

Carbon particle induced foaming of molten sucrose for the preparation of carbon foams



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ABSTRACT

Activated carbon powder was used as a foaming and foam setting agent for the preparation of carbon foams with a hierarchical pore structure from molten sucrose. The rheological measurements revealed the interruption of intermolecular hydrogen bonding in molten sucrose by the carbon particles. The carbon particles stabilized the bubbles in molten sucrose by adsorbing on the molten sucrose–gas interface. The carbon foams obtained at the activated carbon powder to sucrose weight ratios in the range of 0–0.25 had a compressive strength in the range of 1.35–0.31 MPa. The produced carbon foams adsorb 2.59–3.04 mmol/g of CO₂ at 760 mmHg at 273 K and absorb oil from oil–water mixtures and surfactant stabilized oil-in-water emulsions with very good selectivity and recyclability.

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1. Introduction

Carbon foams are new generation materials used in high temperature thermal insulations, ablative structures in thermal protection systems, acoustic absorption, electromagnetic interference shielding, catalyst support and electrodes due to their superior thermal, electrical and electrochemical properties [1–8]. They are light-weight fire resistant materials used as the core in sandwich composite structures for aerospace and ship building applications [9]. Recently, the carbon foams with large surface area and tunable surface chemistry find a number of environmental applications [10]. The carbon foams with hydrophobic surface are demonstrated to absorb oil from oil–water mixture and dyes from solutions, etc. [11,12]. Preparation of carbon foams from petroleum and coal derived pitches is well reported in the literature [2,13,14]. The carbon foams are also prepared by carbonization of organic polymer foams which leave a considerable amount of carbon during pyrolysis. Polymers such as phenol–formaldehyde, melamine formaldehyde, polyfurfural, polyimide, poly(arylacetylene) and cyanate ester resin are used for the preparation of carbon foams [14–17]. The polymer foams for the preparation of carbon foams are obtained by foaming and setting of the respective organic resins using a suitable blowing agent and a surfactant. In this, the low boiling liquids such as diethyl ether, hexane and heptane are used as

the blowing agent to create bubbles in the organic resin medium and the bubbles are stabilized by the adsorption of the surfactant molecules at the resin–gas interface.

Replacement of petroleum based raw materials with natural renewable resources for the preparation of industrial products is very important for sustainable development. Natural renewable materials such as tannin, sucrose and lignin, are studied for the preparation of carbon foams [7,18–26]. Processing of sucrose to carbon foams by foaming of aqueous sucrose resin and molten sucrose is studied [18,21,22]. In this, the water vapour produced by the acid catalyzed –OH to –OH condensation is used for the blowing of the sucrose resin and the generated bubbles are stabilized by the viscosity increase due to the –OH condensation. Nitric acid, aluminium nitrate and boric acid are used as catalyst for the –OH to –OH condensation for the foaming and setting of sucrose [18,19,21,22]. The carbon foams produced from sucrose have relatively low density and compressive strength due to the low carbon yield of sucrose. Incorporation of activated carbon (AC) powder in aqueous sucrose resin is reported to increase the carbon yield and to improve the density and mechanical strength of carbon foams [23]. Though the preparation of carbon foams from AC powder dispersions is simple in small scale, our attempts to scale up for the preparation large foam bodies met with the following problems even at 1 kg level. The evaporation of the 50% water present in the resin took long time which resulted in the agglomeration and sedimentation of finer AC particles. Sedimentation of the agglomerated AC particles led to inhomogeneous foam structure. The bottom portion of the foam bodies had large and distorted cells

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compared to the top portion. Moreover, very large spherical voids of size more than 1 cm were randomly observed in the foam bodies. These problems can be overcome using a melt based process.

Stabilization of gas–liquid and liquid–liquid interfaces using particles are increasingly used for the preparation of foams and emulsions [27–33]. In this, the particles with suitable wetting characteristics irreversibly adsorb on the air–liquid and liquid–liquid interfaces. The possibility to control the wetting characteristics of the particles by modifying their surface using amphiphilic molecules further widen the scope of particle stabilized foams. The particle stabilized foams and emulsions are more stable than the surfactant stabilized counterparts. The particle stabilized foams are resistant to foam drainage and bubble coalescence by Ostwald ripening [31]. The stabilization of bubbles by particles is used for the foaming of powder suspensions for the preparation of ceramic and polymer foams [34–36]. The foaming of polymer melts by bubble stabilization using polymer particles of suitable wetting characteristics is also reported [35]. In the present work, the foaming of molten sucrose by activated carbon particles is used for the preparation of carbon foams. The AC particles not only stabilize the bubbles by adsorbing on the molten sucrose–gas interface but also increase the carbon yield. In addition, the acidic surface functional groups present in the AC particles catalyze the –OH to –OH condensation leading to faster foaming and setting of the molten sucrose. The carbon foams produced showed good CO₂ adsorption and oil absorption from oil-in-water emulsion.

The molten sucrose based process expected to have a number of advantages over the aqueous sucrose resin based process [21,22]. Primarily, the molten sucrose based process avoids the aqueous sucrose resin preparation step. The thermo-foaming step consumes less amount of energy in molten based process as large amount of energy is being spent for the evaporation of water present in the aqueous sucrose resin. Further, the high viscosity of molten sucrose prevents the agglomeration and sedimentation of AC particle. This results in a homogenous foam structure from top to bottom, even in large foam bodies, unlike the aqueous sucrose resin based process. In addition, the molten sucrose based process is green compared to all reported processes for the preparation of carbon foams from sucrose as it avoids the harmful additives such as nitric acid, aluminium nitrate and boric acid used in the earlier processes [18,19,21,23].

2. Experimental

2.1. Materials

The analytical reagent grade sucrose, acetone and AC powder used were procured from Merck India Ltd., Mumbai. The wood derived AC powder used was amorphous and contains particles in the size range of few micrometres to few hundred micrometres. The AC powder received from the supplier was planetary ball milled in acetone medium for 1 h using zirconia jar and zirconia grinding balls. The AC powder to the ball weight ratio used was 1:10. The average particle size (measured using a particle size analyzer) and surface area (measured using a surface area analyzer) of the ball milled AC powder was 7 μm and 883 m²/g, respectively.

2.2. Preparation of carbon foams

The flow chart for the preparation of carbon foam is shown in Fig. 1. The sucrose (300 g) and various amounts of the AC powder were intimately mixed in a mixer-grinder for 10 min. The sucrose–AC powder mixtures were heated in glass trays at 170 °C to melt the sucrose and then stirred well with a glass rod to achieve uniform dispersion of the AC particles in the molten sucrose. Thus

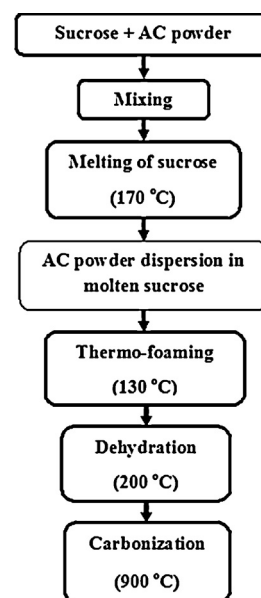


Fig. 1. The flow chart for the preparation of carbon foam.

obtained AC powder dispersions in molten sucrose were heated in an air oven at 130 °C for 36 h for foaming and setting. The organic foams obtained were dehydrated at 200 °C in an air oven for 3 h. The heating rate used was 0.5 °C/h. The dehydrated foams were carbonized by heating in the highly pure argon atmosphere at 900 °C for 2 h. The heating rate used was 2 °C/min. The samples were unloaded after cooling the furnace to room temperature. The inert atmosphere was maintained throughout the cooling.

2.3. Characterization of carbon foams

The functional group characterization of the carbon foams was carried out by using Fourier transform infrared spectroscopy (FTIR) (Perkin Elmer, USA) with a resolution of 4 cm⁻¹ over the spectral range of 4000–400 cm⁻¹. The samples were thoroughly mixed with dry KBr and made into pellets by using a hydraulic press at 5 kN for 5 min. Boehm titration was used to characterize the acidic oxygen surface functional groups on the activated carbon [37,38]. The carbon samples of mass 1.5 ± 0.001 g each was added to 50 ml of 0.05 M solution of NaOH, NaHCO₃ and Na₂CO₃ taken in different iodine flasks. The solutions were closed tightly and shaken using wrist shaker for 24 h. After 24 h, the contents in the iodine flasks were filtered through Whatman filter papers to collect the solutions. 10 ml of aliquot taken from each solution was acidified using 50 ml of 0.05 M HCl and then back titrated with 0.05 M standardized NaOH solution using phenolphthalein indicator. The carboxylic, lactonic and phenolic functional groups present on the AC powder surface were calculated from the titre values as reported in the literature [38].

The viscosity measurement of the AC powder dispersions in molten sucrose was carried out at various shear rates in the range of 1–1000 s⁻¹ at 130 °C using a rheometer (MCR 102 Modular Compact Rheometer, Anton Paar, USA) with a cone and plate measurement system. The foam rise (a measure of foam volume) was calculated as the ratio of the initial height of the AC powder dispersion and the final height of the foam. The samples were physically inspected during foaming and foam setting at an interval of 1 h to determine the approximate foaming and foam setting time. The densities were calculated from the weight and dimension of rectangular carbon foam bodies. The microstructure of the carbon foams was observed using a Scanning Electron Microscope

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