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Fabrication and characterization of silica aerogel blanket as an excellent thermal insulating material

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Abstract. The main application of silica aerogel is as an insulating material. However, basic forms of silica aerogel have limited application. Silica aerogel can be embedded in fibrous materials to achieve a mechanically reinforced and continuous form. This aerogel blanket can be easily used in many applications. In this study, a silica aerogel blanket has been fabricated through sol–gel process. Aerogel precursors undergo the synthesis steps within the fibreglass matric. The thermal conductivity of the fabricated aerogel blanket was 0.021–0.077 W m^{-1} K^{-1} over 20–600°C. The blankets were hydrophobic with a contact angle of 138°. The linear length shrinkage and linear width shrinkage were obtained at 0.12 and 0.22%, respectively. Values of compressive resistance of blankets were 149, 549 and 1700 kPa for 30, 50 and 70% deformation, respectively. The results of hot surface performance, flexibility or rigidity, maximum exothermic temperature rise, and resistance to vibration analyses confirm the excellent thermal and mechanical performance of the fabricated aerogel blankets.

Keywords. Silica aerogel blanket; sol–gel; insulation; thermal and mechanical properties.

1. Introduction

Aerogel is an excellent option for use as thermal insulating material. Aerogels have the lowest thermal conduction among the solids and their conductivity even can be lower than the air conductivity [1,2]. This excellent insulating performance of the aerogels is due to their unique structure in which very small nanoparticles (2–5 nm) are connected to each other in a 3D network and form a highly porous structure. Aerogels are the lightest commercial solids with very high porosity of up to 99% and with pores size in the range of 2–50 nm [2–6].

Convection heat transfer is carried out through the materials pores. This heat transfer mechanism will be considerable for porous materials with pore size larger than 1 mm, in which the air molecules can move freely. Pore size of aerogels is lower than the mean free path of the molecules as well as the pores have a tortuous path, therefore, the convection of the air molecules within the aerogel pores will be difficult, which remarkably restricts the heat convection. Heat conduction will be done through the solid section of aerogels or silica nanoparticles. However due to the high porosity of aerogels, only 1–10% of the aerogel volume is solid, and very low mass and volume of the nanoparticles heat conduction through solid section of aerogel is very low. Primary nanoparticles are interconnected in different directions and form a 3D and tortuous path with nanometric sizes and also in many cases the end of these solid paths is a dead end and does not reach the other side of the aerogel, therefore, the heat transfer through these solid nanoparticles path will be negligible [4,7–12].

Basic forms of silica aerogel, including granule, powder or monolith, are fragile, segregated and difficult to handle, which limits their application as continuous and integrated coverage on insulating surfaces. To overcome this drawback and reinforce the mechanical properties and facilitate silica aerogel utilization as an insulation material, it can be embedded within fibrous matrices. In this method, silica aerogel is supported by flexible and non-woven fibre matrices, in which the resulting composite is an aerogel blanket [1,3,7,13–16].

Silica aerogel blankets are flexible and can be cut easily and used on different industrial equipment or building sectors. They have excellent properties in terms of fire resistance, thermal conductivity, mechanical strength and flame spread indexes [3,14,17–19]. They are hydrophobic, which leads to remarkable abatement of corrosion under insulation (CUI). They do not settle or loose over time. In addition to attractive performance properties, aerogel insulations have many health and environmental benefits. Long service life, high recyclability and much lower consumption volume of aerogel blanket than conventional insulations drastically reduce the amount of waste of these insulators. The potential of ozone depletion and global warming and

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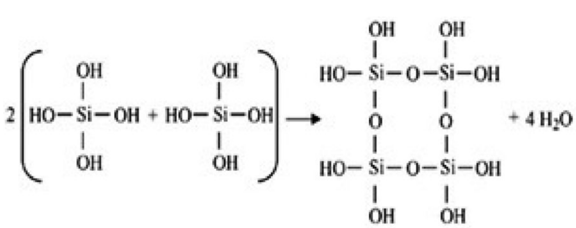
toxicity of this insulation material is near zero [3]. Silica aerogel blankets have been commercially produced by some manufacturers such as Aspen Aerogel, Inc., Cabot Corporation, Svenska Aerogel AB, Acoustiblok UK Ltd., Active Space Technologies, and Airglass AB [3]. However, lowering the production cost can have a great impact on the development of the silica aerogel blanket market and its widespread application in different industries [13]. This work aims to manufacture silica aerogel blankets through an economical process. Different characteristics of the produced aerogel blanket such as thermal conductivity, linear shrinkage, acoustic performance, hydrophobicity, maximum exothermic temperature rise, resistance to vibration, flexibility and compressive resistance have been studied and the results showed the high quality of the produced aerogel blanket.

2. Experimental

2.1 *Experimental procedure*

The aerogel blankets used in this study were produced by Pakan Atiyeh Nano-Danesh Company in Iran. Sol–gel method was used to synthesize the silica aerogel blanket. In order to obtain the silica sol, water was added to industrial sodium silicate solution with the 4:1 volume ratio and agitated vigorously. Then, the acid solution was used as a catalyst for acceleration of the sol–gel process. After the sol reached to pH = 4, the fibreglass blanket was completely immersed in the sol. The glass water solution turns into silicic acid as a result of neutralization (reaction 1). Next, the condensation reaction of silicic acid molecules (reaction 2) takes place and Si–O–Si bonds are formed. The step-by-step and gradual condensation of small silicic acid oligomers leads to a continuous structure of silicon atoms and forms small silica particles and chains, which finally results in a silica gel network.

$$Na_2SiO_3 + H_2O \xrightarrow{2H^+} Si(OH)_4 + 2Na^+ \quad (1)$$



(2)

Special fibreglass with excellent mechanical properties was used as fibrous matrices. The soaked fiberglass was aged in order to convert the sol to gel and the fiberglass was completely filled with gel. Then, the obtained fibreglass was washed with water at temperature of about 50°C to remove any salt and impurities from the gel. Finally, the fibereglass-containing gel was placed within the batch-mode high-pressure reactor for supercritical drying of the fibreglass using methanol and isopropyl alcohol as a solvent to achieve the silica aerogel blanket. Supercritical drying was carried out at 65 bar and 240°C. Methoxylation with methanol is the main mechanism for hydrophobicity generation.

2.2 *Experimental characterization*

The different thermal and mechanical characteristics of the fabricated aerogel blanket were investigated. The main required properties of the aerogel blanket for commercial applications such as thermal conductivity, compressive strength, resistance to vibration, linear shrinkage, hot surface performance, maximum exothermic temperature rise and acoustic performance were analysed using the ASTM standard methods.

3. Results

3.1 *Thermal conductivity*

Thermal conductivity of fabricated silica aerogel blanket, IRogel, was determined as ASTM C177-19. Aerogel blanket samples with 30 × 30 cm dimensions were put in heat flow meter apparatus between hot and cold plates with a given temperature difference. After the system reached stable condition, thermal conductivity was calculated using Fourier's law. Since aerogel blanket can be used up to 650°C application, the thermal conductivity values were determined over a wide range of 20–600°C. The obtained values for thermal conductivity for the IRogel were compared with other commercial products, as in figure 1, which shows the acceptable thermal conductivity for the present aerogel blanket. The thermal conductivity of other products is taken from their datasheets, as presented by their manufacturers (https://www.aerogel.com).

3.2 *Hydrophobicity*

Hydrophobicity is an important factor to maintain the insulation efficiency during its lifetime and also to mitigate the CUI. The insulation performance can be lost remarkably as the water penetrates into insulation and is retained within the insulation. It is reported that the insulation efficiency can be reduced up to 15 times as a result of moisture penetration into the insulation material [20]. On the other hand, moisture penetration into the insulation material is the main cause of the CUI. CUI is a serious issue in different industries, such as petrochemical, oil and gas refineries and chemical industries. CUI accounts for about 40–60% of maintenance costs of insulated pipes [20–22].

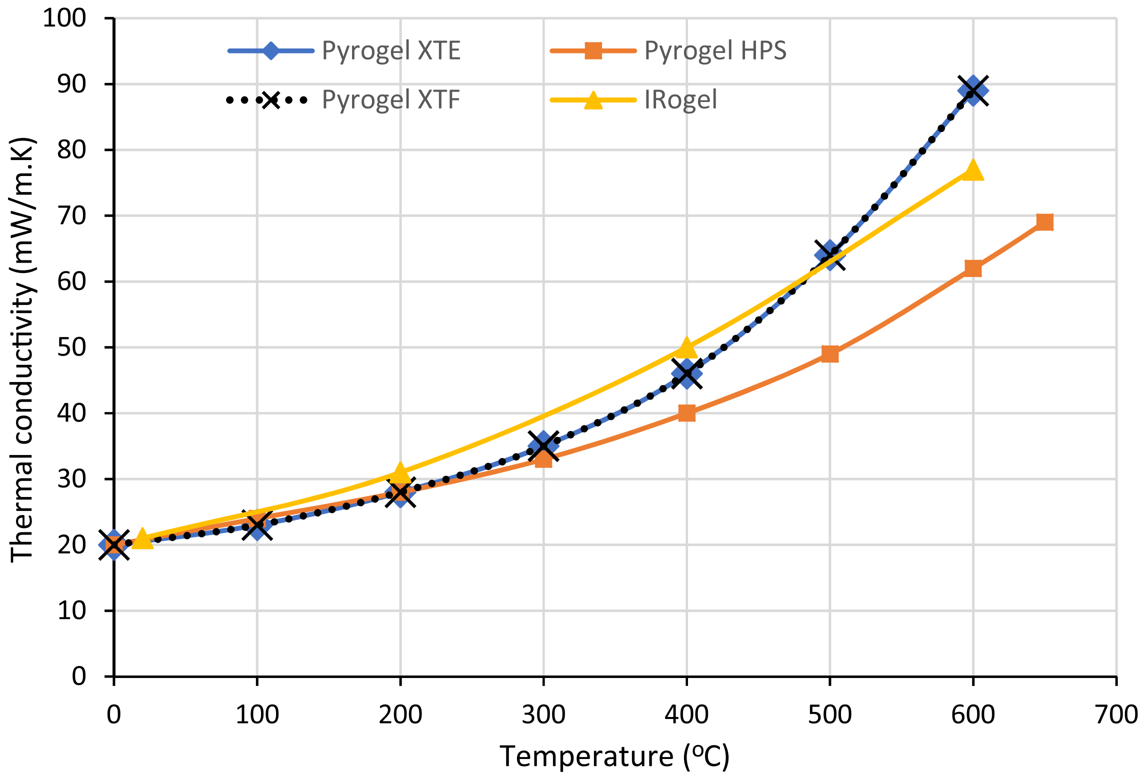


Figure 1. Thermal conductivities of different commercial silica aerogel blankets *vs.* temperature.

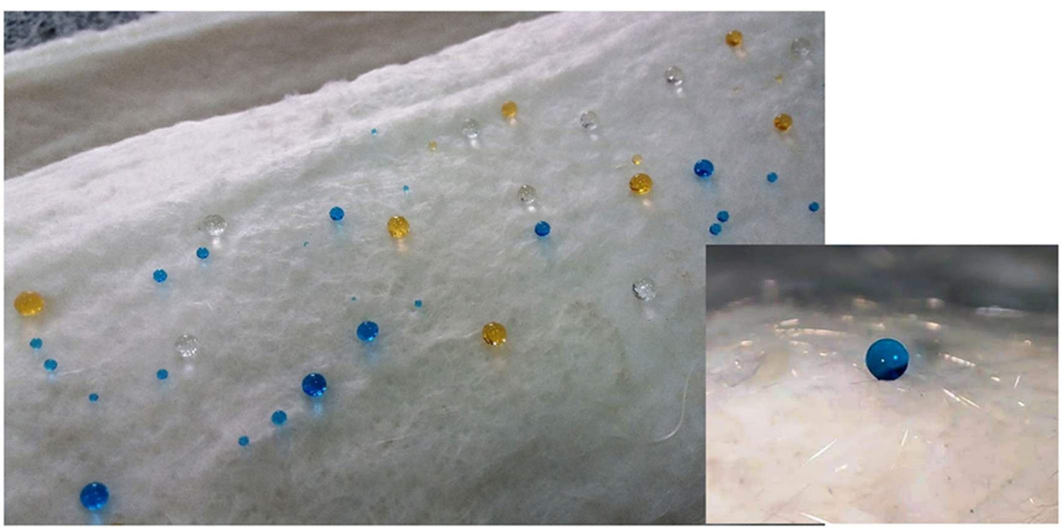


Figure 2. Hydrophobicity of IRogel, water droplets on the surface of IRogel.

The fabricated silica aerogel blanket is hydrophobic, as shown in figure 2. The obtained contact angle is 138°, which restricts the water uptake into insulation materials. The hydrophobicity is due to highly crosslinked alkyl siloxane functional groups, which are covalently bonded to the aerogel backbone and lead to high hydrophobe durability of the silica aerogel blanket. This property of the silica aerogel blanket has an important effect in reducing the CUI and increasing the efficient lifetime of the insulation.

3.3 *Hot surface performance standard*

In this standard, the insulation stability was tested against a hot surface for a given period.

For the fabricated aerogel blankets, this test was conducted as ASTM D1728/ASTM C441/ASTM C447. The blanket samples were placed on a metal surface at 649°C for 96 h. The temperature of the metal surface was kept constant during the test time. After conducting the test, the

Table 1. Linear shrinkage of fabricated aerogel blankets according to ASTM C356.

Sample	Linear length shrinkage (%)	Linear width shrinkage (%)
1	0.08	0.21
2	0.1	0.31
3	0.16	0.17
4	0.13	0.18
Average	0.12	0.22

possible defects in sample are inspected. For the fabricated blankets, the following observations were reported:

- No warping has been observed.
- Ignition, smoke emission or frying of the sample was not observed during the test.
- After test completion, defects such as cracking and flacking of sample were not observed.

Conventional insulations have a high drop in efficiency during operation at temperature above 400°C. Aerogel blanket is an attractive option for insulation of high-temperature applications. On the basis of these test results, the aerogel blanket can keep its initial efficiency for a long time. Various furnaces, gas turbines, reactors in different industries operate in temperatures higher than 400°C.

3.4 *Linear shrinkage*

Different materials experience local melting and volume reduction when exposed to high-temperature conditions. Insulations designed for high temperatures must be resistant at designed temperatures without significant shrinkage.

Standard test methods, ASTM C356–ASTM C1728, have been used to determine the amount of linear shrinkage. In this test, four samples were dried at 121°C for 48 h; then, their dimensions were exactly measured. The samples were placed in a furnace at 649°C for 24 h. Then, the samples were taken out of the furnace and after cooling to atmosphere temperature their length and width were again measured based on test protocol.

After conducting this test on the aerogel blanket samples, no apparent changes were observed in the samples. The results of this test are presented in table 1. The average values of linear length shrinkage and linear width shrinkage were obtained as 0.12 and 0.22%, respectively. The observed linear shrinkages of aerogel blanket were in the acceptable range (< 2%) and showed the high durability of the aerogel blanket samples under continuous exposure at high operating temperatures.

3.5 *Acoustic performance*

It is expected to have a good sound absorption performance for silica aerogel due to its high nano-porosity (2–50 nm pore sizes with about 90% porosity) and large surface area. Sound insulation properties of aerogel blankets can be useful in the abatement of noise pollution in industries and residential areas as an important challenge especially in metropolises. In order to evaluate the sound absorption performance of the fabricated aerogel blanket, the samples are placed in a standard chamber. Sound is generated with different frequencies on one side of blanket and its intensity is measured on both sides of the blanket. The sound intensity reductions at different frequencies when the blankets were placed against sound waves were determined and the obtained values are presented in table 2.

3.6 *Compressive resistance*

The compressive resistance was measured as ASTM C165. The crosshead speed was adjusted to 1.3 mm min^{-1}. Aerogel blanket was used as a specimen for the desired deformations of 30, 50 and 70%. Applied load at any given deformation and compressive resistance were determined and reported in table 3. As seen, the average values of compressive resistance of samples were obtained as 149, 549 and 1700 kPa for 30, 50 and 70% deformations, respectively. Consequently, the aerogel blanket has very high compressive resistance in comparison with conventional insulation, such as rockwool. Conventional insulation generally is loose-fill and collapses under compression which leads to a reduction in their performance. Aerogel blanket can be tightened on the insulated equipment without any reduction in its porosity volume. Due to the high mechanical strength of aerogel blankets, they do not suffer from fuzzing, sagging or collapsing during life time.

Table 2. Sound intensity abatement at different frequencies using an aerogel blanket.

	100 Hz	440 Hz	1000 Hz	10000 Hz
Sound intensity without aerogel blanket (db)	102.3	115.3	113.7	114.7
Sound intensity behind aerogel blanket (db)	81.8	102.2	84.6	65.4
Sound intensity reduction behind aerogel blanket (db)	20.5	13.1	29.1	49.3

Table 3. Compressive resistance of aerogel blanket according to ASTM C165.

Sample no.	Dimension (mm × mm)	Crosshead speed (mm min^{-1})	Sample eight (mm)	Deformation (mm)				Loaded force (N)				Compressive resistance (MPa)			
				10%	30%	50%	70%	10%	30%	50%	70%	10%	30%	50%	70%
1	54.8 × 50.89	1.27	10.08	1.01	3.02	5.04	7.06	390	1338	4099	14139	0.14	0.48	1.47	5.07
2	51.43 × 50.12	1.27	10.26	1.03	3.08	5.13	7.18	464	1495	4047	13120	0.18	0.58	1.57	5.09
3	51.89 × 50.24	1.27	10.81	1.08	3.24	5.41	7.57	452	1409	3908	12735	0.17	0.53	1.47	4.79
4	51.43 × 50.12	1.27	9.40	0.94	2.82	4.7	6.58	486	1539	4295	13858	0.18	0.57	1.59	5.13
Average	0.17	0.54	1.53	5.02											

Table 4. Weight and length changes during resistance to vibration test.

Parameter	Before test	After test
Weight (g)	1393.7	1374.4
Average length (mm)	916.40	912.45

Table 5. Obtained results of the resistance to vibration test.

Value	Result	Acceptance criteria
Weight change (%)	1.38	Max: 15%
Sagging test in length (mm)	3.95	Max: 76 mm

3.7 *Flexibility or rigidity*

The flexibility of hot and cryogenic insulation materials has an important effect on their ease of use and installation. ASTM C1101 test method was used to determine the flexibility or rigidity classification of the aerogel blanket. A sample with a given dimension was bent through an angle of 90° over a pipe with an outer diameter of 21.3 mm (NPS 1/2 in). Then, its outer surface was inspected for visual rupture. No ruptures, cracks or tears on the aerogel blanket insulation surface were observed, but after the insulation is opened, it returns to its original state. Therefore, aerogel blankets can be considered as resilient flexible. This type of aerogel can be easily and quickly installed on the various structures.

3.8 *Maximum exothermic temperature rise*

Polymeric binders have been used in the formulation of the conventional insulations such as rockwool. These polymeric materials can be combusted when exposed at high temperatures, which can lead to a rise in the insulation temperature. Maximum exothermic temperature rise is tested using ASTM D1728/ASTM C411/ASTM C447. For Grade 1 Category A insulation, when the insulation was installed on the surface with the maximum use temperature, its internal temperature should not exceed the hot surface temperature more than 111°C. The aerogel blanket sample was placed on the hot surface at 649°C temperature for 96 h, and its internal temperature was monitored. No temperature rise was observed within the samples. This result showed the thermal stability of the aerogel blanket composition against the maximum use temperature.

3.9 *Resistance to vibration*

Resistance to vibration is one of the main properties required for blanket insulators in industrial applications, especially in equipment with moving parts such as turbines, compressors, dryers and pumps. Vibration can lead to excessive sagging or loss of structural integrity of the insulation and weaken the performance of the insulation over time.

Resistance to vibration was measured by ASTM C592. In this method the sag, settlement, or shakedown of the blanket due to heating or vibrating was determined. The aerogel blanket samples without the attachment of any tie wires or metal mesh coverings with 610 × 914 mm dimensions were prepared and weighed. The vertical distance from the horizontal surface to obtain sag measurement at 40 points was done. Then the specimen was placed in hot-plate at 399°C for 5 h. In the following, the sample was subjected to a vibration test with a frequency of 12 Hz and a total range of 3 mm for 6 h. Finally, the weight and measurements were repeated. The results of this test are shown in tables 4 and 5. On the basis of the obtained results, the silica aerogel blanket sample passed the required criteria and can be applied in industrial applications.

4. Conclusion

Silica aerogel blanket was fabricated through an economical process using sodium silicate as silica precursor and supercritical drying technique. Different thermal and mechanical properties of the silica aerogel blanket were determined using well-defined and ASTM methods. The results showed that the fabricated aerogel blanket passes all the necessary standards, and can be used for insulating the different equipment that have working temperatures up to 650°C. Unique properties such as hydrophobicity, very low thermal conductivity and high thermal stability, good flexibility and mechanic strength, very low required volume compared to conventional insulation materials, sound absorption, ability of the lack of sagging, deformation, weight loss and shrinkage under vibration or hard thermal and mechanical working conditions make the fabricated silica aerogel blanket as an attractive insulating material.

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