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Synthesis of silica cryogel-glass fiber blanket by vacuum drying

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Abstract

Silica cryogel-glass fiber composites with a high specific surface area and high mesoporosity were fabricated via a simple still original drying technique. By applying vacuum at ambient temperature, the system evolution has been monitored and represented in the water phase diagram. The ratio of solvent/silica loading significantly affected the porous structure and thermal insulating properties of the blanket. From the results of BET surface area, apparent density and porosity studies, the microstructures and specific surface areas of the composites were greatly affected by changing the silica amount in the sol. The microstructure of silica cryogel blanket exhibits a porous structure consisting of glass fibers of diameter ~7 μm interconnected with solid silica clusters (5–20 μm). Silica cryogel-glass fiber blankets with low densities from 0.13–0.24 g/cm^3 and thermal conductivity as low as 0.02–0.035 W/mK were obtained using this cost effective, hazardous free and time saving method. The pH of the silica sol influenced the gelling property and the thermal conductivity of the composites.

Keywords: Aerogel; Glass fiber; Mesoporus; Vacuum drying; Thermal insulation

1. Introduction

Aerogels are widely used for thermal insulating applications because of their inherent property of low density, have high porosity from 90% to 99% comprised of about 10 nm sized mesopores. Aerogel functions as thermal insulator primarily by minimizing conduction, convection and radiation. Depending on the formulation they can function well at temperatures of 550 °C and above [1–4]. However the fragile and brittleness of these materials in monolithic state will limit its application inside the laboratory. Much research has been carried out to find a method to over come the mechanical fragility of aerogels, by compositing inorganic aerogels and polymers [5]. One of the interesting research outcomes in making of strengthened aerogel is the composition of aerogel and fiber matrix [6]. Many works have been done to develop aerogel matrix composites of bulk aerogel with fibers dispersed within the bulk aerogel. Fiber matrix plays a role of compartments containing aerogels, which support aerogel and decrease the bulk size of aerogel within aerogel–fiber matrix composite. The fibers were long or short, varying thickness, whiskers, mineral wool, glass wool or even particles [7,8].

Silica aerogels were first prepared by supercritical drying of wet gels and usually using TEOS (tetraethylorthosilicate) or sodium silicate as silica source [9]. However, supercritical drying process is so energy intensive and dangerous that real practice and commercialization are difficult. So it is necessary to synthesize silica aerogels by an alternative drying technique at a reasonable cost [10]. 'Ambient pressure drying' is an alternative method developed for large-scale production of xerogels similar to those of aerogels, but unfortunately, the drying process involves the use of hazardous solvents for tedious solvent exchange and surface modification, which is harmful to humans and the environment [11–14]. Another promising technique is vacuum freeze drying. It is based on sublimation, i.e. direct conversion from the solid to gaseous state, and thus avoids the formation of liquid–vapor meniscus [15–17]. The freeze-dried gels are called 'cryogels'. Inorganic

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cryogels are often synthesized by freezing sol–gel products in an aqueous solution, or a mixed organic/aqueous solution, followed by sublimation using a freeze-dryer [18]. Cryogel synthesis can be considered an alternative means to synthesize materials similar to aerogels. More recently, Su et al. have successfully synthesized nanoporous silica cryogels with prominent thermal insulation properties by vaccum freeze drying of silica-tert-butyl alcohol derived gels [19].

In the present work silica cryogel-glass fiber blankets were prepared by vacuum drying of silica gels derived from an aqueous solvent without any pre-freezing of the gel. We prepared silica cryogel-glass fiber blankets by impregnating the glass fiber board in an aqueous silica sol with varying concentration of silica, gelled and dried by vacuum drying. The first time we reporting the phase diagram of silica gel glass fiber composite during vacuum drying process. We investigated the effect of solvent/silica ratio on the bulk density, porosity, specific surface area and thermal conductivity of the silica cryogel-glass blankets prepared. The properties are compared with panels prepared by freeze drying and ambient drying process.

2. Experimental

2.1. Materials

The chemicals used for the preparation of hydrogels were tetraethyloxysilane (TEOS, SiO_2 > 28.5%), nitric acid (HNO_3) and ammonia (NH_4OH), which were purchased from Sigma Aldrich. The deionized water was used as the solvent.

2.2. Preparation of silica sol

In silica sol preparation, TEOS and distilled water were used as precursor and solvent, respectively. Nitric acid was added to induce the hydrolysis of the precursor, and then the irreversible polycondensation reaction took place in the presence of a base catalyst (NH_4OH). H_2O/TEOS mixtures were prepared with different ratio of TEOS to H_2O (Table 1). A diluted HNO_3 was added into the mixture under continuous stirring for 30 min at 60 °C. Diluted NH_4OH was added drop wise to the acid-catalyzed sols under stirring, until the pH reach around 7.

2.3. Preparation of silica/glass fiber composite

The E- glass fiber board (Eksol-N, bulk density 0.1 g/cc and fibers diameter were 7–10 μm) was first cut into 2 × 2 × 5 in. rectangular pieces and heat-treated at 500 °C to remove the binder. The fiberboards were dipped into the silica sol and squeezed repeatedly to remove the air bubbles and transferred to an aluminum dish, kept for gelling and aging. After aging, the infiltrated glass fiberboards were dried in a vacuum dryer (Edwards MINIFAST). The shelf temperature of the dryer was 16 °C. To monitor the temperature of the sample an external thermocouple was inserted inside the sample. The total production time of the glass fiberboard silica cryogel composite was about 24 h. The vacuum dryer operated at a chamber pressure of 0.3 mbar. An internal thermo-couple was inserted on the sample in order to monitor the temperature of the sample during the drying process. The dried samples after vacuum drying were heat-treated at 500 °C for 30 min. Fig. 1 shows the complete flow chart of the preparation of silica cryogel blanket. In order to compare the properties, samples also prepared by freeze drying and room drying conditions.

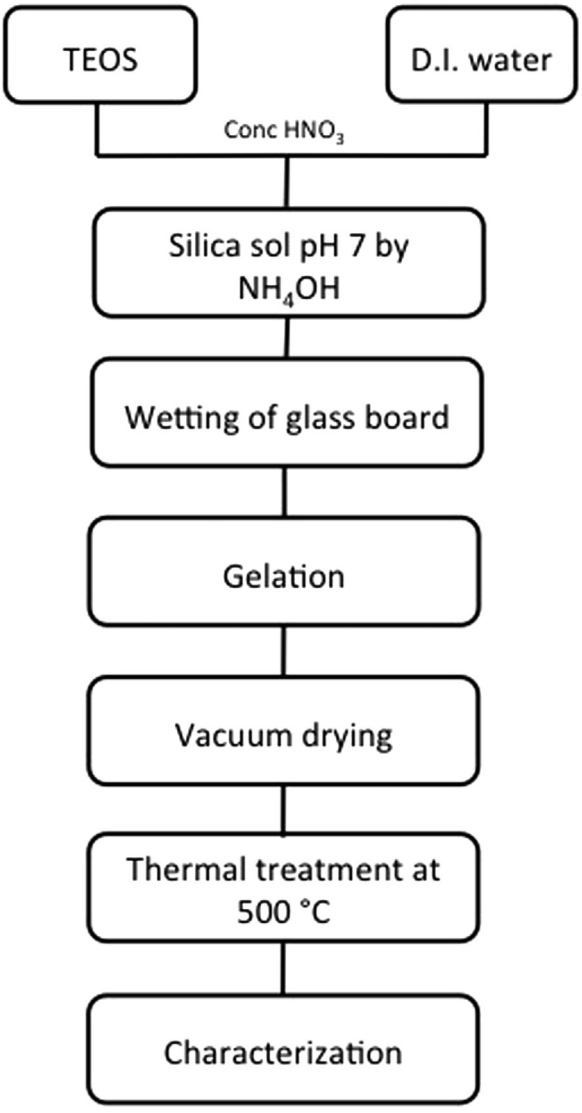

Fig. 1. Flow chart for the preparation of silica cryogel blanket.

2.4. Characterization

The amount of silica in the composite material was calculated by weighing glass fiber board before and after the composite synthesis. Bulk density of the bulk bodies was calculated from the volume and the weight. The porosity of the foams was calculated using the following:

$$P = 1 - \frac{\rho}{\rho_0} \quad (1)$$

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