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Preparation and characterization of carbon foams from cyanate ester mixtures

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ABSTRACT

Carbon foams were prepared by direct pyrolysis of cyanate ester (CE) resins from the mixtures of phenol novolac cyanate ester (PNCE) and bisphenol A cyanate ester (BACE) at ambient pressure. Pyrolysis behaviors of the CE resins were characterized by thermogravimetric analysis (TGA), scanning electron microscopy (SEM) and X-ray diffraction (XRD). In addition, influences of PNCE/BACE mass ratio on the characteristics of the resultant carbon foams were studied. Results show that the CE resin possesses better thermal stability with increasing PNCE/BACE mass ratio, whereas its self-foaming performance becomes worse during pyrolysis. The addition of PNCE to BACE leads to an increase in the bulk density of the resultant carbon foam but a decrease in both its porosity and open cell. Moreover, the compressive strength of the carbon foam increases with an increase in the PNCE/BACE. When the PNCE/BACE mass ratio varies from 0 to 0.75, the compressive strength of the carbon foam prepared increases from 3.25 to 9.6 MPa.

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1. Introduction

Carbon foams exhibit a large variety of excellent properties, such as low density, high surface area, excellent corrosion resistance, high temperature stability, and controllable thermal and electrical conductivity [1], and therefore, have attracted increasing attention in many fields ranging from aeronautics, astronautics, chemicals, electronics, energy, environmental protection to catalysis [2–5]. However, poor mechanical strengths and high production costs of carbon foams hinder their development and application [6–8]. As is well known, the conventional production of carbon foams undergoes the preparation of green foams from pitches, resins, coals or biomass materials followed by lengthy stabilization and pyrolysis [9–14], during which structural defects like cracks easily form deteriorating the mechanical strengths of resulting carbon foams when thermal decomposition causes large shrinkage stress. In order to improve the mechanical strengths of carbon foams, high pressure technique or complex process is usually employed, which no doubt increases their production costs [15–16]. Therefore, it is necessary

to develop an efficient method to improve the mechanical strengths of carbon foams and reduce their production costs [17].

Various approaches have been developed to prepare carbon foams with relatively high mechanical strength [18–21], among which new thermosetting resins are employed to prepare carbon foams with relatively high mechanical strengths [22–24]. In our previous work, we reported that bisphenol A cyanate ester (BACE) resin possessed self-foaming characteristics at high temperatures; the carbon foam with well-developed pore structures could be easily fabricated by direct pyrolysis of the resin [6]. However, its mechanical strength was still low (about 3 MPa), which could be attributed to the fact that the cured resin had a low carbonization yield of about 30% at 1000 °C. Phenol novolac cyanate ester (PNCE) is a kind of cyanate esters with rich methylene and phenyl groups as illustrated in Fig. 1, and meanwhile, it possesses a larger number of cyanate groups than BACE and the cured PNCE resin has higher carbonization yield than cured BACE resin [25]. Therefore, the PNCE resin seems to be a promising precursor for preparing carbon foams with relatively high mechanical strengths. However, it is found that carbon foam cannot be prepared by direct pyrolysis of the PNCE resin with poor self-foaming characteristics at high temperatures, due to its high cross-linking density. In this paper, we attempt the preparation of carbon foams by direct pyrolysis of CE resins from the mixtures of BACE and PNCE, and then structures and properties of the resulting carbon foams were studied.

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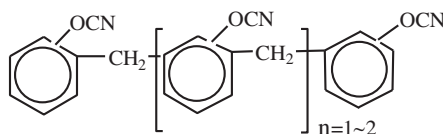


Fig. 1. Chemical structure of PNCE.

Table 1
Some characteristics of the PNCE used in this study.

Property	Value
Appearance	Brown liquid
Solid content	80% (Butanone, 20%)
Gel time	15 min/200 °C
Brookfield viscosity at 25 °C	200 mPa s

2. Experimental

2.1. Raw materials

BACE (99% pure) and PNCE were purchased from Jinliyuan curatorial Co., Ltd., China. Some properties of the PNCE were included in Table 1. *N,N*-Dimethylformamide (DMF) was in analytical grade and supplied by Guoyao Chemical reagent Co., Ltd., China.

2.2. Preparation of CE resins

BACE was mixed with a certain amount of PNCE in DMF under stirring. The DMF was then evaporated with vacuum distillation. The mixture was vigorously stirred at 130 °C for 30 min to obtain a CE prepolymer. The prepolymer was degassed in a vacuum oven at 140 °C for 30 min, and then cast into a steel mold for curing via the procedures of 160 °C/2 h + 180 °C/2 h + 200 °C/2 h + 220 °C/5 h. The resultant CE resins were labeled PNCE/BACE-0, PNCE/BACE-0.25, PNCE/BACE-0.5 and PNCE/BACE-0.75 when the PNCE/BACE mass ratio was 0, 0.25, 0.5 and 0.75, respectively.

2.3. Pyrolysis of the CE resins

The cured CE resin samples were put into a furnace, heated up to 1000 °C at a rate of 5 °C/min, and kept for 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 smooth surfaces of the pyrolysis products were ground off using sandpaper to obtain resultant carbon foams. The carbon foams were labeled CF-0, CF-0.25, CF-0.5

and CF-0.75 when the CE resin used was PNCE/BACE-0, PNCE/BACE-0.25, PNCE/BACE-0.5 and PNCE/BACE-0.75, respectively.

2.4. Characterization

The carbon, hydrogen, and nitrogen weight percentages of samples were determined using a Vario EL III elemental analyzer.

For thermogravimetric analysis (TGA) of the AMB resin, ca 10 mg of sample, placed in a platinum crucible, was heated to 1000 °C at 5 °C min⁻¹ using a TA SDT-Q600 thermogravimetric analyzer under a nitrogen flow of 100 ml min⁻¹.

XRD analysis of the samples was carried out in Rigaku Ultima III diffractometer with Cu K α radiation ($\lambda = 0.154056$ nm). XRD was performed on powder samples with a 40 kV generator voltage and a 35 mA current, and the scan range $2\theta = 10\text{--}70^\circ$.

Morphological measurements of samples were examined by a Hitachi S-3400N scanning electron microscope. The accelerated voltage was 20 kV, and the samples are coated with aurum in vacuum.

The density of the samples was determined by weighing the geometrically shaped foam and dividing the weigh by the measured volume. A Micromeritics AccuPyc 1330 pycnometer was used to measure the true density (helium method) of carbon foam samples. The porosity and percentage of open cell were calculated by the following formula [26]:

$$\text{Porosity (\%)} = \left(\frac{1 - D^*}{D_p} \right) \times 100 \quad (1)$$

$$\text{Open cell (\%)} = \left(\frac{D_s}{D_p} \right) \times 100 \quad (2)$$

where D^* is the bulk density, D_s is the skeleton density of the sample, D_p is the true density of pulverized sample.

For the compressive tests, the samples were machined to cylinders of 12 mm in diameter and 10 mm in height. The tests were carried out at room temperature on an electron universal testing machine (model CMT-5505) and the cross-head speed applied was 2.5 mm/min. The compressive strength was determined from the fracture failure of the samples. All the results were calculated based on the average of five tests.

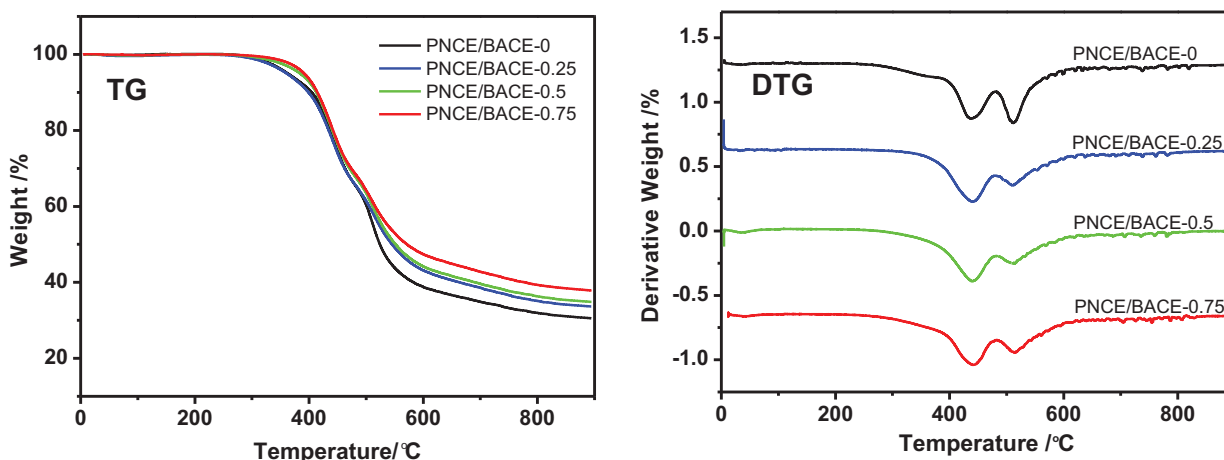


Fig. 2. TG–DTG curves of the CE resins prepared with different PNCE/BACE mass ratios.

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