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Surface modified sodium silicate based superhydrophobic silica aerogels prepared via ambient pressure drying process

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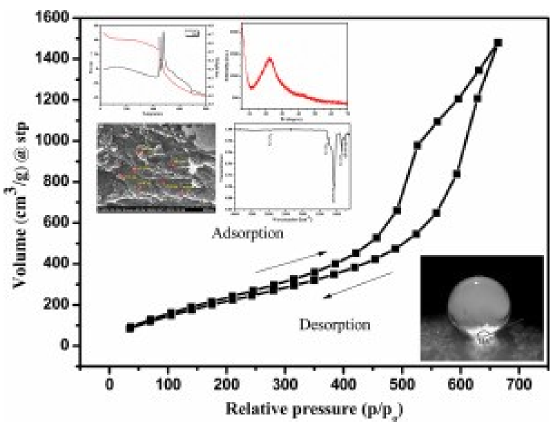
Highlights

- Synthesis of silica aerogel by simple and economic ambient pressure drying method.
- Trimethylchlorosilane (TMCS) used as a silylating agent for surface modifications.
- Prepared aerogels shown high thermal stability with respect to hydrophobicity.
- Brunauer-Emmett-Teller analysis disclosed large surface area of prepared aerogel.
- High contact angle value affirmed the superhydrophobic nature of the aerogel.

Abstract

The silica aerogel was synthesized by simple and cost-effective sol-gel process under ambient pressure drying. The wet gel was modified by using trimethylchlorosilane (TMCS) as silylating agent. The prepared aerogel was characterized by X-ray diffractometer (XRD), thermogravimetric and differential thermal analyzer (TG-DTA), Fourier transform infrared spectrometer (FT-IR), Brunauer-Emmett-Teller (BET) analyzer, field emission scanning electron microscope (FE-SEM) and Ultraviolet-Visible (UV–Vis) spectrophotometer for structural, thermal, functional, surface, morphological and optical properties. The presence of hump in X-ray diffraction pattern revealed the amorphous nature of prepared silica aerogel. Thermal stability of silica aerogel investigated by TG-DTA show a hydrophobic nature up to 478 °C. FE-SEM images confirmed the porous nature of silica aerogel. The surface area and pore radius measured by BET analyzer disclosed as 792.308 m^2/g and 5.779 nm respectively while the total pore volume is 2.289 cc/g. Superhydrophobic nature of silica aerogel sample was affirmed by contact angle measurements. The energy band gap calculated from UV–Vis spectra was found to be 4.25 eV confirming the insulating nature of prepared silica aerogel. The resulting silica aerogel possesses high thermal stability, Superhydrophobicity and large specific surface area which can be useful in various applications such as catalysis, coating materials, oil spill cleanup processes and insulating materials.

Graphical abstract



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Introduction

Silica aerogel materials have scientific and technological significance due to their vast applications in many fields [1]. Aerogels are very light as well as low-density (~0.03–0.5 g/cm^3) materials with a large surface area (500–1000 m^2/g), high porosity (~90%), low thermal conductivity (~0.004 to 0.03 W/mK) and low refractive index (~1.05) [2]. These remarkable properties make them applicable for Cherenkov radiation detector, super thermal insulator for solar energy system, insulated flask and refrigerator, aerogel films of low dielectric constants in large scale integrated circuits [3]. Aerogels are also used in storage media, liquid for rocket propellant and as catalysts [4]. These features of silica aerogel create them to be of great curiosity for scientists to study for variety of fields like space technology, drug delivery, thermal and acoustic insulation, etc.

The drying, in the preparation of silica aerogel is one of the key steps. Generally, silica aerogels are produced via supercritical fluid drying; however supercritical fluid drying involve high pressure process (5-10 MPa) which possesses more safety issues and it is costly. Therefore, supercritical fluid drying limits industrial-scale production of aerogel materials. Now-a-days ambient pressure drying method has emerged as safe and less expensive technique for preparation of silica aerogel at industrial-scale production [5]. Schwerterfeger et al. developed a process for preparation of silica aerogel using sodium silicate in which exchange of solvent and surface modification using trimethylchlorosilane (TMCS)/hexamethyldisiloxane solution were achieved simultaneously by chemical reactions and phase separation mechanism [6]. At the present, the silica aerogels with hydrophobic nature have been proved promising material in various applications like absorption of organic oils/liquid as well as adsorption of toxic substances, transportation of liquid on nanoscale in biotechnological and chemical fields. Wettability plays a vital role in many technological and natural processes. The wettability properties of solid surface can be modified by both geometrical microstructure and chemical composition on the surface. The surface modification of silica wet gel is the most common approach to overcome the capillary tension to produce hydrophobic surfaces. The hydrophobic nature of silica aerogel dried at ambient pressure gives the structure stability against humidity [7].

In the literature, there are some recent developments on surface modification of silica aerogel. A. V. Rao et al. (2018) presented the latest literature about the topic of hydrophobic silica aerogel [8] wherein the use of water glass-based elastic superhydrophobic silica aerogels were investigated. Further, its several scientific and technological applications such as in multidisciplinary subjects dealing with liquid/solid interfacial energies, long-range attractive interactions, frictionless flow of liquids through nano and micro channels, pipes, and storage of mechanical energy was explained. Yu et al. (2015) [9] reported the great potential of high surface area and high porosity of silica aerogel to use as catalyst. A well-tailored intrinsic hydrophobic silica aerogel with an enhanced adsorption property of organic components was investigated by Bhagat et al. (2007) [10]. Huang et al. successfully silylated the surface silica aerogel by hexamethyldisiloxane and trimethylchlorosilane [11]. Rao et al. and Ehsan Ul Haq et al. prepared silica aerogel using different precursors at supercrital drying process and freezing drying process respectively [12,13]. However, there are some reports available about preparation of surface modified silica aerogels by ambient pressure drying. But the removal of Na^+ ions from silica which is barrier to the density and transparency is rarely reported in the literature. It is necessary to eliminate the Na^+ ions ensnared within the pores of the gel network which directly affects to the hydrophilicity and opacity of the aerogels. Usually, unmodified silica aerogels are hydrophilic in nature [14]. To achieve hydrophobicity of aerogel, the hydrogen of the –OH– group must be replaced by some organic group silanol on the surface which get converted into a methylsilyl group and this treatment would change the surface activity. The modification of silica surfaces can be done by the variety of silane couplers and titanium couplers [15]. To make appropriate surface modification, the silica wet gel surface can be changed from hydrophilic to hydrophobic nature using mono, di and tri functional silylating agents.

Thus, in light of the above points, we have made a sincere attempt to prepare silica aerogel by simple, less time consuming and cost effective sol-gel process under ambient pressure drying condition. Sodium silicate was used as a silica precursor as it is inexpensive and abundant in nature. The surface modification of the wet gel was carried out by the trimethylchlorosilane (TMCS). Further, the modified aerogel was characterized by X-ray diffractometer (XRD), thermogravimetric and differential thermal analyzer (TG-DTA), Fourier transform infrared spectrometer (FT-IR), Brunauer-Emmett-Teller (BET) analyzer, field emission scanning electron microscope (FE-SEM) and Ultraviolet-Visible (UV–Vis) spectrophotometer for structural, thermal, functional, surface, morphological, optical etc. properties and results are reported here. The resulting aerogel possess high thermal stability, superhydrophobicity and large specific surface area as compared to the available literature reports [[16], [17], [18], [19]].

Section snippets

Materials

For the preparation of silica aerogels by sol-gel process sodium silicate (Na_2SiO_3) (CAS No. 1344-09-8, M.W.122.06 g/mol, purity ~ 99.9%) solution used as precursor, hexane (C_6H_{14}) (M.W. 86.18 g/mol, purity 85%), methanol (CH_3OH) (M.W. 32.04 g/mol, purity 99.5%) all were purchased from Loba chime and used as received. Ammonium hydroxide (NH_4OH) as a base catalyst (NH_3 was diluted in distilled water), where liquor ammonia purchased from Fisher Scientific (M.W. 17.03). To remove Na^+ ion from…

Mechanism of sol-gel polymerization-solvent exchange and surface modification of silica wet gel

Sol-gel technique is a widely used traditional method for the preparation of silica aerogels involving several parts such as hydrolysis, condensation, gel formation, ageing, silylation, drying etc. [[22], [23], [24], [25]]. Silica aerogels can be cost effectively synthesized with the help of water glass precursors and ambient pressure drying method. During the hydrolysis process, the formation of sodium salt is prime barrier in the transparency and density of the aerogels. Successful removal of …

Conclusions

Hydrophobic silica aerogel was successfully synthesized via ambient pressure drying using sol-gel polymerization of sodium silicate. XRD analysis of prepared silica aerogel confirmed the amorphous nature. These silica aerogels are fond to be thermally stable up to 478 °C with respect to the hydrophobicity; beyond this temperature it shows hydrophilic nature. Surface modification of prepared silica aerogel was confirmed by FT-IR studies. It also confirms the hydrophobic nature of the sample…

Conflict of interest

We wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.…

Author contributions

KMJ conceived the original idea and supervised the whole work. MVK performed all the experimental part, processed the experimental data and performed the analysis. MVK wrote the manuscript with support from KMJ and SBS. MVK and SBS aided in interpreting the results and worked on the manuscript. All authors discussed the results and contributed to the final manuscript. KMJ, MVK and SBS contributed to the revised version of the manuscript.…

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