Preparation and Characterization of Silica Aerogel Incorporated Polypropylene Nonwoven Fabric Composite Dried in Ambient Pressure Drying Method

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ABSTRACT

In recent times, sustainable ecofriendly thermo-insulation materials which is flexible and mechanically robust have grabbed worldwide remark. Nonwoven fabric and aerogel have complementary characteristics needed for desirable thermal insulation.

In this research, silica aerogel/polypropylene (PP) nonwoven fabric composite with desirable properties was synthesized via a two-step sol-gel process through immersing the PP nonwoven fabric into silica sol. After in situ gelation, Silica aerogel-pp Nonwoven Fabric composite gel was hydrophobized with hexamethyldisilazane, and the composites were dried at ambient pressure method. Silica aerogels were distributed inside the pp nonwoven fabric as a composite to act as a supporting skeleton which increased the mechanical property of the silica aerogels. Scanning electron microscopy (SEM), DSC, TGA were used for the characterization of the composites. The contact angle and heat conducting performance of the composites were also determined.

The results show that silica aerogel particles were efficiently covered the surface of the PP non-woven fabric and completely filled the micron size pores of the nonwoven fabric leading to a stronger hydrophobicity and higher thermal insulation performance in the aerogel composite. The findings in this study are significant and can be used for further research in aerogel-treated nonwoven fabrics.

Keywords: SILICA AEROGEL, POLYPROPYLENE NON-WOVEN FABRIC, COMPOSITES, SOL-GEL PROCESS, AMBIENT PRESSURE DRYING, HYDROPHOBIC, THERMAL INSULATION.

Introduction

The term aerogel was first coined less than a century ago by Kistler. Aerogels are defined as gels in which the liquid is replaced by a gas, and the solid gel network remains intact [1,2]. It was regarded widely as the 'miracle material for the 21^{st} century [3] because of its highly nano-porous (>99%) structure, having a network of connected particles like a pearl necklace, with an air volume of 80–99.8% [4]. This unique layout imparts to aerogels the status of the lightest solid materials with first-rate insulation properties [5–9]. Other important attributes of this new-generation material are its high specific surface area (100–1600 m² g⁻¹) and a very low refractive index (1.007–1.240) [4,10], thermal conductivity and possess excellent adsorbing and catalytic properties [11,12].

In recent times, silica aerogels have been used in various advanced applications because of these distinguished characteristics. For instance, adsorbents of harmful compounds [13,14] sensors [15] dielectric materials [16] filtering media [17] Cherenkov detectors [18] kinetic energy absorbers, [19] substrates for catalysts, [20] carriers, [21] extracting agents, [22,23] protective clothing, [24] and even art sculptures [25] are among the reported end-uses of silica aerogels. Undoubtedly, thermal and acoustic insulation are the most significant applications of silica aerogels, [26,27]. However, this material is extremely fragile due to its high porosity and cracking, which creates some difficulties for its widespread conventional applications [28]. Hence, the integration of aerogels with a porous textile structure, including woven and nonwoven, can be considered a facile and fruitful approach through filling the interstitial space among fibers within the structure. Early research on aerogels in the field of textiles was based on the development of spacesuit insulation, funded by NASA [29]. Recent application examples included silica aerogel coating on wool–aramid blended fabric for thermophysiological wear comfort [30] aerogel-encapsulated nonwoven textiles for thermal insulation [28], and polyester/polyethylene nonwoven blankets with aerogel for extreme cold weather [31].

Nonwoven fabrics are generally sheet, web, or batts of entangled fibres, orientated directionally or randomly, bonded together by different methods including mechanical, thermal, and chemical [32]. Unlike other textile structures, for example knitted or woven, the construction of nonwoven fabric consists of individual fibres or layers of fibrous webs rather than yarns [33]. The unique structural features of nonwoven fabrics lead some exceptional functional characteristics, such as high resilience, great compressional resistance, as well as excellent thermal insulating properties [34]. Moreover, the nonwoven structure is highly porous, low weight, and permeable, which can be utilized to develop lightweight clothing with adequate breathability for clothing comfort. For this reason, nonwoven fabrics are extensively used in various technical applications, including protective clothing materials.

Silica aerogels are synthesized through sol-gel chemistry, defined by IUPAC [35] as the "Process through

which formed from solution network is by a progressive change of liquid a precursor(s) into a sol, to a gel, and in most cases finally to a dry network" The sol-gel process is the most commonly employed method for the preparation of silica-based organic-inorganic hybrid materials at the microscale as well as at the molecular level. The advantage of the sol-gel process is that the reaction proceeds at ambient temperature to form hybrid materials, as compared to the traditional methods performed at high temperatures. In this method, a suitable coupling agent is employed to obtain a strongly interconnected network, preventing a macroscopic phase separation. However, aerogels are usually obtained above the supercritical pressure and temperature of the pore liquid. Since processing at such high pressures requires the use of expensive autoclaves, their high production costs restricts their industrial application. Thus, nanoporous silica aerogels prepared under ambient pressure have recently become an interesting subject. However, cracking of silica aerogels is still a major obstacle in the applications due to their poor mechanical properties. Incorporation aerogels in to a fiber matrix such as nonwoven fabrics may support brittle aerogel network and can certainly increase their mechanical properties. Therefore, by following this method the weakness of fragility in the aerogel for industrial applications could be overcome. Hydrophobic silica aerogels dried at ambient pressure method avoid the reduction of adsorption properties of hydrophilic adsorptive material in humid atmospheres, and create the possibility of preparing useful adsorptive composites.

The objective of this study was to prepare hydrophobic silica aerogel-PP nonwoven composites via a sol-gel process by prior dipping and the use of polyethoxydisiloxanes (E-40) as the silicon source. Surface modification is used to realize ambient pressure drying method. The properties of the silica aerogel/polypropylene (PP) nonwoven fabric composite were characterized.

Materials and methods

Materials

Silica aerogels were prepared via the sol-gel process using E-40 as the silica precursor Polyethoxydisiloxane (E-40), ethanol, HF, trimethylchlorosilane (TMCS) hexamethyldisiloxane were purchased from the Maclin Co. Shanghai. All chemicals were used as received. Polypropylene non-woven fabric used as received from Hangzhou Textile Clothing and Manufacturers Pvt. Ltd. Hangzhou, P.R. China. Polypropylene non-woven fabric thickness 0.27 mm and the samples were cut into the size of (30 mm×30 mm). Distillated water was used in experiment.

Methods

'E-40', a commercially available polyethoxydisiloxane used as silica precursor in this experiment which was prepared by the catalytic reaction of ethanol and silicon. Silica sols were prepared via a sol-gel process with an E-40-water-ethanol system and acid- catalyzed by HF. E-40, ethanol, HF and distilled water were mixed

together at room temperature according to the volume proportion 1:10:0.01:0.45, and then were stirred in a magnetic stirring at room temperature for 2 hours. A silane coupling agent was added as an agglomerating agent. Silica sol was obtained 2 hours later. Polypropylene nonwoven fabric was then immersed in the silica sol and stirred for 1 hour; silica aerogel-pp nonwoven fabric composite gels were then formed.

In ambient pressure drying, the composite gels were maintained at room temperature, aging for 48 hours, then dipped into ethanol and then the sample was aged at 70°C for 6 hours. After the composite gels were aged, they were dipped into 10% (volume ratio) trimethylchlorosilane (TMCS) hexamethyldisiloxane solution. After 48 hours, these surface modified composite gels were dried at ambient pressure at 130°C for 6 hours lab oven (Memmert ULE 400), resulting in the hydrophobic silica aerogel-pp non-woven fabric composites.



Figure 1. Ambient Pressure Drying Method

Characterization

The morphology and pore structure of the untreated pp nonwoven fabric and the silica aerogel-polypropylene non-woven fabric composites were characterized by Zeiss ultra-scanning electron microscopy.

A sessile drop method was performed on a Kruss GmbH DSA 10 Mk2 goniometer to measure a contact angle (θ) between the silica aerogel-polypropylene composites and of a 3–5-µL distilled water drop, which was applied to the surface by means of syringes.

Differential scanning calorimeter (DSC) was used to determine the total heat absorption capacity. The tests were conducted under nitrogen purge (20 mL/ min) at a constant heating-cooling rate of 5 °C per minute.

Finally, the mean value of five readings was reported.

Thermogravimetric analysis (TGA) was performed using Mettler Toledo TGA/SDTA851. The temperature was varied from 25 to 600 C with a ramp of 10 C min⁻¹ under nitrogen flow at 50 mL min⁻¹.

Heat conducting performance was measured by using YG606D Flat plate heat retention tester. By determining the variation in the gradient values and in combination with the heating power of baseplate, heat-conducting performance of the fabric was evaluated. The fabric was placed at 25°C and 60% RH for 48 h to adjust the moisture balance. The heating baseplate temperature was set as 36C, and the instrument was preheated for 30 min. The number of test cycle was 5. Under alternating powers, the authors first carried out the empty plate test and recorded the data such as temperature difference and heating power using a computer. Then, the instrument was recovered to the status before the test, successively placing the sample of PP Nonwoven fabric incorporated Silica Aerogel Composite by Ambient Pressure Drying Method to the instrument, and repeated the test thrice under alternating power. The data were recorded for calculating the samples heat transfer coefficient, heat preservation rate, clo value.



Figure 2. YG606D Flat plate heat retention tester

Results and Discussion

Morphological Characterization

To characterize the morphological features of the untreated pp non-woven fabric and the silica aerogelpolypropylene non-woven fabric composite, SEM images were obtained, Figure 3 shows SEM photos of the untreated PP non-woven fabric and the silica aerogel-fabric composite . Figure 3(a) corresponds to the untreated pure Polypropylene non-woven fabric, Figures 3(b, c, d, e) represents the SEM photos of the silica aerogel-pp nonwoven composites by ambient pressure drying at four different magnifications.

From the SEM photo of 3(a) shows smooth surface of untreated pp nonwoven fabric. On the other hand, from the SEM photos of the silica aerogel-pp nonwoven fabric composites by ambient pressure drying method, it is obvious that the uniform deposition of silica aerogels covers much of the surface of the pp nonwoven fabric. North American Academic Research, 4(3) | March 2021 | https://doi.org/10.5281/zenodo.4601626 Monthly Journal by TWASP, USA | 61 It maintains a spongy porous structure and high porosity in the aerogel-pp non-woven composites compared to the untreated pp non-woven fabric, which is beneficial to improve the thermal insulation property. The PP nonwoven fabric acted as a supporting skeleton that could increase the mechanical properties of the silica aerogels.



Figure 3. (a) Untreated PP Nonwoven fabric sample (300); 3.(b,c,d,e) Silica aerogel-pp non-woven fabric composites by ambient pressure drying (300, 500, 1000, 10000) magnification;

(b)

Thermal Stability

The thermal insulation property can be closely connected to the thermal stability. The thermal stability/degradation behavior of the Silica Aerogel incorporated PP Nonwoven fabric composite by Ambient Pressure Drying method was analyzed can be seen in TGA and DTG curves of Figure 4. For relative comparison, the characteristic temperatures of 10% weight loss (T_{10}) and peak degradation (T_{peak}) as well as the residue at 600°C were evaluated, as summarized in the table 1.

Results shows that for ambient dried sample initial degradation begins approximately 400°C, T_{peak} value is 445°C and its residue at 650 °C was 23.2 wt%. So from results, we can say that the tested sample shows excellent thermal stability.



Figure 4. TGA and DTG curves of the Silica Aerogel incorporated PP Nonwoven fabric composite by Ambient Pressure Drying Method.

Sample	T_{10} °C	T _{peak} °C	Residue at 650 °C (wt%)
Ambient dried	397	445	23.2

Table 1. Characteristic thermal degradation temperatures (T₁₀ and T_{peak}) and residue at 600 °C of Silica Aerogel incorporated PP Nonwoven fabric composite by Ambient Pressure Drying Method.

DSC analysis

The differential scanning calorimetry (DSC) technique is the most often used for the thermal performance of textiles. The DSC curves of the PP Nonwoven fabric incorporated Silica Aerogel Composite by Ambient Pressure Drying Method are shown in the Figures 5. As shown in these figures, the DSC curve of the ambient dried sample shows that the main melting transition at 249 °C with a heat of melting of 477.98 J/g.



Figure 5. DSC curve of the Silica Aerogel incorporated PP Nonwoven fabric composite by Ambient Pressure Drying Method.

Heat conducting performance

The data was recorded for calculating the sample heat transfer coefficient, heat preservation rate, clo value, as listed in Table 2. The test results show that the heat transfer coefficient of the PP nonwoven fabric by ambient pressure drying sample was $50.04 \text{ W/m}^2 \text{ C}$, Clue value was 0.12 and heat preservation rate was 30.3%. Results shows that Silica Aerogel incorporated PP Nonwoven fabric composite has excellent heat conducting performance.

Heat Transfer Coefficient	Heat preservation rate(%)	Clo value (0.155C
(W/m ² C)		m ² /W)
50.04	30.3	0.12

 Table 2. Heat-conducting performance of the tested Silica Aerogel incorporated PP Nonwoven fabric

 Composite by Ambient Pressure Drying Method.

Surface property

The contact angle of untreated PP nonwoven fabric and its silica aerogel/pp nonwoven fabric composite with water were presented in Figure 6. The contact angle of the untreated PP Nonwoven fabric was 114°, PP Nonwoven fabric incorporated Silica Aerogel composites with water was 129° for Ambient pressure dried sample which indicated aerogel-pp nonwoven composites have excellent hydrophobic properties. The excellent hydrophobic properties are due to the existence of Si-(CH₃)₃ on the surface, The -CH₃ groups replaced the -OH groups on the surface of aerogels by surface modification. Silylation was the main reaction in the trimethylchlorosilane (TMCS) hexamethyldisiloxane solution modification process. In principle two different reaction can occur:

$$(CH_3)_3SiCl + H_2O \iff (CH_3)_3Si-O-Si(CH_3)_3 + 2HCl$$
(1)

$$HO-Si \equiv + (CH_3)_3 SiCl \iff \equiv Si-O-Si(CH_3)_3 + HCl$$
(2)

TMCS reacts with the pore water to yield hexamethyldisiloxane and HCl in a spontaneous, exothermic reaction 1. Hexamethyldisiloxane can again react with HCl to form TMCS and water. It is a reversible reaction. In the two possible reactions TMCS reacts with the hydroxyl groups on the surface of the silica gel. To avoid the first reaction, the water content is controlled in the preparation process, and hexamethyldisiloxane is the solvent, so the equilibrium of the first reaction is clearly on the TMCS side. It is favorable for the second reaction of TMCS with the hydroxyl groups on the surface of the silica gel to obtain $-Si(CH_3)_3$. It is also useful to dry the silica aerogels at ambient pressure to increase the modification due to the TMCS. The resulting silica aerogel fabric composites have excellent hydrophobicity.





Figure 6. (a) Contact angle of the untreated pure PP Nonwoven fabric(b) Contact angle of the Silica Aerogel incorporated PP Nonwoven fabric Composite by Ambient Pressure Drying.

Conclusions

In a summery, Silica aerogel-pp nonwoven fabric composite was prepared efficiently via sol-gel process and surface modification by ambient pressure drying method. The composites have a typical nano-porous microstructure with hydrophobic properties. Aerogel dispersed uniformly and maintain high porosity in the composites because of the existence of Si-(CH₃)₃ on the surface. The silica aerogel-pp nonwoven fabric composite exhibited the noticeable enhancement of hydrophobic and thermal protection properties compared to the untreated pp nonwoven fabric. The overall performance demonstrated that the Silica aerogel-pp nonwoven fabric composite can be utilized as cold weather protective garments, heat-sensitive devices, pipes, automotive, aircrafts, and buildings for thermal insulation applications.

References

- [1]. R. A. Venkateswara, R. A. Parvathy and M. M. Kulkarni, J. Non-Cryst. Solids, 2004, 350, 224–229.
- [2]. S. W. Hwang, H. H. Jung, S. H. Hyun and Y. S. Ahn, J. Sol-Gel Sci. Technol., 2007, 41, 139–146.
- [3]. A. Taher, The Sunday Times, 2007.
- [4]. N. Husing and U. Schubert, Angew. Chem., Int. Ed., 1998, 37,22-45.
- [5]. D. V. Fomitchev, R. Trifu and G. Gould, in Engineering Construction and Operations in Challenging Environments Earth and Space 2004: Proceedings of the Ninth Biennial ASCE Aerospace Division International Conference, ed. R. B. Malia and A. Maji, ASCE, Texas, 2004, pp. 968–975.
- [6]. A. C. Pierre and A. Rigacci, in Aerogels Handbook, ed. M. A. Aegerter, N. Leventis and M. M.

Koebel, Springer Science+Business Media, New York, 2011, pp. 21-45.

- [7]. Y. Ananthan, K. Sanghamitra and N. Hebalkar, in Nanotechnology for Energy Sustainability, eds. B.Raj, M.Van de Voorde and Y. Mahajan, Wiley-VHC, Weinheim, 2017, pp. 939–966.
- [8]. M. A. Hasan, R. Sangashetty, A. C. M. Esther, S. B. Patil, B. N. Sherikar and A. Dey, J. Inst. Eng. North American Academic Research, 4(3) | March 2021 | <u>https://doi.org/10.5281/zenodo.4601626</u> Monthly Journal by TWASP, USA | 66

(India): Ser. D, 2017,98, 297-304.

[9]. A. V. Rao, M. M. Kulkarni, G. M. Pajonk, D. P. Amalnerkar and T. Seth, J. Sol-Gel Sci. Technol., 2003, 27, 103–109.

- [10]. S. Henning and L. Svensson, Phys. Scr., 1981, 23, 697–702.
- [11]. Hrubesh, L.W. (1998) J. Non-Cryst Solids, 225: 335–342.
- [12]. Schmidt, M. and Schwertfeger, F. (1998) J. Non-Cryst Solids, 225: 364–368.
- [13]. S. Yun, H. Luo and Y. Gao, J. Mater. Chem. A, 2015, 3, 3390–3398.
- [14]. J. P. Vareda, A. J. M. Valente and L. Dur~aes, Adv. Colloid Interface Sci., 2016, 237, 1-15
- [15]. M. S. Jalali, S. Kumar, M. Madani and N. F. Tzeng, in 2013 IEEE Sensors Applications Symposium
- (SAS 2013) Proceedings, IEEE, Galveston, 2013, pp. 133–136.
- [16]. S. S. Prakash, C. J. Sankaran, A. J. Hurd and S. M. Rao, Nature, 1995, 374, 439-443.
- [17]. Z. Pakowski and K. Maciszewska, Inz. Chem. Procesowa, 2004, 25, 1435–1441.
- [18]. G. Poelz and R. Riethmuller, Nucl. Instrum. Methods, 1982, 195, 491–503.
- [19]. K. Ota and J. Inoue, in Extreme Ultraviolet (EUV) Lithography IV, ed. P. P. Naulleau, SPIE, San Jose, CA, 2013, vol. 8679, pp. 86792R-1–86792R-10.
- [20]. O. Orçaire, P. Buisson and A. C. Pierre, J. Mol. Catal. B:Enzym., 2006, 42, 106-113.

[21]. C. C. Li, Y. T. Chen, Y. T. Lin, S. F. Sie and Y. W. Chen-Yang, Colloids Surf., B, 2014, 115, 191– 196.

- [22]. P. C. Thapliyal and K. Singh, J. Mater., 2014, 2014, 10.
- [24]. M. F. Mora, S. M. Jones, J. Creamer and P. A. Willis, Electrophoresis, 2018, 39, 620–625.

[24]. Z. Qi, D. Huang, S. He, H. Yang, Y. Hu, L. Li and H. Zhang, J.Eng. Fibers Fabr., 2013, 8, 134–139.

[25] I. Michaeloudis, can we put the sky into a bottle? www.youtube.com/watch

www.youtube.com/watch?v1/4prWpMLB_2Xw.

[26] Method for production of flexible panels of hydrophobic aerogel reinforced with □bre felts, WO2015016730A2,2015, 1–7.

[27]. J. Feng, D. Le, S. T. Nguyen, V. Tan Chin Nien, D. Jewell and H. M. Duong, Colloids Surf., A, 2016, 506, 298–305.

[28]. Xiong X et al (2018) Thermal and compression characteristics of aerogel-encapsulated textiles. J Ind Text 47(8):1998–2013

[29]. Ho[•]ffele S, Russell SJ, Brook DB (2005) Light-weight nonwoven thermal protection fabrics containing nanostructured materials. Int Nonwovens J 14(4):10–16

[30]. Shaid A, Fergusson M, Wang L (2014) Thermophysiological comfort analysis of aerogel nanoparticle incorporated fabric for fire fighter's protective clothing. Chem Mater Eng 2(2):37–43

[31]. Venkataraman M et al (2015) Novel techniques to analyse thermal performance of aerogel-treated blankets under extreme temperatures. J Text Inst 106(7):736–747

[32]. Wilson A (2007) Development of the nonwovens industry. In: Russell SJ (ed) Handbook of

nonwovens. Woodhead, Cambridge, pp 1-15

[33]. Mao N, Russell S, Pourdeyhimi B (2007) Characterisation, testing and modelling of nonwoven fabrics.

In: Russell S (ed) Handbook of nonwovens. Woodhead, Cambridge, pp 401-514

[34]. Xiong X et al (2016) Transport properties of aerogel-based nano fibrous fabrics. Fibers polym 17(10):1709–1714

[35]. International Union of Pure and Applied Chemistry, IUPAC Compendium of Chemical Terminology (Gold Book), Version 2.3.3, Blackwell Publishing, Oxford, 2014.



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