



# Preparation and properties of carbon foam by direct pyrolysis of ally novolak-modified bismaleimide resin

Qilang Lin<sup>a,\*</sup>, Shaohai Dong<sup>a</sup>, Lijuan Qu<sup>a</sup>, Changqing Fang<sup>b</sup>, Kun Luo<sup>c</sup>

<sup>a</sup> College of Materials Science and Engineering, Fuzhou University, Fuzhou 350116, PR China

<sup>b</sup> College of Printing and Packing Engineering, Xi'an University of Technology, Xian 710048, PR China

<sup>c</sup> State Key Laboratory Breeding Base of Nonferrous Metals and Specific Materials Processing, Guilin University of Technology, Guilin 541004, PR China

## ARTICLE INFO

### Article history:

Received 15 December 2013

Accepted 24 January 2014

Available online 3 February 2014

### Keywords:

Preparation

Carbon foam

Pyrolysis

Properties

## ABSTRACT

Carbon foam was prepared by direct pyrolysis of ally novolak (AN)-modified bismaleimide (BMI) resin at ambient pressure. Pyrolysis behavior of the AN-modified BMI (AMB) resins was characterized by thermogravimetric analysis (TGA), scanning electron microscopy (SEM) and X-ray diffraction (XRD). In addition, influences of AN/BMI mass ratio on the structures and properties of the resulting carbon foams were studied. Results show that the AMB resin possesses self-foaming characteristics at high temperatures. The carbon foams have well-developed pore structures with relatively uniform pore sizes; their bulk density, thermal conductivity and compressive strength increase with a decrease in AN/BMI mass ratio. When the AN/BMI mass ratio is 1:1.1, the bulk density, thermal conductivity and compressive strength of the carbon foam arrive at 0.43 g/cm<sup>3</sup>, 1.02 W/mK and 6.4 MPa, respectively.

© 2014 Elsevier B.V. All rights reserved.

## 1. Introduction

Carbon foams possess unique performances, such as low density, high porosity, excellent corrosion resistance and high temperature tolerance, therefore, have attracted increasing attention in many fields ranging from aeronautics, astronautics, chemicals, electronics, energy, environmental protection to catalysis [1–4]. A process to generate carbon foam from thermosetting resin was first proposed by Ford in 1964, and since then, various processes and techniques, including typical blowing [5], pressure releasing [1] and self-bubbling [6] techniques, have been developed to fabricate carbon foams derived from pitches [7], resins [8], coals [9] or biomass materials [10]. However, the development and application of carbon foams are greatly restricted by their poor mechanical properties and high costs [11]. Some researchers attempt to prepare carbon foams with relatively high mechanical strength via a high pressure technique or a complex process, which no doubt increases the costs of the resultant carbon foams [12,13]. Therefore, it is necessary to develop a simple and efficient method to fabricate carbon foams [14].

Conventional thermosetting resins with high char yield, such as phenolic resin [15] and alcohol resin [16], are often used as precursors for production of carbon foams; however, the carbon

foams usually possess relatively poor mechanical properties. The reason for this is due to that a large number of structural defects like cracks and voids are formed in the carbon foams during preparation process [17–19]. It is known that these thermosetting resins release by-products like water and ammonia compound during curing and easily form a certain amount of void, which would result in formation of structural defects in the resulting carbon foams. On the other hand, their green foams undergo thermal decomposition during carbonization causing a relatively large shrinkage, which would lead to structural defects in the resulting carbon foams and thus deteriorate their mechanical properties [20]. In recent years, new types of thermosetting resins (such as polyarylacetylene and polybenzoxazine) were employed to prepare carbon foams with relatively high mechanical strength [21–23]. These thermosetting resins possess high char yield and especially do not release by-products like water and ammonia compound during curing, which is beneficial to the reduction of structural defects in the carbon foams.

Bismaleimide (BMI) resin is an important resin owing to its outstanding thermal properties [24]. The cured resin, however, is extremely brittle due to its high cross-linking density. One of the most desirable methods for improvement is the copolymerization of BMI with allyl compounds such as ally novolak (AN) [25], and a scheme for the copolymerization of BMI and AN is illustrated in Fig. 1. The AN-modified BMI (AMB) resin has excellent thermal properties and do not release by-products during curing; it possesses excellent formability and can be used to easily fabricate

\* Corresponding author. Tel.: +86 591 22866531; fax: +86 591 22866531.

E-mail address: [linqilang@hotmail.com](mailto:linqilang@hotmail.com) (Q. Lin).

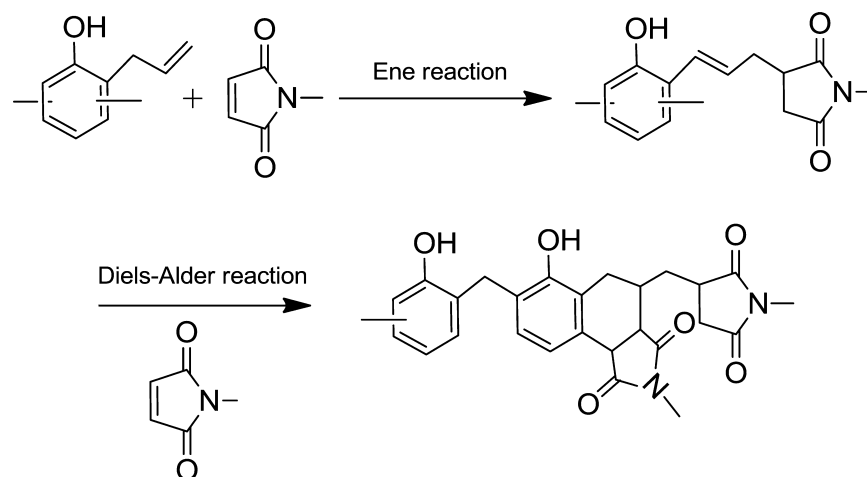


Fig. 1. A scheme for the copolymerization of BMI and AN.

complex components via cast molding and resin transfer molding (RTM) process [25]. Therefore, the AMB resin seems to be a promising precursor for preparing carbon foams with relatively high mechanical strength. In this work, we attempt the preparation of carbon foam by pyrolysis of AMB resin at ambient pressure, and structures and properties of the resulting carbon foam were studied.

## 2. Experimental

### 2.1. Raw materials

Allyl chloride was obtained from Qilu Petrochemical Co., Ltd. (Zibo, China) and used after distillation. Novolak resin (2123), softening point 75 °C, was purchased from Hongtai phenolic plastics Co., Ltd. (Huaian, China). 4,4'-bismaleimidodiphenyl methane, melting point 156–158 °C, was purchased from Fenguang Chemical Co., Ltd. (Honghu, China). Sodium hydroxide and butanol were in analytical grade and supplied by Shanghai Chemical Reagent Corporation (Shanghai, China).

### 2.2. Preparation of allyl novolak (AN)

AN was prepared via Williamson reaction between allyl chloride and novolak resin in the presence of sodium hydroxide catalyst [25]. For preparing AN resin, 1 mol of allyl chloride was used for 1 mol of the phenol group in the resin. The reaction was carried out at 80 °C in a butanol solution for 4.5 h. The degree of allylation of the phenol groups in the novolak resin was 90%.

### 2.3. Preparation of AN-modified BMI (AMB) resin

AN resin was mixed with 4,4'-bismaleimidodiphenyl methane at a certain mass ratio, and then vigorously stirred at 160 °C for 45 min to obtain a homogeneous AMB prepolymer. The prepolymer was degassed in a vacuum oven at 160 °C for 40 min, and then cast into several cylindrical beakers (15 mm in diameter and 12 mm in height) for curing and postcuring via the procedures of 170 °C/2 h + 200 °C/2 h + 220 °C/2 h and 250 °C for 2 h, respectively. The resulting AMB resin was labeled AMB-0.8, AMB-0.9, AMB-1.0, and AMB-1.1 when the AN/BMI mass ratio was 1:0.8, 1:0.9, 1:1 and 1:1.1, respectively. Some properties of the AMB resins are included in Table 1.

### 2.4. Pyrolysis of the AMB resin

The cured AMB resin samples were put into a furnace and heated up to a certain temperature (300 °C, 400 °C, 500 °C, 600 °C, 700 °C, 800 °C, or 900 °C) at a heat rate of 5 °C min<sup>-1</sup> and kept for a certain time (0 or 1 h). After that the samples were cooled naturally to room temperature to obtain resultant pyrolysis products. The nitrogen stream was introduced into the furnace throughout the pyrolysis. The pyrolysis products obtained at 900 °C for 1 h were considered as carbon foams. The resulting carbon foams were labeled CF-0.8, CF-0.9, CF-1.0, and CF-1.1 when the resin used was AMB-0.8, AMB-0.9, AMB-1.0, and AMB-1.1, respectively. The char yield (CY) of the resin was calculated by the following formula:

$$\text{CY}(\%) = \frac{W_1}{W_0} \times 100, \quad (1)$$

where  $W_0$  is the mass of the resin and  $W_1$  the mass of the carbon foam.

### 2.5. Characterization

The volume expansibility of the sample was calculated by the following formula:

$$\text{VE}(\%) = \frac{V_1}{V_0} \times 100, \quad (2)$$

where  $V_0$  is the geometric volume of the sample before pyrolysis and  $V_1$  the geometric volume of the sample after pyrolysis.

The density of the sample was determined by weighing the geometrically shaped foam and dividing the weigh by the measured

**Table 1**  
Characteristics of the AMB resins prepared at four different AN/BMI mass ratios.

Sample	Elemental analysis (wt%)			$T_g$ (°C)	CY (wt%)
	C	H	N		
AMB-0.8	71.75	5.33	5.42	276	31.2
AMB-0.9	71.28	5.17	5.50	289	34.0
AMB-1.0	70.61	5.11	5.56	302	35.8
AMB-1.1	69.94	5.01	5.61	311	36.9

$T_g$ : glass transition temperature.  
CY: char yield.

Download English Version:

<https://daneshyari.com/en/article/1196872>

Download Persian Version:

<https://daneshyari.com/article/1196872>

[Daneshyari.com](https://daneshyari.com)