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Thermal conductivities of silica aerogel composite insulating material

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ABSTRACT

Silica aerogels have interesting physical properties such as hydrophobicity and low thermal conductivity are advantageous for a wide variety of actual applications, such as super thermal insulators. Poor mechanical properties prevented to use silica aerogel directly. In this study, nano silica aerogel and its composite with cotton were synthesized using a water glass precursor by ambient pressure drying method .To modify silica aerogel use trimethyl chlorosilane (TMCS) diluted in n-hexane (1:5 volume ratio). By adding TMCS, –OR groups in silica aerogel structure replaced –OH and silica aerogel became hydrophobic. Thermal conductivity and hydrophobicity of synthesized samples were measured by needle probe method and contact angle meter respectively. Pure silica aerogel contact angel is 166 ° and it shows silica aerogel is super hydrophobic. Silica aerogel added to cotton structure in 20 %, 40 %, 60 % and 80 % wt. Silica aerogel particles attached cotton fibers and changed hydrophobicity properties. Contact angels of composites are 115°, 120°, 128° and 129° respectively. Cotton is hydrophilic and its ability to absorb moisture causes decreasing contact angel in composites. Silica aerogel and cotton thermal conductivities are 0.0134 Wm⁻¹K⁻¹ and 0.0308 Wm⁻¹K⁻¹ respectively. Having low thermal conductivity, pure silica aerogel is known as super thermal. Thermal conductivity of composites was measured 0.0251, 0.0227, 0.0216 and 0.0171 Wm⁻¹K⁻¹ respectively. By increasing silica aerogel ratio, thermal conductivity decreased. In silica aerogel- cotton composite improved structural strength and the thermal conductivity and hydrophobicity were acceptable. Copyright © 2016 VBRI Press.

Keywords: Silica aerogel; nano material; thermal conductivity; composite; cotton.

Introduction

Silica aerogel is lightest porous solid with nano structure. The open cell structures have very interesting properties such as low density (<0.05 g/cm³), high transparency (Because of its nanometer particles and pores size) and surface area $(>500 \text{ g/m}^2)$ [1, 2], ultra-low dielectric constant (~1-2), high porosity (>80 %), environmentally friendly and low thermal conductivity ($< 0.02 \text{ Wm}^{-1}\text{K}^{-1}$) [3]. These properties make silica aerogel appropriate for absorbents, sensor, drug delivery, catalyst and catalytic supports [4], energy, electronic [5, 6], humidity sensor and especially in thermal insulation applications [7]. Because of the increasing cost of energy and limited energy resources, energy savings have become more and more important [7, 8] and Thermal insulation material is a kind of material that can significantly prevent heat loss by reducing the heat flux through it [7, 9]. Using thermal insulation with acceptable operation is one of the best methods to prevent heat lost [10,11]. Thus insulation material is of great importance for many engineering applications, such as energy, building, aeronautics and astronautics and so on. Thermal conductivity is one of the most important parameter that reflects the insulation performance of the thermal insulation material [12]. Aerogels was first discovered by Kistler in the 1930s [13] and can be used as monolith, granules and composite [14]. Silica aerogel with low thermal conductivity is known as super thermal insulation [15,16]. Water thermal conductivity is greater than conventional thermal insulation. So thermal insulation is considered hydrophobic until it does not adsorb humidity and can be used for long time [17]. By adding TMCS, -OR groups in silica aerogel structure replaced by -OH and silica aerogel became hydrophobic [18]. Because of surface modification silica aerogel is super hydrophobic material and can be used in moist environment for long time [19]. Because of pours structure and low density, silica aerogel is fragile, [20, 21] brittleness and weak mechanical strength (Because of the counterpart of the large porosity) [22]. In this study, to improve silica aerogel flexibility cotton is used as matrix. Cotton fibers were incorporated into the sol to gelation and silica aerogel particles attached to the matrix fiber [23,24]. Cotton is flexible and can improve mechanical properties but it adsorbs humidity and becomes hydrophilic. Silica aerogel- cotton composites are flexible with low thermal conductivity. Also silica aerogel particles in composite structure make its hydrophobic. It is expected that the composites of cotton and silica aerogels becomes water-resistant and may improve the performance of the composite as insulation. Hence, in the present work, silica aerogel and silica aerogel-cotton composites were synthesized using water-glass precursor by a two-step sol-gel process followed by ambient pressure drying method. Thermal insulation and the hydrophobic properties of composites at various compositions have been reported.

Experimental

Materials

The chemicals used in this work were: sodium silicate $1.35 \,\mathrm{gm}^{-1}$, Merck Co., Germany), $(Na_2SiO_3,$ trimethylchlorosilane (TMCS) silylating as agent (C₃H₉ClSi, 0.86gm⁻¹, Merck Co., Germany), n-hexane $(C_4H_{14},\ 0.655gm^{-1}$, Merck Co., Germany), tartaric acid $(C_4H_60_6,\ 1.79gm^{-1}$, Merck Co., Germany) and isopropyl alcohol (IPA) (C₃H₈O, 0.786gm⁻¹, Arman Sina Chemical Co., Iran) and distillated water achieved from deionizer (Water Safe Co- Taiwan).

Material synthesis

Sodium silicate solution (water-glass solution) was diluted with the deionized water (by 1:4 volume ratio) in order to obtain the desired weight percentage of silica in the starting material and then mixed with tartaric acid 3.6 n, gel formed after 15 min. Cotton placed in batch reactor. Sol solution adds into cotton with different weight ratio and place at oven and kept at 55 °C for 3h. After strengthening the gel structure, to remove the Na⁺ and sodium tartrate (as byproduct), gel washed with distillated water in 4 times during a 24 h period. In the next step, hydrogel was immersed in isopropyl alcohol and n-Hexane for 24 h at 55 °C subsequently in order to exchange pores water and also strengthen the networks of the gels. To surface modification the wet gels were immersed in an equivolume TMCS/n-Hexane solution (20 % TMCS in n-Hexane) for 24 h at 55 °C. Finally solution was decanted; silvlated gels were washed with n-hexane and kept at ambient temperature for 24 h. In this step residual water removed from silica aerogel / cotton composite [5, 25]. The composite was dried according to the following sequence: 50 °C for 3 h, 70 °C for 3h, and 100 °C for 2 h then was cooled to room temperature and characterized [3, 25, 26].

Device fabrications

The hydrophobicity of the pure silica aerogel and silica aerogel/ cotton composites is tested by contact angle (θ) measurement (PGX, Thwing- Albert. Instrument Co). Water drop placed on samples surface and contact angle measured. The thermal conductivity measured by thermal conductivity measurement (made by Electrical Engineering faculty and Transport Phenomena Research Center (TPRC) of Sahand University of Technology (SUT). In this method probe placed into sample and heated by energy pulse (25.92 mW for 3s). After charging, sample cooled and after a few minutes temperature became constant, then thermal conductivity got measured [27]. In this study Field Emission Scanning Electron Microscopy (FE-SEM, Mira 3-XMU) to surface morphological was used, and structure of pure silica aerogel also dispersion and penetration of silica aerogel powder in cotton was studied. Apparent density of the samples was measured by mass to volume ratio ($\rho = mv^{-1}$) of the aerogel, in which mass was measured by the microbalance with the accuracy of 10^{-5} g and volume of the aerogel was measured by filling aerogel in the measuring cylinder of the known volume [5].





Fig. 1. FE-SEM photos of (a) pure silica aerogel and (b) silica aerogelcotton composite.

Results and discussion

Morphology

Pure silica aerogel FE-SEM image was shown in **Fig. 1(a)**. It is observed that pure silica aerogel exhibits porous network structure which contains nano-scale spherical particles (L = 20-40 nm) with uniform distribution. **Fig. 1(b)** shows the microstructure of silica aerogel-cotton (40 % wt. silica aerogel) composite. Silica aerogel particle penetrated to internal structure of cotton uniformly and also attached to flexible cotton fiber. By added silica aerogel in cotton matrix, the composite properties such as thermal conductivity, hydrophobicity, flexibility and density will change [**20, 23**].

Hydrophobicity analysis

Hydrophobicity is more important requirement in thermal insulation industry [28] to achieve the best application for

long period of time [19]. Water thermal conductivity is more than thermal insulation and absorbing humidity by thermal insulation will decrease its performance and it will need to replace after short time [17, 28, 29]. Also wet thermal insulation provides conditions for growth and reproduction fungi and bacteria that can be dangerous especially in food and drug industry [28, 30]. If the material has a high-energy surface, contact angle will be less than 90° and liquid spreads on the surface and it is known as hydrophilic. On the other hand, if the material has a low energy surface then contact angle is more than 90° and the liquid does not spread on the surface and it is known as hydrophilic (water- resistant) [17, 29, 31]. Silica aerogel can be hydrophobic (Fig. 2a) with θ =166° and hydrophilic with θ = 46° (Fig. 2b) [17].





Wettability of silica aerogel depends on chemical group that cover sample surface. The hydroxyl groups (– OH) in silica aerogel structure are the main reason of hydrophilic properties [32,33]. The hydrophilic surface of the aerogel converted into hydrophobic by surface chemical modification using TMCS surface chemical modification agent (hydrophilic –OH groups were replaced by hydrophobic –CH₃ groups) [1, 34]. Cotton is hydrophilic and absorbs water immediately. Cotton thermal conductivity is low, but its thermal conductivity increases and needs to be changed after a short period of time because of its ability to attract moisture. Silica aerogel/cotton composites is a hydrophobic thermal insulation and contact angel depends on amount of silica aerogel [31, 35]. Hydrophobicity of the silica aerogels and their composite

were tested by measuring the contact angle (θ) with a water droplet placed on the surface using a contact angle meter [22]. A photograph of water droplet on the silica aerogelcotton composites with 20 %, 40 %, 60 % and 80 % wt. silica aerogel is shown in **Fig.** Silica aerogel-cotton composite is not flat and contact angle is affected by silica aerogel, glass wool fibers and air in jagged surface. The contact angle of composites was shown in **Table 1**.

 Table 1. Density, thermal conductivity and contact angel of silica aerogel/ cotton composite.

Sample	Silica aerogel %wt.	Density (kg/m ³)	Thermal conductivity (Wm ⁻¹ K ⁻¹)	Contact angle (degree)
Silica aerogel	100	140	0.01349	166
Silica aerogel/cot ton composite	80	125	0.0171	129
	60	115	0.0216	128
	40	90	0.0227	120
	20	78	0.0251	115
cotton	0	70	0.0308	0



Fig. 3. (a) Curve of thermal conductivity and (b) curve of contact angle vs. silica aerogel percent.

The result shows that contact angel increased by increasing silica aerogel weight ratio. It is evident that hydrophobicity of silica aerogel is more and more than that of cotton. Therefore, it is logical that the hydrophobicity of composite increased with increment of silica aerogel in silica aerogel-cotton composites. Fig. 3b shows that contact angle vs. weight percentage of silica aerogel in silica aerogel-cotton composite. It is evident that hydrophobic silica aerogel-cotton composite ($\theta > 90^\circ$) was obtained by adding a small amount of silica aerogel in cotton matrix (20 % wt. silica aerogel-cotton composite has contact angle of 115°). Also by adding 20 %wt. cotton, the composite contact angle are decreases sharply. That is because cotton is super hydrophilic and changes hydrophobicity properties. There is semi liner relationship between silica aerogel %wt. and contact angel in composites.

Thermal conductivity

In silica aerogel as porous material, total thermal conductivity be recognized as the sum of the solid conduction (k_s) , gaseous (gases trapped in cavities) conduction (k_g) , convective (k_c) , solid and gas radiation (k_r) without any assumptions. At low temperature heat transfer via radiation mechanism is negligible. The pores size are

smaller than air (trapped in cavities) mean free path so convective heat transfer is negligible. So conduction is the main heat transfer mechanism and total thermal conductivity (k_t) of pure silica aerogel is $k_t = k_s + k_g$ [23,36-38]. By adding cotton, energy is transferred by cotton fibers and cotton fibers conduction (k_m) is causes total thermal conductivity to be increased [39]. Thermal conductivity of synthesized samples was measured by needle probe method using the thermal conductivity measurement. To measure pure silica aerogel, cotton and composite thermal conductivity probe of thermal conductivity measurement placed into the sample and heated by energy pulse (25.92 mW for 3s). During charging, probe temperature increased. After a few minutes the temperature decay after discharging a short pulse of energy into a probe placed in the sample is measured. The temperature decay rate depends on thermal conductivity of sample [27]. Fig. 4 show silica aerogel temperature decay via time. The probe temperature increased 31.3 °C. After about 140s decreased to ambient temperature (temperature became constant). Silica aerogel is high porosity [1,40] and there are small amount of solid in silica aerogel skeleton.



Fig. 4. (a) Temperature and (b) noise temperature decay vs. time in pure silica aerogel after discharging pulse.



Fig. 5. Pure silica aerogel thermal conductivity versus time curve for needle probe method.

Also gas (that trapped in captive) conduction is low. So, it is evident that pure silica aerogel thermal conductivity (k_s+k_g) is low. **Fig. 5** shows that temperature of probe became constant and thermal conductivity of silica aerogel was calculated about 0.0135 Wm⁻¹K⁻¹. In this method, thermal conductivity is independent of shape and properties of the probe [**41**].



Fig. 6. (a) Temperature and (b) noise temperature decay vs. time in silica aerogel- cotton composite with 20% wt.

Low thermal conductivity of silica aerogel (lowest than air) causes silica aerogel be known as super thermal insulation. Fragile structure of silica aerogel prevents to use directly. Cotton thermal conductivity was measured 0.0309 $Wm^{-1}K^{-1}$. By adding cotton in silica aerogel structure, mechanical properties improve and composite become flexible. **Fig. 6** shows temperatures versus time in silica aerogel-cotton composite containing 20% wt. silica aerogel. The probe heated by energy pulse (25.92 mW) for 3 sec and temperature decreased from 29.7 °C to about 29.3°C and after 60 sec became constant. It shows that composite thermal conductivity is less than pure silica aerogel. That is because heat loss in composite is faster. Error! Reference source not found.**7** shows that the thermal conductivity of composite with 20 % wt. silica aerogel is 0.0251 Wm⁻¹K⁻¹. The thermal conductivity of synthesized samples and cotton was presented in **Table 1**.



Fig. 7. Silica aerogel- cotton composite with 20% wt. thermal conductivity versus time.

By increasing cotton, composite thermal conductivity increases. The main heat transfer happens along the fibers and energy transfers via cotton fibers that has highest thermal conductivity [39]. On the other hand, cotton fibers fills pores of silica aerogel particles and heat is transferred from fibers paths more quickly. Curve "a" in Fig. shows the change in thermal conductivity of silica aerogel-cotton composite with silica aerogel weight fraction. By increasing of silica aerogel weight percentage, thermal conductivity decreases from 0.0308 to 0.01349 Wm⁻¹K⁻¹. There are semi-liner relationship between composite thermal conductivity and silica aerogel weight ratio. Thermal conductivity sharply decreases after 60 % wt. silica aerogel. That is because cotton fibers become surrounded by silica aerogel particles [23, 39]. Attaching particles prevented direct contact in the fibers and silica aerogel controls heat transfer phenomena. Because conduction as main mechanism in silica aerogel and silica aerogel- cotton composite is very a low heat transfer mechanism, thermal conductivity of sample is low [39, 42].

Conclusion

In this study, silica aerogel and silica aerogel-glass wool composites were synthesized using sodium silicate precursor by ambient pressure drying method. Low thermal conductivity (0.0134 Wm⁻¹K⁻¹) and super hydrophobicity (θ =166°) of silica aerogel made it as thermal insulation that can used for a long time. In order to improve mechanical

properties and flexibility cotton was used as matrix. Silica aerogel particles attached to cotton fibers and changed hydrophobicity and thermal conductivity in composite. Hydrophobicity of composites with 20, 40, 60 and 80 % wt. silica aerogel is 115° , 120° , 128° and 129° respectively. By increasing of silica aerogel weight percentage, thermal conductivity decreases from 0.0308 to 0.01349 Wm⁻¹K⁻¹. That is because silica aerogel particle that attached cotton fiber controls heat transfer.

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