



Mechanically durable superamphiphobic surfaces via synergistic hydrophobization and fluorination

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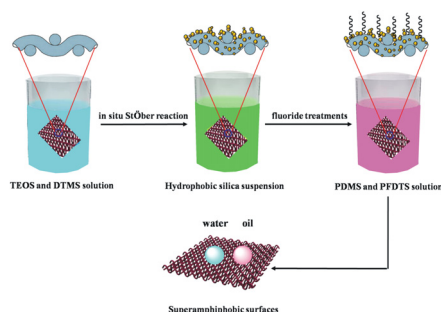
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HIGHLIGHTS

- Superamphiphobic fabrics were fabricated by synergistic hydrophobization.
- The synergistic hydrophobization lowered the amount of fluorinated compound needed.
- The as-obtained fabrics have excellent durability of superamphiphobicity.
- The superamphiphobic fabrics show brilliant resistance to blood staining.

GRAPHICAL ABSTRACT



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ABSTRACT

Superhydrophobic poly(ethylene terephthalate) (PET) fabrics were firstly fabricated by one-pot *in situ* Stober reaction of tetraethylorthosilicate (TEOS) and dodecyltrimethoxysilane (DTMS), in which the as-formed silica particles roughened the fiber surfaces and the hydrolyzed dodecyltrimethoxysilane hydrophobized the fabrics. Then the superhydrophobic fabrics were turned superamphiphobic after modification with perfluorodecyltrichlorosilane (PFDTs), with water and oil contact angles higher than 150°. The synergistic hydrophobization of DTMS, PFDTs and PDMS made the roughened fabrics easy to be superamphiphobic using very low concentration of PFDTs. Meanwhile, the as-obtained superamphiphobic fabric showed excellent chemical robustness even after exposure to different chemicals, such as acid, base, and salt. Importantly, the fabrics were durable to 100 cycles of laundries, 1000 cycles of mechanical abrasion as well as long time exposure to UV irradiation without apparently changing the amphiphobicity. Also, the surface of the superamphiphobic fabrics showed excellent blood stain resistance properties.

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1. Introduction

Superhydrophobic surfaces [1,2] showing a water contact angle (CA) greater than 150° and sliding angle lower than 5° are being

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extensively studied, due to their potential practical applications including self-cleaning [3,4], anti-fouling [5,6], anti-icing [7–10], separating oil from water [11–16], protecting electronic devices [17] and avoiding fluid drag in macrofluidic devices [18,19]. However, most of these surfaces repel water but not oils. Oil contamination has become a challenge for them and has strongly limited their applications in self-cleaning, anti-fouling, etc [20–22] for materials that may face heavy organic contaminations. Therefore, building superamphiphobic surfaces with both

superhydrophobicity and superoleophobicity is urgently needed for practical applications.

Superamphiphobic surfaces are those that are super-repellent to both water and oil, exhibiting CAs above 150° for water as well as various oil [23–25]. However, such superamphiphobic surfaces with low surface energy are difficult to fabricate compared to superhydrophobic surfaces [26–29]. To design superamphiphobic surfaces that resist wetting by low-surface-tension liquids such as hexadecane with surface tension of 27.5 mN/m, both surface energy and surface structure must be well controlled [30–33]. Recently, several groups have attempted different techniques to artificially recreate superamphiphobic surfaces, and some promising results have been reported. The techniques to fabricate superamphiphobic surfaces adopt usually either stochastic or biomimetic bottom-up approaches [34–36] or microfabrication top-down approaches [37]. The latter producing ordered surfaces are useful for basic studies while the former usually produced random, hierarchical rough structure at low cost. Although there is a report about turning a surface superomniphobic by roughness alone [38], fluorinated materials are mostly used in large-scale fabrication of superamphiphobic surfaces. Therefore, reducing the amount of fluorinated substances used to lessen the costs and environmental risks is necessary, however a challenge in preparation of superamphiphobic materials.

In this work, we report the fabrication of superamphiphobic poly(ethylene terephthalate) (PET) fabrics by combination of roughening structures with a coordinated hydrophobic modification. Firstly, PET fibers were rendered superhydrophobic by decoration with silica nanoparticles via one-pot *in situ* Stöber reaction of tetraethylorthosilicate (TEOS) and dodecyltrimethoxysilane (DTMS). Then the superhydrophobic fabrics were modified with perfluorodecyltrichlorosilane (PFDTs) and polydimethylsiloxane (PDMS), as shown in Fig. 1. The synergistic hydrophobization of DTMS, PFDTs and PDMS lowered the concentration of PFDTs to 0.5% required for superoleophobicity and the fabric have excellent superamphiphobicity with strong chemical and mechanical stabilities as well as washing durability. And the surface of the superamphiphobic fabric showed brilliant resistance to blood staining.

2. Materials and methods

2.1. Materials

PET fabric was purchased from a local factory. PFDTs was purchased from Aladdin. TEOS was purchased from Kemiou Chemical

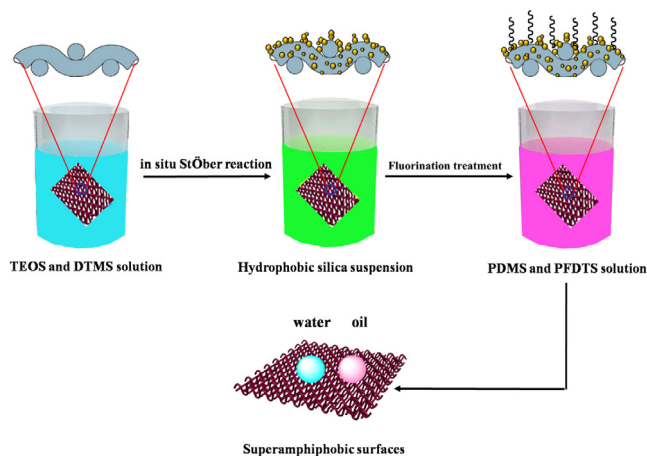


Fig. 1. Illustration of the fabrication of superamphiphobic fabrics.

Reagent Co., Ltd. DTMS was purchased from Jiangnan Reagent Co., Ltd. Polydimethylsiloxane (PDMS, Sylgard 184 Silicone Elastomer Kit with components of PDMS base and curing agent) was purchased from DOW CORNING. Pig blood was purchased from Nanjing Senbeijia Biological Technology Co., Ltd. All other chemicals were used as received without further purification.

2.2. *In situ* introduction of silica nanoparticles onto fibers and pre-hydrophobization

A piece of PET fabric was added to a container with a solution of 100 mL ethanol, 20 mL ammonia, 6 mL TEOS, and 2 mL DTMS. Then the container was sealed and mounted in an infrared-ray heating machine which is commonly used to dye fabrics in our lab as well as in a fabric dyeing manufactory for sampling before large-scale production, and the mixture reacted at room temperature for 5 h. After the Stöber reaction, the PET fabric was washed with ethanol in an ultrasonic bath three times (10 min for each washing) to remove loose particles and then dried under 80°C . The obtained fabric was named as $\text{SiO}_2@PET$.

2.3. Surface fluorination of $\text{SiO}_2@PET$

PDMS precursor part-A (Sylgard 186 elastomer base, 0.5 g) and precursor Part-B (sylgard 186 curing agent, 0.05 g) were dissolved in THF (50 g), then stirred for 10 min to form solution A. 0.5 mL PFDTs was dissolved in ethanol (50 g) to form solution B. Then solution A and B were mixed together at room temperature to form a coating solution. The $\text{SiO}_2@PET$ fabrics were dip-coated with the as-prepared coating solution, dried at 80°C and baked for 5 min at 150°C . The obtained fabric was named as F- $\text{SiO}_2@PET$. For comparison, pure PFDTs solution was obtained by dissolving 0.5 mL PFDTs in 50 g ethanol and mixing with 50 g THF to treat the pristine PET and $\text{SiO}_2@PET$ fabrics. Pure PDMS solution was also obtained by dissolving 0.5 g PDMS precursor part-A and 0.05 g precursor Part-B in 50 g THF and mixing with 50 g ethanol to treat the pristine PET and $\text{SiO}_2@PET$ fabrics.

2.4. Sample characterization

Hitachi S-4800 field emission scanning electron microscope (SEM) was used to examine the surface morphology of the fibers. Contact angles (CAs) were measured with different liquids on a video optical contact angle system (OCA 20, Data physics, Germany) at room temperature. All the CAs were determined by averaging values measured at 6 different points on each sample surface. Atomic force microscopy (AFM) images in ScanAsyst mode were collected on a scanning probe microscope (Dimension Icon, Bruker). TGA data were obtained using NETZSCH STA 409 PC/PG at a heating rate of $10^\circ\text{C}/\text{min}$ in an N_2 atmosphere. Fourier transform infrared spectra in attenuated total reflection mode (ATR-FT-IR) were obtained using a Bruker VERTEX 70. The air permeability of the samples was conducted by YG (B) 461D digital fabric air permeability tester. The sample area setting ring was 20 cm^2 and two side pressure drop of fabric was 100 Pa. The surface composition of the corresponding fabrics was measured by X-ray photoelectron spectroscopy (XPS, Kratos Axis Supra) using $\text{Al K}\alpha$ radiation at a 90° take-off angle. All the binding energy values were calibrated using the reference peak of C1s at 284.6 eV. Spectra were evaluated using the Casa XPS software, version 2.3.15 from Casa Software Ltd. (United Kingdom). The symmetrical GL (30) line shape was applied, which consists of a Gaussian (70%) and a Lorentzian (30%) component.

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