



Carbon foams with a triplex pore structure by compression molding of molten sucrose–NaCl powder pastes



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ARTICLE INFO

Article history:

Received 17 November 2016

Received in revised form

23 March 2017

Accepted 27 March 2017

Available online 30 March 2017

ABSTRACT

A compression molding process for the preparation of carbon foams with a triplex pore structure has been studied. The pastes with moldable consistency, prepared by hot-blending of sucrose–NaCl powder mixtures of NaCl to sucrose weight ratios in the range of 0.7–1.2, set into solid bodies due to the caramelization of molten sucrose during the hot pressing. The caramelization in presence of NaCl results in carbon-enriched polymeric structure due to the enhanced α -elimination reactions along with the –OH condensation as evidenced from torque-time measurements, IR, TGA and elemental analysis. The pyrolysis of compression molded bodies, followed by NaCl removal and further heat treatment results in carbon foams with a hierarchical triplex pore structure. The shrinkage during carbonization (28.5–24.2 vol%) and density (0.26–0.33 g/cm³), compressive strength (2.84–8.37 MPa), Young's modulus (170–370 MPa) and thermal conductivity (0.348–0.257 W/m.K) of the carbon foams depend on the NaCl to sucrose weight ratio. The carbon foams have a high specific surface area of ~400 m²/g and a combination of macropores (414–70 μ m), microcells (2–12 μ m) and micropore texture.

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1. Introduction

Carbon foams are getting renewed interest due to their light-weight, tunable electrical and thermal conductivities, fire resistance, high electromagnetic interference shielding effectiveness and acoustic absorption property [1–5]. They are prepared from either fossil fuel based precursors such as pitches and synthetic polymers or natural renewable molecules of plant origin such as tannin, lignin and sucrose [6–17]. The general strategy for the preparation of carbon foams involves foaming and setting of the precursor materials followed by pyrolysis and graphitization [1–17]. Template based methods are also used for the preparation of carbon foams. Polyurethane foams are used as a template for the preparation of reticulated carbon foams. In this, the carbon precursor materials coated on the webs of polyurethane foam template by impregnation and curing is pyrolyzed to replicate the polyurethane foam structure in carbon [18–22]. Sodium chloride (NaCl) particles are also used as a template for the preparation of carbon foams [23]. In this, a compact of NaCl template particles prepared by pressing is infiltrated with the carbon precursor such as

mesophase pitch. The pitch infiltrated into the interparticle porosity in the NaCl compact is pyrolyzed after an oxidation stabilization heat treatment. The NaCl present in the pyrolyzed body is removed by leaching with water to produce the carbon foam. Preparation of cellular carbon foam from lignin–resorcinol–formaldehyde polymer using poly(methyl methacrylate) particles as pore template is also reported [24]. The majority of the foaming and template based methods reported in the literature produce carbon foams with relatively large cell size [1–25]. The large cell size makes the carbon foams fragile. This results in debris formation during storage and transportation of carbon foam bundles. The debris formation is a serious concern during construction, maintenance and cleaning of carbon foam based structures [26].

Decreasing the cell size is the way to increase the compressive strength and debris formation resistance of carbon foams. Foams with cell sizes in the range of 0.1–100 μ m are called microcellular foams. The processing of microcellular polymer foams is well reported in the literature [27]. There are few methods reported for the preparation of microcellular carbon foams. One of the methods is by the pyrolysis of microcellular poly (acrylonitrile) foam obtained by a phase inversion process [28–30]. Infiltration of phenol–formaldehyde resin into a sintered NaCl powder compact followed by curing, pyrolysis and NaCl removal is another method reported for the preparation of microcellular carbon foam [31,32]. High

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pressure foaming of an ethanol solution of phenol-formaldehyde resin in an autoclave followed by pyrolysis is used for the preparation of microcellular carbon foams with high compressive strength [33]. High pressure foaming of pitch in an autoclave followed by pyrolysis and graphitization is also reported for the preparation of microcellular carbon foams [34,35]. In the present work, we report a compression molding process for the preparation of carbon foams with a triplex pore structure containing large pores (414–78 μm), microcells (2–12 μm) and micropore texture from sucrose and sodium chloride.

2. Experimental

2.1. Preparation of carbon foam

The analytical reagent grade sucrose, NaCl and acetone were procured from Merck India Ltd. Mumbai. The flowchart of the process for the preparation of carbon foams is shown in Fig. 1. Various amounts of sucrose and NaCl powders were mixed by planetary ball milling (Fritsch, Germany) in an acetone medium for 2 h at a speed of 200 RPM using zirconia grinding balls of 10 mm diameter and zirconia jar of 500 ml capacity. The powder to ball and powder to acetone weight ratios were 1:2 and 3:4, respectively. The use of a solvent as a milling medium was essential as the dry milling of sucrose-NaCl mixture yielded a cake-like mass that does not ensure proper particle size reduction of NaCl. The selection of acetone as a medium for ball milling was due to the low solubility of NaCl (0.00042 g/Kg) and sucrose (7 g/Kg) in acetone. The slurry obtained after the ball milling was dried in an air oven at 70 °C in a borosilicate glass tray. The low boiling point of acetone enabled easy drying of the slurry obtained after ball milling. The sucrose-NaCl mixtures thus obtained were heated in an air oven at 185 °C

to melt the sucrose and thoroughly stirred with a wooden ladle to form a paste. The paste of NaCl powder in molten sucrose was transferred into a stainless steel mould with a rectangular cavity of 10 cm \times 10 cm \times 2.5 cm size. The mold was then covered with its lid and gently hot pressed in a hydraulic press for 3 h. The temperature of the upper and lower platens of the press was kept at 195 °C. The sample was removed from the mould after cooling to room temperature. The sample removed from the mold was annealed at 200 °C for 2 h in an air oven. The sample was then heated in an argon atmosphere at 750 °C for 2 h. The heating rate used was 1 °C/minute. The sample was removed from the furnace after cooling to room temperature. The same inert atmosphere was maintained during the cooling. The sample heat treated at 750 °C was immersed in distilled water at 90 °C until the whole NaCl present in the body was leached out. The NaCl removed body was further heat treated at 900 °C in the inert atmosphere furnace for 2 h at a rate of 1 °C/minute. The density of the thus obtained carbon foams was determined from their weights and dimensions. Shrinkage during the heat treatments was calculated from their initial and final dimensions. The skeletal density of the carbon foams was measured using a helium pycnometer (AccuPyc II 1340 Pycnometer, Micromeritics, USA).

2.2. Torque measurement

The variation of torque with time of the NaCl paste was studied at 160 °C using a torque rheometer [Brabender Plasti-Corder, GmbH, Germany]. Nearly 50 cm³ of the sucrose-NaCl powder mixture prepared by planetary ball milling was loaded into the internal mixing unit of the rheometer which was set at a temperature of 185 °C and at a rotor speed of 10 RPM. The temperature was lowered to 160 °C after 5 min at which sucrose in the mixture properly melted and mixed well with the NaCl to form a paste. The torque data was collected with time until it reaches 100 Nm.

2.3. NaCl removal

The compression molded sucrose-NaCl paste after carbonization at 750 °C was immersed in 800 ml distilled water taken in a 1 L beaker. The mouth of the beaker was covered with an aluminium foil and tied with a plastic rope (Fig. S1 in Supplementary information). The beaker containing the carbonized body immersed in water was then heated in an air oven at 90 °C. The water was replaced with fresh water after every 4 h until the removal of the whole NaCl present. The completion of NaCl removal was confirmed by testing the spent water for chloride ion using AgNO₃ solution. The NaCl content in the spent water was estimated by titrating against standard AgNO₃ solution using potassium chromate indicator [36]. The spent water was first made up to 1 L and then appropriately diluted with distilled water before the titration.

2.4. Thermogravimetric analysis

Thermogravimetric analysis (TGA) was carried out in air and nitrogen atmosphere using a thermogravimetric analyzer (Q-50, TA Instruments, USA). The heating rate used was 5 °C/minute. The sucrose polymer sample for TGA was obtained by NaCl removal by soxhlet extraction of sucrose polymer-NaCl composite body followed by drying.

2.5. Infrared spectrum, XRD and elemental analysis

The infrared spectrum of the sucrose polymer was recorded using an FTIR spectrophotometer (Spectrum-150, Perkin Elmer,

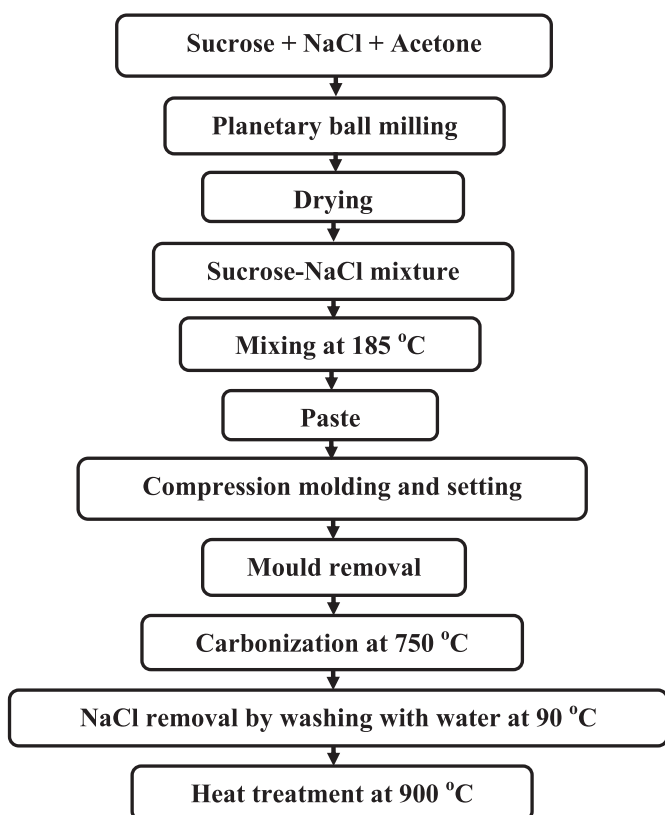


Fig. 1. Flowchart for the preparation of carbon foams.

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