



# Preparation of superhydrophobic poly(ethylene terephthalate) fabric by high-temperature sucrose fatty ester inlaying and esterification



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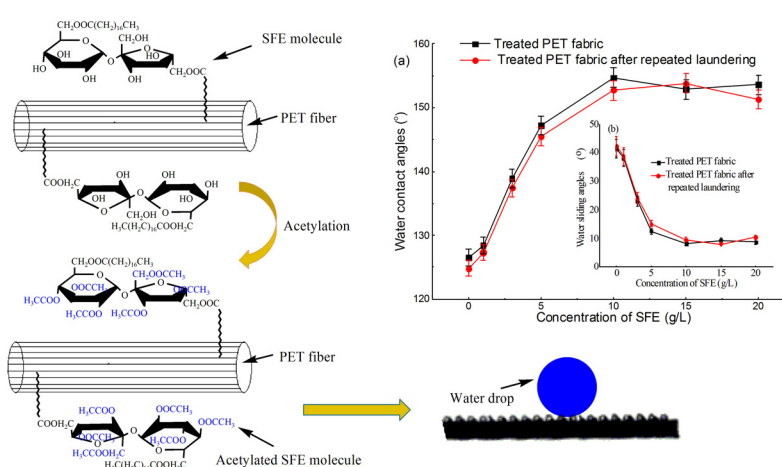
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## HIGHLIGHTS

- The rough surface of PET fiber was made by semi-inlaid sucrose fatty ester.
- The hydroxyl radical was acetylated to lower the surface free energy of PET fiber.
- The water contact angle of PET fabric could reach 154.7°.
- The hydrophobic PET fabric was durable.
- The hydrophobic modification technology was ease of operation and high efficiency.

## GRAPHICAL ABSTRACT



## ARTICLE INFO

### Article history:

Received 10 November 2015

Received in revised form 16 January 2016

Accepted 20 January 2016

Available online 23 January 2016

### Keywords:

Polyester

Sucrose fatty ester

Inlay

Superhydrophobic modification

## ABSTRACT

Poly(ethylene terephthalate) (PET) fabric was modified by high-temperature sucrose fatty ester (SFE) inlaying and acetic anhydride-mediated acetylation at 40 °C to generate a superhydrophobic material. The water contact angle increased from 105.3° to 154.7°, while the water sliding angle decreased from 41° to 8.2° upon modification. In addition, the spray rating increased from 2 to 5°, and the water absorption rate decreased from 97.8% to 26.0%. Multiple washing and abrasion negligibly affected the hydrophobicity of the resulting fabric. Scanning electron microscopy revealed that the hydrophobic fibers were characterized by extremely rough surfaces. Fourier-transform infrared spectra demonstrated that the SFE was inlaid into the PET fibers and that almost all sugar hydroxyl groups were acetylated. X-ray diffraction and differential scanning calorimetry suggested that the chemical process did not alter the main PET fiber structure. Furthermore, the fabric retained its ultimate tensile strength, breaking elongation, elastic wrinkle recovery, whiteness, and rigidity as well as its air and moisture permeability.

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## 1. Introduction

Superhydrophobic solid surfaces exhibiting water contact angles larger than 150° and low sliding angles [1–4] are expected

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to have applications in self-cleaning and impermeable textiles, microfluidic devices, outdoor antennas, and windows. Numerous approaches have been proposed to prepare these surfaces on different solid substrates. Most methods developed to manufacture superhydrophobic fabrics and textiles, such as self-assembly [5,6], electrochemical deposition treatment [7,8], the sol–gel method [9–12], electrospinning [13,14], plasma polymerization [15,16], and coating [17–19], rely on the imitation of natural phenomena such as the lotus leaf effect. The artificial engineering of lotus-type materials requires a substrate with both low surface free energy and high surface roughness. Several materials involve organosilanes bearing long alkyl chains or fluorochemicals as finishing agents to impart low surface energies to the substrates. These materials also fully apply nanotechnology principles to traditional fabrics and textiles such as natural cotton and polymeric fibers. Specifically, adequate concentrations of micron-sized and nanoscale particles have been applied to these fibers to enhance their surface roughness.

Numerous fluorochemicals have been employed to manufacture superhydrophobic and oleophobic textiles because of their unique properties and significant effects on surface wettability, even in small doses. However, most fluorides are costly compared to the most frequently used chemical reagents and pose serious risks to the environment and humans when used extensively. Many alkoxyorganosilane reagents containing long alkyl groups, such as hexadecyltrimethoxysilane, have also been used as water-repelling agents in functional textile finishing, but their prices are almost comparable to those of fluorides [20–23].

Typically, poly(ethylene terephthalate) (PET) fabrics have been widely used in the textile industry because of their outstanding durability as well as wash-and-wear and attractive crease-free properties. In this study, an effective method involving a short and simple modification process was developed to prepare superhydrophobic PET fabrics. In this process, the PET fabric was inlaid with sucrose fatty ester (SFE) at a high temperature and pressure to produce fibers with rough surfaces before being subjected to esterification in the presence of acetic anhydride to reduce its surface free energy. The modified superhydrophobic PET fabric displayed durability. Furthermore, this modification did not require any special equipment and readily produced several materials, making it attractive for potential industrial applications.

## 2. Experimental

### 2.1. Materials

Commercial plain-weave PET fabric bought from a Chongqing market (China) (231 g/m<sup>2</sup>, 604 × 323, 15 tex × 15 tex) was used as a substrate. A SFE consisting of two eighteen-carbon alkyl chains was purchased from the Liuzhou Food Additive Company (China). Analytical-grade acetic anhydride was acquired from Chongqing Chuandong Chemical Group Co., Ltd. (China). Anhydrous sodium carbonate and sodium dodecyl benzene sulfonate (SDBS) were supplied by the Chongqing Chemical Reagent Factory (China). All

chemical reagents were used as received. Water was distilled in our laboratory before being used in the experiments and tests.

### 2.2. Polyester fabric preconditioning

To remove surface impurities, the fabric materials were immersed at 60 °C for 10 min in aqueous sodium carbonate (1.0 g/L) with a bath ratio of 1:30, rinsed with distilled water, and dried at room temperature.

### 2.3. Superhydrophobic modification of PET fabrics

Table 1 shows the superhydrophobic finishing of PET fabrics. The fabric specimens were treated by using a high-temperature dyeing machine (Nantong Hongda Experiment Instrument Co., China) in a SFE bath (liquor ratio: 1:10) at concentrations ranging from 1 g/L to 20 g/L at 130 °C for 60 min. Subsequently, they were esterified with acetic anhydride (liquor ratio: 1:20) at 40 °C for 20 min, washed by distilled water, and dried at 60 °C.

### 2.4. Characterizations

#### 2.4.1. Hydrophobic performance tests

All contact angles were analyzed before and after modification at different locations more than five times to obtain average values. The water contact angles (WCAs) were evaluated by using a JGW-360A instrument (Hebei Chenghui Testing Machine Co., Ltd., China). These static measurements were performed by using individual water droplets of 2 μL. The water sliding angles (WSAs) were measured according to a previously reported procedure [17,18]. The modified sample durability was evaluated in a 25 °C water bath by using a soaping color fastness tester (Roaches Co., England). The abrasion resistance was performed on a QS-M304 friction instrument for textiles (Beijing hengao technology Co., Ltd., China). The spray rating performance was examined by using a YB813 tester (Wenzhou Darong Textile Instrument Co., Ltd., China). The water absorption rate was determined by a weighing method. The PET fabric with a dry weight  $W_1$  was immersed in distilled water for 5 min, hung in air for 2 min, and weighed again. The water absorption rate was calculated by using the difference between this final weight  $W_2$  and  $W_1$  as:

$$\text{Water absorption rate \%} = \left[ \frac{(W_2 - W_1)}{W_1} \right] \times 100 \quad (1)$$

#### 2.4.2. Structural characterization of PET fabrics

The PET fiber surface morphologies were examined before and after hydrophobic modification with a Sirion 200 field-emission scanning electron microscopy (SEM) (FEI Co., Netherlands). Fourier-transform infrared (FTIR) spectra were obtained between 5000 and 400 cm<sup>-1</sup> by using a Spectrum GX apparatus (PerkinElmer Co., USA) and KBr pellets. The crystallinity was investigated by using X-ray diffraction (XRD) to examine the structure at scattering angles of 5–60° and the walk width of 0.02° ( $\lambda = 1.5406 \text{ \AA}$ ) by using

**Table 1**  
The superhydrophobic finishing of PET fabrics.

Number of PET fabric specimens	a	b	c	d	e	f	g
Number	1	2	3	4	5	6	7
SFE concentration (g/L)	0	1.0	3.0	5.0	10.0	15.0	20.0
Liquor ratio of inlay	1:10	1:10	1:10	1:10	1:10	1:10	1:10
Inlay temperature (°C)	130	130	130	130	130	130	130
Time of inlay process (min)	60	60	60	60	60	60	60
Acetic anhydride	AR	AR	AR	AR	AR	AR	AR
Esterification temperature (°C)	40	40	40	40	40	40	40
Liquor ratio of esterification	1:20	1:20	1:20	1:20	1:20	1:20	1:20
Time of esterification process (min)	20	20	20	20	20	20	20

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