



Preparation and characterization of hydrophobic silica aerogel sphere products by co-precursor method



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ARTICLE INFO

Article history:

Received 22 June 2015

Received in revised form

22 July 2015

Accepted 5 August 2015

Available online 8 August 2015

Keywords:

Silica aerogel ball

MTES/TEOS

Sol–gel

Hydrophobic

Thermal stability

ABSTRACT

In the present paper, silica aerogel balls were prepared using methyltriethoxysilane (MTES) and tetraethoxysilane (TEOS) co-precursor with different MTES/TEOS molar ratio (I) by two-step acid-base catalyzed sol–gel process and molding technology followed by the carbon dioxide supercritical drying. The physical properties of various silica aerogels whose I varied from 0 to 1.0 were studied by BET and SEM. Approving aerogel ball was obtained by using acetone as exchanging solvent at I of 0.8. Better properties are less crack, little shrinking percentage (17%), low apparent density (0.103 g/cm^3), large surface area ($996.35 \text{ m}^2/\text{g}$) and high pore volume ($3.32 \text{ cm}^3/\text{g}$). Characterized by contact angle measurements and thermal stability, the characterizations of aerogel spheres were strongly affected by the MTES/TEOS molar ratio. Hydrophobic property increased with the increase in I value, and silica aerogels at $I = 0.8$ and 1.0 have a superhydrophobic characterization with the highest contact angle (152°). The TG-DTA analysis shows that the silica aerogel spheres transform hydrophobic to hydrophilic at around 450°C , which is due to oxidation of Si–CH₃ to Si–OH groups. The surface chemical modification was confirmed by FT-IR spectrums, which demonstrated that Si–CH₃ groups be beneficial to molding of silica aerogels.

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1. Introduction

In recent years, silica aerogels are the most highly porous nanostructured materials, with a three-dimensional network of silica particles, and have a low bulk density ($0.003\text{--}0.5 \text{ g cm}^{-3}$), small pore size (1–100 nm), low index of refraction (~ 1.05), high optical transmission ($\sim 93\%$), extremely high porosity ($\sim 99\%$) and large surface area ($\sim 1600 \text{ m}^2 \text{ g}^{-1}$) [1–5]. Due to their superior characteristics above, silica aerogels are potential application materials for scientific and technological, such as environment clean up and protection [6–8], internal confinement fusion (ICF) targets for thermonuclear fusion reactions [9], efficient catalysts and catalytic supports [10], efficient thermal insulator for solar energy systems, refrigerators, thermal flasks, building, aeronautical and aerospace domains [11–15], and others [16,17]. Therefore, molding problem of silica aerogels as a filling material is extremely important requirements for different aspects of the applications.

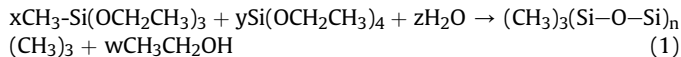
Despite these advantages, silica aerogels are highly susceptible to fracture or collapse of their pore structure during the drying stage and difficult to prepare for large-scale industrial production setting due to their weak mechanical properties. Several approaches have been investigated in order to improve the surface mechanical properties for unbroken silica aerogel products. Columnar flexible superhydrophobic silica aerogels have been prepared using a flexible precursor followed by supercritical drying [18,19]. Synthesis of hydrophobic silica aerogel beads have been achieved using trimethylchlorosilane (TMCS) via simultaneous solvent exchange and surface modification [20]. Products were block or powder with different sizes in previous reports, and failure to satisfy the requirement of the special filler material. Compared with the ambient pressure drying, carbon dioxide supercritical drying avoids the collapse and shrinkage caused by the surface tension force associated with the removal of the fluid effectively, because of almost zero surface tension under supercritical pressures [21]. Working pressure and medium concentration exclude the external force of rapid evaporation of solvent during drying.

The surface of unmodified silica aerogel covered with hydroxyl groups (–OH), which are sensitive to moisture. This can be achieved by modifying the surface chemistry of the silica network with

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appropriate functional groups and then growing the polymer from the modified surface. Methyltriethoxysilane (MTES, $\text{H}_3\text{C}-\text{Si}(\text{OCH}_2\text{CH}_3)_3$) served as a modifier and precursor has overcome inadequacies such as fragility, collapsibility, and hydrophilic [19,22]. In general, the surface of silica alcogels modified by MTES occurs predominantly according to the following reaction:



In the present work, we prepared hydrophobic silica aerogel balls via two-step acid-base sol-gel process with MTES precursor and molding technology followed by carbon dioxide supercritical drying. We investigated the effects of synthesis conditions on the properties and surface characterization of silica aerogel balls, in addition to acquiring a correlation between the synthesis procedure and the surface properties of the obtained aerogel balls. This paper is mainly to solve the molding problem of the large size aerogel.

2. Experimental procedure

2.1. Preparation of silica aerogels

Silica aerogels were prepared using tetraethoxysilane (TEOS) and methyltriethoxysilane (MTES) co-precursor by two-step acid-base catalyzed sol-gel process followed by the carbon dioxide supercritical drying, ethanol (EtOH) and acetone as solvent, hydrochloric acid (HCl) and ammonium hydroxide (NH_4OH) as catalyst. The stepwise experimental scheme is represented in Fig. 1.

Initially, the co-precursor of TEOS and MTES was hydrolyzed by mixing with EtOH, H_2O and HCl controlling a pH range from 4.0 to 5.0 at 50°C . The mixed solution was stirred for 1.5 h and hydrolyzed for 4 h. In order to investigate the effect of different MTES/TEOS molar ratio (I) on the characterization of aerogels, the molar ratio was respectively designed at 0, 0.1, 0.2, 0.4, 0.6, 0.8 and 1.0. The molar ratio of TEOS-MTES:EtOH: H_2O was kept constant at 1:10:4. Secondly, the remaining EtOH and NH_4OH were

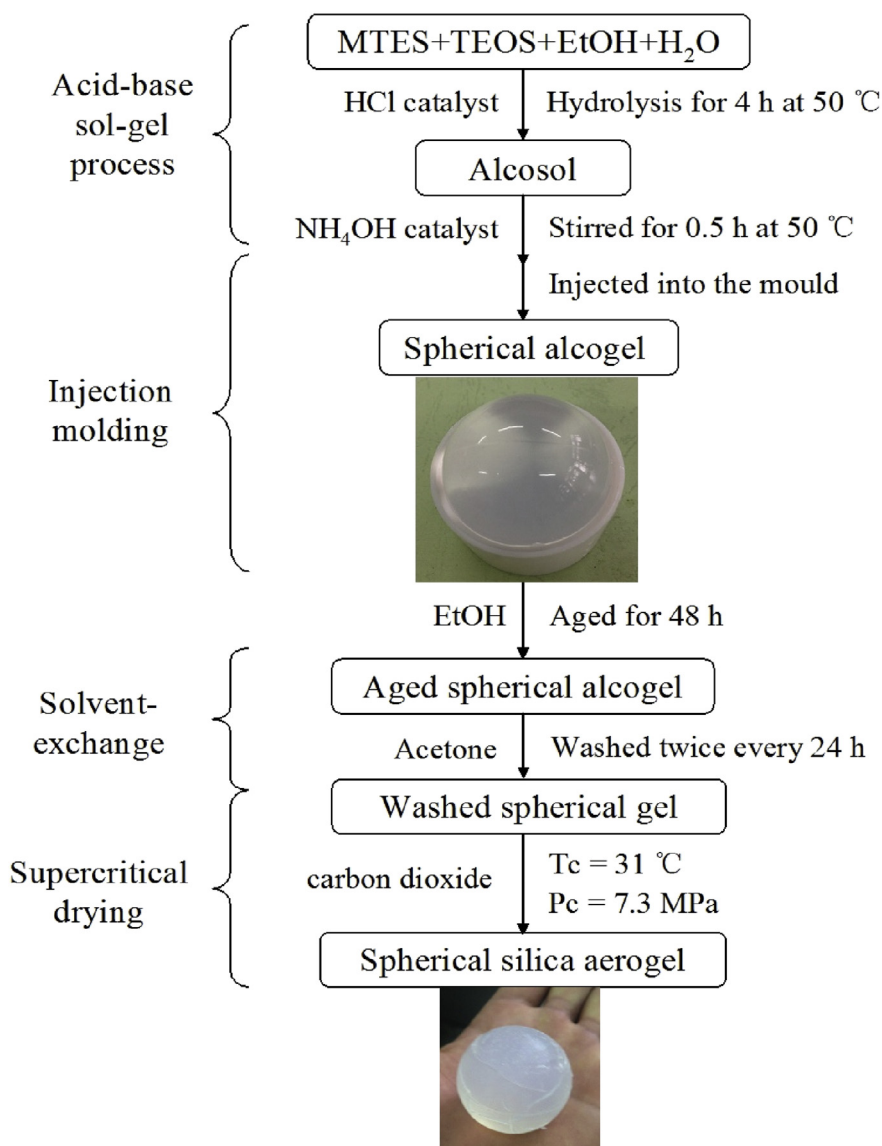


Fig. 1. Experimental scheme for the synthesis of silica aerogel balls.

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