



Freeze gelcasting of naphthalene-in-aqueous alumina slurry emulsions for the preparation of macroporous alumina ceramics

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Abstract

An emulsion freeze gelcasting process has been studied for the preparation of macroporous alumina ceramics. The naphthalene-in-aqueous alumina slurry emulsions prepared at 90 °C from a 30 vol% alumina slurry undergo gelation on cooling in a mould due to the solidification of naphthalene and physical cross-linking of the carrageenan present in the aqueous slurry. The medium internal phase emulsions with naphthalene to alumina slurry volume ratios in the range of 1 to 1.86 show shear thinning flow behavior with low viscosity and yield stress, suitable for casting. The room temperature drying of the gelled bodies followed by naphthalene removal at 70 °C and sintering at 1550 °C produces macroporous alumina ceramics with porosity in the range of 65 to 78%. The macropore morphology depends on the emulsifying agent (sodium dodecyl sulphate) concentration and naphthalene to alumina slurry volume ratio. The average macropore size (35.1 to 4.9 μm) decreases with an increase in the emulsifying agent concentration and naphthalene to alumina slurry volume ratio. The macroporous ceramics obtained have high compressive (7 to 28.75 MPa) strength and Young's modulus (553 to 1230 MPa).

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1. Introduction

Macroporous ceramics are increasingly used in applications such as high temperature thermal insulation, catalyst support, electrodes in solid oxide fuel cells, membrane substrate, molten metal filtration, bio-implants and pre-form for polymer ceramic and metal–ceramic composites [1–10]. There are mainly three general methods for the preparation of macroporous ceramics from ceramic powders: (1) using fugitive pore template (2) polymer foam replication and (3) foaming and setting of ceramic powder suspensions [11,12]. In the first method, suitable fugitive particles incorporated in the ceramic powder assembly during processing are burnt off before sintering to create the macropores. Carbon particles, polymer beads, short polymer fibers, wheat particles, starch, crystals of organic molecules, etc, are used as pore templates [13–21]. Though this method is simple, it produces low level of

porosities. The second method involves the coating of flexible polymer foam with a ceramic powder suspension followed by burning off the polymer foam template and sintering to create a ceramic replica of the polymer foam [22–24]. The method produces highly porous reticulated ceramic foams. However, the mechanical strength of these foams is low due to the porous struts. In the third method, a suspension of the ceramic powder is first foamed by using a blowing agent. The foamed suspension cast in a mould is then set either by in situ polymerization of the organic monomers present in the suspension medium or by the coagulation of the suspension [25–30]. The wet foam bodies removed from the mould are subsequently dried and sintered to produce macroporous ceramics of high porosity and interconnected pore structure. Surfactant molecules or particles with suitable wetting characteristics are used for the interim stabilization of bubbles which aid the foaming of the suspensions [30–35].

In addition to these methods, freeze casting of ceramic powder suspensions is widely studied for the preparation of macroporous ceramics [36–40]. In this, a ceramic powder

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suspension cast in a mould is set by freezing the solvent medium. Subsequent removal of the frozen solvent by sublimation followed by sintering, results in macroporous ceramics of a wide range of porosities. The directional solidification by unidirectional cooling of the suspensions produces macroporous ceramics with unidirectionally aligned pores.

Preparation of macroporous polymer and carbon materials using emulsion droplets as pore template is well reported [41–43]. The emulsion templating method is also studied for the preparation of macroporous ceramics through sol–gel route. This involves the deposition of ceramic over emulsion droplets through sol–gel chemistry using alkoxides followed by the removal of the emulsion droplets by drying and sintering, which will result in macroporous ceramics [44–47]. Recently, Barg et al. reported the preparation of macroporous ceramics from powder suspensions through a high alkane phase emulsion based process using decane as the oil phase [48–50]. An emulsion templating method based on liquid paraffin as pore template and collagen as consolidating agent has also been reported [51]. In the present work, we report an emulsion templating method for the preparation of macroporous alumina ceramics from aqueous powder suspension using naphthalene as the oil phase. Herein, the gelation of the emulsion cast in a mould is achieved by the solidification of naphthalene and the physical cross-linking of carrageenan gelling agent present in the aqueous alumina slurry.

2. Experimental

α -Alumina powder of average particle size $0.34\ \mu\text{m}$ and specific surface area $10.4\ \text{m}^2/\text{g}$ was procured from ACC Alcoa, Kolkata, India. Analytical reagent grade naphthalene and sodium dodecyl sulphate (emulsifying agent) were procured from Merck India Ltd., Mumbai. A 35 wt% aqueous ammonium poly(acrylate) solution (Darvan 821A) used as dispersant was received from Vanderbilt Company Inc., USA. The carrageenan used as gelling agent was procured from Aldrich Chemical, USA. Distilled water was used for the preparation of alumina powder suspensions.

The flow chart of the emulsion gelcasting process is shown in Fig. 1. A 50 vol% aqueous alumina powder suspension was prepared by dispersing 100 g alumina powder in 25 ml water using 1 g ammonium poly(acrylate) dispersant. The slurry was ball milled in a polyethylene bottle using zirconia grinding media on a roller ball mill for 12 h. The slurry was diluted to 30 vol% using distilled water and transferred to a 500 ml round bottom flask. The suspension was heated in a water bath at $90\ ^\circ\text{C}$ with mechanical stirring using a Teflon paddle after adding 0.75 g of carrageenan. The stirring was continued for 30 min to completely dissolve the carrageenan. Naphthalene and sodium dodecyl sulphate taken in a beaker were melted in a water bath at $90\ ^\circ\text{C}$. Molten naphthalene containing the emulsifying agent was added to the alumina slurry containing the gelling agent and stirring was further continued for another 30 min. The mixing speed used was 350 rpm. The emulsion thus obtained was cast in cylindrical glass moulds and allowed to cool naturally to room temperature for gelation. The gelled

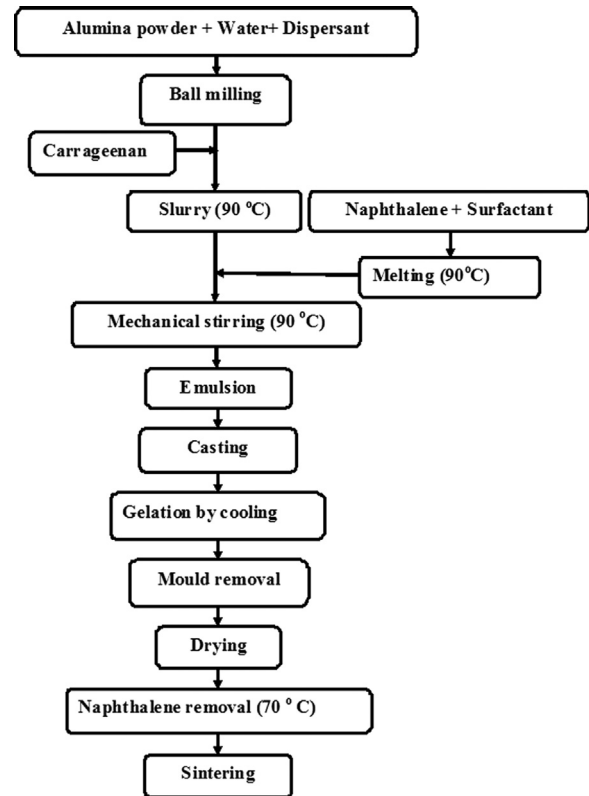


Fig. 1. Flowchart for the preparation macroporous alumina by freeze gelcasting of naphthalene-in-aqueous alumina slurry emulsions.

bodies removed from the mould were dried at room temperature in an open air atmosphere. The dried bodies were kept in an air oven at $70\ ^\circ\text{C}$ for the removal of naphthalene. The naphthalene removed bodies were heated in an electrically heated sintering furnace at a rate of $2\ ^\circ\text{C}/\text{min}$ up to $600\ ^\circ\text{C}$ and then at $5\ ^\circ\text{C}/\text{min}$ up to $1550\ ^\circ\text{C}$. A holding time of 2 h was given at the final temperature.

Viscosity of the emulsions was measured using a RVT model Brookfield viscometer (Brookfield Engineering Inc., Middleboro, MA) with a small sample adapter and a cylindrical spindle (SC-21). The temperature of the emulsion was maintained at $90\ ^\circ\text{C}$, during the viscosity measurements, with the help of a thermostol accessory along with the viscometer. The gelation time of the emulsions was determined from the cooling curve. The cooling curves were drawn from the temperature of 80 ml emulsion taken in a 100 ml beaker, monitored at regular intervals during cooling using a thermometer. The slurry was stirred with a glass rod until its gelation.

The drying kinetics of the gelled emulsion bodies and naphthalene removal kinetics of the dried emulsion bodies were studied by measuring the weight losses at regular time intervals in an air atmosphere at room temperature and at $70\ ^\circ\text{C}$, respectively. Cylindrical bodies of 22 mm diameter and 50 mm length were used for the drying and naphthalene removal studies. The shrinkages during drying and sintering were calculated from the initial and final dimensions of the cylindrical samples. Density of the sintered samples was determined from their weights and dimensions. The porosity of the sintered ceramics was calculated from their density

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