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Pore structure and fractal analysis of Ximeng lignite under microwave irradiation

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HIGHLIGHTS

- The changes in pore structure during microwave drying were nonlinear.
- The evolution mechanisms of pore structure during microwave drying were obtained.
- The changes in D_1 were similar to those in average pore diameter development.
- The changes in D_2 were consistent with those in specific surface area development.
- D_2 can reflect the volumetric roughness of mesopores in Ximeng lignite.

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ABSTRACT

The pore structure and fractal analysis of Ximeng lignite (XL) under microwave irradiation were investigated. Effects of drying temperature, microwave irradiation time, and microwave power level were studied. The pore structures of XL were obtained by N_2 adsorption/desorption at 77 K. Two fractal dimensions, D_1 and D_2, at relative pressures of 0–0.5 and 0.5–1, respectively, were calculated with the fractal Frenkel–Halsey–Hill model. The specific surface area of the microwave-treated XL differently decreased, whereas its average pore diameter and total pore volume differently increased. However, the evolution mechanisms of pore structures during microwave drying were similar. These mechanisms included the collapse of pore structures caused by shrinkage forces that resulted from the removal of moisture, open and cross-linking of blind and closed pores, and thermal decomposition of organic macromolecular structures under a high temperature. The changes in D_1 were similar to those in average pore diameter development, whereas the changes in D_2 were consistent with those in specific surface area development. The reliability of the relationship between D_2 and mesopores was examined with some scanning electron microscope images and D_2 can reflect the volumetric roughness of mesopores.

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1. Introduction

Coal is an organic macromolecular solid material that contains different inorganic mineral impurities and is structurally heterogeneous, porous, and amorphous. The surface morphology and pore structure of coal show capillaries, cracks, and open and closed pores and are thus very complex and irregular [1]. This complicated microscopic pore structure of coal samples not only affects their physical properties (i.e., mechanical properties, wetting characteristics, and adsorption properties) but also controls the reactant input and product output during coal processing and utilization. Therefore, accurately and quantitatively describing the microscopic pore structure of coal samples is important. However, the traditional Euclidean geometry is limited by its theory in describing irregular objects, and it fails to analyze the irregularity and disorder of surface morphology and pore structure. New theories and methods should be developed to describe irregular objects with mathematical rigor [2]. Fractal geometry has been developed in the current case.

The initial concepts and possible applications of fractals were popularized by Mandelbrot in 1975 [3], and fractal geometry has rapidly developed since then. Nowadays, fractal geometry is widely used in many natural and social science fields and is proven to be a powerful and reasonable tool that quantitatively describes irregular or disordered self-similar fractals in nature [4–6]. Notably, not all irregular objects can be defined as fractals; the application of fractal models to the fractal analysis of disordered objects involves major limitations and can only be applied over a certain range of scales [4].

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Hausdorff dimension (D) is generally used in the fractal characterization of the microscopic pore structure. For porous solids, the fractal dimension, D, can be a non-integer value that varies between 2 and 3: $D = 2$ for a smooth surface and $D = 3$ for a rugged surface that tends to fill the space in which it resides. As a magnitude that describes the surface irregularity or roughness of disordered fractals, a fractal dimension that is high indicates an irregular surface of disordered fractals. Therefore, an increase in fractal dimension indicates an increase in the roughness of the surface, whereas a decrease in fractal dimension indicates smoothing of the surface. The fractal dimension of fractals can be determined with different methods, including small-angle X-ray scattering, scanning electron microscope (SEM), high-resolution transmission electron microscopy, mercury porosimetry, small-angle neutron scattering, nuclear magnetic resonance spectroscopy, and gas adsorption [7–12]. Gas adsorption can be the simplest and most convenient method that is widely used to analyze the pore structure and fractal characterization of objects [13–16].

Fractal geometry provides a new method and perspective to study the microscopic pore structure of coal samples. The pioneering work in applying fractal geometry to the study of coal micropore structures by Bale and Schmidt involved calculating the surface fractal morphology in Beullah lignite coal and the surface fractal dimension of the coal sample [17]. Johnston et al. [18] used small-angle X-ray scattering to examine the pore structure of Victorian brown coal in unprocessed and different processed states. Results show that fractal concepts can be used to describe the pore surfaces of Victorian brown coal. The surface fractal dimension, D_s, and the surface measure, N_0, of the coal sample processed under different conditions differently changed and were very sensitive to the processing conditions. Mahamud and Novo [19] investigated the fractal characterization of four bituminous coals and the corresponding coal sample oxidized in air at 543 K for different time periods. Different methods based on mercury porosimetry data were used to analyze the evolution of the fractal dimensions of the coal sample. Fractal dimensions calculated with different methods similarly changed and were sensitive to oxidation treatment. The evolution of the fractal dimension of coals with oxidation was controlled by two dominant mechanisms: the oxidation of the pore surface and the access of mercury to previously non-accessible regions. Hu et al. [20] analyzed the surface structures of parent coal and char with different burn-off ratios, as well as experimentally and theoretically investigated the fractal characteristics of coal/char structure. The particle surface fractal dimension (D_{ps}) was determined by SEM image analysis, whereas the internal surface fractal dimension (D_s) was determined by N_2 isotherm adsorption/desorption. With increasing carbon burn-off ratios, the D_{ps} first increased and then decreased. The changes in D_s of three Chinese coals were similar to their S_{BET} development.

Microwave drying technology offers several advantages compared with conventional drying, such as its rapid and uniform heating, high safety, and superior dried-product quality. Because of these unique features, microwave has been applied to heat and modify lignite [21–23]. The microscopic pore structure of lignite has important effect on its slurrying ability and would change during drying process [24–26]. However, few reports have focused on the evolution mechanisms and fractal characteristics of the microscopic pore structure of lignite under microwave irradiation. The relationship between fractal dimension and pore structure is still unclear.

China has abundant lignite resources, and the proven lignite reserves in Inner Mongolia account for 77.55% of the total lignite reserves in China [27]. Thus, investigation of Inner Mongolia lignite, especially Ximeng lignite (XL), will allow for better utilization of China's lignite resources. Wang et al. [28] investigated the effects of acid pretreatment on the structure and pyrolysis performance of XL. The structure of XL was not significantly affected by the HF treatment process, but an obvious organic structure change was found in the coal samples further treated by HNO_3 solution. The fractional step acid treatment had effects on the pyrolysis performance of XL. Bai et al. [29] studied on the effects of demineralization on phenols distribution and formation during XL pyrolysis and found that the inherent mineral of XL had catalytic activity on the conversion of catechol to phenol. Zhu et al. [30] systematically investigated the thin-layer drying and reabsorption characteristics of XL at different coal particle sizes and microwave power levels. The drying rate increased and drying time decreased with increasing particle size or microwave power level, while the equilibrium moisture of XL decreased with increasing microwave output power or decreasing particle size. The Midilli–Kucuk model provided a better fit for all applied drying conditions and could be used to estimate moisture in XL at any time during the microwave-drying process.

In order to utilize XL cleanly and efficiently, it is important to dehydrate and modify XL prior to its deep processing and further utilization. The medium- to low-temperature thermal treatment and hydrothermal dewatering were employed to dehydrate XL and the dewatered products were used to prepare coal water slurry [25,31,32]. After dehydration, the coal rank and heat value of XL increased as the inherent moisture decreased, the oxygen functional groups in XL decreased because of decomposition. Therefore, the maximum solid concentration of coal water slurry prepared from XL increased.

XL was processed and dehydrated with microwave in this study. The effects of isothermal drying (drying temperature) and non-isothermal drying (including microwave irradiation time and microwave power level) on the microscopic pore structure and fractal characteristics of XL were investigated by N_2 adsorption/desorption at 77 K. The goal is to provide references for processing and utilizing XL.

2. Experimental

2.1. Material

The lignite used in this study came from the Ximeng region in Inner Mongolia, which is the largest producer of lignite in China. Naturally dried XL was ground in a bowl mill and sieved through a 100-mesh sieve. Proximate and ultimate analyses of raw coal are shown in Table 1. The particle size distribution of raw coal is shown in Fig. 1.

2.2. Methods

2.2.1. Microwave-drying experiments

Microwave-drying experiments were performed in a microwave reaction workstation (MAS-II; Sineo Microwave Chemistry Technology Ltd., Shanghai, China) with a microwave frequency of 2450 MHz (wavelength: 12.24 cm). The workstation is equipped with a non-contact infrared thermometer (maximum working temperature: 250 °C, precision: ±1.5 °C) which is connected to the microwave generator, and using frequency conversion, the power level is automatically adjusted for temperature. A schematic illustration of the microwave irradiation experimental apparatus is shown in Fig. 2.

During the isothermal drying experiments, 10 g XL was weighed with a digital balance (Sartorius BS 224 S, precision: 0.1 mg) and then placed in a 50 mL three-necked flask. The left neck was used as an air inlet, the middle was used as a non-contact infrared thermometer inlet, and the right neck was used as an air outlet. Before the experiments, high-purity nitrogen (99.99%) was

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