



# Preparation of alumina foams by the thermo-foaming of powder dispersions in molten sucrose

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## Abstract

The alumina powder disperses in molten sucrose due to the hydrophilic interaction between the particle surface and sucrose hydroxyls. The thermo-foaming of the dispersions is due to the bubbles created by the water vapour produced by the  $-OH$  condensation at  $150^\circ C$  which are stabilized by the alumina particles adsorbed on the gas–liquid interface as well as the increase in viscosity. The foaming time, the foam setting time and the foam volume depend on the alumina powder to sucrose weight ratio. The alumina foams have interconnected cellular microstructure and the cells are having a near spherical morphology. The porosity (97.84–93.29 vol.%) decreased and the average cell size (0.54–1.2 mm) increased with the increase in alumina powder to sucrose weight ratio (0.4–1.4). The alumina foams with density in the range of 0.239–0.267 g/cc showed compressive strength in the range of 1.02–1.47 MPa.

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## 1. Introduction

Ceramic foams are increasingly used in various applications such as molten metal filtration, catalyst support, thermal insulation, environmental pollution control, biomedical implants, pre-form for composites *etc.*<sup>1–5</sup> They are generally prepared either by the polymer foam replication method or by the foaming of the ceramic powder suspensions followed by setting. In the former method, the ceramic replica of a reticulated polymer foam is prepared by impregnating the polymer foam with the ceramic powder suspension followed by drying, burnout of the polymer foam and sintering.<sup>6–9</sup> In the latter method, gas bubbles are stabilized in the ceramic powder suspensions using suitable surfactants and the foamed suspensions are then set into gelled bodies. The subsequent drying of the gelled bodies followed by the polymer burnout and sintering produces ceramic foams with interconnected cellular structure.<sup>10–14</sup> *In situ* polymerization of the organic monomers dissolved in the suspension medium is used for the setting of the foamed suspensions.<sup>10–14</sup>

The non-oxide cellular foam ceramics are prepared by the foaming and setting followed by the pyrolysis of the pre-ceramic polymers.<sup>15,16</sup>

The current processing methods for the preparation of ceramic foams use synthetic organic polymers and monomers, surfactant molecules and solvents, which are produced from petroleum, as the processing additives. Replacement of these processing additives with non-toxic naturally renewable molecules is of great economic and environmental importance. Natural renewable molecules such as proteins and polysaccharides are studied for the foaming and setting of aqueous powder suspensions for the preparation of cellular ceramic foams.<sup>17–19</sup> Pradhan and Bhargava used soapnut, a naturally occurring surfactant, for the foaming and gum augur for the setting of alumina powder suspensions for the preparation cellular alumina foams.<sup>20</sup> Sucrose is another naturally renewable molecule of agricultural origin used in the preparation of ceramic foams. Dhara et al. used sucrose to control the drying shrinkage of wet-alumina foams prepared from ovalbumin-based aqueous alumina slurries.<sup>21,22</sup> Prabhakaran et al. reported the foaming of alumina powder suspensions in an aqueous resin prepared by heating acidic sucrose solutions using aluminium nitrate as blowing agent.<sup>23</sup> Jana and Ganesan reported the preparation of alumina foam with 98.5% porosity by foaming and

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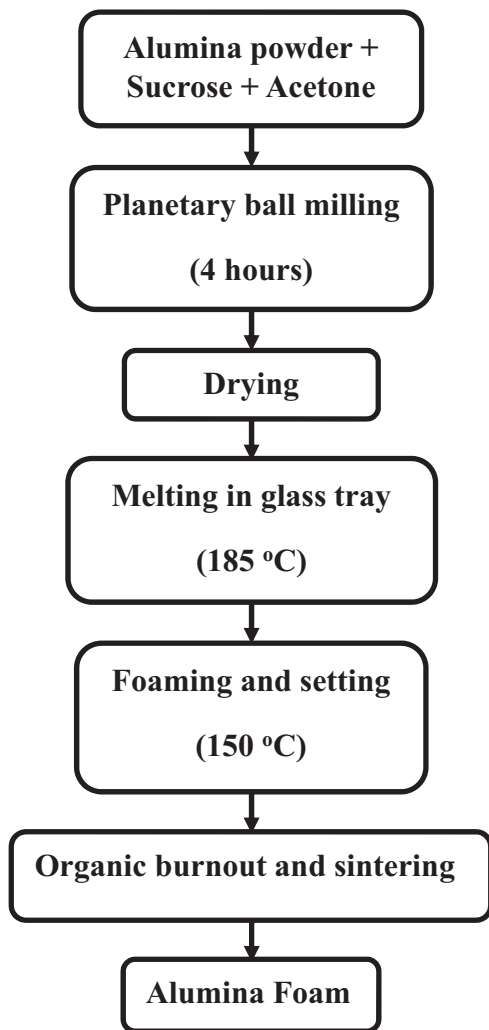


Fig. 1. Flowchart of the thermo-foaming process for the preparation of alumina foams.

the setting of a resin prepared by heating sucrose and aluminium nitrate followed by organic burnout and sintering.<sup>24</sup> The aluminium nitrate used as blowing agent as well as a precursor for the alumina produces nitrous fumes during foaming and setting which are hazardous to the environment.<sup>23,24</sup> The present work reports the thermo-foaming of alumina powder dispersions in molten sucrose for the preparation of alumina foams with a range of porosities. This is a solvent and surfactant free process for the preparation of alumina foams which avoid the use of all synthetic and toxic processing additives.

## 2. Experimental

$\alpha$ -Alumina powder (A16SG, ACC Alcoa, Kolkata) of average particle size  $0.34\ \mu\text{m}$  and specific surface area  $10.4\ \text{m}^2/\text{g}$  was used. Analytical reagent grade sucrose and acetone used were procured from Merck India Ltd., Mumbai. The process flowchart for the preparation of alumina foams is shown in Fig. 1. The sucrose (200 g) and alumina powder in various weight ratios were intimately mixed by planetary ball milling

(Fritsch, Germany) in acetone medium for 4 h using zirconia grinding media of 10 mm diameter in 500 ml zirconia jars. The sucrose to acetone and sucrose to zirconia balls weight ratios were 1:3 and 1:6, respectively. The ball milling speed was 200 rpm. The slurries thus obtained were dried in glass trays at  $80\ ^\circ\text{C}$  in an air oven. The sucrose–alumina powder mixtures were heated in 2.5 l borosilicate glass trays at  $185\ ^\circ\text{C}$  in an air oven for the melting of sucrose. The melt was stirred well with a glass rod to get uniform dispersion of the alumina powder in the molten sucrose. The alumina powder dispersions in the molten sucrose were kept in the air oven at  $150\ ^\circ\text{C}$  for foaming and setting. The foams were cut into rectangular bodies of  $6\ \text{cm} \times 6\ \text{cm} \times 4\ \text{cm}$  and heated in an electrically heated furnace up to  $1600\ ^\circ\text{C}$  for the removal of the organics and sintering. The heating rates used were  $30\ ^\circ\text{C}/\text{h}$  and  $120\ ^\circ\text{C}/\text{h}$  from room temperature to  $600\ ^\circ\text{C}$  and from  $600\ ^\circ\text{C}$  to  $1600\ ^\circ\text{C}$ , respectively. A holding time of 2 h was given at  $600\ ^\circ\text{C}$  and  $1600\ ^\circ\text{C}$ . The volume shrinkage of the bodies was calculated from the initial and final dimensions. The density of the alumina foam bodies was calculated from their weights and dimensions.

The amount of sucrose dissolved in acetone during ball milling was estimated by evaporating off acetone from the supernatant liquid separated from the sucrose–alumina slurry by centrifugal method. The static contact angle of molten sucrose on an alumina surface was measured by the sessile drop method using a Goniometer (OCA 20, Dataphysics, Germany). The sample for contact angle measurement was prepared by melting a small grain of sucrose on a polished alumina surface in an air oven at  $180\ ^\circ\text{C}$ . The viscosity measurement of the alumina powder dispersions in molten sucrose was carried out at various shear rates in the range of  $2\text{--}210\ \text{s}^{-1}$  at  $150\ ^\circ\text{C}$  using a rheometer (MCR 102 Modular Compact Rheometer, Anton Paar, USA) with a cone and plate measurement system (CP-25, 25 mm diameter and angle  $2^\circ$ ). Small amount of the alumina powder dispersions collected from the glass trays before keeping at  $150\ ^\circ\text{C}$  for foaming was used for the viscosity measurements. The alumina powder dispersion samples solidified when cooled to room temperature. The frozen samples were loaded on the viscometer plate and the temperature was raised to  $150\ ^\circ\text{C}$ . The samples were kept at  $150\ ^\circ\text{C}$  for 2 min for forming a uniform melt before starting the viscosity measurements. The thermal decomposition of the polymerized sucrose from the foamed body was studied by thermogravimetric analysis in air atmosphere at a heating rate of  $10\ ^\circ\text{C}/\text{min}$  using a thermogravimetric analyzer (Q-50, TA Instruments, USA). The microstructure of the foams was observed using a scanning electron microscope (SEM, FEI Quanta FEG200). The strut thickness was measured on the magnified image of the alumina foams observed using a vision inspection system with a CCD colour camera (Vision 300 GL, TESA Technologies, Switzerland). The values reported were average of ten measurements. The compressive strength of the alumina foams was measured according to the ASTM standard C365/C365-05 using rectangular samples of  $25\ \text{mm} \times 25\ \text{mm} \times 12\ \text{mm}$  size in a Universal Testing Machine (Instron 5500, Instron, USA) at a loading rate of  $0.5\ \text{mm}/\text{min}$ .

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