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# Regulation of pore cell structures of coal-based carbon foams based on the nucleation mechanism of microcellular polymer

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## ABSTRACT

It is widely accepted that the performance of carbon foams can be regulated by the tailoring of pore cell structures to meet the requirements of various applications. However, no theory has been used to date for guiding such regulation. In this work, carbon foams were prepared by saturating vitrinite concentrate with nitrogen gas under high pressure. The influence of key factors on the pore cell structure of carbon foams was investigated systematically. The results showed that the mean cell diameter and the bulk density of carbon foams can be regulated, respectively, in the ranges 140–440  $\mu\text{m}$  and 0.29–0.75  $\text{g}/\text{cm}^3$ , which indicates that vitrinite concentrate separated from fat coal is highly suitable for the preparation of carbon foams. The variation trends of the pore cell structures were well explained by combining the homogeneous nucleation mechanism of microcellular polymer with the viscosity of the fusant formed from vitrinite concentrate. The inherent reason for all the variation trends is related to the gas nuclei density and viscosity of the fusant. More importantly, a strategy is suggested to successfully accomplish the design and regulation of the pore structure of carbon foams by taking into account the homogeneous nucleation mechanism and viscosity of the fusant.

## 1. Introduction

Carbon foams are a lightweight, porous, and monolithic carbon material with a three-dimensional structure. They have the same nature as carbon materials. Moreover, their bulk density, thermal conductivity, electrical conductivity, and compressive strength are adjustable by regulating the pore structure [1,2]. Therefore, they can be used as a thermal management material [3,4], an electromagnetic shielding material [5,6], a wave-absorbing material [7], an electrode material [8], a gas adsorption agent [9,10], a catalyst carrier [11], a biological material [12,13], and a lightweight anti-ablation material [14]. According to the microstructure of the pore wall, carbon foams fall into two categories: graphite carbon foam and nongraphitized carbon foam, both of which can combine with metals or nonmetals to form composite materials with multiple excellent properties [15,16]. Therefore, the preparation [17], modification [18], and application of carbon foams attract much attention. However, the regulation of the pore cell structure of carbon foams is essential for realizing the regulation of

performance and this is actually also one of the bottlenecks for batch production [1,2]. To regulate the pore structure of carbon foams, many research works have been carried out in the past few years.

Changing the rheological characteristics of the precursor is a means to realize the regulation of the pore structure of carbon foams [2]. Chong Chen et al. [19] studied the effect of different precursors on the density of carbon foams. They found that the bulk density of carbon foams decreased with the increase in the maximum Giseeler fluidity and the swelling degree of the precursor. Calvo et al. [20] used different volatile bituminous coal as raw materials to prepare carbon foams by saturating nitrogen under high pressure. They also found that the bulk density of carbon foams decreased with the increase in the maximum Giseeler fluidity of the bituminous coal. Xu et al. [21] adopted a heavy medium flotation method to separate vitrinite concentrate from medium-degree metamorphic coal to improve the rheological properties of the precursor. In this way, they realized the control of the pore cell structure of coal-based carbon foam in the range 150–350  $\mu\text{m}$ .

Adjusting the versatile process parameters (foaming temperature,

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pressure, time, etc.) is another means to regulate the pore structure of carbon foams [2]. Wang et al. [22] used mesophase pitch from petroleum as raw materials to prepare carbon foams by self-foaming. They studied the effect of the process parameters on the structure of carbon foams in detail and found, with the increase in foaming pressure, the pore cell diameter of carbon foams reduced, the pore cells became more regular, and the bulk density increased. With the increase in the foaming temperature, the pore cell diameter of carbon foams increased and the open porosity increased. Since then, Wang et al. [23] used AR asphalt as a raw material to prepare carbon foams by saturating nitrogen under high pressure. They studied the influence of the holding temperature time on the pore structure of carbon foams in detail and found the pore diameter of carbon foams achieved by prolonging the constant temperature time increased, the porosity increased, the bulk density was reduced, and the compressive strength was also enhanced. Calvo et al. [24] used good plastic bituminous coal as a precursor to prepare carbon foam by two steps of heat treatment at different foaming temperatures and pressures. It was found that the pore size decreased with the increase in pressure, and the pore volume increased with the increase in temperature. Xiong et al. [25] adopted AR mesophase pitch as a raw material to prepare carbon foam by self-foaming. At the initial foaming pressure of 3 MPa and the foaming temperature between 390 and 450 °C, they investigated the influence of the foaming temperature on the structure and properties of carbon foams. It was found that, with the increase in the foaming temperature, the bulk density of carbon foams first decreased and then increased. At 410 °C, the bulk density was the minimum. Shen et al. [26] studied the preparation, structure, and properties of mesophase pitch-based carbon foams. They found that, with the increase in foaming pressure in the range of 1–10 MPa, the bulk density of carbon foams decreased. Studies on the effect of temperature and pressure on the properties of foams from petroleum pitch and naphthalene pitch were also conducted by Klett et al. [27]. They found that the process pressure and temperature affected the foam structure to a higher degree than the properties of the raw material used.

The available previous research works provided some valuable information for regulating the pore structure of carbon foams based on simple factor experiments, in which regulating the pore structure was carried out by either changing the raw material with various rheological properties or by changing the partial process parameters for the same raw material. However, little has been thoroughly presented on the influences of key factors on the pore structure of coal-based carbon foams. In addition, the essential physical chemistry revealing the change in the pore structure of carbon foams has still not been determined.

With the above in mind, we used the vitrinite concentrate from fat coal as the precursor to prepare carbon foams by saturating nitrogen under high pressure and thoroughly presented the influences of key factors on the pore structure of coal-based carbon foams. Furthermore, we attempted to explain the inherent reason for the pore structure variation of carbon foams with the key parameters changing based on the nucleation theory of microcellular polymers.

## 2. Experimental

### 2.1. Preparation and analysis of precursor

The fat coal, air dry basis, was crushed into a 60–80 mesh of pulverized coal. Zinc chloride solution with a density of 1.35 g/cm<sup>3</sup> was used as a specific gravity liquid. The pulverized coal and zinc chloride solution were intensively blended and then the resultant turbid liquid was centrifuged for 30 min. The substances in the upper layer were taken out for filtration, washing, and drying to get vitrinite concentrate of fat coal, which was used as a precursor. The proximate and ultimate analysis of fat coal and its vitrinite concentrate were tested. The Giseeler fluidity of the vitrinite concentrate was measured in

accordance with ASTM D2639-98. A thermogravimetric analyzer (Diamond TG/DTA6300) was used to analyze the weight loss of the vitrinite concentrate during heating. Samples in the range 15–17 mg were taken for subsequent use. The flow rate of N<sub>2</sub> gas was controlled at 100 mL/min. The samples were heated at 2 °C/min up to 1000 °C.

### 2.2. Preparation of carbon foam

Seventy grams of vitrinite concentrate of fat coal was placed in a metal mold. A certain pressure, which is defined as the stamping pressure, was exerted by a press machine to adjust the gap of adjacent particles. The mold was then put into an autoclave and heated at 2 °C/min up to the softening point temperature. Nitrogen gas was filled to meet the requirements of the saturating pressure (foaming pressure) and then the outlet valve was closed. The temperature continued to rise at 2 °C/min up to the saturating temperature (foaming temperature), and was held for a certain period, which is defined as the foaming time. The foaming pressure started to release at a rate of 2 MPa/min before finishing the holding temperature, and the moment when the foaming pressure is released to atmospheric pressure must be the moment of finishing the holding temperature to nucleate and grow in a closed isothermal system in chemical equilibrium. Then, the temperature continued to rise at 2 °C/min up to 550 °C, after which it was held constant for 20 min and cooled naturally to 25–30 °C. Thus, a green type of carbon foam was achieved. The preparation device is shown in Fig. 1. The green carbon foam was transferred to a tube-typed furnace for the treatment of carbonization. Under the protection of high-purity nitrogen gas, the furnace was heated at 2 °C/min up to 1000 °C, after which the temperature was held constant for 120 min and cooled to 25–30 °C naturally. Finally, the coal-based carbon foam was successfully prepared.

### 2.3. Characterization of carbon foams

#### 2.3.1. Bulk density and porosity of carbon foam

Carbon foam was ground into particles smaller than 0.2 mm. The true density ( $\rho_T$ ) was measured using the Pycnometer method (GB/T217 21996). A rectangular block of approximate length, width, and height of 20 mm, 20 mm, and 10 mm, respectively, was processed. A caliper was used to accurately measure the length (a), width (b), and height (c). The mass (M) of the sample was determined. Then, the volume density ( $\rho_V$ ) was calculated in accordance with formula (1) and the porosity (P) with formula (2), as follows:

$$\rho_V = \frac{M}{abc} \quad (1)$$

$$P = 1 - \frac{\rho_V}{\rho_T} \quad (2)$$

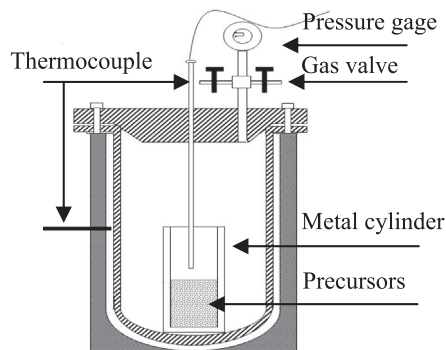


Fig. 1. Schematic diagram of set-up for preparation of carbon foams.

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