ORIGINAL ARTICLE

Highly flexible, thermally stable, and dust-free fiber-embedded nanoporous Silica aerogel blanket for spacecraft applications

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Received 18 August 2023; revised 07 October 2023; accepted 07 October 2023; available online 10 October 2023

Abstract

The highly lightweight and thermal insulator polyvinyl alcohol (PVA) doped fiber-embedded silica aerogel blankets was prepared with different types of fibers. The performances of PVA-doped silica aerogel blankets were compared. The silica aerogel blankets were prepared by integrating different fibers with encapsulated the PVA during the aging process. The synergic effect of PVA doping-derived silica aerogel blankets on the thermal conductivity, surface physical characteristics and young modulus were investigated. Consequences revealed that PVA-encapsulated blankets could diminish the thermal conductivity, porosity, and density of the aerogel blanket. The prepared blankets have shown further properties intensely without significant reduction. Experimental outcomes exhibited the properties of improvement in thermal conductivity (in the range of 0.023-0.035 W/m.K), density (in the range of 0.051 - 0.118 g/cm³), and Young Modulus regarding the pure silica aerogel. The prepared PVA-based aerogel blanket has prospective in various sectors such as the production of spacecraft apparel, military jackets, clothes for fire-fighting and hilly areas people from low-temperature protection.

Keywords: Ambient Pressure Drying; Dust-Free Silica Aerogel Blanket; Nanoporous; Polyvinyl Alcohol (PVA) Encapsulation; Thermal Insulator, Mechanical Robust.

How to cite this article

Bakul Jadhav S., Makki A., Hajjar D., Bhikaji Sarawade P. Highly flexible, thermally stable, and dust-free fiber-embedded nanoporous Silica aerogel blanket for spacecraft applications. Int. J. Nano Dimens., 2023; 14(4): 320-330.

INTRODUCTION

Silica aerogels, consequential from the solgel method, have an exclusive nanoporous structure. These are fascinating properties such as low density, high specific surface area, high porosity, and low thermal conductivity [1, 2], etc. Such properties have drawn significant attention in aerospace applications, thermal insulation, catalytic supports, and energy conservation fields [3, 4]. It should be observed that pure silica aerogels are extremely fragile structures, which restrict the characteristics of their realworld potential applications. However, several researchers applied different strategies to * Corresponding Author Email:

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strengthen silica structures that have previously been developed, and several innovative ones are approaching [5, 6]. Some of them are the aging process of the wet-gel [7-10], the usage of silica precursors that assured additional flexibility of the silica network [11-13], or the embedding of polymers into silica networks [14]. On the other hand, there is a well-known research method in which fibers are in silica structures. Different types of biological and non-biological fibers such as glass fibers [14-15], mullite fibers [16], aramid fibers [17] and ceramic fibers [18] can improve mechanical strength. Another severe concern, that restricts the aerogels' application, is the loose dusty silica particles contaminate as surface modification process with the hazardous organic solvent used

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while solvent replacement and surface alteration process in the APD method. An alternative method for surface modification is the co-precursor method. However, it has limitations due to the free silica particles of the fiber-reinforced aerogel blankets which affected the contamination of the other hardware parts [19].

Previous studies discovered the prospect of using different fibers based on silica aerogel overcomes the fragile nature of silica aerogel. The resultant nanocomposite can be a real-world application for thermal insulation purposes with good mechanical strength. However, extreme protection with adequate thermal insulation and loose silica particles remained a challenging concern that requires more attention. The key cause of the external surface of the fiber cannot be covered by silica particles in the aerogel blanket owing to the immense variance of its diameters [20]. The change in dimension set up the void places and networks linking fibers ensuing in a way for the passage of heat. Therefore, the resultant thermal conduction of nanocomposite is amplified. Furthermore, the dispersion of granules of silica in the nanocomposite is widespread due to the lack of chemical linkage among the fibers and silica aerogel [21]. Polyurethane (PU) based silica aerogel integrating an organic protecting cover on cotton fiber [22] had certain limitations such as high wideness and toughness, besides less permeability of air and humidity due to PU binder. In the Silica aerogel blanket, silica particles are incorporated with thermally attached nonwoven fibers [23] verified chemically and thermally protected with sufficient air and humidity absorptivity. However, the highly wide and bulky caused by the numerous dusty silica granules are uncomfortable to wear and reduce the cloth flexibility.

One of the water-soluble biopolymers is polyvinyl alcohol (PVA) [24]. PVA is a stable element, highly incombustible and economical [25], but restricted due to its high density and thermal stability [26]. In this impact, we aim to encompass our research by developing a novel strategy of dust-free and flexible silica aerogel blanket via integrating fibrous mat before gelation, with the aging process carried out in PVA solution. Thus, the purpose of this work was to develop a dust-free thermally stable silica aerogel blanket with better tensile strength. In the present work, we developed a commercial technique to prepare a PVA-based recron-based silica aerogel blanket with mechanical robustness, low density and significant thermal insulation. A 3D silica network structure of silica aerogel was formed with a small number of fibers as reinforcement before gelation. After gelation, the aging process is performed in the PVA solution to strengthen the silica structure. The solvent exchange process carried out in ethanol solution can improve the inner structure of the hydrogel. The one-step surface alteration was obtained through ethanol: n-hexane: TMCS (40 : 40 : 20) solution to avoid the lengthy process of surface alternation. The ambient pressure drying process is accomplished at different temperatures for different time slots. Moreover, owing to the different types of fibers reinforced in hydrosol and aging in PVA solution. The resultant silica aerogel blanket revealed the outstanding performance of mechanical strength and optimized thermal conductivity. The encapsulation of the PVA by the porous silica particles was affected by the interior and exterior factors. The resultant dustfree silica blanket could show better performance in spacecraft applications without contamination of other parts.

EXPERIMENTAL SECTION

Materials

The fibers for insulation blankets were purchased from commercially obtainable recron fiber and cotton. The variation in the width of the fiber mat used for support of silica sol. Glass fibers used as reinforcement was purchased from Sigma-Aldrich. The recron fibers are forced to form the proper size mat to be suitable for the mold. Industrial grade sodium silicate (Na_SiO_) and sulphuric acid (25%, H_SO_) were obtained from Romest Silichem Pvt. Ltd. India. Trimethylchlorosilane (>95%, TMCS) was provided by Sigma-Aldrich. Ethanol, and cyclo-hexane (>99%) were supplied from Duksan Chemical. The prepared 10% of 25% H₂SO₄ (aq.) and 15% Na₂SiO₃ (aq.) were utilized as acid and base catalysts respectively in the sol-gel process. All reagents were utilized as received without additional refinement. Distilled water (DW) was used throughout the research. The fibers were washed away using ethanol and then desiccated at 50°C.

Experimental Methods

Three types of fibers used in silica aerogel blankets were explored: inorganic glass fiber,

recron fiber, and cotton fibers based silica aerogel blankets. The silica aerogel blankets improvement was obtained with the sol-gel process. Initially, PVA solution was made by melting PVA powder in the water at ~90°C for around 2-3h in concentrations of 200 mg/mL. The homogeneous PVA solution was prepared under constant stirring (~300 rpm). Then, the solution was refrigerated to ambient temperature. In the first step, 20 ml diluted sodium silicate and diluted sulphuric acid are added drop-wise to an acid catalyst to support the condensation process for the formation of sol. The pH of the hydrosol was balanced up to 5 for carrying out the hydrolysis process by adding diluted H₂SO, acid under constant stirring (300 rpm) at atmospheric temperature. At the same time, silica hydrosol poured on the fibers mat of 3 wt.% of hydrosol (Recron fiber, Glass fiber, and Cotton fiber) was placed down on a squareshaped plastic container. Gelation takes place within 20 min. The aging process occurs in PVA

solution for 24 hrs. Subsequently aging occurs in ethanol for 48 hrs. The leftover pore solvent is extracted via a one-step solvent exchange method with ethanol: n-hexane: TMCS (40:40:20) for 72 hrs. The samples were washed out with fresh cyclo-hexane polar solvent. Finally, ambient pressure drying of the aerogel takes place at different temperatures, 50 °C for 40 hrs, 80 °C for 2 hrs, and 120 °C for 3 hrs. to obtain monolithic nature. The prepared samples are recron reinforced silica aerogel blanket (RSAB), cotton reinforced silica aerogel blanket (CSAB) and glass fiber reinforced silica aerogel blanket (GSAB). The samples of the blanket were 200 mm × 200 mm with 20 mm thickness for RSAB, 10 mm thickness for GSAB and 7 mm for CSAB. Fig. 1 shows the synthesis process and Fig. 2 demonstrates the preparation stages of PVA-based dust-free silica aerogel blankets. Fig. 3 reveals the schematic representation of the PVA-based silica aerogel blanket structure.



Fig. 1. Flowchart of synthesis process for PVA-based dust-free silica aerogel blanket.



Fig. 2. Schematic illustration of preparation stages for PVA-based dust-free silica aerogel blanket.



PVA based Silica Aerogel Blanket

Fig. 3. Schematic representation of PVA-based silica aerogel blanket structure.

Methods of characterization

The physical parameters of the PVA-based various fibers reinforced silica aerogel blankets were explored. The structure of the dustless silica aerogel blanket of the different fibers was experimental with a field emission scanning electron microscope (FESEM, Quanta 200 ESEM). The bulk density $(\rho_{\rm b})$ was calculated with the proportion of mass to the volume. The volume of the aerogel was obtained using the drainage process whereas the mass was calculated with an analytical microbalance. The specific surface area was determined using N2 adsorptiondesorption isotherms (BET, Model Micromeritics ASAP 2000) measured at 77 K of the degassed silica aerogel blanket samples (150 °C for 3 hrs.). Pore size distribution (P_d) and Pore volume (P_d) were investigated using the BJH method [27] (P consists of the measurement of open pores in the

range of 1.7 to 300 nm). The chemical functional group of silica aerogel blankets was verified using a Fourier transform infrared spectroscopy (FTIR, Model number 760) within 4000 and 400 cm⁻¹. The thermal conductivity of silica aerogel blankets was determined by a thermal constants analyzer (TCA, Hot-Disk 2500) at 28 °C. The thermal stability study was carried out by TGA (STA 2500 Regulus) through a heating rate of 10 °C/min from ambient temperature to 800 °C in air. The young modulus test was performed on a universal testing machine (UTM) at a speed of 1 mm/min to 30% strain and these testing plates were detained for 10 seconds before the subsequent compression cycle. Young's modulus (E) values were evaluated to define the elastic nature of the aerogels. The computations as per the formula were accomplished subsequently:

 $E = \frac{Stress}{Strain} = slope \ of stress \ against \ strain \ curve$



Fig. 4. Microstructure of PVA-based silica aerogel blankets.

RESULTS AND DISCUSSION

The morphology of pure silica aerogel and PVA-based reinforcement of different fibers silica aerogel blankets such as RSAB, GSAB, and CSAB as displayed in Figs. 4a, b, c and d. In Figs. 4b, c, and d, the exterior of the blankets is fluffy and tighter which can be ascribed to the nano-sized silica particles that are well bonded to the fibers with better interfacial adhesion due to the PVA particles. It also displays the structure of different fibers with small amounts of PVA. When the fibers were 3 wt%, the hydrosol has been exhibited large support structures. The prepared silica blankets appearance was dust-free due to the molecular series entanglement being sufficient in the PVA solutions with low concentration. The agglomeration of a large amount of silica aerogel particles indicates part of the plasticizer plays a role in increasing strength. In other words, the growth of agglomeration decreases the surface area of the silica aerogel particles. The density increases caused by the accumulation of silica particles thoroughly together as shown in Fig. 4. Therefore, the prepared PVA-based silica blankets

were subjected to better mechanical strength.

The isotherms of nitrogen adsorptiondesorption of the PVA-based silica blanket are described in Fig. 5a. All arcs did not upsurge abruptly in the low relative pressure region (P/ P_{o} < 0.5), indicating that there was an absence of microspores in the samples. The rising slope of the curve in the range of 210~235 A^o mesoporous region of the PSDs (2-50nm) [28]. Fig. 5a, the isotherms of the adsorption-desorption curve of the silica aerogel blankets are of type IV and are appropriate to the mesoporous region with a hysteresis loop at $P/P_0 > 0.6$. Isotherms growth is seen in high relative pressure regions $(P/P_0 =$ 0.95-1.0) due to the liquid condensation denoting the existence of macrospores [29]. Also, the pore size distributions (PSDs) measured using a Barrett-Joyner-Halenda (BJH) technique direct that maximum numbers of the pore diameter (P_{d}) dispersed near 170 nm for the PVA-based silica blankets as shown in Fig. 5b. The blankets were not enclosed with microspores due to the particles of PVA strengthened by the tightening of the silica particles and fibers, which affected the collapse of



Fig. 5. a) N, adsorption-desorption isotherms, and b) pore size distributions of PVA-based silica aerogel blankets of various fibers.



Fig. 6. Influence on surface area and density of PVA on silica aerogel blanket.

the interior pores in the silica aerogel blanket. This method may offer a different methodology for improving the mechanical robustness of aerogel blankets. As a consequence, it is promising to develop mechanical robustness without trailing a few of the most valued features of pure silica aerogel, such as pore diameter, specific surface area, bulk density, and low thermal conductivity. Fig. 6 represents the effect of density is inversely proportional to the surface area.

The chemical bonding of RSAB, GSAB, and CSAB was analyzed using FTIR. Fig. 7 shows the FTIR spectra of the four silica aerogel blankets. The strong adsorption peaks observed near 1100 cm⁻¹ and 800 cm⁻¹, which corresponded to Si–O–Si groups asymmetric, symmetric vibrations attributed [30]. The peaks existed near 3450 cm⁻¹ and 1640 cm⁻¹ related to stretching vibrations owing to the residual Si-OH groups [31]. The

appearance of small bending about 2973 cm⁻¹ and 2911 cm⁻¹ can be ascribed as the stretching vibration of -CH, groups [32, 33]. The peaks at 1274 cm⁻¹ and 918 cm⁻¹ indicate the existence of Si-CH, clusters which approached due to the TMCS surface modification, which produced the hydrophobic surface. The bending around 960 cm⁻¹ confirmed the broadening vibrations of the Si-OH association produced from the nontransformed silicate Si-OH. The FTIR range of PVAbased blankets indicates distinctive peaks around 3332 cm⁻¹ (O-H group), 1645 cm⁻¹ (C=O group), and 1029 cm⁻¹ (C–O–C group) [34]. The broadening bending vibration band of the -OH group at 3332 cm⁻¹ represents the development of hydrogen bonds among the two components. Broad and prominent hydroxyl (-OH) groups are significant in the hydrophilicity of the PVA [35]. The fibers and silica particles are encapsulated in PVA coating





Fig. 7. FT-IR spectrum of the PVA-based silica aerogel blankets of various fibers.



Fig. 8. TGA curvatures of the pure silica aerogel and PVA-based silica aerogel blankets of various fibers.

took place. In FTIR spectra in Fig. 7, each chemical functional group was steady during the production of the PVA-based silica aerogel blanket structure.

TGA is commonly used to calculate the thermal stability at higher temperatures. The thermal permanence of the PVA-based aerogel blankets was investigated in the blowing air is discovered by the TGA analysis as comprehended in Fig. 8. Table 1 outlines the weight loss related to each degradation stage and the absolute residual weight. %. The residue rate of fiber-based silica aerogel blankets is higher (~25%) as compared to pure silica aerogel and other PVA-based silica aerogel blankets. The overall weight loss is observed in all blankets in the range of temperature of 100-800 °C. The weight degradation method can be categorized into three phases. The first phase, which happens in the temperature range of 100 °C and 220 °C,

includes the removal of water cohesion on the silica aerogel's blanket superficial. In this phase, the weight degradation is around 0.5%, which signifies hydrophobic performance subsequently the APD method. The rate of weight loss is negligible in the temperature range of 220 °C and 370 °C in the second weight-degradation phase demonstrating the lesser quantity of -OC₂H₂/-OCH, clusters are residual in the APD method. In the last phase, the resultant to the temperature beyond 440 °C is affected by the oxidation of -CH₂ bonds on aerogel and additional residual bonds in the aerogel network [36]. As an increase in temperature affects minor weight loss that impact on the aerogel blankets becomes hydrophilic [37]. The elevated temperature of degradation and lesser weight loss show the improvement in the thermal permanence of the blankets owing to

Sample	Weight loss at 380ºC	I st degradation		II nd degradation		Decidual wt% at
		Temperature Range	Wt%	Temperature Range Wt%		= Residual wt% at 800 ºC
Pure Silica Aerogel	1	-	-	500-800 ºC	1.5	2
RSAB	5	380-485 ºC	13	500-800 ºC	6	4
CSAB	3	380-480 ºC	10	500-800 ºC	5	3
GSAB	4	380-440 ºC	8	440-800 ºC	4	5

Table 1. Thermogravimetry analysis of PVA-based silica aerogel blankets.



Fig. 9. The thermal conductivity of the pure silica aerogel and PVA-based silica aerogel blankets of various fibers.

the PVA encapsulation of the silica particles and fibers. It can be assumed that the carbonization of the surface of the silica blanket by the degradation of the PVA coating at a low-temperature range enhanced the thermal stability. The carbon layer placed on the surface of the blanket acts as a better performance of the thermal insulator and reduces the degradation rate at a higher temperature. These results propose that the owing to the PVA coating of silica granules and fibers can improve the thermal permanence of blankets.

The influences of the PVA and the fibers on the thermal conductivity of the silica aerogel blankets are revealed in Fig. 9. Fig. 9 shows that the thermal conductivity of the silica aerogel blankets was reduced initially and improved later growing for different fibers. Associated with the silica aerogel blanket with different fibers, pure silica aerogel and RSAB exhibited lower thermal conductivity. The addition of fibers enhances the thermal conductivity of the blanket in contrast to pure silica aerogel. This caused macro-pores of silica aerogel to merge with fibers which resulted in the size of pores of silica being reduced. Reduction in space of the enormous pores produced due to the growth of silica particles which causes weak heat transfer within the gas. The reaction was carried out among the silanol clusters of silica precursor and the hydroxyl (-OH) cluster of PVA through hydrogen or covalent bonds [38]. Due to the formation of covalent bonds, there is a reduction in heat transfer and restricts the decay of the silica blanket [39]. The microstructure variations of the silica aerogel blanket were deliberated in an earlier part. The purpose behind is that the thermal conductivity was generally affected due to the PVA and the fibers. Thus, the thermal conductivity of the PVA-based fiber-based silica aerogel blankets increased.

Fig. 10 is the strength of strain-stress curvatures of the blankets obtained by a Universal Testing Machine (UTM). The stress was improved with the accumulative strain for different fibers. The strain was observed from 20 to 35%, and the curvature linearity up to 20%. The stress at the strains of 20% and 35%, respectively, and the variation in Young's modulus of the blankets were shown in Fig. 10. The existence of the silica particles plays a vital part in



Fig. 10. Stress-strain curves of the pure silica aerogel and PVA-based silica aerogel blankets of various fibers.

Table 2. Physical, thermal, and mechanical parameters of the as-prepared PVA-based silica aerogel blankets with different fibers.

Sample	Pore diameter (Aº)	BET Surface area (m²/g)	Density (g/cm ³)	Thermal conductivity (W/(m·K))	Young's modulus (E)(MPa)
Pure Silica Aerogel	190	1604.23	0.052	0.023	20
RSAB	165	605.54	0.098	0.028	35
CSAB	160	585.50	0.102	0.032	33
GSAB	152	575.25	0.118	0.035	37

performing the stiffness of the blanket. The strain is stable at 20%, and the extreme stress is at about 15 MPa before the cycle, demonstrating that the aerogel blanket shows very low Young's modulus and great elasticity. The extreme stress is a little bit reduced to 14 MPa later in the compression cycle investigation. This result indicates that a prepared dust-free flexible silica aerogel blanket can be an ultimate material cast off as a super-thermal insulator in various applications such as spacecraft, catalyst barriers, and intellectual sensors. The Young's modulus (E) values of different blankets were intended from the rectilinear section of the stress-strain curves (Fig. 10).

SPACECRAFT THERMAL INSULATOR APPLICATIONS

After the earlier research works, it was detected that due to the loose silica particle detaching nature of fiber-reinforced silica aerogel blanket, not safe while implemented in spacecraft applications. The contamination of flight hardware due to dusty silica particles could be avoided with the encapsulation of a PVA-based fiber-embedded silica aerogel blanket which overcomes the limitation in the spacecraft field. Rocha et al.[40] have recommended the outline with aluminized Kapton sheets of silica aerogel blanket on the upper and bottommost surfaces with wrapped boundaries, to restrict contamination and harm to the other spacecraft modules due to the dusty silica particles. Despite wrapping edges with Kapton tape, due to the small applied pressure while launching also damaged the sealed tape. To resolve such an issue, this approach of dust-free silica aerogel blanket is appropriate for spacecraft application without contamination of other parts of the spacecraft. The Physical and thermomechanical properties of dust-free silica aerogel blankets are summarized in Table 2.

CONCLUSION

This research work demonstrates the dust-free lightweight silica aerogel blanket with preserved pores of the silica network with low thermal conductivity and better Young Modulus of the blankets. The proposed method was obtained by aging of fiber-reinforced silica gel in PVA liquid followed by ambient pressure drying. It was shown that a dust-free silica aerogel blanket could enhance tensile properties without significantly altering density, textural properties, and thermal insulation. Investigates demonstrated that a PVA-based silica aerogel blanket with 3 wt.% fibers showed the highest thermal insulation and better flexibility with well-preserved pores. The pores in the silica network are well conserved, and the surface area of the silica particles in the composites plays a vital physical factor responsible for the thermal conductivity and the mechanical strength of the silica composite. The proposed dust-free, lightweight, better thermal insulator and enhanced Young Modulus of PVA-based fiberreinforced blanket have potential applications in fields in which weight factors are significantly considered such as the development of apparel in spacecraft, military jackets, fire-fighting, and hilly areas people which is essential dustfree, lightweight, and highly thermal insulator properties.

CONFLICTS OF INTEREST

The authors declared do not have any conflicts with the publication.

ACKNOWLEDGMENTS

The authors greatly acknowledge the Deanship of Scientific Research (DSR), the University of Jeddah under the research project grant (UJ-02-020-ICGR), and the DST-SERB project grant (EEQ/2020/0002980) for their technical and financial support. The authors also gratefully acknowledge the Rajiv Gandhi Science and Technology University of Mumbai under a major research project grant from University of Mumbai.

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