



Preparation and characterization of carbon foam derived from pitch and phenolic resin using a soft templating method



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ARTICLE INFO

Article history:

Received 30 March 2014

Accepted 11 September 2014

Available online 28 September 2014

Keywords:

Carbon Foam

Thermal insulation

Phenolic resin

Porosity

ABSTRACT

Carbon foam has been developed by soft templating method with phenolic resin and coal tar pitch as matrix precursor and polyurethane (PU) foam as an organic sacrificial template. Micron sized powdered pitch was mixed well in a diluted resin and the PU foam was soaked in it. The impregnated foam was dried at 70 °C for 6 h. This impregnation–stabilization (IS) process was repeated for four and six times. A slow curing cycle with hold steps at 80, 110, 140 and 2000 °C was used for the sample being pressed isostatically in a steel mold. Carbonization at 800 °C in a reduced and inert environment resulted in a geometric density of 0.48 and 0.60 g/cm³ for IS cycles of four and six respectively. The carbon foam was characterized by porosity calculations, thermogravimetric analysis, compressive strength and scanning electron microscopy. The developed carbon foam showed an open porosity of 63–67%, compressive strength of 13.3–19.5 MPa and pore size distribution of 10–500 μm. An experimental setup was designed for the testing of carbon foam as a high temperature thermal insulation. The carbon foam specimen was heated to 975 °C on the one face and the other face was monitored. The experiment was repeated with a carbon-felt specimen and the insulation indices compared. The carbon foam showed a higher insulation index but lower insulation density index due to higher density than that of the carbon felt.

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1. Introduction

Carbon foams are rigid and porous materials with certain attractive features like being lightweight, having high temperature stability (~3000 °C in inert atmosphere), moderate compressive strength and tailorable thermal and electrical conductivities [1–3]. Typically, carbon foams are prepared from organic precursors like phenolic resin, coal or mesophase pitches [4–7]. The precursor and the process with controlled process parameters determine the properties of the resulting foam. The template carbonization process has been regarded as the most effective approach to produce carbon foam with controlled physical and chemical properties. The development of mesoporous and macroporous, as well as microporous carbon foam is of great importance in many analytical applications like adsorption, separation, sieving, catalysis, purification and ion exchange etc. [8,9]. Hard templating offers a rigid and nano-casting mold to replicate in a porous interconnected network. The resulting carbon foam is very homogeneous with a

narrow pore size distribution on a nano or micro scale. On the other hand, the soft templating method is a cooperative formation of replicating agent and the organic precursor [10]. The soft template itself converts into carbon foam during carbonization of the organic precursor and hence the control of properties is somewhat difficult. Impregnation of a precursor into the template, the degree of cure and the temperature are the key controls, which offer a great challenge especially for thick PU samples. The commonly used organic precursor is a phenolic resin. Carbon foam derived from phenolic resin can be used for the high-strength thermal insulations in aerospace, nuclear, cryogenics and building industry [11–13]. Other foam insulations like polyurethane (PU) foams, polyethylene foams and polystyrene foams are the most popular and the cost effective thermal insulations, but easy to ignite. Carbon felt is a practical solution for most of the industrial high temperature insulations such as degassing, brazing, annealing, sintering carburizing and graphitizing furnaces but it tears off at high gas velocities. Moreover, the thermal and mechanical properties of a carbon felt are dependent on the fiber properties, which is non-isotropic in nature. Carbon foam provides the similar insulation properties with added advantage of structural rigidity and isotropy. Ultramet prepared carbon foam by impregnating PU foam with phenolic resin and then carbonizing at high-temperature [14–16].

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Table 1
Characteristics of coal tar pitch used as filler in carbon foam.

Density (g/cm ³)	Softening Point (°C)	Melting point (°C)	Carbon content (wt%)	Volatile (wt%)	QI (wt%)	TI (wt%)	Sulfur (wt%)	Ash contents (wt%)
1.25	85	125	>90	62–57	<12	~30	0.75	0.2

The PU foam used for impregnation should be open celled consisting of a network of interconnected struts. Coal derived and pitch based carbon foams usually have high thermal conductivities and lower structural strengths [17]. No reference is available in literature where both phenolic resin and pitches are used simultaneously for the preparation of carbon foam using PU a soft template. Chen et al. [18] used first time PU foam as template with a high softening point pitch and organic binders. Yadav et al. [19] attempted to develop the graphite foams of different densities by impregnating mesophase pitch and its water slurries in the presence of polyvinyl alcohol into the PU foams of different densities as a template material and then carbonizing and graphitizing the same up to 2400 °C. The aim of the present investigation is to develop a relatively simple method for hybrid resin/pitch based carbon foam offering potential improvements in terms of both mechanical strength and insulating performance. The effect of adding pitch can be further investigated by high temperature treatment above 1400 °C or graphitization at ≥ 2000 °C. Pitches have a high carbon content and are graphitizable after high-temperature treatment (HTT) [20–23].

2. Experimental procedure

In this work, slurry of powdered pitch in a diluted phenolic resin was prepared and a semi-rigid PU foam (commercially available, yellow color, 0.07 g/cm³ density, 0.40 mm average pore size, 8–12% carbon contents as determined by TGA) was soaked in it. After curing, the green foam (GF) was stabilized/post cured at 200 °C and carbonized at 800 °C. The carbon foam thus prepared was highly insulating and characterized for density, porosity, compressive strength, thermal stability and finally, on a pilot-scale, as a high-temperature thermal insulation. An experiment was designed in which a laboratory-scale furnace was used to create the thermal gradient in the carbon foam sample. Cylindrical shape carbon foam was fixed in the exhaust cavity such that the lower face was exposed in the heating chamber. Thus, the furnace temperature was the temperature of the inserted lower face, hereafter called as hot-face (HF), of the carbon foam. A thermocouple was used to record the upper face, hereafter called the cold-face (CF), temperature. The temperature-time data for the HF and the CF was plotted and compared the trends with that of the commercially available carbon felt.

2.1. Raw materials

Commercially available semi-rigid PU foam, after washing and drying, was tested for density and carbon contents. The phenolic resin was prepared by the polymerization of industrial-grade phenol and 37% analytical grade formaldehyde (mole ratio 1:1.6) at 96–100 °C with barium hydroxide as a catalyst. Processing details can be found in [24–27]. The phenolic resin has a number-average molecular weight of preferably 300–600. Properties of coal-tar pitch (Wuhan, China) used to increase the carbon yield of carbon foam during the carbonization process are shown in Table 1. Analytical grade isopropanol (purchased from Sinopharm Chemical Reagent Beijing Co., Ltd.) was used to lower the viscosity of the phenolic resin/powdered pitch impregnating mixture.

2.2. Development of impregnation and curing scheme for green foam

Experimental activities were first focused on the homogeneous impregnation of the PU foam. For this, impregnation cycles with diluted resin/pitch mixtures, pre-curing stabilization at 70 °C, compression of PU foam isotropically and curing temperatures were carefully monitored and controlled. Diluted resin/pitch mixture was made by mixing 50 gm of resin in 50 gm isopropanol and 10 gm pitch powder. A slow speed mixer was used to avoid agglomeration and ensure homogeneous mixing. Rectangular shaped PU foam with dimensions 100 mm \times 50 mm \times 50 mm was immersed completely in the slurry for homogeneous wetting. Then the excess resin was squeezed out gently using a roller mill and the wetted foam was placed in a forced air circulation oven at 70 °C for 6 h. The sample was placed on an absorbing mat and after every 1 h, it was turned upside down for avoiding uneven settling of the resin. This impregnation–stabilization (IS) process was repeated 4–6 times depending upon the final properties of the carbon foam, for example more cycles for the higher final density.

After the IS process, the volume of the foam increased about 8–10%, but its appearance was kept porous. It was then placed in a steel container which was already fabricated as per size of the unimpregnated PU foam. Therefore, when the expanded IS foam was put in the container, a slight isostatic pressure was applied on it. The temperature and time of the curing cycles are the key parameters for avoiding cracks in the stiffened green foam [28,29]. The viscosity of the resin reduced to minimum at about 95–100 °C and due to the presence of the solvent, the first holding step was selected at 80 °C for 12 h. No hold at 80 °C resulted in excessive bubble formation and non-homogeneous cured green foam. The temperature was then increased to 110 °C at a very slow rate and held for 2 h. This step ensured polymerization of the resin with a rubbery appearance. Finally heating up to 140 °C ensured stiffening of the foam. After cooling, the green foam (GF) was easily demolded as it shrank to about 2–5% in original size of the PU foam. For post-curing and stabilization, the demolded GF was heat treated at 200 °C for 4 h in air atmosphere.

2.3. Carbonization

This step involved the decomposition of the pitch, resin and PU foam, eliminating CO₂, CO, H₂, O₂, CH₄ etc., leaving behind the fixed rudimentary pore structure [30]. The process was carried out in both inert and reducing environment. In the coking furnace, the samples were buried in the coke breeze (three times the volume of the GF) and the process was carried out at 800 °C in a continuous stream of N₂. At the first stage of the carbonization process, a slow heating rate of 10°/h was selected until 450 °C. The slow rate minimized the risk of cracks produced due to evolution of volatiles from the sample. Some of the pores formed at this stage were blocked by the tarry products formed during pyrolysis [31]. After 450 °C, the rate was increased to 40°/h up to 800 °C and held for 4 h. The coke breeze, being reusable, served multiple functions other than producing a reduced environment. It controlled the flow of volatiles, even temperature distribution, slight pressurization on the sample and preserved heat during the cooling cycle. In some trial carbonizations, the samples carbonized without coke breeze showed some cracks on the surface of the samples. After

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