

Improvements of reinforced silica aerogel nanocomposites thermal properties for architecture applications



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ABSTRACT

An 1,4-cis polybutadiene rubber/carboxymethyl starch (CMS)-based silica aerogel nanocomposites as a insulation material was developed that will provide superior thermal insulation properties, flexibility, toughness, durability of the parent polymer, yet with the low density and superior insulation properties associated with the aerogels. In this study, reinforced 1,4-cis polybutadiene–CMS–silica aerogel nanocomposites were prepared from a silica aerogel with a surface area $710 \text{ m}^2 \text{ g}^{-1}$, a pore size of 25.3 nm and a pore volume of $4.7 \text{ cm}^3 \text{ g}^{-1}$. The tensile properties and dynamic mechanical properties of 1,4-cis polybutadiene/CMS nanocomposites were systematically enhanced at low silica loading. Similar improvements in tensile modulus and strength have been observed for 1,4-cis polybutadiene/CMS mesoporous silica aerogel nanocomposites.

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1. Introduction

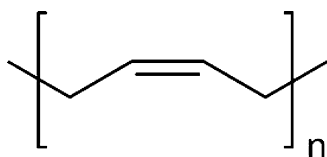
Aerogels are advanced materials almost like solid smoke, an aerogel resembles a hologram, appearing to be a projection rather than a solid object. They consist of more than 96 percent air. The remaining four percent is a wispy matrix of silicon dioxide [1]. Aerogels, consequently, are one of the lightest weight solids ever conceived. An aerogel is made by the so called “sol–gel process”. During this process, organic compounds containing silica undergo a chemical reaction producing silicon oxide (SiO_2) [2]. This mixture is a liquid at the creation of the reaction, and becomes more and more viscous as the reaction proceeds. When the reaction is completed, the solution loses its fluidity and the whole reacting mixture turns into a gel. This gel consists of a three-dimensional network of silicon oxide filled with the solvent [3]. During the special drying procedure, the solvent is extracted from the gel body leaving the silicon oxide network filled with air. This product is called aerogel. Silica aerogels (SiO_2) are unique porous materials composed of more than 90% air and less than 10% solid silica in the form of highly cross-linked network structures, which results in low thermal conductivity, and a large surface area ($500\text{--}1000 \text{ m}^2 \text{ g}^{-1}$), pore size (5–100 nm) and pore volume ($1.5\text{--}4.5 \text{ cm}^3 \text{ g}^{-1}$) [4–6]. The pore structure of aerogels is comparable to that of large pore

mesostructures. Therefore, aerogels that have an open-pore structure can be readily adapted to polymer nanocomposites as reinforcing agents. An organic polybutadiene (PBD) rubber-based aerogel insulation material was developed that will provide superior thermal insulation in the architecture, exhibiting the flexibility, toughness, and durability typical of the parent polymer, yet with low density and superior insulation properties associated with the aerogels [7]. The rubbery behaviors of the PBD rubber–CMS based aerogels are able to overcome the weak and brittle nature of conventional inorganic and organic aerogel insulation materials [8,9].

Inorganic aerogels such as silica aerogels demonstrate many unusual and useful properties. There are several strategies to overcoming the drawbacks associated with the weakness and brittleness of silica aerogels. Development of flexible fiber-reinforced silica aerogel composite blanket has proven one promising approach, providing a conveniently fielded from factor that is relatively robust toward handling in industrial environments compared to silica aerogel monoliths [10–14]. However, the flexible silica aerogel composites still have a brittle, dusty character that may be undesirable, or even intolerable, in certain applications [15–19]. Although the cross-linked organic aerogels such as resorcinol–formaldehyde (RF), polyisocyanurate, and cellulose aerogels show very high impact strength, they are also very brittle with little elongation [20,21]. In the present study, we report mechanical reinforcement of hyperbranched 1,4-cis polybutadiene/CMS nanocomposites using a silica aerogel as a reinforcing agent [22]. The PBD-based rubber aerogel nanocomposite are very

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Scheme 1. The chemical structure of 1,4-cis polybutadiene.

flexible, no-dust, and hydrophobic organics that demonstrated the following ranges of typical properties such as density, shrinkage factor and thermal conductivity [23,24]. The second component of polymeric system is 1,4-cis polybutadiene. For many applications, it is becoming increasingly popular to reinforce the concrete with small (Scheme 1).

2. Materials and methods

2.1. Materials

Butadiene as a monomer was purchased from Sigma–Aldrich with an obtained purity of $\geq 99\%$. Chlorobenzene ($\geq 99.7\%$) and hexane ($\geq 99\%$) also were purchased from Merck and were dried using molecular sieve and then distilled. Toluene was initially dried with sodium metal for 24 h and then distilled in the presence of metallic sodium. Cobalt dialkylthiocarbamate as a main catalyst was purchased from Sigma–Aldrich. To synthesize the silica aerogel, sodium silicate ($\text{Na}_2\text{O} = 9\text{--}19\%$, $\text{SiO}_2 = 28\text{--}38\%$, Merck Co.) was used as a silica source and ethyl trimethoxysilane (ETMS) (Aldrich) was used as a surface modifier of the silica.

2.2. Instruments

Transmittance through the samples was measured by obtaining UV–vis spectrum with Shimadzu UV-1601PC spectrophotometer. Simultaneous differential thermal and thermogravimetric analysis (DT/TGA) was carried out by using Seiko TG/DTA-320. Heating rate was $3^\circ\text{C}/\text{min}$, and rate of air flow over the sample was $30\text{ mL}/\text{min}$. A N_2 adsorption–desorption isotherm was obtained at 77.3 K on an ASAP 2420 volumetric adsorption analyzer. The silica aerogel was outgassed at 90°C for 30 min, followed by 230°C for 3 h. The microstructure and morphology of the silica aerogel were then observed by Philips XL-30 E SEM scanning electron microscope (SEM) at 30 kV (max.). The samples were prepared by physical vapor disposition method. The gold layer thickness was about 100 \AA at these samples. The nanocomposite samples were obtained by Freeze dryer Model FD-10 (Pishtaz Engineering Company). The samples were examined to determine the mean diameter and size distribution.

2.3. Preparation of carboxymethylstarch (CMS)

Firstly, the 0.5 g corn starch and 120 mL 2-propanol were placed in a 500 mL vessel and stirred for 2 h. The 5 g sodium hydroxide was added and reacted for 1 h at $78\text{--}80^\circ\text{C}$. After that, the 10 g chloroacetic acid was added to the vessel and stirred for another 2 h at 50°C . The product was filtered and washed several times with ethanol, then dried under vacuum. The resulting CMS was crushed in a mortar [degree of substitution (DS) = 0.49].

2.4. Preparation of silica aerogel

A silica aerogel was prepared using sodium silicate as the starting material. The sodium silicate was diluted with deionized water (sodium silicate:deionized water (weight ratio = $1:3$)) and 1.0 M aqueous HCl solution was used to modify the pH of the silica sol

to 5. The obtained silica was then stirred for 1 min, after which the sol suspension was aged until gelation occurred. To remove the sodium, the hydrogels were washed with deionized water three times, after which the silica hydrogel was collected. Next, butanol solution (pH adjusted to 2 by HCl solution) containing ETMS (5 wt% to the hydrogel) was added to the hydrogel. The mixture was then refluxed at 110°C ($\sim 10\text{ h}$) until the pore water was exchanged with butanol. The water was removed using a Dean-Stark trap. Finally, the ETMS-modified alcogel was dried at 130°C for 3 h in a vacuum oven.

2.5. Preparation of nanocomposites

To prepare the 1,4-cis polybutadiene polymer nanocomposites, a pre-determined amount of silica aerogel was added to the butadiene monomer and mixed at 25°C for 10 min. A stoichiometric amount of the Cobalt Dialkylthiocarbamate catalyst ($2.0 \times 10^{-4}\text{ M}$) in the toluene solvent was then added to the mixture and mixed at 25°C for another 10 min. The resulting slurry was out-gassed under vacuum and transferred to a silicon mold. Pre-curing of nanocomposite was conducted at 25°C for 2 h to complete the crosslinking. Then, CMS mixed at 25°C for 30 min.

2.6. Particle size

After drying at 37°C for 48 h, the mean diameter of the dried nanocomposite was determined by a sieving method using USP standard sieves. Observations are recorded.

3. Results and discussion

3.1. Morphology of reinforced silica aerogel

A solvent exchange step is required during the synthesis of silica aerogels to dry the hydrogels. Hexane with low surface tension have generally been used to exchange pore water even when a supercritical CO_2 drying tool is used to remove the organic solvent confined in the silica pores. A surface modification process is also necessary to induce hydrophobicity on the silica aerogel surface to avoid water adsorption. The hydrophobic aerogel can preserve its low thermal conductivity due to the absence of water in the pores. In this study, a silica aerogel was synthesized via simultaneous solvent exchange and surface modification in a butanol solution containing ETMS. Fig. 1 shows the SEM morphology of the silica aerogel. The synthesized silica aerogel exhibited a porous network structure with $5\text{--}100\text{ nm}$ pores. The particle size of aerogels is measured (Table 1).

Table 1

The average particles sizes of 1,4-cis polybutadiene-CMS silica aerogel nanocomposites.

Variables	Values	Average particle size (nm)
Aerogel content (wt%)	0	25.79 ± 0.12
	1	28.34 ± 0.06
	3	32.76 ± 0.23
	5	45.00 ± 0.16
Polymerization time (h)	0.5	20.16 ± 0.12
	1	23.76 ± 0.08
	1.5	29.76 ± 0.20
	2	35.02 ± 0.16
Temperature ($^\circ\text{C}$)	25	23.65 ± 0.16

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