



Low cost porous alumina with tailored gas permeability and mechanical properties prepared using rice husk and sucrose for filter applications



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ABSTRACT

The present study demonstrates a cost effective way to produce porous alumina with 39–70 vol% porosity and 64–516 μm avg. pore size (length), using 20–40 wt% rice husk (RH) of <75 μm , 75–180 μm , 180–355 μm , 355–420 μm , and 420–600 μm sizes as pore former and sucrose as binder as well as a pore former. Microstructure of samples revealed interconnected pore microstructure consisting of mixture of coarse elongated pores and fine pores (avg. size 4 μm), created during burnout of RH and sucrose, respectively. Mechanical properties such as three point bending strength (98–14 MPa) and compressive strength (82–6 MPa) of the developed samples were strongly dependent on their porosity and pore size. Also, the Darcian permeability (k_1) and non-Darcian permeability (k_2) of porous alumina were a strong function of pore microstructure. The Darcian permeability ranges from 0.38×10^{-10} to $9.15 \times 10^{-10} \text{ m}^2$ which is in the order of magnitude of gas filters. The non Darcian permeability ranges from 0.33×10^{-5} to $3.92 \times 10^{-5} \text{ m}$. Experimental results agree closely with predictions made based on Forchhemier equation. The developed porous alumina is considered potentially useful in filtration and gas purging applications.

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1. Introduction

Porous ceramics find technological applications in diverse areas as gas/liquid separator, fluid filter, catalyst carrier, heat exchanger, thermal insulation, solid oxide fuel cell, combustion burner, biomedical implant [1–7] etc., owing to their distinct properties such as low density, high porosity, high surface area, good thermal shock resistance, low thermal mass, etc. Furthermore, porous ceramics are ideal substitutes for conventional porous media in various fields because they offer some advantages over beds of packed particles [8]. Recently, applications of porous ceramics as filters have generated considerable interest in technological applications like metal refinement, diesel combustion, hot gas filtration, residues burning etc. [9–11] due to their resistance at high temperature (usually >1000 °C). For high temperature gas

filtration, the ceramic filters are designed to satisfy the stringent environmental laws in the control of particulate emission.

Ideally, the ceramic filter should be able to remove maximum impurities with minimum resistance to the flow of fluids [12]. One of the most important properties of porous ceramics for filtration applications is permeability. Higher permeability is obtained by increasing the volume fraction porosity, pore size, pore connectivity etc. [13–16] Also, higher porosity though increases permeability, usually reduces mechanical properties. Therefore, optimization between permeability and mechanical strength is a basic requirement for adequate operation of porous ceramics as a filter.

Both permeability and strength are strongly affected by the porous microstructure [17–24]. Though large pores favour good permeability but reduce the collection efficiency of particles. On the other hand, while small pores increase the collection efficiency, reduce permeability of the filter. Optimization of both properties is best realized through achieving ideal combination of pore size, porosity and extent of pore interconnection [25]. Thus, microstructural tailoring of porous ceramics via processing techniques is

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in demand in order to obtain controlled permeability and mechanical properties for filter applications.

Fabrication of porous compacts with a range of permeability has been reported by different researchers. Isobe et al. prepared porous alumina with unidirectionally aligned cylindrical pores through extrusion and permeability of the resulting sample having 39% porosity was $3.9 \times 10^{-3} \text{ m}^2$ [25]. Yan et al. reported fabrication of porous cordierite with interconnected pores through an in-situ technique, and the permeability of sample with a porosity of 40.3% was $2.31 \times 10^{-12} \text{ m}^2$ [26]. A comparison of permeability of samples having above mentioned two types of pore structures with almost similar porosity shows that those with interconnected pores have relatively higher gas permeability. Thus, porous ceramics with interconnected pores and adequate strength can be preferred for filter application. Accordingly, selection of fabrication technique and optimization of processing parameters is important to obtain porous shapes with controlled microstructures.

Among various processes, burning of pore forming additives to create pores in the sample is a popular approach for fabrication of porous ceramics. Pore formers with biological origin are popular because their burnout is harmless from ecological and hygiene point of view. Accordingly, various pore formers such as starch, saw dust, cotton thread, wheat flour etc. were successfully used in fabrication of porous ceramics [23,27–29]. Rice husk, an agricultural waste, is used by various researchers as pore former for fabrication of porous ceramics [30–33]. But the reported porosity and pore sizes are limited within a narrow range. Also, they did not emphasize on tailoring of permeability and mechanical properties through control of microstructure of pores.

From the view point of producing cost effective ceramic filters, the objective of present work was to develop porous alumina having wide range of permeability through microstructural tailoring, using rice husk as pore former. Sucrose played its dual role as binder and pore former. Though, sucrose was used as binder and rheology modifier during wet processing of ceramics [34–38], its use in dry processing has not been reported. Attempts are made to tailor the porosity, pore size and pore inter connections by varying RH content and its particle size in the composition. The influence of porosity and pore size on permeability and strength of samples was examined. The experimental results of permeability were compared with the predicted values obtained using the standard Forchheimer's equation. Although the present investigation is focused on alumina ceramics, we would like to emphasize that the results of this study can be valid for all ceramic materials.

2. Theory

The permeability of a porous medium relates to the pressure drop, which is developed across the thickness of the material as any fluid flows through it. The interaction between the fluid and the porous medium which results in pressure drop varies with fluid flow velocity. The permeability at room temperature can be described by well known Forchheimer's equation (Eq. (1)) [39], which displays a parabolic trend of the pressure drop with the superficial fluid velocity (v_s). An ASTM standard (C 577 – 99) has defined the method for measuring permeability of porous refractory materials. Accordingly, higher permeability materials would result in lower pressure drops across the thickness for a certain flow rate. For low fluid velocity ($Re = (\rho v_s / \mu) * (k_1 / k_2) \ll 1$), the equation originally proposed by Darcy (1856) for packed granular beads, which can be used for calculating the permeability of a porous medium is given as [40,41].

$$\frac{\Delta P}{L} = \left(\frac{\mu}{k_1} \right) \times v_s \quad (1)$$

where ΔP = pressure drop, L = thickness of the sample (parallel to the fluid flow), μ = Dynamic fluid viscosity, k_1 = Darcian permeability and v_s = fluid velocity

The Darcy's equation accounts for only the viscous effects of the fluid. The above equation was modified by Reynolds (1900) and Forchheimer (1901) to include both the viscous and inertial effects, which can be expressed as [42].

$$\frac{\Delta P}{L} = \left(\frac{\mu}{k_1} \right) \times v_s + \left(\frac{\rho}{k_2} \right) \times v_s^2 \quad (2)$$

where $\Delta P = P_i^2 - P_0^2 / 2P_0$ with P_i and P_0 being the pressure values at the entrance and exit respectively, k_1 and k_2 are the Darcian and non-Darcian permeability, μ is the dynamic viscosity of nitrogen (in this work, $\mu = 1.75 \times 10^{-5} \text{ Pa s}$ at 25°C and ρ is the density of nitrogen (1.13 kg/m^3 at 25°C), v_s is the gas velocity, k_1 & k_2 are Darcian and non-Darcian permeability constants, respectively.

Both Darcian and non-Darcian permeabilities depend exclusively on the porous structure. The first term of the right hand side of Eq. (2) represents a linear term, which expresses the contribution of the attrition effects to flow resistance (viscous regime), and the latter part represents a quadratic term, which describes the contribution of kinetic effects on the pressure drop inertial force (turbulent regime). Both k_1 and k_2 have a non-linear dependence on the porosity and pore size.

Ergun in 1952 defined the permeability constants (k_1 , k_2) directly in terms of the flow through a granular packed bed as [39,43].

$$k_1 = \frac{\epsilon^3 d_p^2}{150(1 - \epsilon)^2} \quad (3)$$

$$k_2 = \frac{\epsilon^3 d_p^2}{1.75(1 - \epsilon)} \quad (4)$$

where ϵ is the porosity of the granular bed and d_p is the mean particle diameter of the granular medium.

To apply these Ergun's equations in consolidated materials with configured cell (pore) structures such as porous ceramics, the particle diameter (d_p) in Eqs. (3) and (4) has been replaced with average pore size by some researchers [39]. As per the above equations, both Darcian (k_1) and non-Darcian permeability (k_2) have a non-linear dependence on the porosity and pore size. When the kinetic effects are negligible, Eq. (2) reduces to Darcy's law, which displays only the linear dependence of the gas velocity on the pressure gradient. Commercial ceramic filters exhibit Darcian permeability typically in the range of 10^{-10} – 10^{-8} m^2 and compressive strength varying from 0.5 to 2 MPa [10].

3. Experimental procedure

3.1. Processing and characterization of rice husk and sucrose

RH (unprocessed) was thoroughly cleaned using distilled water for complete removal of dust, clay and sand adhered to the surface. Then, wet RH was dried in ambient atmosphere for 2 h followed by drying in a preheated oven at 150°C for 24 h. Drying temperature was chosen based on trial experiments related to easy grinding of rice husk. Fully dried RH was ground in a mixer machine (Kenstar Prince Royal, Videocon Industries Limited, India, 18,000 rpm) for

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