

## Surface modification of polyester nonwoven fabrics by Al<sub>2</sub>O<sub>3</sub> sol-gel coating

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**Abstract** Nonwoven polyester (PET) fabrics have been extensively studied for various applications. However, the nonwoven PET fabrics have poor wettability. In this work, nonwoven PET fabrics were impregnated in a stable and transparent alumina sol that was prepared by the hydrolysis of aluminium isopropoxide using the sol-gel technique. The Al<sub>2</sub>O<sub>3</sub> particles were coated on nonwoven PET fabrics after the rolling-drying process. The surface morphology of modified nonwoven PET fabrics was characterized by scanning electronic microscopy (SEM) and atomic force microscopy (AFM). The SEM and AFM observations revealed the formation of the Al<sub>2</sub>O<sub>3</sub> particles on the fiber surface. The Al<sub>2</sub>O<sub>3</sub> coating was also confirmed by Fourier transform infrared (FTIR). The mechanical properties of the coated nonwoven PET fabrics were investigated using a tensile strength test, and the results showed that the mechanical properties were improved after surface sol-gel coating. The effect of Al<sub>2</sub>O<sub>3</sub> on the wetting behavior of the fabric was also significantly improved.

**Keywords** Sol-gel, Nonwoven PET fabrics, Characterization, Morphology, Wettability

### Introduction

The sol-gel process involves the transition of a system from a chemical solution (mostly colloidal) into an integrated network. The precursors used in the

preparation of the “sol” are usually inorganic metal salts or metal organic compounds such as metal alkoxide. The precursor is subjected to a series of hydrolysis and polymerization reactions to form a colloidal suspension, or a “sol.” The sol then evolves towards the formation of an inorganic continuous network containing a liquid “gel” phase. The drying process is also required to remove the liquid phase from the gel, thus forming a porous material, and the thermal treatment may be performed to favor further polycondensation and enhance mechanical properties.<sup>1-5</sup> The sol-gel technique has been increasingly used to produce the functional coatings on various forms of materials, such as steel,<sup>6</sup> glass,<sup>7</sup> and polymer.<sup>8</sup>

There is a growing interest in the use of sol-gel technology for the surface modification of textile substrates for a variety of antibacterial, UV protection, and wrinkle-free applications.<sup>9</sup> PET is a type of hydrophobic fiber, and its moisture regain is only 0.6–0.8% even at 100% relative humidity,<sup>10</sup> which hinders the expanding applications of this fiber. PET fibers have also become one of the most commonly used fibers in the nonwoven industry. Surface modification using various techniques has been performed to improve the wettability of nonwoven PET fabrics.<sup>11</sup> The sol-gel technique provides an effective means of modifying nonwoven fabrics, and it has been proven to be simple, effective, nonpolluting, and provide many other advantages.<sup>12,13</sup>

In this paper, nonwoven PET fabrics were modified by Al<sub>2</sub>O<sub>3</sub> sol with a rolling-drying processing. The surface morphology of the modified nonwoven fabrics was examined by scanning electron microscopy (SEM) and atomic force microscopy (AFM). Fourier transform infrared (FTIR) was used to confirm the deposition of Al<sub>2</sub>O<sub>3</sub> on the PET fibers. The effect of Al<sub>2</sub>O<sub>3</sub> by sol-gel technique on the mechanical properties of the nonwoven PET fabrics and wetting behavior were investigated in this work.

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## Experiment

### Materials

Fabrics used in this study were nonwoven PET fabrics (1.4 dtex, 70 g/m<sup>2</sup>). The fabric samples were first washed in ethanol followed by two rinses in distilled water, and then were dried at 40°C in an oven.

### Sol-gel coating

#### Preparation of Al<sub>2</sub>O<sub>3</sub> sol

The Al<sub>2</sub>O<sub>3</sub> sol was prepared by mixing the precursor aluminum isopropoxide (Al(OCH(CH<sub>3</sub>)<sub>2</sub>)<sub>3</sub>) with the ethanol-water mixture at about 80°C with high-speed stirring. The ethanol and water were mixed in a volume ratio of 1:1. The water and aluminum isopropoxide were mixed in a molar ratio of 1:1. The reaction time was set for about 1 h. A kind of thick transparent liquid was formed after the reaction. The temperature was then increased up to 90°C, and the pH value of the liquid was adjusted to 2.5 using hydrochloric acid. Polyvinyl alcohol (PVA, molecular weight from 84,000 to 89,000) was added to the mixture, and the PVA and aluminum isopropoxide were in a weight ratio of 2:1. The stable and transparent sol were then acquired after 12 h of mixing.

#### Fabric coating

The samples of the nonwoven PET fabrics were immersed in the prepared sol for 10 min and rolled on a very smooth surface plate with a glass bar. These steps were repeated twice. The fabric samples were then dried in the oven at 90°C for 40 min.

### Surface characterization

#### SEM and AFM observation

The structures and surface morphology of the modified nonwoven PET materials were observed with a SEM (Hitachi S-4800, Japan). The magnification was set at 700, 1000, and 1700. An AFM (CSPM4000, Benyuan Company) was also used to observe the surface morphology of the materials. Tapping mode was chosen in this study. All samples were scanned at room temperature in the atmosphere.

#### FTIR analysis

The surface chemistry of the modified nonwoven PET fabrics was examined by FTIR spectroscopy (Nicolet NEXUS470) in the range of 4000–400 cm<sup>-1</sup> using the ATR-Ge method. The absorption spectra were recorded with 64 scans at a resolution of 4 cm<sup>-1</sup>.

### Mechanical and wettability tests

The mechanical tests were performed on a YGA026A electronic tensile strength machine. The breaking strength and elongation at fracture were tested in this work. Three samples were used for each test, and the average of the three test results was reported.

The wetting tests were performed on a DSA100-droplet shape analyzer (Kruss, Germany). Deionized water was dropped onto the sample from a needle onto a microsyringe during the test. A picture of the drop was taken 10 s after the drop set onto the sample. The contact angles were calculated by analyzing the shape of the drop.

## Results and discussion

### Surface morphology analyzed by SEM and AFM

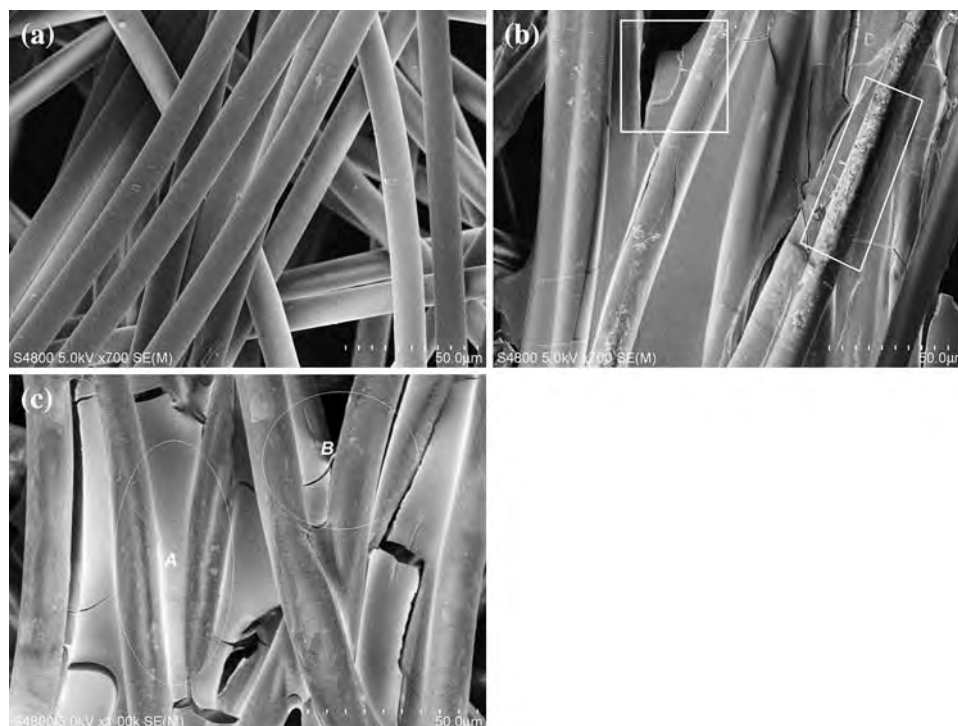
#### Characterization by SEM

The nonwoven PET fabrics were made by spun-bonding, and the fibers in the nonwoven fabrics looked circular with clean and smooth surfaces.<sup>14</sup> The fibers were distributed in random orientation, as indicated in Fig. 1a. It can be seen from Fig. 1b that most of the PET fibers were covered with a layer of coated gel. The porous structures of the nonwoven fabrics remained unchanged, but the coating on the fiber surfaces was clearly visible, as revealed in Fig. 1b. It is also clearly observed that the coating layer was not very even, as marked by the rectangle in Fig. 1b. The sol-gel coating covered the fibers and formed the coating film among the fibers, as indicated in Fig. 1c. It clearly reveals that the coating film formed on the fiber surface looked smooth, without any cracks, as marked "A" in Fig. 1c, but coating film between fibers showed some cracks, as marked "B" in Fig. 1c. The cracks formed between fibers were attributed to the larger space between fibers.

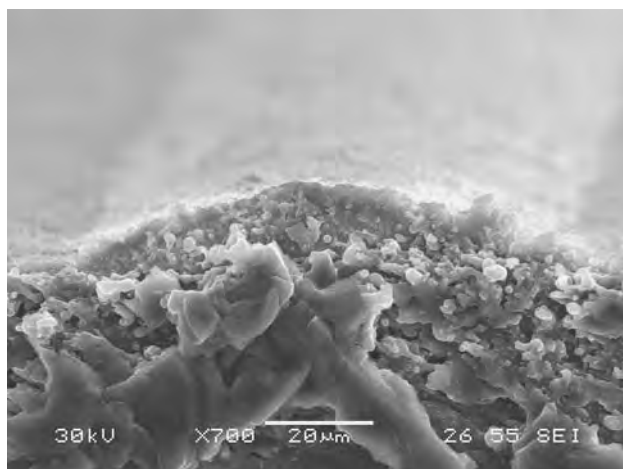
The SEM image in Fig. 2 shows the structures of Al<sub>2</sub>O<sub>3</sub> gel particles collected on a glass plate for comparison. It can be seen that the Al<sub>2</sub>O<sub>3</sub> gel particles formed a dense network and that pores existed among the particles. The porous structure was formed by a large number of spherical aggregates that had sizes in the range of a few nanometers to over 1000 nanometers. The coating layer formed on the fibers looked different from the gel particles on the glass plate due to rolling during the coating of the fibers.

#### Characterization by AFM

The AFM image of a 6.0 μm × 6.0 μm scan in Fig. 3 reveals the surface morphology of the modified PET fibers. As can be seen from the image, the fiber surface was covered by a thin layer of granular material. The sizes of these particles look quite even and the AFM



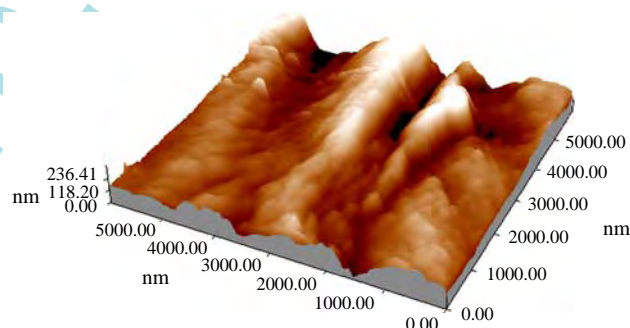
**Fig. 1: SEM images: (a) original PET fibers; (b) PET fibers after sol-gel coating; and (c) coating on the nonwoven PET fabrics and between fibers**



**Fig. 2: SEM image of pure  $\text{Al}_2\text{O}_3$  gel particles**

image reveals that their height was just around 100 nm. That means the coating did not obviously affect the appearance of nonwoven fabric. The AFM observation also confirms the formation of a dense network of  $\text{Al}_2\text{O}_3$  gel particles on the fiber surface, as presented in Fig. 3. The coating layer was integrated with the fiber surface, forming an interfacial adhesion between the coating layer and the fiber substrate.

The AFM image of a  $3.0 \mu\text{m} \times 3.0 \mu\text{m}$  scan in Fig. 4 shows the surface morphology at a higher magnification. As can be seen from the image, clearly recognized particles were deposited on the fiber's surface. The



**Fig. 3: AFM image of coated nonwoven PET fabrics at  $6.0 \mu\text{m} \times 6.0 \mu\text{m}$  scan**

deposited particles form a dense network and porous structures on the fiber surface. The particle aggregates show varying sizes that range from a few nanometers to over 100 nanometers, as revealed in Fig. 4.

The AFM observations reveal that most of the particles covered the surface of the fiber, but some particles may penetrate into the fiber matrix due to roll pressing. This effect may contribute to the adhesion between the particles and fibers.

#### **Surface chemistry analyzed by FTIR**

The effect of surface modification on the surface chemistry was analyzed by FTIR, as presented in Fig. 5. The high peaks from  $1716.37 \text{ cm}^{-1}$  to  $600 \text{ cm}^{-1}$

demonstrated the organic signals, such as a characteristic spectra of the stretching vibration band of C=O at the peak of  $1730\text{ cm}^{-1}$  and C–O–C stretching vibration band at the peak of  $1130\text{ cm}^{-1}$  and  $1260\text{ cm}^{-1}$ . All these peaks confirmed the existence of esters. The peak between  $700\text{ cm}^{-1}$  and  $900\text{ cm}^{-1}$  indicated the existence of Benzene Ring, and the peak at  $1502\text{ cm}^{-1}$  showed the symmetric structure of Benzene Ring. However, there was a weak peak at  $3540\text{ cm}^{-1}$ , representing the unreacted stretching vibration of –OH.

The ‘b’ sample curve reflected the information on the modified nonwoven PET fabrics using the  $\text{Al}_2\text{O}_3$  coating. It was observed that the wide peak existed at

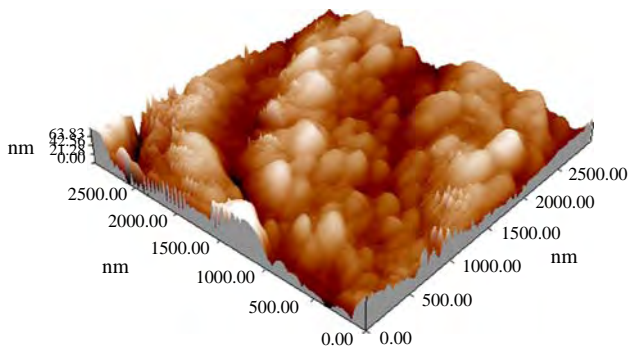


Fig. 4: AFM image of coated nonwoven PET fabrics at  $3.0\text{ }\mu\text{m} \times 3.0\text{ }\mu\text{m}$  scan

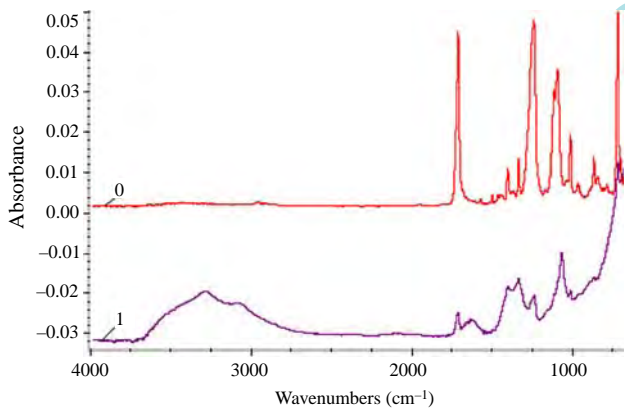


Fig. 5: FTIR spectra of both original and coated nonwoven PET fabrics

about  $3500\text{ cm}^{-1}$ , indicating the information of –OH of  $\text{Al}_2\text{O}_3$  gel.<sup>15</sup> The –OH and the small amount of –OH in PET may form the hydrogen bonds. The strong peak at  $700\text{ cm}^{-1}$  revealed the Al–O structure,<sup>16</sup> and some characteristic peaks of PET could also be observed from  $1730\text{ cm}^{-1}$  to  $1000\text{ cm}^{-1}$ .

### Mechanical properties of the fabrics

The results of the mechanical tests are listed in Table 1. As can be determined from the table, the average breaking strength of original nonwoven PET fabrics was about 196 N with an average elongation of 22.6% at the break. The average breaking strength of the coated nonwoven PET fabrics increased to 211 N with an average elongation of 23.9% at the break. The breaking strength and elongation were both improved by about 7.9% and 5.7%, respectively.

The solvent evaporation resulted in the  $\text{Al}_2\text{O}_3$  gel particles in the form of a network covering on the fiber surface during the drying process. As shown in Fig. 4, particles were deposited and integrated with the PET fibers. When the fibers were stretched by the external force, the  $\text{Al}_2\text{O}_3$  network also shared some force and gave some flexibility, leading to a small increase in breaking strength and elongation at break.

### Wetting behavior of the fabrics

Water droplets on the surface of original nonwoven PET fabrics formed a contact angle of about  $79^\circ$  after 20 s, as shown in Fig. 6a. The contact angle appeared

Table 1: Results of mechanical tests

Items	Samples	
	Nonwoven PET fabrics	Modified nonwoven PET fabrics
Average breaking strength (N)	196	211
Average elongation at fracture (%)	CV%: 8.5%	CV%: 5.8%
	22.6	23.9
	CV%: 10.4%	CV%: 9.1%

Note: CV coefficient of variation

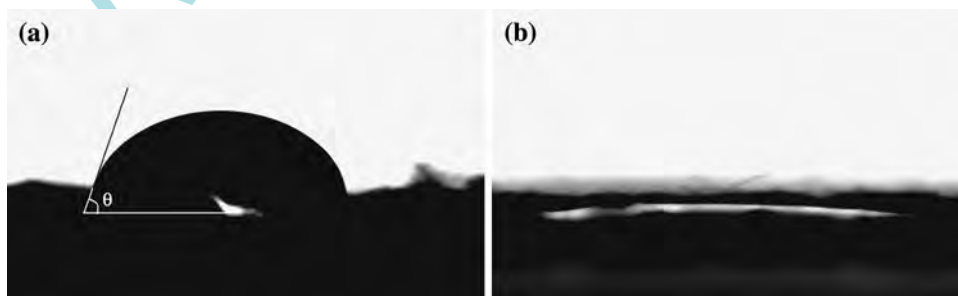


Fig. 6: Wetting behavior: (a) original nonwoven PET; (b) coated nonwoven PET



because of the poor wetting behavior of PET fibers.<sup>17</sup> PET fibers possessed hydrophobic properties, but the existence of fibrous and porous structures made the contact angle of the fabric lower than that of the fibers.<sup>18</sup>

The wetting behavior of the coated nonwoven PET fabrics is shown in Fig. 6b. When a water droplet set on the surface of fabrics, the water drop penetrated into the fabric rapidly, indicating the improved wetting properties of the fabric. The Al<sub>2</sub>O<sub>3</sub> particles deposited on the surface of fabrics might form hydrogen bonds with water when they contact water. The porous network of the Al<sub>2</sub>O<sub>3</sub> particles may also facilitate the adsorption of water, thus the water adsorption of nonwoven PET fabrics was significantly improved by the surface modified with alumina sol-gel coating.

## Conclusion

This work has investigated the surface modification of nonwoven PET fabrics by Al<sub>2</sub>O<sub>3</sub> sol-gel coatings. The SEM observation revealed the deposition of the Al<sub>2</sub>O<sub>3</sub> particles on the PET fibers, but the fabric structures remained unchanged. The dense network and porous structures of the Al<sub>2</sub>O<sub>3</sub> particles formed on the fiber surfaces were observed by AFM scans. The Al<sub>2</sub>O<sub>3</sub> coating on the PET fiber surface was also confirmed by FTIR. The Al<sub>2</sub>O<sub>3</sub> coating contributed to the mechanical properties of the nonwoven PET fabrics. The wettability of the fabric was significantly improved by using the Al<sub>2</sub>O<sub>3</sub> coating.

**Acknowledgments** The financial support from the Special Fund from the Ministry of Science and Technology (NCSTE-2006-JKZX-015), the Program for New Century Excellent Talents in University (No. NCET-06-0485), and the Program for Innovation Team of Jiangnan University (PIRT Jiangnan) is gratefully acknowledged.

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