

Preparation and characterization of coal-based carbon foams by microwave heating process under ambient pressure

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ABSTRACTS

To reduce significantly the preparation time and cost in energy of coal-based carbon foams and to develop facile synthesis condition, a microwave heating process (MHP) for fabricating coal-based carbon foams was suggested. The preparation time, the structure and the compressive strength of carbon foams fabricated respectively by MHP and carbonization in nitrogen atmosphere at high pressure (CNHP) were contrastively studied. The results show that MHP exhibited three distinct characteristics in contrast to CNHP. First, the preparation time of green foam is shortened by 55.2% based on the microwave heating principle. Secondly, the preparation conditions are more moderate, only under ordinary pressure instead of high pressure. Thirdly, the resultant carbon foams display small pore diameter with narrow pore size distribution and high compressive strength. More importantly, the strategy of coal-based carbon foam by microwave heating process can be applied to other thermoplastic precursor foaming to obtain macro-porous materials.

1. Introduction

Carbon foams are non-toxic, highly porous, light monolith materials which demonstrate a wide range of superior properties, such as high mechanical strength, refractory, as well as adjustable thermal conductivity and electrical conductivity [1,2]. They can be used as thermal management materials [3], electromagnetic shielding materials [4], wave-absorbing materials [5], electrode materials [6,7], gas adsorption agents [8], a catalyst carrier [9] and biological materials [10]. Generally, carbon foams fall into two categories: graphite and non-graphitized carbon foams, both of which can combine with metals and nonmetals to obtain composite materials with multiple excellent performances [11,12]. Therefore, carbon foams have been attracting an extensive attention worldwide since 1960s.

Compared to pitch-based and resin-based carbon foams, coal-based carbon foam is considered as the most promising monolithic porous material in the future due to the wide availability and low cost of coals [1,2]. In 1999, Stiller and coworkers [13] first synthesized coal-based carbon foam by carbonization in nitrogen atmosphere at high pressure (CNHP). In this work, asphaltene was extracted from coal and used as a precursor to prepare isotropic, anisotropic, and anisotropy-adjustable carbon foams. However, it is complicated and expensive to achieve asphaltene from coal. In order to reduce the preparation cost, Rogers and coworkers [14] prepared carbon foams by CNHP using low-ash and

low-sulfur coal as the precursor. It should be noted that the coals used as precursors in the above work possess the limited range of free swelling index i.e. 3.5–5.0. The as-obtained carbon forms have densities of 0.1 g/cm³–0.8 g/cm³, and the aperture of 300 μm–500 μm. In another work, Calvo and coworkers [15] prepared carbon forms using a good plastic bituminous coal as the precursor by CNHP. It was found that the foaming temperature and pressure determined the average diameter of the pore cell and the number of the pores. The pore size decreased with the increase of pressure, and the pore cumulative volume increased with the increase of temperature. Furthermore, Calvo and coworkers [16] prepared carbon foam by CNHP using bituminous coals with different volatilities as the raw materials. The bulk density of the as-prepared carbon foam decreased with the increase of fluidity of the coal precursors. It was also found that the average pore size of the carbon foam increased with the increase of the volume fraction of the exinite, but decreased with the increase of the volume fraction of the vitrinite in the precursor. In 2016, our group [17] prepared carbon foam by a CNHP process using vitrinite-concentration extracted from coal as the precursor and carefully investigated the effect of preparation parameters on the porous structure of the as-prepared carbon foam.

Although the CNHP process has been widely applied to prepare coal-based and pitch-based carbon foam, this process has several inherent drawbacks. Firstly, CNHP is a high pressure process. Secondly, this process is complex and time-consuming, which greatly increases

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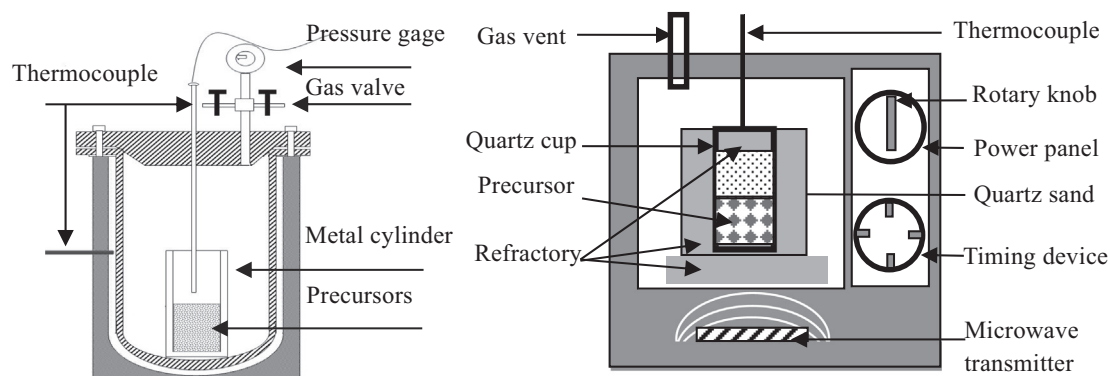


Fig. 1. Schematic diagram of experimental set-up for preparation of carbon foams by CNHP (a) and MHP (b).

Table 1

Proximate analysis of fat coals and its vitrinite concentrates.

Precursors	$M_{ad}/\%$	$A_d/\%$	$V_{daf}/\%$
Fat coal	1.60	8.52	38.29
FCV	1.63	3.5	43.8

Notes: ad —air dry basis, d —dry basis, daf —dry ash free basis.

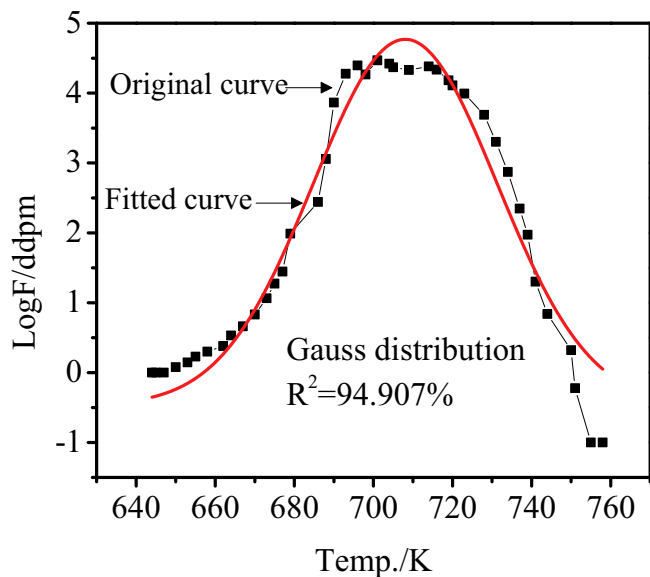


Fig. 2. The logarithm of Gieseler fluidity as a function of testing temperature.

Table 2

The processing variables of carbon foams by different methods.

Process	FCV size/mesh	Mechanical pressure/MPa	Foaming tem./K	Foaming pressure/MPa	Foaming time/hour
CNHP	60–80	10	706	6	30 min
MHP	60–80	10	706	0	30 min

the preparation cost. Thirdly, the pore size of the as-prepared products is not even, which usually show a wide pore size distribution. These disadvantages are mainly due to the limitation of the CNHP process. In the CHNP process, the precursor was gradually carbonized i.e. from the surface layers to the interior layers. Since the main way of heat transfer is via the conduction process, this consumes more time and leads to the uneven distribution of pore sizes. Therefore, CNHP process is not favorable for the batch production of carbon foam. To circumvent the

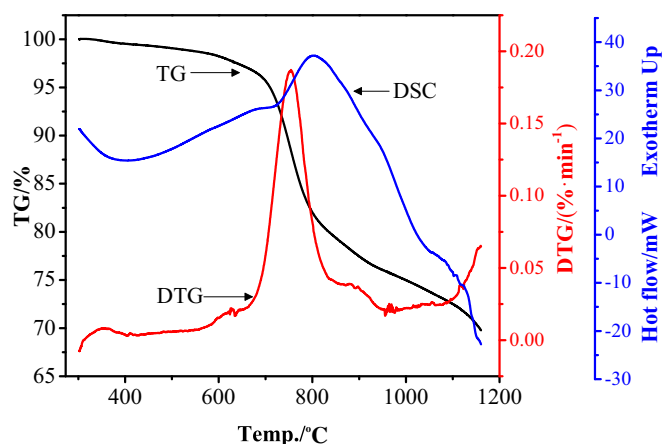


Fig. 3. TG, DTG and DSC curves of FCV.

problems above, it is intriguing and urgent to develop a new strategy of preparing coal-based carbon foams in a large scale.

In this work, a microwave heating process (MHP) is developed to prepare carbon foams at ambient pressure using coal vitrinite concentrates as the raw material. The unique heating principle of microwave heating process makes all the precursors melt, foaming and carbonizing simultaneously, which greatly shortens the preparation time and improves the homogeneity of the as-prepared products. For comparison, carbon foams were prepared by CNHP and MHP processes, respectively. The preparation time, the pore structure and the compressive strength of the resulting products were carefully examined to illustrate the advantages of the MHP process.

2. Experimental

2.1. The precursor

The vitrinite concentrates were separated and enriched from fat coal (FC) by the gravity flotation, and the specific process has been introduced in the literatures [18]. The fat coal vitrinite concentrates (FCV) obtained were used as the precursor to prepare carbon foam. The moisture, ash and volatile components of the precursor were analyzed. According to the ASTM D2639-98 standard, the Gieseler fluidity of the precursor was analyzed by using PL-2006A double furnace Gieseler Plastometer. The TG-DSC (SDT-Q600) was used to analyze the weight loss and thermal effect of the precursor during the heated process. The testing conditions were as follows: the sample 15–17 mg was taken, nitrogen gas flow rate was $100 \text{ mL}\cdot\text{min}^{-1}$, and then the temperature rose up to 1173 K at the rate of 20 K/min.

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