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FABRICATION AND CHARACTERIZATION OF POROUS SILICA CERAMICS BY DIRECT FOAM & SACRIFICIAL TEMPLATE METHOD

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ABSTRACT

This research work comprises of fabricating highly porous silica ceramics by two different fabrication routes such as Direct Foaming Method and Sacrificial Template Method (saw dust is used as porgen). The as-fabricated porous ceramics were characterized for physical, structural and mechanical properties for their end use as thermal insulators in various applications. In this investigation, the results shows that the porosity of porous silica ceramics by direct foaming method and sacrificial template method as 86% and 78% respectively. Compressive strength which was found from universal testing machine (UTM) is 25 x 10^{-2} MPa & 14 x 10^{-2} MPa for silica ceramics by direct foaming method and sacrificial template method respectively.

KEYWORDS: Compressive Strength, Porgen, Porosity, UTM

INTRODUCTION

Significant number of processing methods have been developed in the last few years to manufacture porous ceramics . This paper presents a new approach for the preparation of porous silica foams through direct foam method in which small amount of CTAB act as foaming agent and PVA (Poly Vinyl Alcohol) as binding agent. Where as in sacrificial template method saw dust (is used as porgen). The microstructure, mechanical and thermal property of the resultant foams has also been discussed.

Porous silica tiles made by direct foaming method, in that pores are observed as irregular and less compressive strength is observed. In sacrificial template method saw dust is used as porgen so that a small pore is developed for little higher compressive strength.

In present market, Porous ceramics materials are in great demand due to their intrinsic properties like low mass, high permeability, high surface area, low specific heat and low thermal conductivity. Hence it is used in various applications in catalysis, separation, lightweight structural materials, thermal insulation and biomaterials etc. [1].

EXPERIMENTAL PROCEDURE

Raw Materials

The raw materials used for this study were fused silica powder (99% pure, d50 = 38μm), Alumina powder (99% pure, d50 = 1.5μm), PVA (Poly Vinyl Alcohol, molecular wt. 1, 25, 000, S.D. Fine Chemicals Ltd.), CTAB (Cetyl Trimethyl Ammonium Bromide, S.D. Fine Chemicals Ltd.), Saw dust (~425 microns) & Distilled water.

- **Equipments Used for Processing Porous Ceramics:** Pot Mill, Hot Air Oven, Raising Hearth Sintering Furnace
- **Equipments Used for Characterization of Porous Ceramics:** Particle Size Analyzer, SEM, Thermal Conductivity Tester.

- **Preparation of Porous Ceramics by Direct Foam Method:** The processing of silica foams was carried out by following several steps (Figure 1). In this process, the first step involves slurry preparation. The slurry was prepared in an order; first PVA solution (4 wt. %) was mixed with calculated amount of water, then slowly the silica powder & then alumina balls were mixed. This was followed by addition of the alumina powder and at last CTAB powder was also added to it. Alumina balls of ~8 mm diameter were used as milling media in 1:1 powder-to-media ratio. The as-prepared mix was milled in a polystyrene bottle in a pot mill at 60rpmfor 18 h and then the foaming was done for 3 h by changing the direction of polystyrene bottle to pot mill axis.

The second step was the casting of the foamed slurry in the greased polystyrene molds of 60 x 50 x 15 mm dimension. The as-cast porous ceramics in the molds was kept for 24 h at room temperature (25ºC & 65% RH) for the aging process and initial drying at RT (Room Temperature). During the casting process, the mold was periodically tapped to distribute the foam uniformly. The green strength of the foams was optimized through careful selection of ~52.64% solids loading. To arrive at the optimum solids loading, separate experimental trials were conducted involving different process parameters such as (i) varying amounts of PVA, CTAB, alumina powder, (ii) mixing, (iii) foaming, and (iv) sintering durations. Based on such trial studies, it was found that the composition including ~52.64% overall solids loading, 4 wt% of PVA, 5mg of CTAB had formed kinetically stable foam.

The third step was the controlled drying in a convention hot air oven at temperature up to 100ºC in steps. First the oven was kept to hold at 50ºC for 5h. This step was followed by release of the partially dried samples from polystyrene molds and then the temperature of the oven was increased to 80ºC for further drying of the samples. At 80ºC, the samples were dried for 2h and then 100ºC for 1h finally in order to get completely dried green porous bodies. The green density and drying shrinkage of the sample was thus calculated by weighing the sample and measuring the sample dimensions.

The fourth step was sintering of the green samples at 1100ºC for 3h in a raising hearth sintering furnace. The fifth step consisted of the physical, micro-structural and mechanical characterization of the samples through density calculation, SEM and UTM. The pore sizes were calculated by using image analysis software (Image Tool).

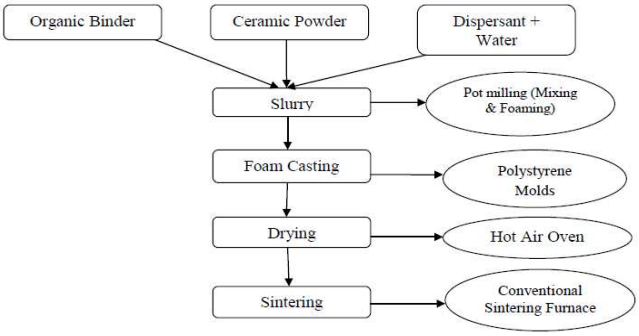

Figure 1: Flow Chart for Direct Foaming Method

- **Preparation of Porous Ceramics by Sacrificial Template Method:** The processing of silica foam through sacrificial template route (porogen saw dust) was carried out following several steps (Figure 2). In this process the first step involves slurry preparation. The slurry was prepared in an order; first PVA solution (4 wt. %) was mixed with calculated amount of water, then slowly silica powder & alumina balls (~8 mm diameter) were mixed in a polystyrene container. Alumina balls were used as milling media in 1:1 powder-to-media ratio. This was followed by addition of the alumina powder, saw dust particles & finally CTAB powder was also added to it. The as-prepared mix was shaked well and then milled in a polystyrene bottle in a pot mill at 60 rpm for 18h and no foaming was further carried out as saw dust was used as pore forming agent in this process. The second step was

casting of the as-milled slurry in the greased polystyrene molds of 60 x 50 x 15 mm dimensions. The as-cast porous ceramics in the molds were kept for 24h at room temperature (25ºC & 65% RH) for the aging process and initial drying at RT. During the casting process, the mold was periodically tapped to distribute the slurry casting uniformly.

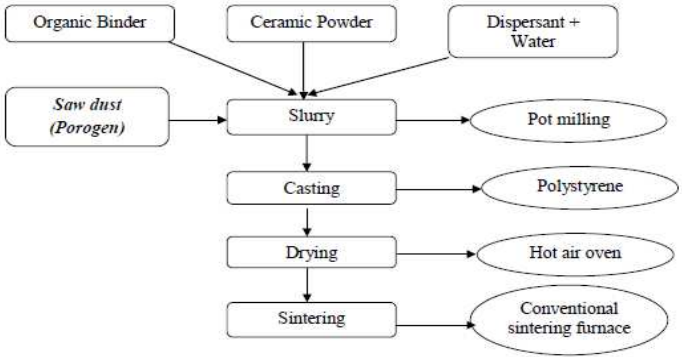

Figure 2: Flow Chart for Sacrificial Template Method

The green strength of the foams was optimized through careful selection of ~ 47.7% solids loading. To arrive at the optimum solids loading, separate experimental trials were conducted involving different process parameters such as (i) varying amounts of PVA, CTAB, alumina and saw dust (ii) mixing, and (iii) sintering durations. Based on such trial studies, it was found that the composition including ~47.7% solids loading & 4 wt. % of PVA, 5 mg of CTAB formed kinetically stable foam.

The third step was the controlled drying of as-cast samples in a convention hot air oven at temperature up to 100ºC in steps. First the oven was kept to hold at 50ºC for 5h. This step was followed by release of the partially dried samples from polystyrene molds and then the temperature of the oven was increased to 80ºC for further drying of the samples. At 80ºC the sample were dried for 2h and then 100ºC for 1h finally in order to get completely dried green porous bodies. The green density and drying shrinkage of the sample was thus calculated by weighing the sample and measuring the sample dimensions. The fourth step was sintering of the green samples at 1100ºC for 1h in a raising hearth sintering furnace. The fifth step consisted of the physical, micro-structural and mechanical characterization of the samples through density calculation, SEM and UTM.

CHARACTERIZATION OF POROUS CERAMICS

Physical Characterization

The *mass density* or *density* of a material is defined as its mass per unit volume. The symbol most often used for density is ρ (the Greek letter *rho*). Mathematically, density is defined as *mass* divided by *volume*: $\rho = m / V$. Where ρ is the density, m is the mass, and V is the volume. *Porosity* is a reverse of density in any material and is a measure of the void spaces in a material.

Porosity is calculated as 1- (Sintered density / Bulk density) (1)

In this study, the as-cast porous ceramic samples from each composition were dried at room temperature for 24 hours to ensure partial water loss. The green samples were then measured for their wet dimensions. Then the samples were kept in hot air oven for 50^0C for 3 h and then slowly temperature was increased to 80C for 2h and at 100^0C for 1h. The samples were then fired in a resistance furnace to temperature in the range of 1100ºC. After cooling, the samples were weighed and sample dimensions were measured which is known as sintering weight and sintered dimension. Shrinkage data was generated for 3-5 different samples for each system, and the average was calculated for it. The drying shrinkage,

firing shrinkage and the total shrinkage (%) were calculated for each sample using the following formula [6].

Avg.Drying Shrinkage = (OLDL)/OL*100 (2)

Avg.Firing Shrinkage = (DLFL)/FL*100 (3)

Total Shrinkage = (OLFL)/OL*100 (4)

Microstructural Characterization

Compressive strength is measured on a Universal Testing Machine (UTM) ranging from 20 N to >50 MN capacity. Measurements of compressive strength are affected by the specific test method and conditions of measurement. The formula used for calculating the compressive stress and strain is as follows;

Compressive Strain = Compressive extension (mm) / Height of the Sample (mm) (5)

Compressive Stress (N/mm2) = Load (N) / Surface Area of the Sample (mm2) (6)

Where, Surface Area of Sample (mm2) = Width (mm) x Length (mm) of the sample in eq. (5&6).

For measuring compressive strength in this study, the sample preparation process involved resin impregnation of the sintered foam samples followed by drying at 60°C for 30 minutes and then curing at 135°C for 10 minutes, so that the sample could be handled without breakage during its testing and analysis. The resin impregnated porous ceramic samples were cut into 10 x 10 x 10 mm cubes using diamond cutting tool machine. The resin was further burnt off (650°C/1h) from the samples after cutting or machining them into desired shapes and size for testing purpose. The as-machined and fired foam samples were compression tested by using Universal Testing Machine (Instron 5500R) at a crosshead speed of 0.5 mm & load cell of 100kN (Figure 3).

At every close interval, instantaneous load and extension data was measured & recorded. For each sample, 3 tests were performed and based on the average result; a graph was plotted between compressive stress and compressive strain.

Figure 3: Universal Testing Machine (UTM) Used for Compressive Strength Testing

RESULTS AND DISCUSSIONS

Physical Properties

By measuring the weight and dimensions of porous silica ceramics samples by direct foaming method (Si-DF) and sacrificial template method (Si-Sd) after drying and sintering operations, the porosity was calculated by using the formula given in eq.(1)

Table 1: Porosity of Silica Porous Ceramic Sample as Fabricated in this Study

Material System	Bulk Density (g/cc.)	Sintered Density (g/cc.)	Porosity (%)
Si-DF	2.2	0.36	86%
Si-Sd	2.2	0.52	78%

Linear Shrinkage

The dimensions of the porous ceramics after drying and sintering were measured and the linear shrinkage table was prepared for porous silica ceramics are studied, as listed in Table 2 &3. The linear shrinkage was calculated from the formula in eq. (3&4).

Table 2: Linear Shrinkage Data for as-Fabricated Porous Silica Ceramics

Material System	Wet Dimension (mm) L x W x H	Green Dimension (mm) L x W x H	Sintered Dimension (mm) L x W x H
Si-DF	6.3x5.2 x1.3	6.25x5.15x1.25	6.2x 5.1x1.2
Si-Sd	6 x 5 x 1.5	5.95 x 4.9 x1.45	5.9x 4.85x1.3

Table 3: Total Linear Shrinkage Data for as-Fabricated Porous Silica Ceramics

Dry Shrinkage (%)			Sintered Shrinkage (%)			Total Shrinkage (%)			Sintered Temperature (°C/hr)
L	W	H	L	W	H	L	W	H	
0.8	0.8	3.8	0.8	0.97	4	1.6	1.7	7.8	1100 °C/3h
0.8	2	3.3	0.8	1.0	10.3	1.6	3	13.6	1100 °C/1h

Microstructural Characterization

By examining the SEM micrographs, for porous silica ceramics made by DF method (Figure 4) pores were found to be nearly spherical in shape and almost uniformly distributed, hence the average pore sizes was calculated by image analysis of the micrographs and shown in Table 4.

Table 4: Description of Image Analysis Data in the Porous Silica Ceramics

Material System	Strut Thickness (µm)	Minimum Pore Size (µm)	Maximum Pore Size (µm)	Average Pore Size (µm)
Si-DF	100	300	800	500
Si-Sd	--	220	624	400

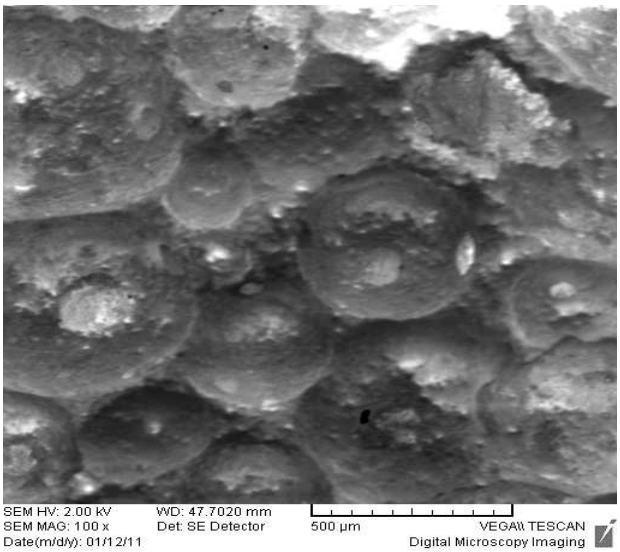

Figure 4: SEM Micrographs of Porous Silica Ceramics Made through Direct Foam Method

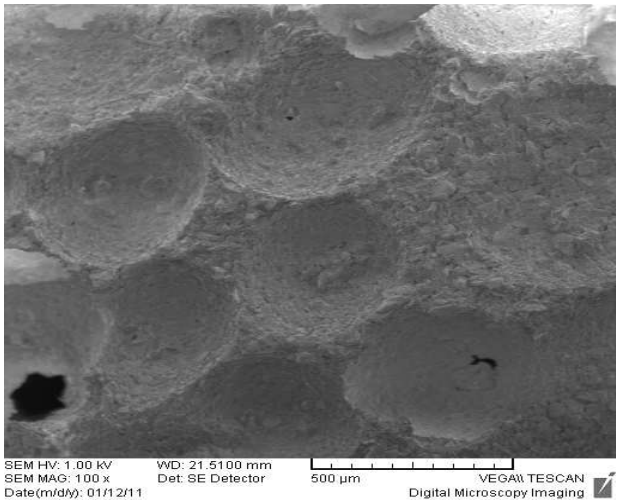

Figure 5: SEM Micrographs of Porous Silica Ceramics Made through Sacrificial Template Method

Mechanical Properties: Compressive Strength

The average compressive strength was calculated by using formula in eq. (6) & (7). The Universal Testing Machine (UTM) was used for calculating average compressive strength and the results of mechanical testing porous ceramics have been listed in Table 5.

Table 5: Average Compressive Strength Data for Porous Silica Ceramics System

Material System	Sample Dimensions (mm)	Porosity (%)	Average Compressive Strength (MPa)
Si-DF	10 x 10 x 10	86%	25 x 10-2
Si-Sd+C	10 x 10 x 10	78%	14 x 10-2

The stress-strain graph has been plotted by considering compressive strength and strain at every stage. The stress-strain graphs for silica porous ceramics have been shown in Figure 6 & 7.

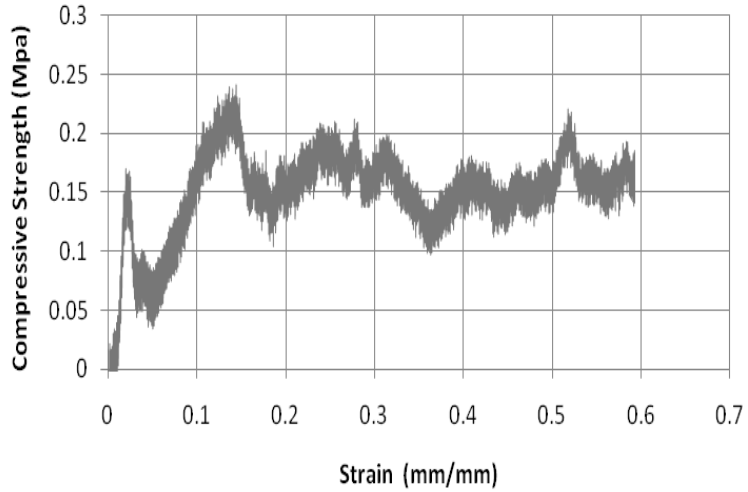

Figure 6: Compressive Stress-Strain Curve for Silica Porous Ceramics Made by Direct Foam Method

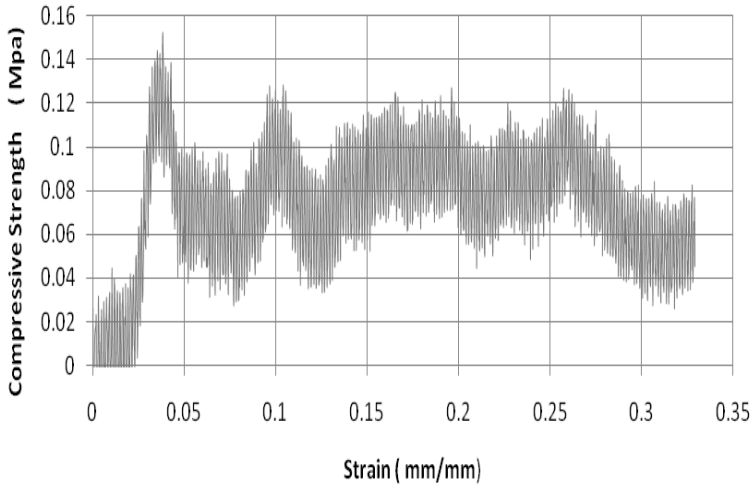

Figure 7: Compressive Stress-Strain Curve for Silica Porous Ceramics Made by Sacrificial Template Method

CONCLUSIONS

In this investigation, the results show the porous ceramics by direct foam method has more porosity than sacrificial template method. Since the porosity is more so the compressive strength was less in porous ceramics by direct foam method than sacrificial template method. Almost uniform pore size is observed in direct foam method. Maximum linear shrinkage is achieved in height for two methods since upper surface is exposed to atmosphere.

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