Zhenrong Zheng[1,2,*],
Hongmei Wang[1],
Nannan Zhang[1],
Xiaoming Zhao[1]

[1] **College of Textiles,
Tianjin Polytechnic University,**
Tel.: +86 22 83955355
*E-mail: tianjinzhengzr@163.com

[2] **Key Laboratory of Advanced Textile Composites
of Ministry of Education,
Tianjin Polytechnic University,**
Tianjin, 300387, China

Preparation of Self-cleaning Polyester Fabrics by Chemical Vapor Deposition of Methyltrichlorosilane/ Dimethyldichlorosilane

Abstract

Self-cleaning polyester fabrics were prepared by a simple gas phase deposition procedure in which a layer of polydimethylsiloxane nanofilaments was grown onto textile fibers. Superhydrophobic and self-cleaning properties, tensile breaking strength, mechanical stability and permeability of polyester fabrics were investigated. The results showed that the fabrics deposited had superhydrophobicity, and the contact angle and sliding angle of the fabric surface were 159° and 1.7°, respectively. The self-cleaning test showed that dust particles adhere to rolling water and shed from the surface of polyester fabric, leaving an extremely clean surface. In addition, the polyester fabric deposited still has excellent breaking strength and permeability. This approach is simple, inexpensive and has little effect on the mechanical properties of the fabric.

Key words: *superhydrophobic, polyester fabric, self-cleaning, permeability.*

Introduction

A surface with a contact angle greater than 150° and sliding angle lower than 10° is usually called a superhydrophobic surface [1-2]. Many surfaces in nature are highly hydrophobic and have a self-cleaning property, such as the lotus leaf [3-5], cabbage [6-7] and the wings of a butterfly [8-10]. Numerous studies have validated that this combination of micro-and nano-scale roughness, along with a low surface energy material leads to the self-cleaning property of a surface [11-14]. At present, great interest has been focused on the preparation of self-cleaning fabrics. Preparation technologies are reported such as the sol-gel process, electrospinning, the wet chemical process and coatings [15-18]. Polyester fabric is one of the most significant and extensively used fabrics which would greatly benefit from possessing superhydrophobic and self-cleaning properties. These polyester fabrics have significant potential practical application in high performance textiles such as superhydrophobic fabrics, self-cleaning fabrics, frictionless flow water pipes, snowsuits, tents, decorative materials and so on [19-23].

In this work, gas phase deposition from a mixture of MTS/DDS solution was used to fabricate self-cleaning textiles. Based on fabric roughness woven from yarns, low surface energy material can be fabricated on a fabric surface with micro-and nano-scale roughness by a chemical method to obtain a superhydrophobic and self-cleaning fabric.

Firstly polyester fabric was treated with alkali solution to increase the surface roughness. Methyltrichlorosilane (MTS) and dimethyldichlorosilane (DDS) belong to volatile organic chemicals. Moreover volatile gases have a self-hydrolysis property due to their molecule structure. The silicon-chlorine groups of DDS and MTS can easily hydrolyse to form silanol groups. The interaction and condensation of silanol groups are attributed to the formation of silicon-oxygen-silicon bonds. Under different conditions, organosilane structures obtained by self-assembly are different. Hence the react condition is critical to the surface roughness of the polyester fabric.

Experimental

Treating with alkali solution

To increase the surface roughness of polyester fibers, polyester fabrics (Jiaxing Rongsheng Textile Co. Ltd, China) with a plain weave (450 ends/270 picks, yarn number 12 tex) were immersed in 20 g/l NaOH solution for 60 min at 90°C. Then the polyester fabrics were rinsed with water and dried at 100°C.

Chemical vapour deposition

$(CH_3)_2SiCl_2$ (DDS) and CH_3SiCl_3 (MTS) (Tianjin Guangfu Chemical Research Institute, China) were mixed and placed in a closed container. Because of the strong volatility, DDS and MTS gases can diffuse in the container (glass desiccator from Tianjin Tianbo Instrument Manufacturing Co. Ltd, China). After treating with alkali solution, polyester fabrics were placed in the DDS/MTS gas for grafting for 90-180 min under 80% relative humidity at 25°C.

Characterization

Water contact angle measurements were carried out with a 50μl droplet at room temperature by means of a contact angle goniometer (JY-82, Hebei Chengde Testing Machine Co. Ltd, China). To illustrate the self-cleaning property of polyester fabric, carbon dust was sprayed onto the surface of the fabric. Subsequently 0.3 ml water droplets were sprayed onto fabric, and the fabric surface was tilted at a certain angle to the horizontal to test the sliding angle and self-cleaning property of the fabric. Surface morphologies of the polyester fabrics were investigated with a scanning electron microscope (SEM, TM-1000 Hitachi High-technologies Co. Ltd, Japan) and field emission environmental scanning electron microscope (FESEM, Hitachi S-4800 Hitachi High-technologies Co. Ltd, Japan). The surface chemical compositions of the fabrics were measured by X-ray photoelectron spectroscopy (XPS, Kratos Axis Ultra DLD, Japan). The tensile breaking strength and permeability of the polyester fabrics were tested according to the China National Standards GB/T 3923.1-1997 and GB/T5453-97, respectively. The abrasion resistance of the polyester fabric was measured by a rubbing-crock meter. After the fabrics were rubbed 20 times with cotton fabric under a certain pressure, the contact angles of the fabrics were tested to investigate the mechanical stability of the self-cleaning fabric obtained.
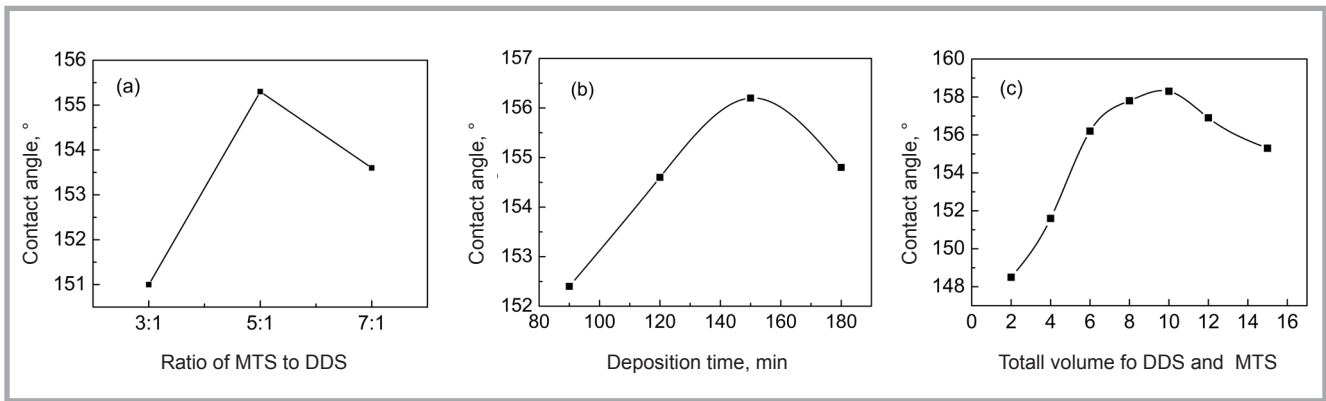
Figure 1. Effects of major influence factors on contact angle of polyester fabric.

Results and discussions

Influential factors of chemical vapour deposition

A total volume of MTS and DDS of 6ml and deposition time of 150 min were kept constant. **Figure 1.a** shows that the water contact angle of polyester fabric rose from 151° to 156.2° at a ratio of MTS to DDS increasing from 3:1 to 5:1, and then the contact angle decreased to 153.6° when the ratio of MTS to DDS was 7:1. MTS can hydrolyze with water to form three silanol groups, which can graft in three dimensions with the fabric or other silanol groups to form filaments on the fabric surface. However, DDS was used as an end-capping agent, and its dimethyl groups inhibited further reaction near them [25]. When the ratio of MTS to DDS was increased, more MTS gases were introduced and more depositon materials were formed; consequently, the contact angle of the fabric increased. But when the ratio of MTS to DDS was 7:1, there was not enough DDS to inhibit the silanol groups hydrolyzed from MTS; some silanol groups (-Si-OH) were exposed on the fabric, and hence the contact angle decreased.

In order to explore the influence of the deposition time on the hydrophobicity of the fabric, a total volume of MTS and DDS of 6ml and a ratio of MTS to DDS of 5:1 were kept constant. **Figure 1.b** shows that the water contact angle of the polyester fabric gradually rose from 152.4° to 156.2° as the deposition time increased from 90 to 150 min, and then the contact angle decreased to 154.8° when the deposition time was prolonged to 180 min. Therefore the optimum deposition time is 150 min.

A deposition time of 150 min and ratio of MTS to DDS of 5:1 were applied to investigate the influence of the total volume of MTS and DDS on the hydrophobicity of fabric. As shown in **Figure 1.c**, the water contact angle of polyester fabric was 158.3° when the total volume of MTS and DDS was 10 ml. The contact angle decreased to 155.3° when the total volume of MTS and DDS was increased to 15 ml. The materials deposited on the fabric surface may be too dense and cover the fabric roughness woven from yarns. When water dropped on this fabric surface, the air entrapped between the water and fabric surface decreased, leading to a decrease in the contact angle.

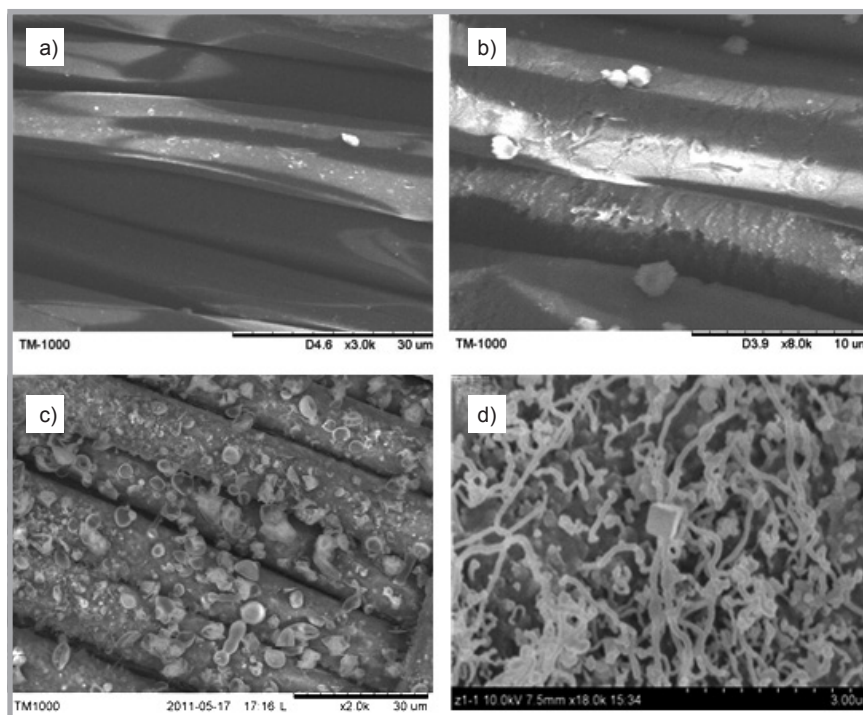Furthermore the orthogonal design of experiment (L_9 (3^4)) was performed to optimise the chemical vapour deposition

Table 1. Factors and levels.

Levels	Factors		
	Ratio of MTS to DDS	Deposition time, min	Total volume of MTS and DDS
1	4:1	120	8
2	5:1	150	10
3	6:1	180	12

Figure 2. SEM images of (a) original polyester fabric(3000×); (b) fabric treated with alkali (8000×); (c) deposited fabric (5000×); FESEM image of (d) deposited fabric (18000×).

conditions. The three factors mentioned above were chosen as shown in **Table 1**.

The results of this experiment show that when the ratio of MTS to DDS was 6:1, the deposition time was 180 min, the total volume of MTS and DDS 12 ml, the water contact angle of polyester fabric 159°, and the sliding angle of the fabric surface was 1.7°.

Surface morphology of polyester fabric

The surface morphology of polyester fabric was investigated by SEM and FE-SEM. It can be seen from **Figure 2.a** that the surfaces of fibers from the original polyester fabric were smooth. However, **Figure 2.b** shows that there are many cracks in the surface of the alkali treated fibers, and the surface roughness of the polyester fibers has been enhanced. **Figure 2.c** shows that many pellets of 1-5 μm diameter were distributed over the fabric deposited. The enlarged views in **Figure 2.d** display clearly that the surfaces of the fabric deposited were well wrapped by filaments of nano-scale size. Hence, filaments, pellets and fabric roughness woven by yarns constructed the multiple surface roughness of the polyester fabric.

XPS spectrum of deposited materials

The composition of materials deposited on polyester fabric was investigated by XPS. **Figure 3** shows that three elements (Si2s at 153eV, Si2p at 102.4 eV, C1s at 284.8 eV, and O1s at 530.4 eV) were observed. The Si2s, Si2p, C1s and O1s components originated from the Si-O, Si-(CH$_3$)$_2$, CH$_3$-Si and O-Si groups, respectively. Combined with the condensation theory of silanol groups, it was deduced that the deposition materials were polymethysiloxane and polydimethylsiloxane. Because DDS was used as the end-capping agent, the surface material of the fabric deposited was mainly polydimethylsiloxane.

Superhydrophobicity of polyester fabric

The wetting angle is used to characterise the hydrophobic properties of a fabric surface. A surface with a contact angle greater than 150° and sliding angle lower than 10° is usually called superhydrophobic. And this surface has a self-cleaning property. **Figure 4** displays water droplets residing on the polyester fabric deposited. When water droplets were dripped on the original polyester fabric, they were absorbed completely by

Figure 3. *XPS spectrum of the polyester fabric deposited with MTS/DDS.*
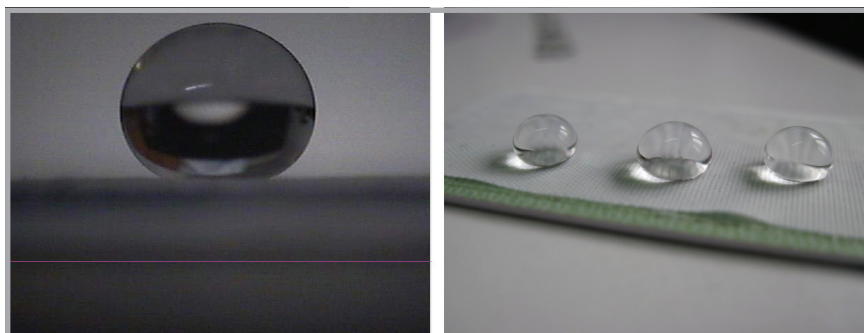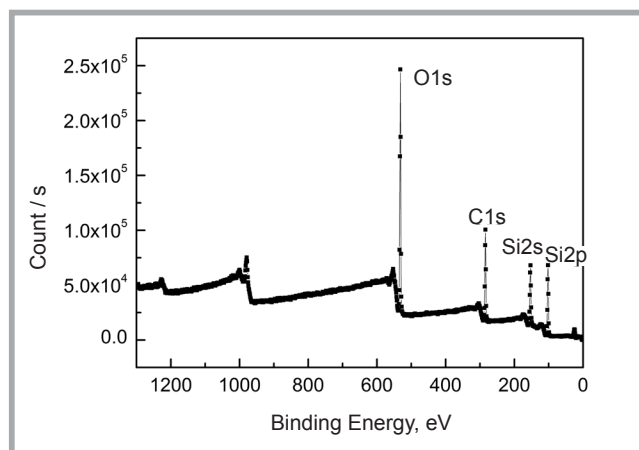
Figure 4. *Water droplets resided on the deposited polyester fabric.*

it. However, when water droplets were dripped on the polyester fabric deposited, they jumped up as balls immediately and rolled on the surface. The water contact angle of the fabric deposited was 159°, and the sliding angle was 1.7°.

Since polyester fabric is a porous substrate, and the organosilane deposited on this fabric were filaments of nano-scale size, as shown in **Figure 2.c** and **Figure 2.d**, the polyester fabrics deposited are considered as conforming to the Cassie model (air pockets at the interface). In the Cassie model, the apparent contact angle (θ_{CB}) is the sum of all the contributions of solid and air phases, as described in the following **Equation (1)** [26].

$$\cos\theta_{CB} = f_1\cos\theta_1 + f_2\cos\theta_2 \qquad (1)$$

Where f_1 and f_2 are the surface fractions of solid and air and θ_1 & θ_2 are the contact angles of solid and air on the corresponding smooth surfaces, respectively. Given f is the solid fraction, then the air fraction is $(1-f)$. With $\theta_2 = 180°$ for air, the resulting θ_{CB} can be calculated by the following **Equation (2)**:

$$\cos\theta_{CB} = f(\cos\theta_1 + 1) - 1 \qquad (2)$$

In this model, to obtain a superhydrophobic surface, the solid part (f) should be

as small as possible. For the polyester fabric deposited, the contact angle of the fabric surface (θ_{CB}) was as high as 159°, validating that the solid part coming into contact with water was very small and that lots of air was trapped between the fabric and water. Therefore the adherence strength between the water and fabric surface was greatly reduced, and hence the sliding angle of the fabric deposited was only 1.7°. At the same time, the fabric surface area in contact with dirty particles was also small. Because organosilanes are low surface energy materials, the adherence strength between dirty particles and the fabric surface is lower than that between water and dirty particles. Hence the dirty particles will be easily taken away by rolling water droplets.

Self-cleaning property of polyester fabric

The self-cleaning test of the original polyester fabric is shown in **Figure 5.a** (see page 124). Water droplets were absorbed by this fabric and carbon dust was adhered to it. However, when water droplets dripped on the polyester fabric deposited, they formed spheres and could easily roll off with dirty particles when tilting 1.7°, leaving an extremely clean surface, as shown in **Figure 5.b**.
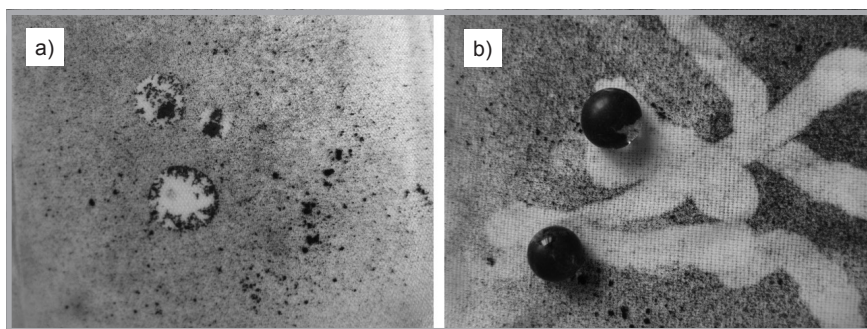
Figure 5. Self-cleaning test of the polyester fabrics: (a) original and (b) deposited with DDS/MTS.

Table 2. Other physical properties of polyester fabric.

Polyester fabric	Tensile breaking strength, N	Permeability, mm/s	Contact angle before abrasion resistance, °	Contact angle after abrasion resistance, °
Original	473	148.3	0	0
Deposited	454.5	247.0	159	150

This validates that the polyester fabric deposited has excellent superhydrophobic and self-cleaning properties.

Other physical properties of polyester fabric

Physical properties such as the tensile breaking strength, permeability and abrasion resistance are very important for all kinds of fabrics. These properties of polyester fabrics were shown in *Table 2*.

The tensile breaking strength is an important parameter of fabric, which has a direct influence on the use performance. During the preparation process of a super-hydrophobic fabric surface, the chemical reagents may affect the tensile breaking strength. Hence the breaking strengths of the original and treated fabrics were tested. The tensile breaking strength of the original polyester fabric was 473N/5cm. It decreased to 454.5N /5cm after the fabric was deposited with MTS/DDS, and the retention of breaking strength was 96.1%. This indicated that the polyester fabric deposited still has excellent breaking strength. The decrease in breaking strength may be caused by the alkali corrosion of fibers used to increase the surface roughness of the polyester fibers. Hence more and more air can permeate the fabric through the gaps between polyester fibers. Compared with the original polyester fabric, the air permeability of the polyester fabric deposited greatly increased to 247.0 mm/s. Some fabric surfaces with the super-hydrophobic property were usually prepared by the coating method or a method forming a thin film. These surfaces of fabrics were usually airtight materials. In this work, the permeability of the super-hydrophobic fabric was much better than that of the original one (148.3 mm/s). The water contact angle of the fabric deposited was 159°. After a long abrasion, the contact angle of the fabric deposited reduced to 150°, indicating that the fabric still has superhydrophobicity, and the abrasion resistance of the fabric deposited should be improved in a further study.

■ **Conclusion**

In summary, self-cleaning polyester fabrics were successfully prepared using the chemical vapor deposition technique. When the ratio of MTS to DDS was 6:1, the deposition time was 180min, the total volume of MTS and DDS 12ml, the water contact angle of the polyester fabric 159°, and the sliding angle of this fabric surface was 1.7°. Filaments, pellets and fabric roughness woven by yarns constructed the multiple surface roughness of the polyester fabric. It was shown that dust particles adhere to rolling water and shed from the surface of polyester fabric, leaving an extremely clean surface. In addition, the polyester fabric deposited still has excellent breaking strength and permeability.

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