Facile Fabrication of Non-fluorinated Durable Superhydrophobic Cotton Fabric

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Abstract: The superhydrophobic surface is easy to lose the superhydrophobicity in the practical applications, due to the fact that the micro- and nanoscale rough structure is easily damaged. Therefore, it is one of the challenges to endow solid surface with durable superhydrophobic property. In this work, the environment-friendly superhydrophobic cotton fabric was prepared by dip-coating methodology. Meanwhile, the cotton fabric was endowed with durable superhydrophobicity. Herein, the octadecylamine was utilized to reduce the surface free energy of cotton fabric. However, octadecylamine was only adsorbed on the fabric surface. Therefore, the hydrophobic resin was employed to supply enough adhesion force. As a result, the limitation that traditional methods frequently utilize additional nanoparticles to construct hierarchical rough surfaces was broken. The water contact angles of the coated cotton fabric at room temperature and 70 ^{o}C reached 154.4±0.6 ^{o} and 120±1.1 ^{o}, respectively. Moreover, the coated cotton fabric still reserved superhydrophobic property after 30th cycle's abrasion and exhibited excellent hydrophobicity after 500th cycle's abrasion. In addition, the surface free energy of original cotton fabric reduced by 54.7 % so that the coated cotton fabric had outstanding repellency to hydrochloric acid, sodium hydroxide solution, coffee and sodium chloride solution, respectively.

Keywords: Superhydrophobic, Mechanical abrasion, Octadecylamine, Hydrophobic resin, Surface free energy

Introduction

Inspired by lotus leaf, the surface wetting properties of materials has been rapidly developed in recent years [1,2]. The superhydrophobic surface has the characteristics of self-cleaning [3,4], anti-corrosion [5,6], anti-fouling [7], anti-bacterial [8-10] and anti-fog [11]. Therefore, it has high potential application value and a wide range of applications, such as energy transport [12], drug release [13], oil-water separation [14-16], anti-fouling [17], drag reduction [18], etc. Superhydrophobic surface means that the static contact angle of the solid surface with water is greater than 150 ^{o} and the sliding angle is lower than 10 ^{o}. Moreover, micro and nanoscale hierarchical roughness structure and low surface energy materials are two key factors to constructure the superhydrophobic surfaces [19]. At present, methods for preparing superhydrophobic surfaces were employed, including sol-gel method [20], template method [21], electrochemical oxidation [22], deposition method [23], self-assembly [24], phase separation method [25], plasma technology [26]. However, most of these methods have drawbacks. For instance, the preparation process is complicated and the equipment is expensive. Therefore, it is difficult to meet the demand of large-scale industrial production. Besides, the micro and nanoscale hierarchical roughness is easily damaged by the external forces, resulting in the loss of superhydrophobic properties of the solid surface [27,28]. As a result, it is one of the challenges to endow the surface of solid materials with stable and long-lasting superhydrophobicity.

In the 1950s, DuPont pioneered the use of fluorochemical finishes to prepare superhydrophobic or superoleophobic surfaces. However, fluorinated materials cannot be degraded by microorganisms in natural ecological environment and it is also bio-accumulative so that it has potential harm to human and the environment [29]. In addition, fluorine-containing superhydrophobic finishing agents are expensive and not suitable for industrial large-scale applications. Therefore, the development of environmentally friendly and cost-effective superhydrophobic surfaces is promising in the future [30,31].

Conventional methods for preparing superhydrophobic surfaces frequently utilize the extra nanoparticles to construct the hierarchical surface. For instance, Lakshmi *et al*. constructed the superhydrophobic surface by adding perfluoroalkyl methacrylic acid copolymer nanoparticles into the SiO$_2$ mixed sol-gel matrix [32]; Wu *et al*. prepared the self-healing superhydrophobic surface with antibacterial property by coating nano-silver particle [33]; Cai *et al*. employed R974 of industrial hydrophobic nanoparticle and epoxy resin to prepare the superhydrophobic coating [34]. Unfortunately, the interaction adhesion between nanoparticles and substrate surface is extremely weak so that the external abrasion makes nanoparticles gradually fall off, resulting in the hierarchical rough structure easily damaged. The substrate is completely immersed in a beaker containing the functional coating liquid. Subsequently, the substrate is taken out of the beaker and extruded by the padder under a certain pressure. This method is called dip-coating methodology. The merit of dip-coating is high production efficiency, simple operation and low coating liquid loss. Moreover, the dip-coating is suitable for fibers, fabrics, sheets, and complex equipment or electrical insulator materials. Therefore, it is a classic

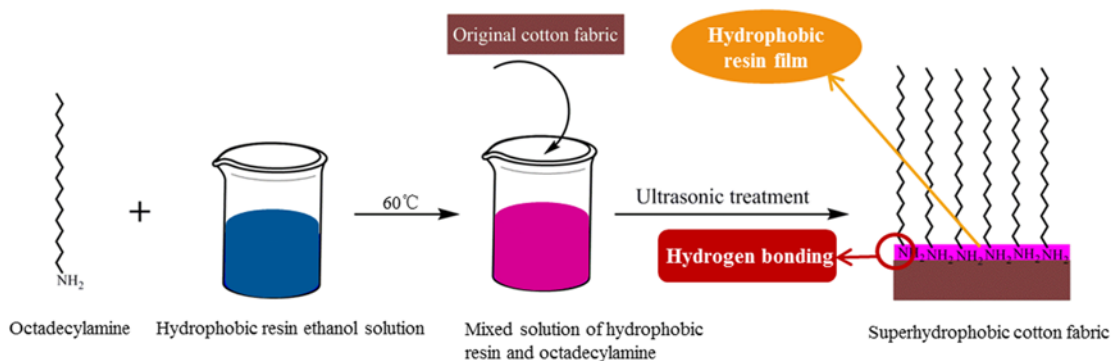*Corresponding author: zhangdan@jiangnan.edu.cn

Figure 1. Preparation process of superhydrophobic cotton fabric.

method for fabricating various multifunctional surfaces. For example, Lee *et al*. endowed the objects surface with multifunctional polymer coatings by simple dip-coating in an aqueous solution of dopamine [35]. Wang *et al*. synthesized boron nitride (BN) coatings on the surface of silicon carbide (SiC) fibers by dip-coating method [36].

Based on above analysis, an environmentally friendly superhydrophobic cotton fabric was fabricated. The preparation process of coated cotton fabric can be seen from Figure 1. First, based on the property that octadecylamine is able to self-aggregate at a high concentration, it was applied to the surface of the cotton fabric and formed a micro-nano layered structure. However, octadecylamine was only adsorbed on the fabric surface. Therefore, the hydrophobic resin was utilized as the interlayer so that the octadecylamine was firmly adhered onto the surface. As a result, the purpose of constructing the hierarchical rough surface structure and reducing surface free energy was achieved. Meanwhile, the hydrophobic resin could naturally solidify to form a hydrophobic film in air, thereby the hydrophobicity was enhanced and the weak interfacial adhesion between the low surface energy materials and the solid surfaces was solved. The superhydrophobicity of coated cotton fabric can be obtained from the Video-1 in the Supplementary Material. As a result, the contact angles of the coated cotton fabric at room temperature and 70 ^{o}C reached 154.4±0.6 ^{o} and 120±1.1 ^{o}, respectively. The contact angles at room temperature reached 151.5±0.4 ^{o} and 145.7± 0.8 ^{o} after 30th and 500th cycle's abrasion, respectively. Moreover, the surface free energy of cotton fabric was reduced by 54.7 % so that the coated cotton fabric had excellent repellency to hydrochloric acid, sodium hydroxide solution, coffee and sodium chloride solution, respectively.

Experimental

Materials

Cotton plain weave (yarn linear density: 20 tex×20 tex, warp density: 547/10 cm, weft density: 139/10 cm, weight:

114.2 g/m^2) was obtained from Wuxi First Cotton Textile Co. Ltd.. It was then impregnated with acetone and deionized water, and then dried at the temperature of 85 ^{o}C. Octadecylamine (purity, 97 %) was supplied by Jinan Chiyuan Chemical Co., Ltd.. Hydrophobic resin (SJ-35) was supplied by Sanjin Pigment of Zongyang Co., Ltd.. Absolute ethanol, hydrochloric acid, sodium hydroxide and sodium chloride were supplied by Sinopharm Chemical Reagent Co., Ltd..

Fabrication of Superhydrophobic Finishing Agent

The appropriate amount of hydrophobic resin, ODA and absolute ethanol were weighed into a beaker, and then the beaker was sealed with plastic wrap. Subsequently, the beaker was placed into a water bath at 60 ^{o}C until the ODA completely dissolved. The concentration of hydrophobic resin was 1.0 wt%, and the concentrations of ODA were 1.5 wt%, 2.0 wt%, 2.5 wt%, 3.0 wt%, 3.5 wt% and 4.0 wt%, respectively. The optimal concentration of ODA was investigated. Subsequently, a series of uniformly mixed finishing agents were prepared according to above method to investigate the optimal concentration of hydrophobic resin.

Preparation of Superhydrophobic Cotton Fabric

The clean cotton fabric was immersed in the mixture solution of hydrophobic resin and ODA for 15 min under ultrasonic treatment, and then placed in a blast drying oven at 60 ^{o}C. Meanwhile, a beaker containing deionized water was also placed in the oven to provide an environment with humidity. Eventually, the cotton fabric was dried for 40 min.

Characterizations

Infrared Spectra Measurements

The infrared spectra of the surfaces of the cotton fabric samples before and after finishing were measured by the attenuated total reflectance method using the infrared conversion spectrometer (Nicolet iS50, Thermo Fisher Scientific, America).

Surface Morphology of Cotton Fabric

The cotton fabric samples before and after finishing were

equilibrated in a desiccator at room temperature for 24 h. Subsequently, the 3 mm×3 mm samples were attached to the sample stage, vacuum-sprayed, and observed through using the scanning electron microscope (Su1510, Hitachi Co., Japan) at 20 kV.

Elements on the Surface of Cotton Fabric Measurements

The cotton fabric samples before and after finishing were equilibrated in a desiccator at room temperature for 24 h. Subsequently, the 3 mm×3 mm samples were placed on the sample stage, vacuum-sprayed, and the elements on the surfaces of the cotton fabric samples were detected using energy dispersive X-ray spectroscopy (EDS, Bruker, Germany) at 15 kV.

Calculation of the Surface Free Energy of Superhydrophobic Cotton Fabric

The surface free energy of superhydrophobic cotton fabric was calculated via the contact angle s of the tested liquids on the cotton fabric, equations (1) and (2) [37].

$$\gamma_x(1 + \cos\theta) = 2(\sqrt{\gamma_1^d \gamma_x^d} + \sqrt{\gamma_1^p \gamma_x^p}) \qquad (1)$$

$$\gamma_x = \gamma_x^d + \gamma_x^d \qquad (2)$$

where $\theta(^o)$ are the contact angles of the tested liquids on the cotton fabric, γ_x is the surface free energy of tested liquids, γ_x^d is the non-polar component, and γ_x^p is the polar component. The information of the tested liquids is listed in Table 1.

Contact Angles and Sliding Angles Measurements

The coated cotton fabric samples were flatly attached to the glass slide, and its static water contact angle was measured using the drop shape analyzer (JC2000DM, China). The droplets (5 μl) were contact with the cotton fabric for 10 s, the samples were photographed. In addition, 20 μl of the droplets volume was dropped on the surface of the cotton fabric samples, and then adjusted the tilt angle. When the droplets began to slide, the tilt angle value was the sliding angle. Each cotton fabric sample was measured at 5 different points and averaged.

Characterization of Friction Resistance of Cotton Fabric

The samples were cut into 200 mm×50 mm, and 50 mm× 50 mm square standard cotton fabric was warped on the friction head. The contact angles of the friction surface of the cotton fabric with water were measured after color fastness friction meter (Y(B)571-II, China) reciprocated rubbing 100 mm in the longitudinal direction of the samples

Table 1. Surface free energies of tested liquids (20 °C)

Tested liquids	Surface free energy	Non-polar component	Polar component
	γ_x (mJ/m^2)	γ_x^d (mJ/m^2)	γ_x^p (mJ/m^2)
Deionized water	72.8	21.8	51.0
Glycerol	64.0	34.0	30.0

for 20, 30, 50, 100, 300 and 500 times, respectively. Each sample was measured 5 times and averaged.

Breaking Strength Test of Cotton Fabric

The cotton fabric samples before and after finishing was cut into 30 cm×5 cm along the warp and weft direction, respectively. The breaking strength of cotton fabric samples were measured by fabric breaking strength machine (YG(B)026D-250, China).

Whiteness Test of Cotton Fabric

The cotton fabric sample was folded into 4 layers, and the whiteness of the cotton fabric sample before and after finishing was measured by automatic whiteness meter (WSD-III, China).

Results and Discussion

Controlled Factors in the Preparation Process of Superhydrophobic Cotton Fabric

Effect of ODA Concentration on the Surface Wettability of Cotton Fabric

Figure 2 shows that the concentration of ODA has great influence on the wetting property of the fabric surface. Previous reports have shown that ODA has a very low surface energy, and it is able to spontaneously aggregate to form a lamellar structure when the concentration reached a certain value [38], as shown in Figure 3(b). It was the lamellar structure significantly improved the roughness of the cotton fabric surface and reduced the surface free energy. However, ODA was only adsorbed on the fabric surface. Therefore, the hydrophobic resin was employed to supply enough adhesion. Hydrophobic resin has the strong adhesion property and it is able to firmly hold the ODA onto the cotton fabric surface. In addition, hydrophobic resin is able to naturally solidify in the environment with humidity to form a hydrophobic film. As shown in Figure 3(a), the position indicated by the arrows was obviously formed by the deposition of hydrophobic resin. The experimental tests
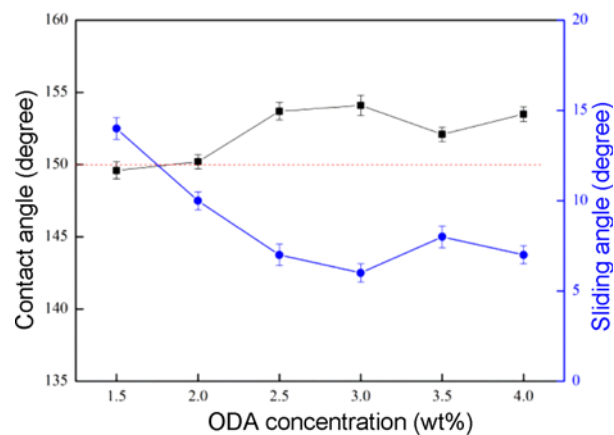

Figure 2. Water contact angles of the concentration of ODA coated on the cotton fabric as a function of coating amount.
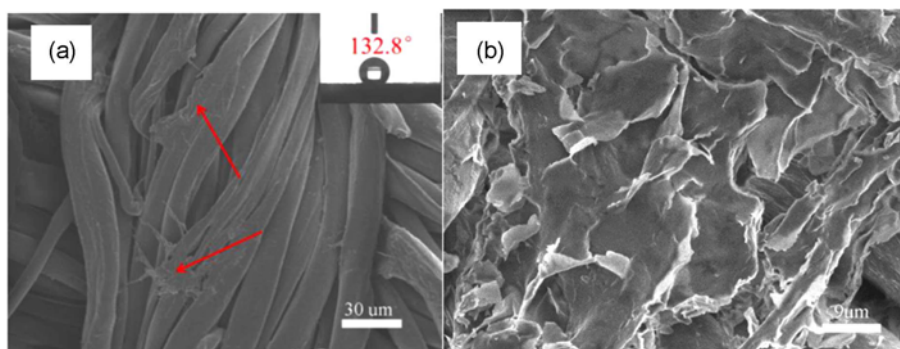
Figure 3. Field emission scanning electron microscopic images of the cotton fabric modified by hydrophobic resin of 2.5 wt% (a) and superhydrophobic cotton fabric (b).

demonstrated that the contact angle of the cotton fabric surface with water droplet was only 132.8 °. Fortunately, the original cotton fabric was coated by the mixed solution of hydrophobic resin and ODA, and the water contact angle reached 154.4±0.6 ° and the sliding angle was 6 °, when the concentration of ODA was 3.0 wt% (see Figure 2). With the increasing of ODA concentration, the hydrophobicity of the cotton fabric not significantly improved. Therefore, the optimal concentration of ODA was 3.0 wt%.

Effect of Hydrophobic Resin Concentration on the Surface Wettability of Cotton Fabric

With the increasing of hydrophobic resin concentration, more lamellar ODA was adhered on the cotton fabric surface so that the roughness was greatly improved. However, the stiffness of the cotton fabric was also promoted. The cotton fabric surface had excellent superhydrophobic property (see Figure 4), when the concentration of hydrophobic resin was 2.5 wt%. When the concentration of hydrophobic resin was more than 2.5 wt%, the water contact angle gradually decreased, due to the fact that the hydrophobic film reduced the roughness of the cotton fabric surface. Overall, the
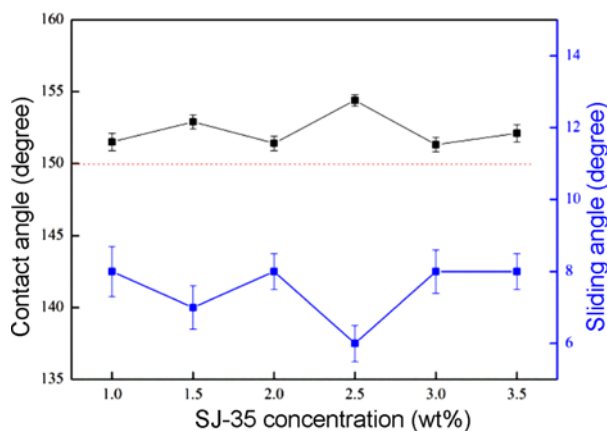
optimal concentration of hydrophobic resin was 2.5 wt%.

Infrared Spectra Analysis of Cotton Fabric Surface

Figure 5 reveals that the chemical structure of the coated cotton fabric surface has altered. The symmetric stretching vibration of -CH_3 and the asymmetric stretching vibration absorption peak of -CH_2 appeared at 2915 cm^{-1} and 2847 cm^{-1}, respectively. At 1567 cm^{-1}, a medium-intensity absorption peak composed of -CN stretching vibration and -NH bending vibration coupling appeared, indicating that ODA was successfully immobilized on the cotton fabric surface. In addition, a medium-intensity absorption peak and a weak absorption peak of -C=C- appeared at 1465 cm^{-1} and 820 cm^{-1}, respectively, demonstrating that hydrophobic resin was adhered on the cotton fabric surface.

Analysis of Surface Morphology of Cotton Fabric

Figure 6(a-f) illustrated that the surface roughness of superhydrophobic cotton fabric was remarkably improved.
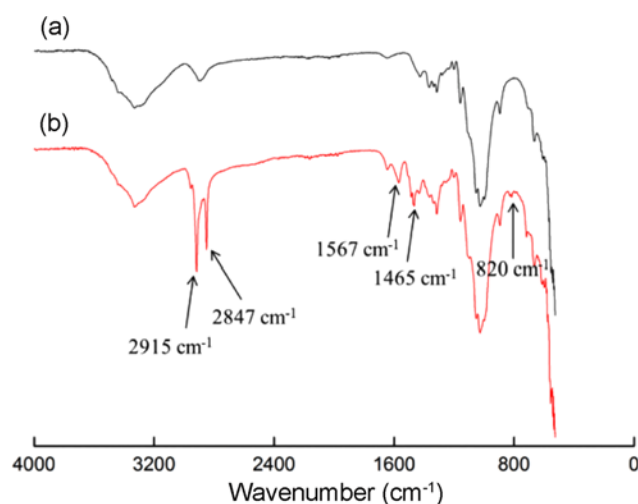

Figure 4. Water contact angles of the concentration of hydrophobic resin (SJ-35) coated on the cotton fabric as a function of coating amount.

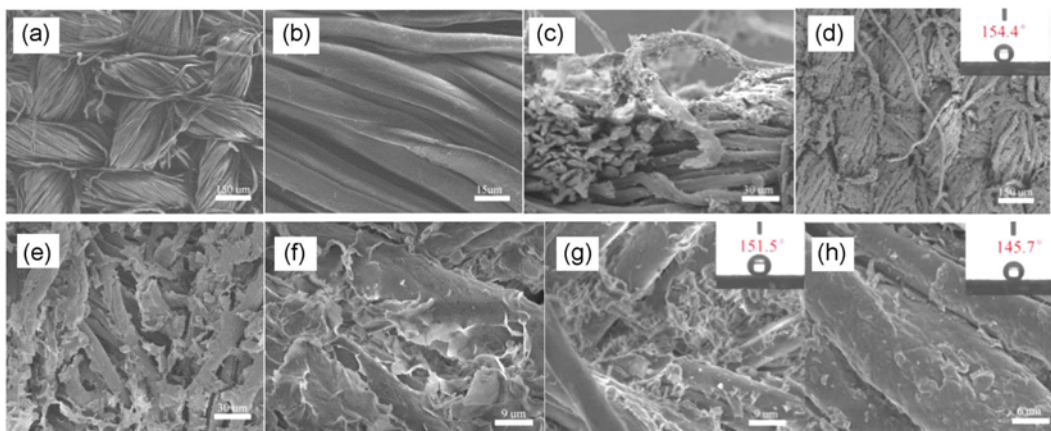**Figure 5.** Infrared spectra of original cotton fabrics (a) and superhydrophobic cotton fabrics (b).

Figure 6. Field emission scanning electron microscopic images of original cotton fabric (a, b), superhydrophobic cotton fabric (c, d, e, f), and superhydrophobic cotton fabric after 30th cycle's abrasion (g) and 500th cycle's abrasion (h).
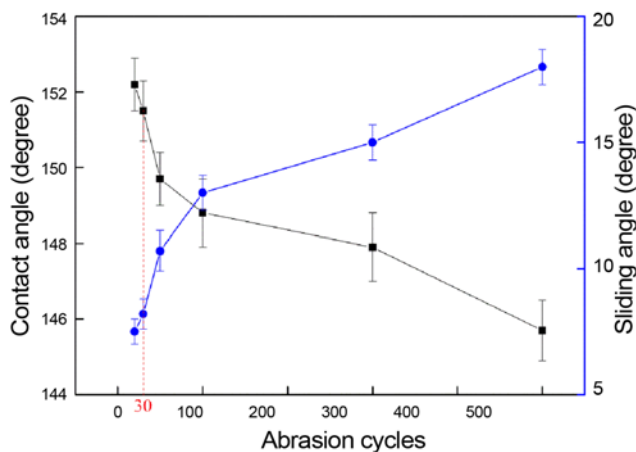

Figure 7. Water contact angle of superhydrophobic cotton fabric surface as a function of abrasion cycles.

Moreover, the cotton fibers surface and the fibers gaps were covered by hydrophobic resin and ODA. Meanwhile, the contact angle of the cotton fabric surface at room temperature reached 154.4±0.6 °, as shown in Figure 6(d-f). The contact angle was still greater than 150 ° as the coated cotton fabric was 30 times of mechanical abrasion, as shown in Figure 6(g) and Figure 7. However, the surface roughness of the coated cotton fabric significantly reduced after 500th cycle's abrasion, as seen from Figure 6(h). Meanwhile, the melted octadecylamine was adhered on the cotton fabric surface and the contact angle was only 145.7 °. However, the Figure 6(b), 6(f), 6(g) and 6(h) demonstrated that ODA molecules gradually fell off under mechanical abrasion, leading to the surface roughness decreased. Therefore, the contact area of water droplets with the cotton fabric surface was enlarged. Meanwhile, the adhesion between the cotton fabric surface and the water droplets was gradually improved as the ODA molecules fell off. Besides, the cotton fibers were gradually broken under the mechanical abrasion so that the fiber ends

were exposed on the surface. Finally, the contact angle was decreased and the sliding angle was increased (seen Figure 7).

Surface Elements Analysis of Cotton Fabric

Figure 8 reveals that the silicon was detected when cotton fabric was only treated with hydrophobic resin at the concentration of 2.5 wt%. Moreover, the nitrogen was detected when cotton fabric was coated with mixed solution of hydrophobic resin and ODA. In addition, the carbon content increased, while the oxygen content decreased (see Table 2).

Calculation of the Surface Free Energy of Superhydrophobic Cotton Fabric

The surface free energy (γ_x), non-polar component (γ_x^d) and polar component (γ_x^p) of superhydrophobic cotton fabric were calculated based on the data of Table 1 and the static contact angles of Table 3. The surface free energy of superhydrophobic cotton fabric was significantly reduced (see Table 3 and Figure 9) and it was less than a quarter of surface free energy of deionized water and glycerol, respectively. Therefore, the superhydrophobic cotton fabric had the outstanding repellency to the deionized water, coffee, hydrochloric acid, sodium chloride solution and sodium hydroxide solution, respectively (see Figure 10).

Repellency Performance of Cotton Fabric Surface to Water at Different Temperature

The superhydrophobicity of solid surface at room water has attracted much attention in recent years. However, the superhydrophobicity of solid surface to water at different temperature was rarely reported. Furthermore, the research of superhydrophobic cotton fabric to hot water can greatly broaden its range of application and protect the body from the hurt caused by hot water, especially children. Therefore, the research has potential value in daily life.

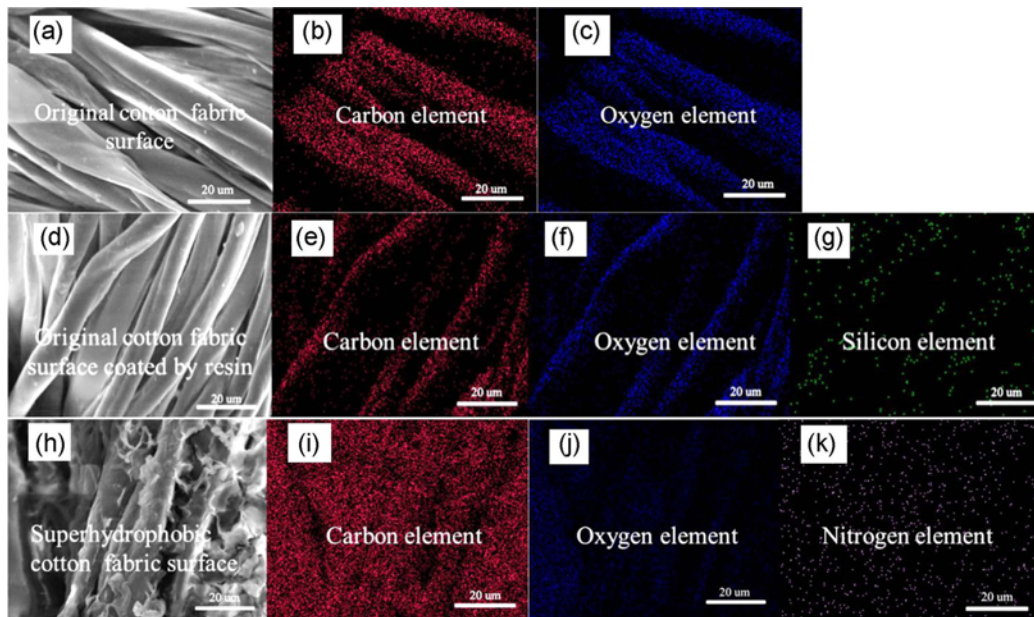The surface roughness of cotton fabric is mainly composed

Figure 8. Elements were detected on the surface of original cotton fabric (a, b, c), cotton fabric coated by hydrophobic resin (d, e, f, g) and superhydrophobic cotton fabric (h, i, j, k).

Table 2. Elements were detected on the surface of cotton fabric

Sample	Elements (weight/%)			
	C	O	Si	N
Original cotton fabrics	45.0	55.0	0	0
Cotton fabric coated by resin	46.8	53.0	0.2	0
Superhydrophobic cotton fabric	58.9	38.9	0	2.2

of self-aggregation of octadecylamine, and the melting temperature of octadecylamine is between 55 and 60 °C. Figure 11 shows that the contact angle was 128 ° when the hot water temperature reached 55 °C. In Figure 3, the water contact angle was 132.8 ° when the cotton fabric was treated only with the hydrophobic resin. The above analysis proved that octadecylamine had begun to melt at 55 °C, resulting in a decrease in the surface roughness of the cotton fabric. The contact angle was 124 °which was lower than 128 ° when the water temperature was 60 °C. It demonstrated that the octadecylamine greatly melted at 60 °C, resulting in a significant reduction in the surface roughness. According to the Table 4, the slope values ($|K|$) of the contact angle drop
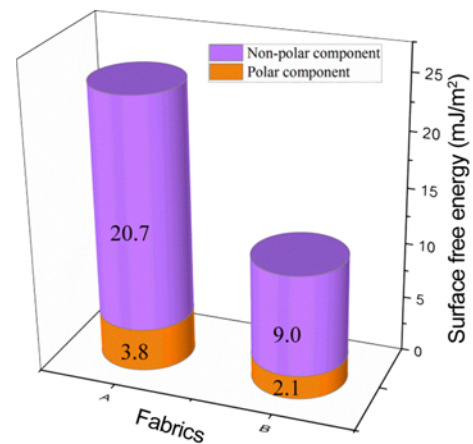

Figure 9. Surface free energy (a) of original cotton fabric (A) and superhydrophobic cotton fabric (B).

were 0.87, 0.80 and 0.40, when the hot water temperature was between 25-55 °C, 55-60 °C and 60-70 °C, respectively. Therefore, it indicated that the repellency of the coated cotton fabric surface to hot water was mainly provided by

Table 3. Static contact angles and surface free energy of cotton fabric samples

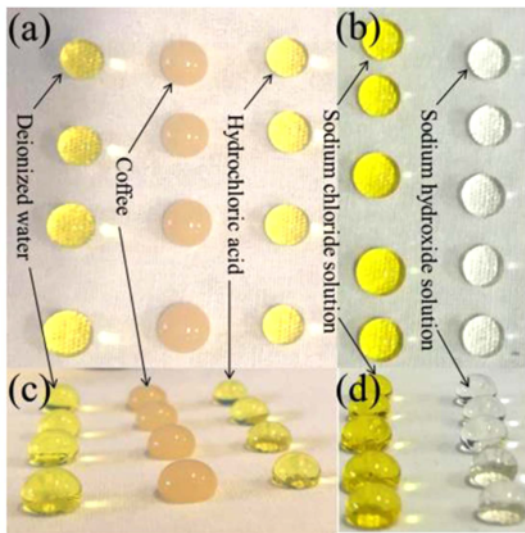| Sample	Static contact angles (θ/°)		γ_x (mJ/m^2)	γ_x^d (mJ/m^2)	γ_x^p (mJ/m^2)
	Deionized water	Glycerol			
Original cotton fabric	0	0	24.5	20.7	3.8
Superhydrophobic cotton fabric	154.4	135.0	11.1	9.0	2.1
Variation	+154.4	+135.0	-54.7 %	-85.5 %	-44.7 %

Figure 10. Liquid droplets (50 u*l*) on the surface of superhydrophobic cotton fabric. The samples were deionized water, coffee (0.5 wt%, Alicafe), hydrochloric acid (1 mol/*l*), sodium chloride solution (3.5 wt%) and sodium hydroxide solution (0.1 mol/*l*), respectively (b).
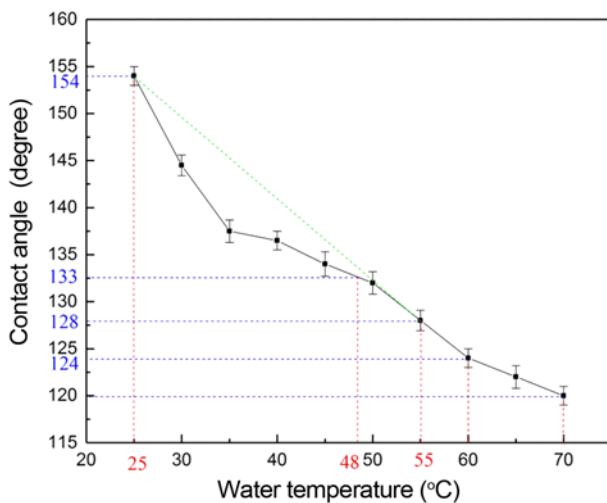

Figure 11. Variations in the contact angle with the temperature of water droplets on the cotton fabric surfaces.

Table 4. $|K|$ value at different temperature of water

	Water temperature ($^{\circ}$C)	$|K|$
1	25-55	0.87
2	55-60	0.80
3	60-70	0.40
4	25-48	0.91
5	48-55	0.71
6	48-60	0.75

Table 5. Physical and mechanical properties of cotton fabric samples

Sample	Whiteness (%)	Breaking strength (N)	
		Warp	Weft
Original cotton fabric	94.0±1.8	630.9±8.0	336.0±6.2
Coated cotton fabric	95.3±1.7	591.6±8.3	308.1±5.4

hydrophobic resin, when the water temperature was between 60 and 70 $^{\circ}$C. Besides, the water at 48 $^{\circ}$C was difficult to adhere to the surface of coated cotton fabric. For more details, please refer to Video-2 in the Supplementary Material. Subsequently, the slope values of the contact angle drop was calculated and it reached 0.91, 0.71 and 0.75 when the hot water temperature was between 25 and 48 $^{\circ}$C, 48 to 55 $^{\circ}$C and 55 to 60 $^{\circ}$C, respectively. As a result, when the water temperature was between 48 and 55 $^{\circ}$C, the change of the contact angle was slight, indicating that the cotton fabric had the repellency to water at 55 $^{\circ}$C. In addition, the coated cotton fabric was still great resistant to water at 70 $^{\circ}$C and the range of applications of cotton fabric was greatly enlarged.

Analysis of Physical and Mechanical Properties of Cotton Fabric

Table 5 shows that the warp and weft breaking strength of the coated cotton fabric were decreased by 6.2 % and 8.3 %, respectively. However, the whiteness was increased by 1.3 %. Because ethanol had some etching effect to cotton fibers and caused the loss of cotton fiber strength. Meanwhile, hydrophobic resin was fully covered between the gaps of cotton fibers so that the interaction force between the individual fibers was strengthened. However, the loss of cotton fibers strength by etching effect was higher than the interaction adhesion of hydrophobic resin to cotton fibers. As a result, the breaking strength of the coated cotton fabric was reduced. In addition, the hydrophobic film formed on the surface of the cotton fabric was colorless, and the lamellar structure formed by ODA was white. Therefore, the whiteness was slightly increased.

Conclusion

In this work, the durable superhydrophobic cotton fabric is successfully fabricated by one step. The coated cotton fabric not only has the superhydrophobic property at room temperature, but also has the excellent repellency property at 70 $^{\circ}$C, thereby the research on superhydrophobic cotton fabric to hot water has been expanded. Moreover, the surface free energy of cotton fabric is significantly reduced by 54.7 % after finishing the coating. As a result, the coated cotton fabric has outstanding repellency to hydrochloric acid, sodium hydroxide solution, coffee and sodium chloride solution, respectively.

Owning to the strong adhesion from hydrophobic resin,

octadecylamine is firmly adhered onto the cotton fabric surface, indicating that the weak interfacial adhesion between low surface energy materials and solid surfaces is overcome. Meanwhile, the application of extra nanoparticles is avoided to construct the micro and nanoscale hierarchical surfaces. Due to the property of a hydrophobic film naturally solidify from hydrophobic resin, the superhydrophobic finishing agent is promising to endow other substrates (not limited by its shape and surface structure) with super-hydrophobic property, including ceramic, steel, plastic and glass. Furthermore, the overall preparation process is facile, environmental friendly and cost-effective.

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