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

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Abstract

In recent times, sustainable ecofriendly thermo-insulation materials which are 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 phase was hydrophobized with hexamethyldisilazane, and the composites were dried at ambient pressure method. 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 beused for further research in aerogel-treated nonwoven fabrics

Keywords: Silica aerogel • Polypropylene non-woven fabric • Composites • Ambient pressure drying • Hydrophobic

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 21st century [3] because of it's 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 $m^2 g^{-1}$) and a very low refractive index (1.007–1.240) [4-10], thermalconductivityand possessexcellent adsorbing and catalytic properties [8,9].

In recent times, silica aerogels have been used in various advanced applications because of these distinguished characteristics. For instance, adsorbents of harmful compounds [10,11] sensors [12] dielectric materials [13] filtering media [14] Cherenkov detectors [15] kinetic energy absorbers, [16] substrates for catalysts, [17] carriers, [18] extracting agents, [19,20] protective clothing, [21] and even art sculptures [22] are among the reported end-uses of silica aerogels. Undoubtedly, thermal and acoustic insulation are the most significant applications of silica aerogels, [23,24]. However, this material is extremely fragile due to its high porosity and cracking, which creates some difficulties for its widespread conventional applications [25]. 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 [26]. Recent application examples included silica aerogel coating on wool–aramid blended fabric for thermo physiological wear comfort [27] aerogel-encapsulated nonwoven textiles for thermal insulation [25], and polyester/polyethylene nonwoven blankets with aerogel for extreme cold weather [28].

Nonwoven fabrics are generally sheet, web, orbatts of entangled fibers, orientated directionally or randomly, bonded together by different methods including mechanical, thermal, and chemical [29]. Unlike other textile structures, for example knitted or woven, the construction of nonwoven fabric consists of individual fibers or layers of fibrous webs rather than yarns [30]. 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 [31]. 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 [31] as the "Process through which a network is formed from solution by a progressive change of liquid 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 micro scale 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, Nano-porous 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

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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. 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. All chemicals were used as received. Distilled water was used in experiment.

Sample preparation

'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 h. 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-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 the in ambient pressure drying the sample was aged at70°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 6 for hours lab oven (Memmert ULE 400), resulting in the hydrophobic silica aerogel-pp non-woven composites.

Characterization

The morphology and pore structure of 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 3-5μl 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.

Thermo gravimetric analysis (TGA) was performed using Mettler Toledo TGA/SDTA851. The temperature was varied from 25 to 600°C with aramp 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 1).

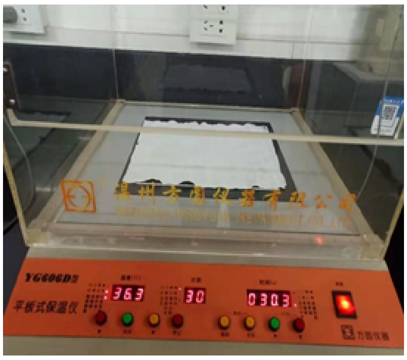

Figure 1. YG606D Flat plate heat retention tester.

Results and Discussion

Morphological characterization

To characterize the morphological features of the silica aerogel-polypropylene non-woven fabric, SEM images were obtained, Figure 1 shows SEM photos of the PP non-woven fabric and the silica aerogel-fiber composites. Figure 1(a) corresponds to the untreated pure Polypropylene non-woven fabric, Figures 1(b, c, d, e) show SEM photos of the silica aerogel-pp nonwoven composites by ambient pressure drying at four different magnifications. From the photos of the silica aerogel-pp nonwoven composites, it is obvious that the silica aerogels cover much of the surface of the pp nonwoven fabric and maintain 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. PP nonwoven fabric acted as a supporting skeleton that could increase the mechanical properties of the silica aerogels.

Figure 1 SEM photos of the aerogel-pp woven fabric composites with different magnifications: 1. (a) Untreated PP Non woven fabric sample (300);

1. (b, c, d, e) silica aerogel-pp. non-woven composites by ambient pressure drying (300,500,1000,10000) magnification;

Thermal stability

The thermal insulation property can be closely connected to the thermal stability. The thermal stability/degradation behavior of the PP Nonwoven fabric incorporated Silica Aerogel Composite by Ambient Pressure Drying Method was analyzed can be seen in TGA and DTG curves of Figure 2. 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 we can say both tested sample shows excellent thermal stability (Figure 2).

DSC analysis

The DSC curves of the the PP Nonwoven fabric incorporated Silica Aerogel Composite by Ambient Pressure Drying Method are shown in the Figures 3. 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. the DSC curve of the pad dry cure sample shows that the main melting transition at 259°C with with a heat of melting of 735.89 J/g (Figure 3).

Heat conducting performance

The data were recorded for calculating the samples 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 W/m^2C, Clue value was 0.12 and heat preservation rate was 30.3%. Results shows that PP Nonwoven fabric

Table 1. Characteristic thermal degradation temperatures (T_{10} and T_{peak}) and residue at 600°C of PP Nonwoven fabric incorporated Silica Aerogel Composite by Ambient Pressure Drying and Pad Dry Cure Method.

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

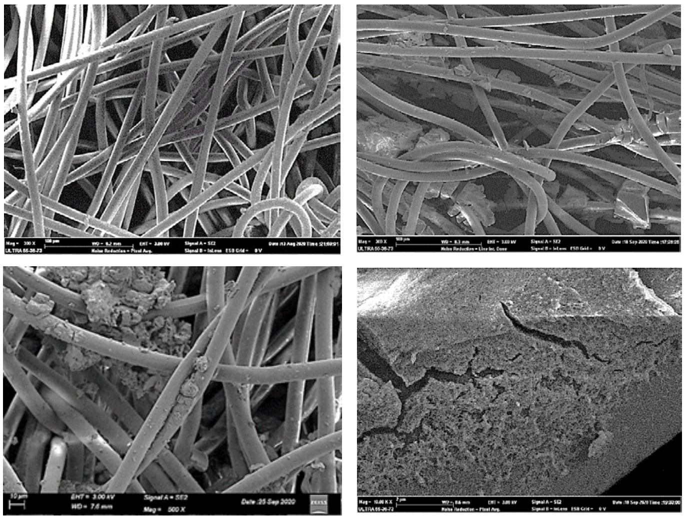

Figure 2. TGA and DTG curves of PP Nonwoven fabric incorporated Silica Aerogel Composite by Ambient Pressure Drying and Pad Dry Cure Method.

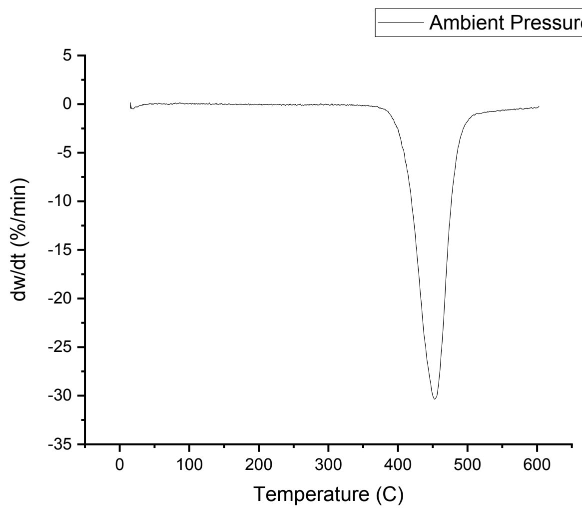

Figure 3. DSC curve of the PP Nonwoven fabric incorporated Silica Aerogel Composite by Ambient Pressure Drying.

Table 2. Heat-conducting performance of the tested PP Nonwoven fabric incorporated Silica Aerogel Composite by Ambient Pressure Drying Method.

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

incorporated Silica Aerogel Composite has excellent heat conducting performance (Table 2).

Surface property

The contact angle of untreated PP nonwoven fabric and its silica aerogel composite with water were presented in Figure 4. 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

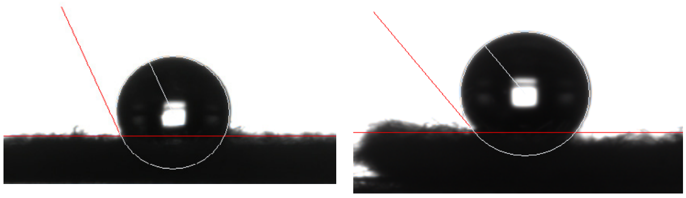

Figure 4. (a) Contact angle of the untreated pure PP Nonwoven fabric (b) Contact angle of the PP Nonwoven fabric incorporated Silica Aerogel Composite by Ambient Pressure Drying.

hydrophobic properties. The excellent hydrophobic properties are due to the existence of Si-$(CH3)_3$ on the surface, the -CH_3 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 reactions can occur.

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$(CH3)_3$. 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 4).

Contact Angle 114°

Contact Angle 129°

Conclusion

In a summery, Silica aerogel-pp nonwoven fabric composite was prepared efficiently via sol-gel process and surface modification by ambient pressure drying. 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.

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