

# Robust and Durable Superhydrophobic Cotton Fabrics for Oil/Water Separation

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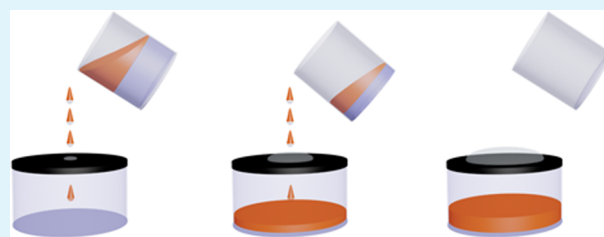
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## S Supporting Information

**ABSTRACT:** By introducing the incorporation of polyaniline and fluorinated alkyl silane to the cotton fabric via a facile vapor phase deposition process, the fabric surface possessed superhydrophobicity with the water contact angle of 156° and superoleophilicity with the oil contact angle of 0°. The as-prepared fabric can be applied as effective materials for the separation of water and oil mixture with separation efficiency as high as 97.8%. Compared with other materials for oil/water separation, the reported process was simple, time-saving, and repeatable for at least 30 times.

Moreover, the obtained fabric kept stable superhydrophobicity and high separation efficiency under extreme environment conditions of high temperature, high humidity, strong acidic or alkaline solutions, and mechanical forces. Therefore, this reported fabric has the advantages of scalable fabrication, high separation efficiency, stable recyclability, and excellent durability, exhibiting the strong potential for industrial production.

**KEYWORDS:** superhydrophobicity, cotton fabric, oil/water separation, recyclability, durability



## 1. INTRODUCTION

Superhydrophobic surfaces, characterized by a high water contact angle ( $>150^\circ$ ), have attracted worldwide attention for their potential applications, including antifouling, microfluidics, energy conversion, and water treatment.<sup>1–5</sup> With the increase of industrial oily wastewater and oil spill accidents, the development of superhydrophobic materials for selective absorption or direct separation of oil from water is greatly desired, due to their high separation efficiency and stable recyclability.<sup>6–8</sup> A variety of routes have been developed to fabricate superhydrophobic materials for separation of water and oil, including chemical etching,<sup>9</sup> electrospinning techniques,<sup>10</sup> hydrothermal methods,<sup>11</sup> self-assembly processes,<sup>12,13</sup> and others.<sup>14–16</sup> All these approaches can be categorized as the following strategies: creating micro/nanostructures on hydrophobic substrates or chemically modifying a micro/nanostructured surface with materials of low surface energy. Although great successes have been made in the preparation of superhydrophobic surfaces, challenges still exist for one-step production technique that is realizable for industrial use, relatively mild processing conditions, and large scale fabrication on diverse substrates.<sup>17</sup>

Most superhydrophobic surfaces have poor durability, especially when they are exposed to an environment of high temperature, strong light, external forces, or corrosive substances.<sup>18,19</sup> These extreme conditions may cause the loss of surface superhydrophobicity permanently, which will

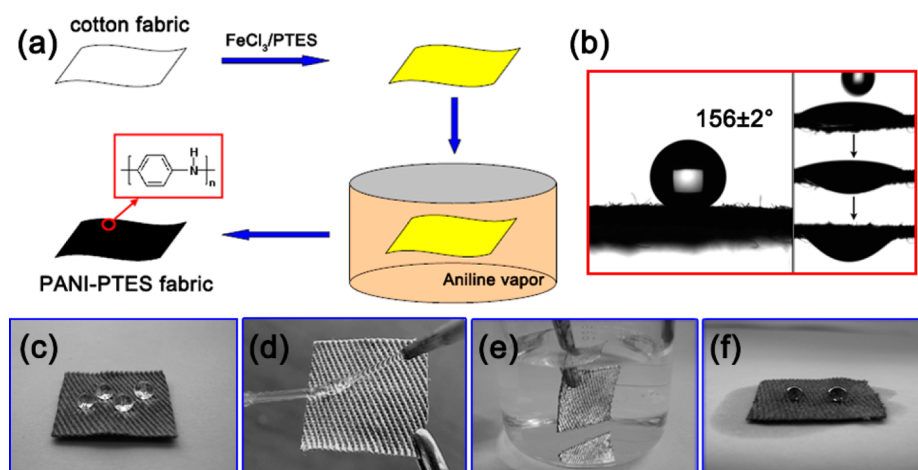
seriously hinder the practical applications of superhydrophobic materials. Recently, a large number of studies have been made to improve the durability of synthetic superhydrophobic surfaces.<sup>20–22</sup> For instance, Lin et al. prepared a self-healing superhydrophobic and superoleophobic surface, which showed excellent durability to acid, UV light, machine wash, and abrasion.<sup>23</sup> Seeger et al. obtained superhydrophobic textiles with long-term water resistance as well as durability upon machine washing by applying the silicone nanofilament coating onto the textile substrates.<sup>24</sup> However, to the best of our knowledge, no work has been reported about the environmental durability of superhydrophobic materials for oil/water separation. The lifetime of these separation devices can be largely extended if they are durable toward severe environmental conditions.

Herein, we report a single-step fabrication method for creating robust superhydrophobic cotton fabrics by in situ vapor phase deposition (in contrast to solution-based method<sup>25,26</sup>). A composite coating was obtained on the textile with particular wettability, repelling water completely while allowing the permeation of oil. Since water stayed exclusively on the textile surface, oil could be successfully separated from the mixture. The as-prepared fabric maintained high separation

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**Figure 1.** (a) Schematic of the fabrication of superhydrophobic cotton fabrics through vapor phase deposition process. (b) Water droplet sitting on the superhydrophobic cotton fabric and hexadecane droplet spreading on and permeating through the fabric. (c) Water droplets on the coated textile, (d) a jet of water bouncing off the surface, (e) the textile immersed in water by an external force, (f) water droplets on the oil contaminated textile.

efficiency and stable recyclability during the repeated separating experiments. Furthermore, we have investigated, for the first time, the environmental durability of the material for oil/water separation. Such superhydrophobic fabrics could withstand various harsh conditions such as high temperature, high humidity, acidic or alkaline solutions, and mechanical forces. So, our approach described here is versatile, simple, and can be applied to create functionalized materials with durable superhydrophobicity under extremely practical conditions.

## 2. EXPERIMENTAL SECTION

**2.1. Materials.** Raw cotton fabrics were purchased from a local fabric store. Aniline (C<sub>6</sub>H<sub>7</sub>N) was purchased from Xilong Chemicals Co., Ltd., China. 1H,1H,2H,2H-perfluorooctyltriethoxysilane (C<sub>14</sub>H<sub>19</sub>F<sub>13</sub>O<sub>3</sub>Si) (PTES) was obtained from Sigma-Aldrich. FeCl<sub>3</sub>·6H<sub>2</sub>O was purchased from Tianjin Kermel Chemical Reagent Co., Ltd., China. All other chemicals were analytical-grade reagents and used as received.

**2.2. Sample Preparation.** The preparation process of polyaniline (PANI) coated cotton fabric via vapor phase deposition is similar to the previous reports.<sup>27,28</sup> First, a piece of 12 × 12 cm<sup>2</sup> cotton fabric was cleaned with acetone and deionized water to remove possible impurities. 0.7 g FeCl<sub>3</sub>·6H<sub>2</sub>O and 0.14 g PTES were dissolved in 14 mL ethanol to form a homogeneous orange solution. The clean fabric was then steeped in the solution for approximately 2 min, removed, and dried at room temperature. The vapor phase deposition of aniline was performed by placing the treated fabric into a small chamber filled with aniline vapor at 70 °C for 30 min. After that, the fabric was thoroughly rinsed in anhydrous ethanol and deionized water to get rid of all the residuals. Finally, the fabric was dried in an oven at 100 °C for 30 min to remove the solvent. The obtained fabric was cut into pieces of different sizes according to different characterization methods.

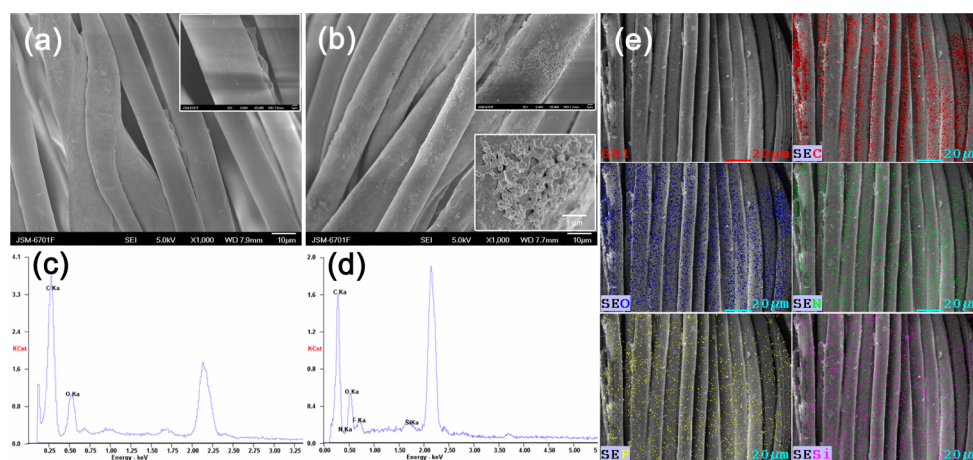
**2.3. Characterization.** Static and dynamic contact angle measurements were carried out using a Krüss DSA 100 (Krüss Company, Ltd., Germany) apparatus. An average of five measurements was made to determine the surface wettability, and the volume of the individual droplet was 5 μL. The surface morphology of the cotton fabric was observed by a field-emission scanning electron microscope (JEOL JSM-6701F FESEM). X-ray photoelectron spectroscopy (XPS) characterization was conducted on a PHI-5702 electron spectrometer using an Al Kα line excitation source with the reference of C 1s at 285.0 eV. The takeoff angle of XPS was 90°. The conductivity of the fabric at room temperature was measured by a standard four-probe method, using a SZT-2B four point probe tester (Suzhou Tongchuang

Electronic Co., Ltd., China). The high temperature and high humidity test was carried out in an alternating temperature and humidity test chamber (Wuxi Jinhua Testing Equipment Co., Ltd., China). The axial tensile force was provided by an electronic universal material testing machine WDW-200 (Shenzhen Sans Testing Machine Co., Ltd., China), and the force was kept for 30 s for each test.

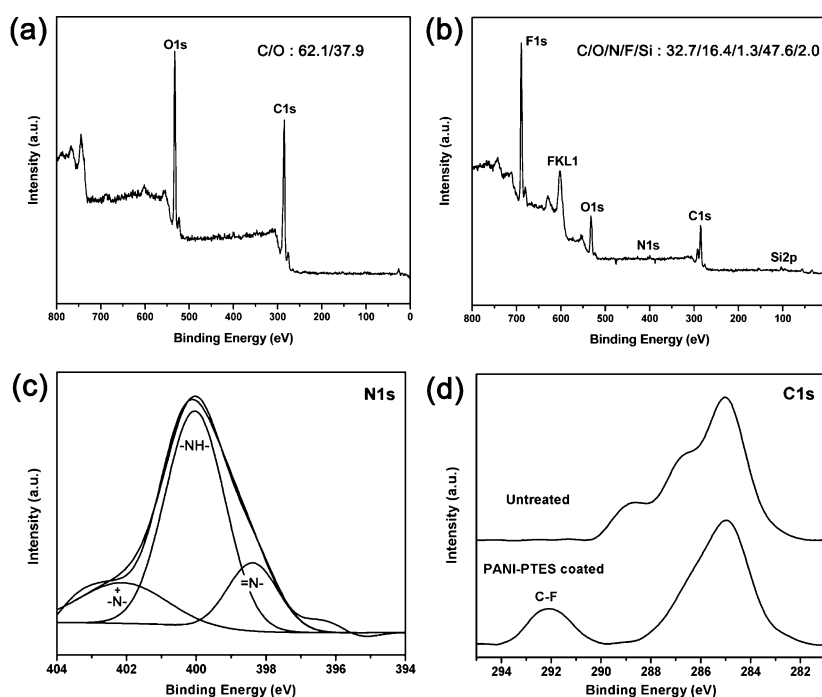
## 3. RESULTS AND DISCUSSION

The procedure for preparing PANI-PTES cotton fabrics through the traditional vapor phase deposition as described in the Experimental Section is illustrated in Figure 1a. A cotton fabric specimen was treated by the FeCl<sub>3</sub>/PTES solution, then exposed to the aniline vapor, and finally rinsed and dried. This obtained fabric was demonstrated to be superhydrophobic with a water contact angle of 156 ± 2°, as shown in Figure 1b. By contrast, hexadecane, a liquid with low surface tension ( $\gamma_{lv} = 27.5$  mN/m), spread quickly on the coated fabric and permeated thoroughly within 10 s, exhibiting the superoleophilicity. This is because the surface tension of oil is commonly much smaller than that of water. When the surface tension of the solid substrate lies between those of water and oil, hydrophobicity and oleophilicity can be realized. Additionally, a bright and reflective surface could be observed underneath the water droplet in Figure 1c, which was a signature of trapped air and the establishment of a composite solid–liquid–air interface. The corresponding wetting regime was in the Cassie–Baxter state.<sup>29</sup> The water jet from a pipet could bounce off the fabric surface without leaving a trace, indicating the weak interaction between water and the surface (Figure 1d). Upon immersion into water, the fabric surface appeared mirror-like due to the existence of trapped air between water and the superhydrophobic fabric (Figure 1e). Most of the area beneath water was the liquid/air interface and the ratio of liquid/solid interface was quite small. After the textile was taken out of water, its surface remained completely dry. When hexadecane droplets were dripped onto the textile surface in Figure 1f, they were rapidly absorbed and the position contaminated by oil still showed repulsion to water. All the results mentioned displayed a stable superhydrophobicity of the PANI-PTES fabric.

The superhydrophobic property of the as-prepared fabric results from both the surface morphological structure and the



**Figure 2.** FESEM images of the pristine (a) and the superhydrophobic (b) textile surfaces. Insets are the corresponding high magnification FESEM images. EDS spectra of the pristine (c) and the superhydrophobic (d) textile surfaces. (e) FESEM-EDS mapping of the coated fabric.



**Figure 3.** XPS survey spectra of the pristine (a) and the coated (b) fabrics. (c) N1s high resolution spectrum of the coated fabric. (d) C1s high resolution spectra of the pristine and the coated fabrics.

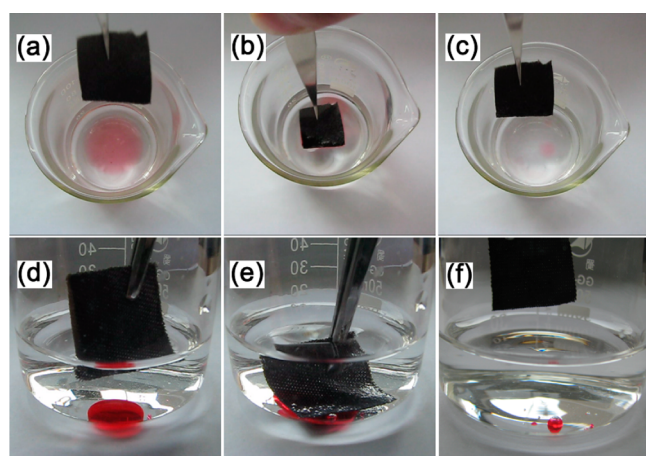
chemical composition.<sup>30</sup> Figure 2a presents the representative FESEM image of the pristine cotton fabric possessing uniformly oriented three-dimensional microfibers with an average diameter of 13  $\mu\text{m}$ . The surface of the individual fiber is smooth, as shown in the inset image of Figure 2a. After the in situ vapor phase deposition at 70  $^{\circ}\text{C}$ , a compact coating with a number of nanoscale granules randomly distributed on the fiber surface (Figure 2b), which suggested the accomplishment of aniline polymerization on the cotton fabric. The particulate morphology provided roughness at the nanoscale to complement the microscale roughness inherent in the fabric weave. This hierarchical micro- and nanoscale roughness of the coated fabric surface is an essential necessity to obtain the superhydrophobicity. Energy dispersive spectroscopy (EDS) analysis has been performed to examine the chemical composition of the textile. As shown in Figure 2c, only peaks of C and O are detected on the pristine fabric, and no other impurities can be

observed (The signal of Au element comes from the gold spray). However, on the coated fabric, N, F, and Si can be observed besides C and O (Figure 2d). The element N is mainly from PANI, while F and Si are from PTES. From the FESEM-EDS elemental mapping of C, O, N, F, and Si in Figure 2e, all the five elements are distributed on the entire fabric surface, suggesting the uniform coverage of PANI-PTES coating on the treated fabric.

XPS measurement was used to further investigate the surface chemical composition of the cotton fabrics. For the pristine fabric, only peaks corresponding to C and O were observed (Figure 3a), which was in good agreement with the EDS results. After the deposition process, new peaks with binding energies of 689, 601, 400, and 104 eV appeared in Figure 3b, which were attributed to F 1s, F KL1, N 1s, and Si 2p, respectively. The ratio of C/O/N/F/Si was 32.7/16.4/1.3/47.6/2.0%. Three deconvoluted peaks of N1s high resolution

spectrum appearing around 398, 400, and 402 eV, were found for the coated fabric (Figure 3c), which belonged to characteristic components of quinonoid imine ( $=N-$ ), benzenoid amine ( $-NH-$ ) and positively charged nitrogen ( $-N^+$ ) of PANI,<sup>31</sup> respectively. The C1s high resolution spectrum had new peaks around 292 eV (Figure 3d), typically from the fluorinated carbon moieties of PTES. This measurement confirmed the existence of PANI and PTES on the coated fabric, which might be linked by a large number of strong hydrogen bonds. PTES has a very low surface energy due to its high content of fluorinated carbon groups. Accordingly, the combination of extremely low surface energy and the hierarchical roughness endows the fabric with stable superhydrophobicity. It is well-known that the surface resistance of the raw fabric is infinity due to its insulation. However, in the case of the PANI coated fabric, the resistance was measured to be around  $0.4 \text{ K}\Omega\text{-cm}^{-1}$ . This indicated that PANI formed a continuous conductive network on the cotton fibers, and the conductive fabrics might find their potential applications as electromagnetic interference shielding materials in the future.

As mentioned, the coated fabric showed simultaneous superhydrophobicity and superoleophilicity, which made it very promising as the material for removal of oil from water. Hexadecane dyed with oil red was dropped onto the middle of the water surface to form a thin layer, and then a piece of coated cotton textile was brought into contact with the hexadecane layer. The hexadecane was fully absorbed within a few seconds, leaving a transparent region on the water surface (Figure 4a–c). In addition, the absorption of chloroform from

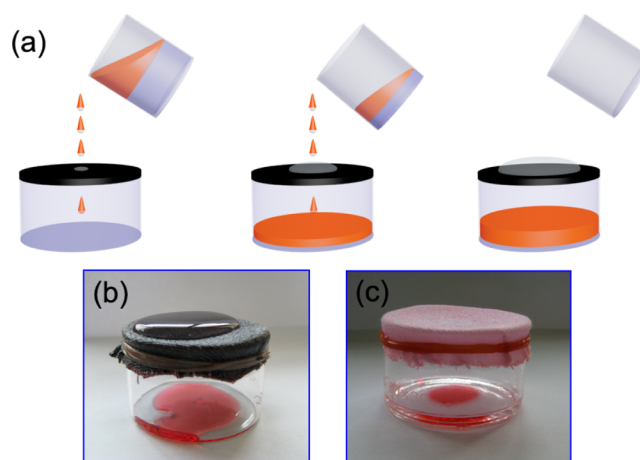


**Figure 4.** Images of the removal processes of hexadecane (a–c) and chloroform (d–f) from water using the coated fabric.

water was carried out to verify the use of the fabric with organic chemicals that had higher densities than water. When the superhydrophobic textile was inserted into water to approach chloroform, the chloroform droplet could be immediately sucked up by the textile underwater (Figure 4d–f). Most of the chloroform droplet could be removed by taking out the absorbed textile from water. Nevertheless, due to the inherent poor uptake capacity of cotton textile, the application of this superhydrophobic textile for the absorption of oil from water might be restricted.

Only absorbing oil from water is not enough for the solutions to oil spill accidents, and it is still necessary to separate oil–water mixtures toward preparing the oil skimmer and separation devices.<sup>32</sup> When a mixture of hexadecane and

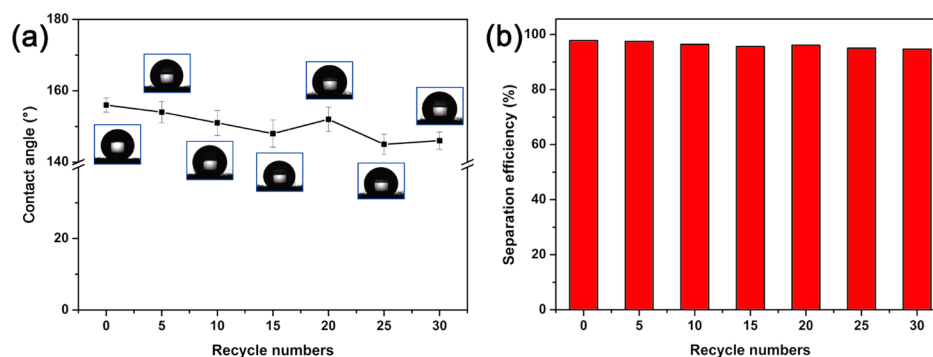
water was poured onto the PANI-PTES cotton fabric, only hexadecane could pass through the fabric and drop into the container below. Water was retained above the surface thanks to the superhydrophobic and low water adhesion properties of the fabric (see Figure 5a and Movie S1, Supporting



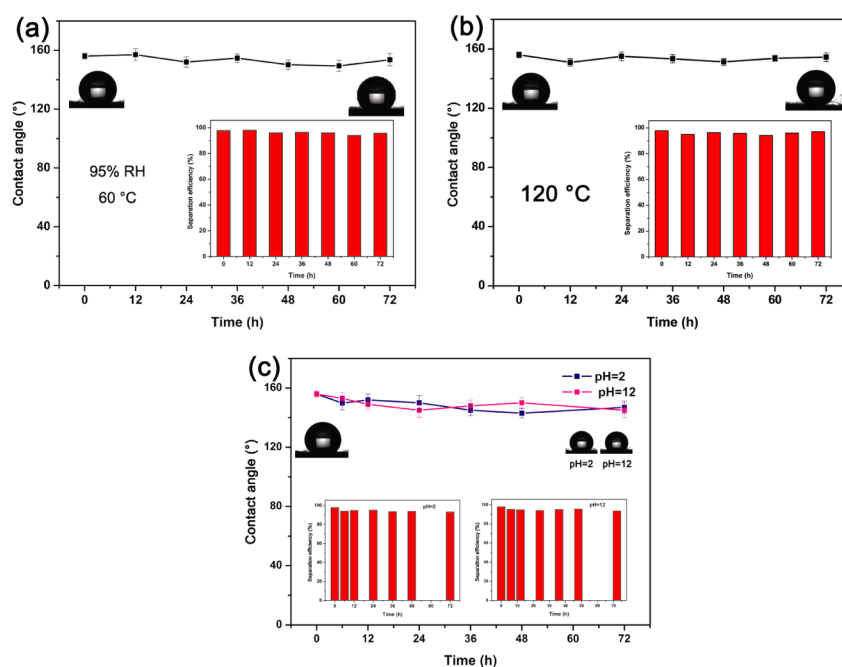
**Figure 5.** (a) Schematic representation of the separation process for hexadecane–water mixture using the superhydrophobic cotton fabric. Images after pouring the hexadecane–water mixture onto the superhydrophobic fabric (b) and the raw fabric (c).

Information). This result implied that the coated fabric could separate oil–water mixtures with a high oil ratio by a simple filtering method. The separation efficiency of the textile was further investigated. Separation efficiency was given by the ratio between the weight of water collected and that initially added to the mixture.<sup>33</sup> The separation efficiency of the fabricated superhydrophobic textile was calculated up to 97.8% for the hexadecane–water mixture with a volume ratio of 1:1. On the contrary, when the mixture was poured onto the raw fabric, hexadecane and water could both penetrate through the fabric (Figure 5c). Hence, our superhydrophobic fabrics could be utilized as filter membranes to realize highly effective separation of oil–water mixtures.

As important criteria for practical applications, the recyclability and durability of the obtained superhydrophobic cotton textile were also evaluated. After the oil/water separation experiment, the contaminated textile was rinsed thoroughly by alcohol and water to remove the absorbed oil. Subsequently, the clean textile was dried in an oven at  $70^\circ\text{C}$  for half an hour, and the superhydrophobicity could be easily recovered. This separation and recovery process could be repeated for at least 30 times. The line in Figure 6a shows the relationship between the surface wettability and the recycle numbers: the water contact angle on the fabric decreased slowly from  $156^\circ$  to  $146^\circ$  after 30 separation cycles, which might be caused by the residual oil adhered to the textile. The separation efficiency for the oil–water mixture varied slightly during the repeated experiments and always stayed above 94% (Figure 6b), which indicated good recyclability of our material. FESEM image of the coated fabric after 30 cycles of oil/water separation showed that the hierarchical nanostructure was mostly preserved (Figure S1a, Supporting Information), which was of great importance for the fabric to maintain long-term superhydrophobicity. XPS spectrum revealed that the F element could be detected from the fabric even after 30 separation cycles (Figure S1b, Supporting Information). A



**Figure 6.** Variation of the water contact angle (a) and the separation efficiency (b) of the coated fabric versus the recycle numbers of oil/water separation.



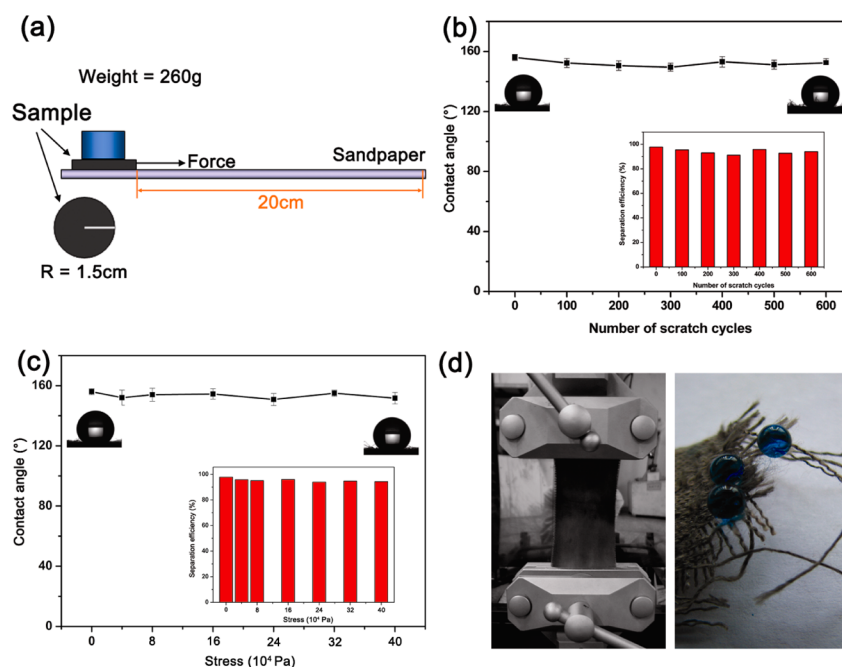
**Figure 7.** Variation of water contact angles on the coated fabric (a) under 95% RH at 60 °C, (b) at 120 °C, (c) exposed to acidic and alkaline solutions. Insets are the corresponding separation efficiencies for the oil–water mixture.

quantitative analysis of XPS spectrum indicated that atomic percentage of the F element slightly decreased. However, PTES still remained on the fabric surface, maintaining the superhydrophobicity and high separation efficiency of the fabric.

Evaluation of the durability was conducted under various rigorous conditions. When the coated fabric was placed in an environment with a temperature of 60 °C and a relative humidity (RH) of 95% for 72 h, its contact angle for water remained unchanged within experimental error and the separation efficiency for the oil–water mixture was almost constant (Figure 7a). Similarly, the fabric did not lose superhydrophobicity, and the separation efficiency remained unaffected after exposure to an extremely hot environment at 120 °C for 72 h (Figure 7b). Thus, the coated fabric shows rather stable superhydrophobicity toward high environment temperature and high humidity. The chemical durability of the coated fabric was studied by immersing them in strong acidic or alkaline solutions. As shown in Figure 7c, water contact angles were in the range 143–156° when the fabric was immersed into the strong acidic (pH = 2) or alkaline (pH = 12) solutions for 72 h. Even after attack by strong acid or alkali, our fabric was

still capable of effectively separating the oil–water mixture. The surface morphology and components of the fabric were all maintained after the chemical treatment as shown in Figure S4 and S5, Supporting Information. These results demonstrate that the obtained superhydrophobic surface is tolerant to acidic and alkaline conditions.

To check the robustness of the fabric against mechanical forces, an abrasion test and a stretch test have been performed. Figure 8a illustrates the method of the abrasion test: 800 mesh sandpaper served as an abrasion surface. The coated fabric was subjected to a 260 g weight and was kept in close contact with the sandpaper. Then the fabric sample was dragged in one direction with a speed and abrasion length of 4 cm/s<sup>-1</sup> and 20 cm, respectively. It was found that the water contact angle was higher than 150° and the separation efficiency remained above 93% after 600 scratch cycles (Figure 8b). These results indicate that the coated fabric has excellent durability against abrasion. Figure 8c displays the effect of tensile forces on the property of the coated fabric. It is clear that the fabric preserves the superhydrophobic property and high separation efficiency throughout the strain levels investigated. When the stress



**Figure 8.** (a) Schematic illustration of the methodology of the abrasion test. The variation of water contact angles of the coated fabric (b) scratched by the sandpaper, and (c) stretched by axial tensile forces. Insets are the corresponding separation efficiencies for the oil–water mixture. (d) Images of the stretched textile (left) and water droplets on the broken yarns (right).

caused by the axial tensile force reaches  $4.8 \times 10^5$  Pa, the fabric specimen suffers from severe fracture. Interestingly, the broken cotton yarns sustain the superhydrophobicity (Figure 8d). That is because the microfibers of the textile are coated with PANI and PTES, including those deeply imbedded inside, which indicates that the mobility of the reactive precursors in the gas phase is sufficient to penetrate into the three-dimensional textile structure. Mechanical stretch would not destroy the microstructure and surface component of the fabric and thus hardly affects the hydrophobicity. It is crucial to design and fabricate robust superhydrophobic materials for oil/water separation, especially durable under the extreme environment conditions such as high temperature, humid atmospheres, corrosive substance, mechanical forces, and so forth. This can greatly extend the application of the separation devices and, more importantly, inspire new attention to equipment design and mechanism revolution.

#### 4. CONCLUSIONS

In summary, we have demonstrated a facile and versatile approach to prepare superhydrophobic and superoleophilic cotton fabrics for oil/water separation. In the coating process, each individual textile fiber is coated with a compact layer of hydrophobic nanostructure. The coated fabric can effectively separate the oil–water mixture with high separation efficiency, which is up to 97.8%. This process can be repeated for at least 30 times with stable superhydrophobicity and constant separation efficiency. Besides, the obtained fabric can withstand the severe environment conditions such as high temperature, humid atmospheres, corrosive substance, and mechanical forces. Thus, our functionalized fabrics are expected to become sustainable and highly effective materials for oil-spill cleanup, as the materials can be recycled many times with remarkable durability.

#### ■ ASSOCIATED CONTENT

##### Supporting Information

FESEM images, EDS results, and XPS spectra of the superhydrophobic cotton fabrics after the repeated oil/water separation for 30 times and under various severe environment conditions, and a video of the separation procedure using the superhydrophobic fabric. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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##### Notes

The authors declare no competing financial interest.

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