air dried basis; A_d, ash yield, dry basis; V_{daf}, volatile matter, dry ash-free basis; FC_{ad}, fixed carbon; BET, Brunauer-Emmett-Teller multi-molecular adsorption formula; LTNA, low-temperature nitrogen adsorption; MP, Mercury porosimetry.

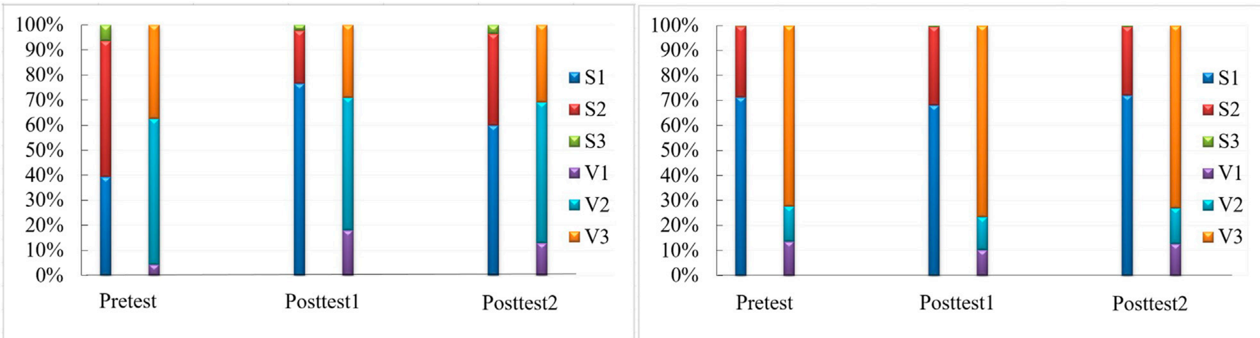

FIGURE 2 | Percentage of specific surface area and pore volume in each pore size interval (left panel: LTNA; right panel: MP). S1~S3 and V1~V3 are specific surface area ratios and the pore volume ratios of micropores, minipores, and meso-macropores, respectively.

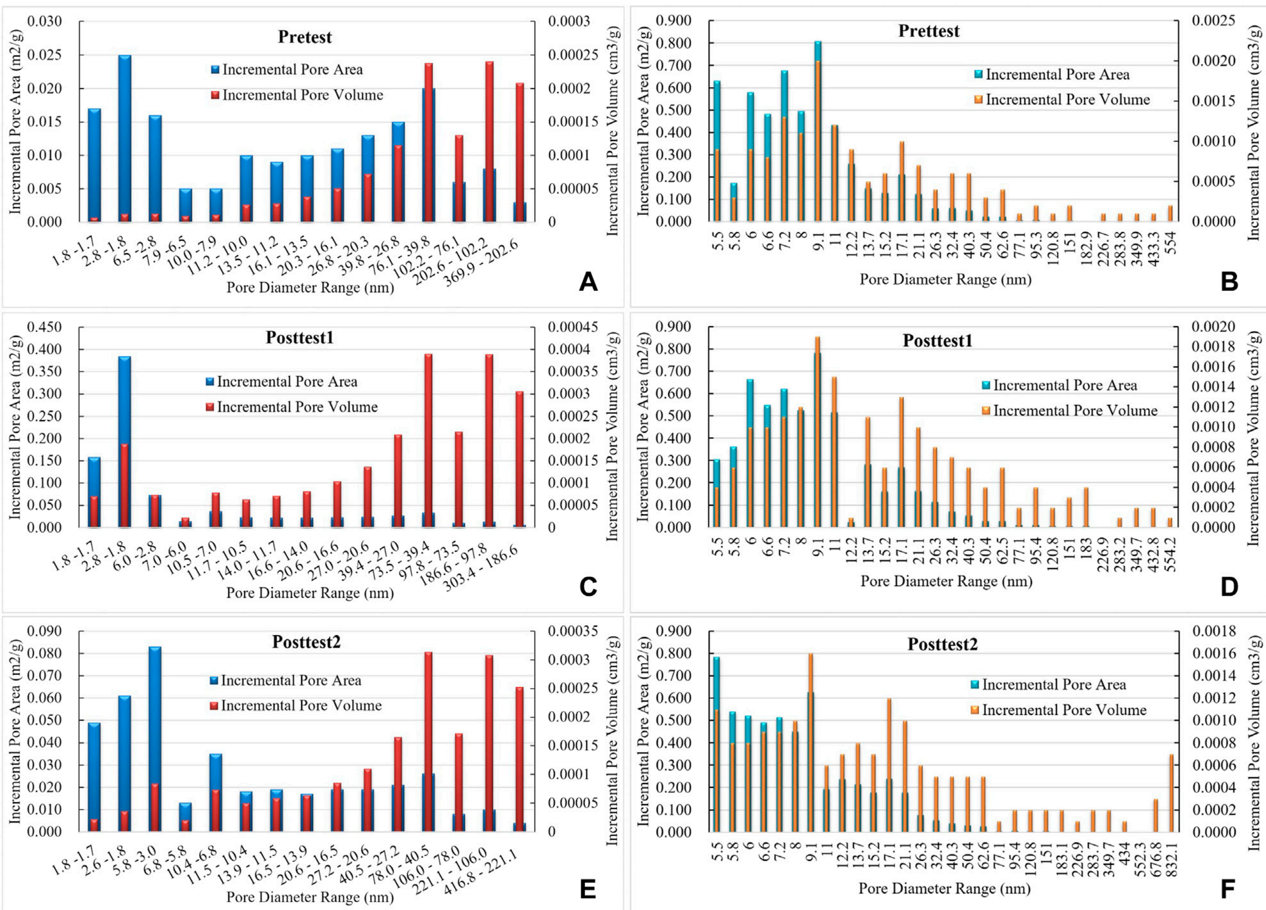

FIGURE 3 | Specific surface area and pore volume distribution characteristics based on pore distribution change according to LTNA measurements for **(A) (C) (E)** and MP measurements for **(B) (D) (F)**; the left ordinate is the incremental pore area (m^2/g) and the right ordinate denotes the incremental pore volume cm^3/g).

bicarbonate, and sodium sulfide. The experiment lasted for 47 days (until 72 h no gas production was noted). Two experimental devices produced 10,449 and 11,120 ml of gas respectively, of which the methane content was 4,013 and 4,583 ml.

Test Methods

An Automatic Proximate Analysis Instrument was used to perform proximate analysis (the moisture, ash, and volatile and fixed carbon contents) experiments, as per ISO 17246-2010. A Micromeritics

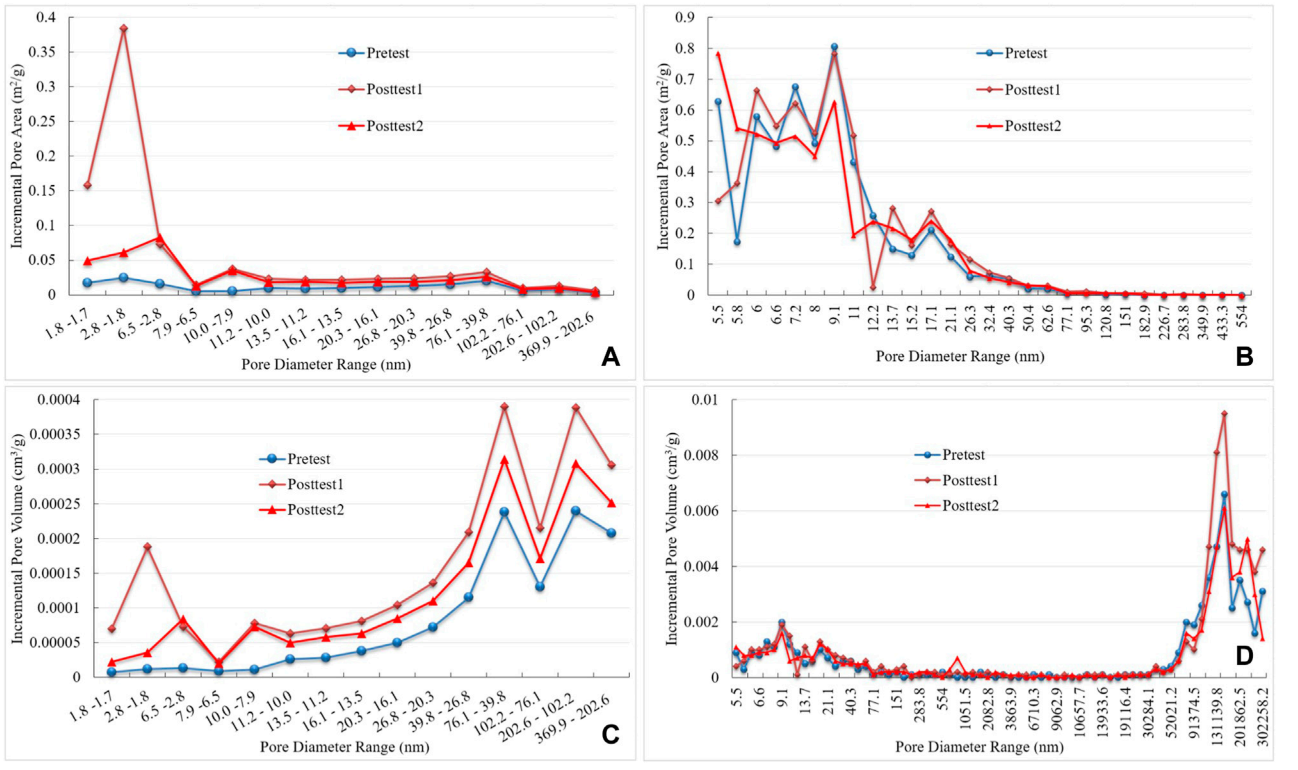

FIGURE 4 | The changes in specific surface area and pore volume based on pore size change [**(A,C)**: LTNA; **(B,D)**: MP].

ASAP-2020 automated surface area analyzer (United States) was adopted to perform the low-temperature nitrogen adsorption/desorption experiments, as per the ISO 15901.2-2006 test method over a pore diameter analysis range of 0.35–300 nm. An Automated Isothermal Gas Adsorption/Desorption Experiment System TerraTek ISO-300 (United States) was used to conduct the isothermal adsorption/desorption experiments, as per Chinese National Standard GB/T 19560-2008. All the above tests were completed at the State Key Laboratory of Coal and Coalbed Methane Co-Extraction in China. A Micromeritics Autopore IV 9500 Instrument (United States) was used to perform mercury porosimetry experiments, according to the ISO 15901-1-2005 test method, the particle size of the coal sample used was 4.75–3.35 mm over a pore-diameter analysis range of 7.5 nm–360 μm. The pore characteristics of coal and methanobacteria were observed under FEI Quanta 250 FEG (United States) field emission scanning electron microscopy (these two tests were conducted at Henan Polytechnic University in China).

RESULTS AND DISCUSSION

Pore Properties

In this study, the following Ходот criteria developed in the former USSR were adopted: the pores in a coal specimen were divided into: micropores (<10 nm), minipores (10–100 nm), mesopores (100–1,000 nm), and macropores (>1,000 nm) (Hudot, 1966; Cheng et al., 2021).

From the distribution of specific surface area (**Figure 2**), the specific surface area of coal samples comprised mostly micropores and minipores. The specific surface area of micropores and minipores of coal samples increased from 93.6% to 97.8% and 96.5% after bioconversion under LTNA test conditions, and the specific surface of micropores and micropores of coal samples decreased from 99.7 % to 99.5% under mercury injection test conditions. From **Figures 3**, **4A,4B**, the specific surface area of micropores of coal samples was found to increase significantly after bioconversion, while that of the minipores increased slightly, and the overall specific surface area increased from 1.79 to 4.01 m^2/g on average after LTNA testing; however, the overall specific surface area decreased slightly, from 5.398 to 5.246 m^2/g under mercury injection test conditions.

Different from the specific surface area, the contribution of pore volume arose mainly from minipores, mesopores, and macropores (**Figure 3**), the contribution of meso-macropores increased from 72.4 % to 76.7% and 73.1% after bioconversion under MP test conditions (**Figure 2**). The contribution of micropores to the pore volume increased from 4.34 % to 18.0% and 12.98% under the LTNA test, although the contribution of micropores to the increase in pore volume after the experiment was large, the proportion of micropores to the increase in the pore volume remained small. As can be seen from **Figures 4C,D**, the pore volume of coal samples increased

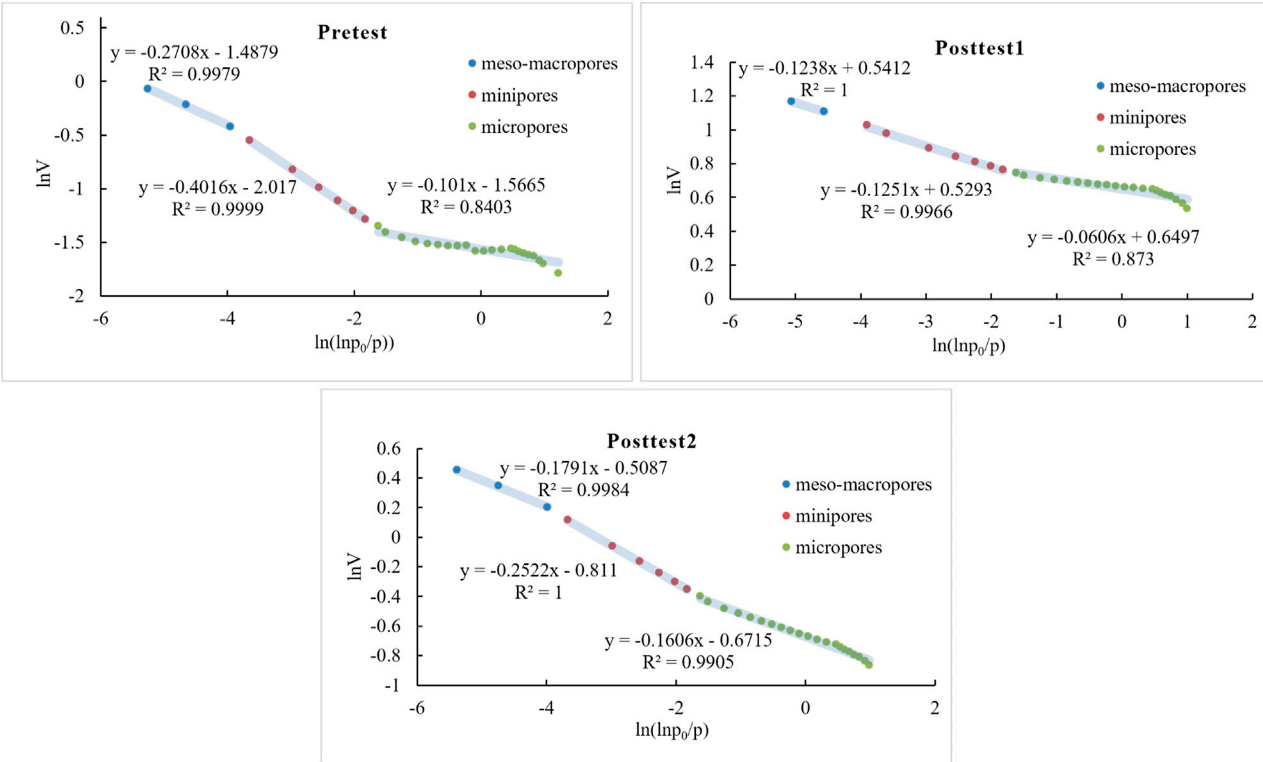

FIGURE 5 | Relationship between ln *V* and ln (ln (P_0/*P*)) based on LTNA.

from 0.0018 cm^3/g (LTNA) and 0.0530 cm^3/g (MP) before testing to 0.0051 cm^3/g (LTNA) and 0.0626 cm^3/g (MP) after testing in LTNA and MP conditions, respectively (**Table 1**).

The differences in the data of specific surface area and pore volume between the LTNA and MP tests methods are mainly based on two reasons: one is that MP focuses on macropores and mesopores, while LTNA is more advantageous for micropores and minipores; the other is that according to the experimental results, microbial modification of mesopores is greater, which reduces the specific surface area of mesopores and increases the pore volume.

Surface Fractal Dimension

The pore structure of coal has strong heterogeneity and anisotropy, with fractal characteristics (Zhang et al., 2017), therefore, the fractal dimension is often used to quantify and characterize the pore structure in coal reservoirs (Wang et al., 2021). In this study, the Frenkel-Halsey-Hill (FHH) fractal model was used to estimate the fractal dimension from the LTNA data. The FHH equation can be expressed as (Lowell et al., 2004; Zhang et al., 2014):

$$\ln \frac{v}{v_o} = C + A\left[\ln\left(\ln \frac{p_o}{p}\right)\right] \quad (1)$$

$$\text{Either } A = (D-3)/3 \quad (2)$$

$$\text{Or } A = D-3 \quad (3)$$

where v is the sorption capacity; v_o denotes the volume of monolayer coverage; D is the fractal dimension; A represents the pre-exponential factor, which is dependent on D; P_0 is the saturated vapor pressure; C is a constant.

According to **Eq. 1**, there must be a linear relationship between ln *V* and ln (ln (P_0/*P*)) when the pores have fractal characteristics. As can be seen from **Figure 5**, the bilogarithmic coordinates show

TABLE 2 | Fractal dimension calculations based on the FHH model.

Sample ID	d ≤ 10 nm			10 nm < d < 100 nm			d ≥ 100 nm		
	A	*D*	R^2	*A*	*D*	R^2	*A*	*D*	R^2
Pretest	−0.2708	2.7292	0.84	−0.4016	2.5984	1.0	−0.1010	2.899	1.0
Posttest1	−0.0606	2.9394	0.87	−0.1251	2.8749	0.99	−0.1238	2.8762	1.0
Posttest2	−0.1606	2.8394	0.99	−0.2522	2.7478	1.0	−0.1791	2.8209	0.99

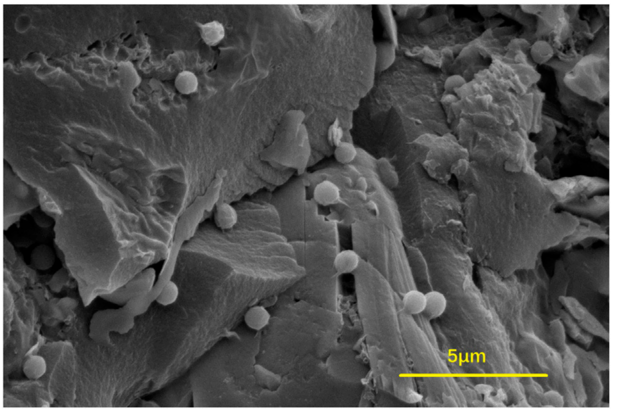

FIGURE 6 | SEM image of spherical microbial attachment to the coal after microbial treatment.

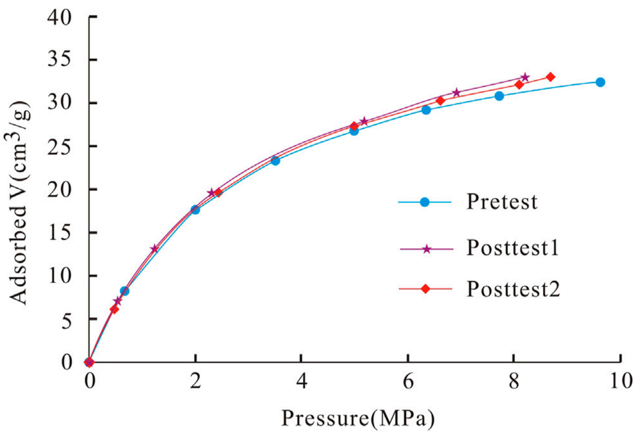

FIGURE 8 | Curves of methane isothermal adsorption on samples.

a significant linear relationship, indicating that the pores of coal samples can be characterized by fractal theory. It is noteworthy that there are two different formulae available for calculating D with A based on the different situations: **Eq. 2,3** (Pfeifer and Avnir, 1984; Avnir and Jaroniec, 1989; Ismail and Pfeifer, 1994; Yao et al., 2008; Dou et al., 2021). **Eq. 2** is applicable to the membrane/gas interface controlled by van der Waals force, and **Eq. 3** is applicable to the interface controlled by liquid–gas tension. In this study, many of these D values are less than 2 as calculated using **Eq. 2**, which is unrealistic, so D is calculated using **Eq. 3** (**Table 2**).

To study the effect of microbial action on pore structure of coal, the data are divided into three regions to calculate the fractal dimension, $d \leq 10$ nm, 10 nm $< d <$ 100 nm, $d \geq 100$ nm. The fractal results showed that when $d \leq 10$ nm, the fractal dimension of coal after microbial treatment was increased from 2.73 to 2.89, when 10 nm $< d <$ 100 nm, the fractal dimension D was increased from 2.60 to 2.81, however, when $d \geq 100$ nm, the fractal dimension D was reduced from 2.90 to 2.85, The results indicated that the surface irregularities of mesopores and macropores decreased, the pores became smoother and the pore volume decreased after the bioconversion (**Figure 6**). This finding was consistent with the aforementioned research (the pore structure evolution model is shown in **Figure 7**).

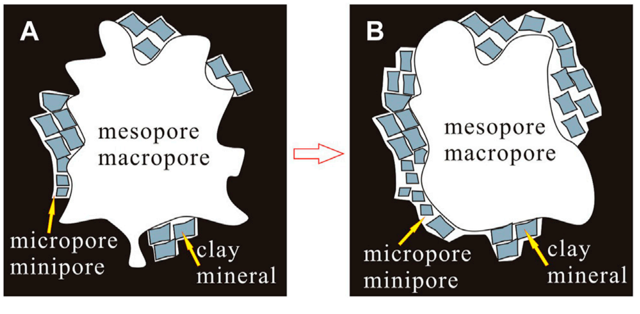

FIGURE 7 | A conceptual pore structure evolution model with microbial treatment. **(A)** Pore structure before microbial treatment. **(B)** Pore structure after microbial treatment.

Proximate Analysis and Adsorption Characteristics

From the perspective of industrial analysis (**Table 1**), compared with pre-test values, the moisture content, ash yield, and clay content of coal samples increased from 2.99%, 13.13 % and 4.13%–5.53%, 15.65 % and 7.37%, respectively, while the volatile matter and fixed carbon contents reduced from 6.68%, 78.63 %–5.09% and 75.63%, respectively. This showed that microorganisms participated in the degradation of coal reservoir and promoted the generation of methane gas.

The curves of the methane adsorption on samples can be drawn according to the Langmuir model **Eq. 4**:

$$V = \frac{v_L P}{P_L + P} \tag{4}$$

where V is the adsorption volume, V_L denotes the Langmuir volume, P is the equilibrium pressure, P_L is the Langmuir pressure (Zhang et al., 2017; Chen et al., 2018). It can be concluded from **Figure 8** that the Langmuir volume was increased after bioconversion, from 34.84 to 36.34 cm^3/g (mean average), and the Langmuir pressure was increased from 2.73 to 3.28 MPa (mean average) after bioconversion based on the isothermal methane adsorption experimental data (**Table 1**). This result showed that transformation of coal by microorganisms is more beneficial to the adsorption and production of CBM.

CONCLUSION

In this study, No. 3 coal seams in Qinshui Basin were taken as the research objects to observe the pore structure characteristics after microbial treatment, and the following conclusions can be drawn.

The contribution of micropores and minipores to the specific surface area of coal samples reached more than 93%, and the contribution was further increased after microbial treatment. After bioconversion, the specific surface area increased from 1.79 to 4.01 m^2/g under LTNA test conditions, while the specific surface area decreased from 5.398 to 5.246 m^2/g under MP test conditions. The fractal dimension of micropores and minipores increased from 2.73 and 2.60 to 2.89 and 2.81, respectively, but the fractal dimension of meso-macropores decreased from 2.90 to 2.85.

The pore volume of coal samples arose mainly from minipores, mesopores, and macropores, and the contribution of minipores, mesopores, and macropores to the increase in the pore volume was more than 70%. The pore volume of coal samples increased from 0.0018 cm^3/g to 0.0530 cm^3/g to 0.0051 cm^3/g to 0.0626 cm^3/g after testing under LTNA and MP test conditions, respectively.

The volatile matter and fixed carbon were both reduced from 6.68 % to 78.63% to 5.09 % to 75.63%, and the Langmuir volume and Langmuir pressure increased from 34.84 cm^3/g and 2.73 MPa to 36.34 cm^3/g and 3.28 MPa, respectively.

The results demonstrated that microorganism participated in the degradation of coal reservoir and promoted the production of methane gas. The meso-macropores were more obviously modified by the microorganisms, so that the pore diameter stabilized, the pores became smoother, the specific surface area decreased, and the pore volume increased. After microbial treatment, the coal body was more conducive to the adsorption of methane gas. The increase of Langmuir pressure was more conducive to the subsequent production of CBM.

DATA AVAILABILITY STATEMENT

The raw data supporting the conclusion of this article will be made available by the authors, without undue reservation.

AUTHOR CONTRIBUTIONS

DG searched the literature; DG and BG analyzed the data and wrote the paper; KT and HR treated samples and performed the experiments; HG processed some data and drew some maps.

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