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Research Article

Study on the Cotton Fabrics with Photoinduced Reversibly Switchable Wettability

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Superhydrophobic cotton fabric with photoinduced reversibly switchable wettability was prepared by a coating of the hydrophobic copolymer and α-Fe_2O_3 nanoparticles. The surface morphology of the fabric was observed by scanning electron microscope (SEM). The wettability of the surface was tested under UV illumination and after storage in the dark. The chemical composition of the cotton fabric surfaces before and after UV illumination was analyzed using an X-ray photoelectron spectroscope (XPS) and FTIR. The experimental results showed that the prepared cotton fabric exhibited the excellent superhydrophobic property with a contact angle (CA) of $157.3 \pm 2.9^{\circ}$, and became superhydrophilic after UV illumination for 64 h. The surface wettability reverted back to its initial superhydrophobic state after being stored in the dark for 30 d. Based on the XPS and FTIR analyses, the possible mechanism was discussed, and the switchable wettability was caused by the content change of the hydroxyl groups on the α-Fe_2O_3 surface. Moreover, the superhydrophobic cotton fabric also became superhydrophilic after sunlight illumination for 120 h.

1. Introduction

Surface wetting behavior is an important property of solid materials. In the past decades, materials with special wettability have attracted enormous research interest [1]. For example, superhydrophobic materials with water CA greater than 150° are very meaningful in numerous applications, such as self-cleaning [2, 3], antifreezing [4], oil/water separation [5], and corrosion-resistance [6]. With the development of research, it was found that the wettability could be changed by external stimuli [7]. Due to their unique properties, stimuli-responsive surfaces with switchable wettability have great potential in many technological applications, including smart textiles [8], drug delivery [9], and smart separation [10]. Up to now, various external stimuli, such as light illumination [11], electric field [12], pH value [13], ion exchange [14], and heating [15] have been used to change the wettability of surfaces. Among them, light is one of the most commonly used techniques, and TiO_2 [11], ZnO [16], WO_3 [17], azobenzene [18], and spiropyran [19] were applied for the preparation of surfaces with photoinduced switchable wettability.

However, there are still some issues, which restrict the practical application. For example, some materials, which are used to fabricate superhydrophobic surfaces are expensive and toxic [18, 19], and some preparation techniques are sophisticated and costly [20]. Therefore, it is essential to develop facile, low-cost, and environment-friendly methods to manufacture surfaces with photoinduced switchable wettability.

The cotton fabric is inexpensive, biodegradable, plentiful in nature, and widely used in daily life. Therefore, it was applied as the substrate material of superhydrophobic surfaces in this study. α-Fe_2O_3 is a commonly-used semiconductor photocatalyst that has the advantage of being cheap and environment-friendly [21]. There is no report on the fabrication of surfaces with switchable wettability until now.

In this study, we employed a facile wet chemical method to fabricate wettability switchable cotton fabrics. The

hydrophobic emulsion was synthesized by the copolymerization of butyl acrylate (BA) and vinyl-substituted MQ silicone resin (SR). The superhydrophobic cotton fabric was prepared by dip-coating of the hydrophobic emulsion and α-Fe_2O_3 dispersion. The surface morphology of the fabric was observed by SEM. The wettability of the surface was tested under UV illumination and after storage in the dark. The chemical composition of the surfaces before and after UV illumination was analyzed using XPS and FTIR. The possible mechanism was discussed on the basis of the results obtained in the present study.

2. Materials and Methods

2.1. Materials. The chemicals such as ferrous sulfate, ethanedioic acid, sodium hydroxide, 3-glycidoxypropyltrimethoxysilane (KH-560), BA, Span-20, sodium dodecylbenzene sulfonate, ammonium persulfate, and absolute alcohol were of analytical grade and purchased from Aladdin Chemistry Company (Shanghai, China). SR was of industrial grade and purchased from Jinan Silicon Technology New Material Company (Jinan, China). Cotton fabric was purchased from the local market. Water was deionized, doubly distilled.

2.2. Preparation of Hydrophobic Emulsion. 1 g Span-20, 1 g sodium dodecylbenzene sulfonate, 50 mg ammonium persulfate, and 100 mL distilled water were added into a three-necked round bottomed flask, and stirred for 1 h. Subsequently, 18 g BA and 5 g SR were added into the flask and reacted for 7 h at 80°C. Then, the hydrophobic BA-SR copolymer emulsion was prepared.

2.3. Preparation of Iron Oxide Dispersion. 20 g ferrous sulfate and 20 g ethanedioic acid were dissolved in 200 mL distilled water, followed by 1 h magnet stirring at ambient temperature. The precipitated FeC_2O_4 was separated by centrifugation and washed with distilled water. The residue of FeC_2O_4 was dried at 60°C for 6 h and calcined in a muffle furnace at 400°C for 3 h. Then, the iron oxide particles were prepared.

5 g as-prepared iron oxide particles, 0.25 g KH-560, and 150 mL absolute alcohol were added into a flask and stirred for 3 h at 80°C. The particles covered with KH-560 were obtained and then washed with absolute alcohol, in which the particles were isolated from water by centrifugation. This washing-centrifugation procedure was repeated five times. Then, 2 g iron oxide particles obtained in the above process was mixed with 100 mL absolute alcohol under vigorous stirring until a stable suspension was formed.

2.4. Dip-Coating of Superhydrophobic Cotton Fabrics. Cotton fabrics (4 cm × 4 cm) were degreased in a sodium hydroxide solution (2 g/L) for 1 h in an ultrasonication bath, then thoroughly washed by distilled water, and dried for further use.

The pretreated fabrics were dipped in the as-prepared hydrophobic emulsion and dispersed under an ultrasonic bath for 5 min, followed by drying in an oven for 6 h at 50°C. This coating-drying procedure was repeated two times. Then, the resulting fabrics were immersed in the iron oxide dispersion for 5 min and dried at 50°C. This coating-drying procedure was repeated two times, and then the superhydrophobic cotton fabrics were obtained.

2.5. Characterization. The crystal structure of the iron oxide was evaluated by X-ray diffractometer (XRD, Dmax-Rapid II, Rigaku, Japan). The morphological feature of iron oxide particles was observed by transmission electron microscopy (JEM-F200; JEOL, Japan). The surface morphology of cotton fabrics was observed by SEM (Quanta-450, FEI, USA), which were operated at an accelerating voltage of 10 kV. The chemical composition of cotton fabrics was analyzed by energy dispersive X-ray spectrometer (EDS, X-MAX50, Oxford, UK) attached to the SEM. FTIR spectra were reported in the range of 4000-400 cm^{-1} with a spectral resolution of 4.0 cm^{-1} by the transmission approach using a FTIR spectrophotometer (Nexus 670, Nicolet, USA). The chemical compositions of the samples surfaces were analyzed using XPS (Axis Ultra, Kratos, UK). The CA was measured using a contact angle measuring system (DSA100, Kruss GmbH, Germany) at ambient temperature, and the CA was determined by averaging the measurements taken from five different positions on the samples with the distilled water droplet of 6 μL.

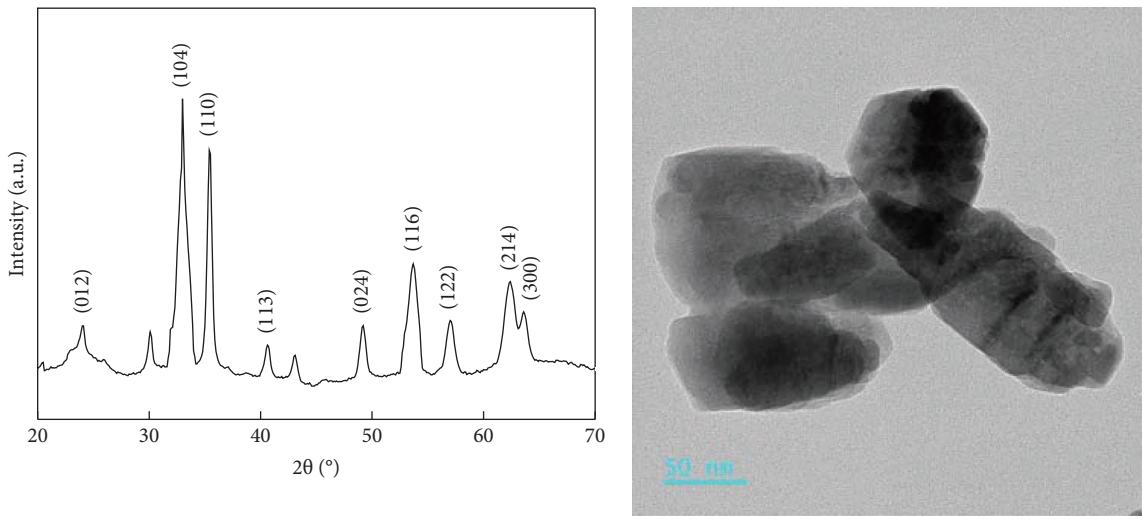
3. Results and Discussion

3.1. Characterization of Iron Oxide. The XRD analysis was applied to examine the crystal structure of the prepared iron oxide particles. In Figure 1(a), the peaks at $2\theta = 24.1°$, 33.2°, 35.5°, 40.7°, 49.1°, 53.8°, 57.4°, 62.4°, and 63.6° of iron oxide are related to (012), (104), (110), (113), (024), (116), (122), (214), and (300) planes of α-Fe_2O_3, respectively. All diffraction peaks are agreed with the hexagonal α-Fe_2O_3 phase (JCPDS 33-0664) [22]. Furthermore, it was calculated that the average crystallite size of the α-Fe_2O_3 particles was 27.3 nm by using the Scherrer equation.

The morphological feature of α-Fe_2O_3 particles was observed by TEM. Figure 1(b) showed that the particles were polygonal and their average diameters were 70–100 nm.

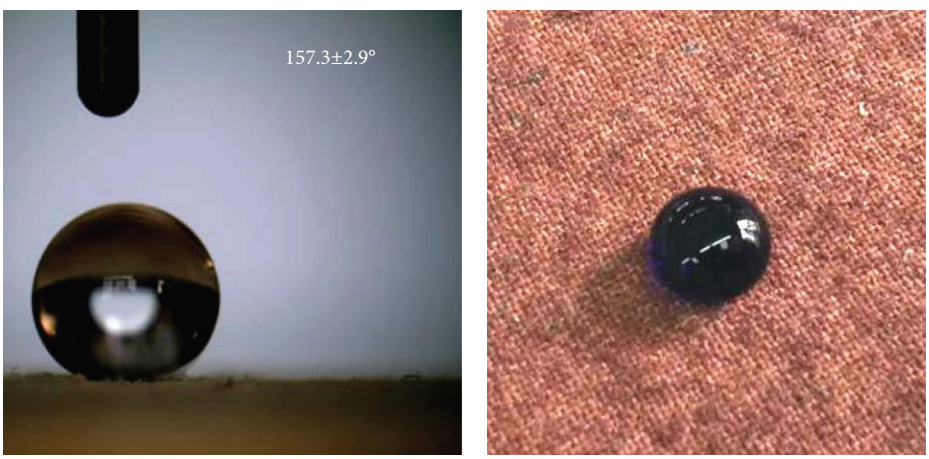
3.2. Characterization of the Superhydrophobic Cotton Fabric. Photographs of CA and water droplet (dyed with methylene blue) on the as-prepared cotton fabric are shown in Figure 2. As can be seen in Figure 2(a), the CA was measured as $157.3 \pm 2.9°$, which indicated that the fabric exhibited superhydrophobicity after the dip-coating of hydrophobic emulsion and α-Fe_2O_3 dispersion. Moreover, the water droplet was nearly spherical on the fabric surface (Figure 2(b)), confirming the cotton fabric's excellent water repellency.

It is well recognized that the wetting behavior of the solid surface was determined by both surface morphology and



(a) (b)

Figure 1: XRD diffractogram (a) and TEM image (b) of iron oxide.



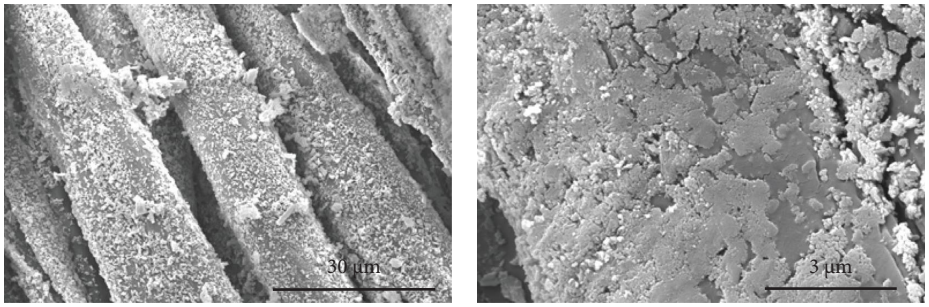
(a) (b)

Figure 2: Photographs of contact angle (a) and water droplet (b) on the superhydrophobic cotton fabric surface.

chemical composition [23]. Therefore, the surface morphology of the superhydrophobic cotton fabric was observed by SEM. As shown in Figure 3(a), the fiber surface was covered with close-packed BA-SR copolymer and α-Fe_2O_3 particles. The diameters of the individual fibers were approximately 15 μm to 20 μm. The image in Figure 3(b) confirmed a uniform distribution of α-Fe_2O_3 nanoparticles on the substrate. Consequently, the micro-nano hierarchical rough structure was formed and endowed the fabric surface with the capability of trapping air, which was one of the essential characteristics of superhydrophobic surfaces.

The element composition of the superhydrophobic cotton fabric surface was investigated by EDS and is shown in Figure 4. The C, O, Si, and Fe elements were detected in the EDS spectrum. It confirmed that the BA-SR copolymer and α-Fe_2O_3 nanoparticles were incorporated on the fabric surface. Therefore, the existence of the Si element could significantly reduce the surface energy of the cotton fabric and effectively improve its water repellency [1]. In this study, the superhydrophobic cotton fabric was fabricated by combining micro-nanohierarchical rough structure and low-surface energy materials containing Si.

3.3. Wettability Switching by UV Illumination and Storage in the Dark. To investigate its wettability switchable performance, the superhydrophobic cotton fabric was directly illuminated under a UV lamp ($\lambda = 254$ nm, 32 W) at a working distance of 20 mm, and the CAs were recorded every 8 h. The effect of illumination time on the CAs of the fabric is shown in Figure 5(a). With the increase in



(a) (b)

Figure 3: SEM images of the superhydrophobic cotton fabrics under different magnifications: (a) ×3000 and (b) ×40000.

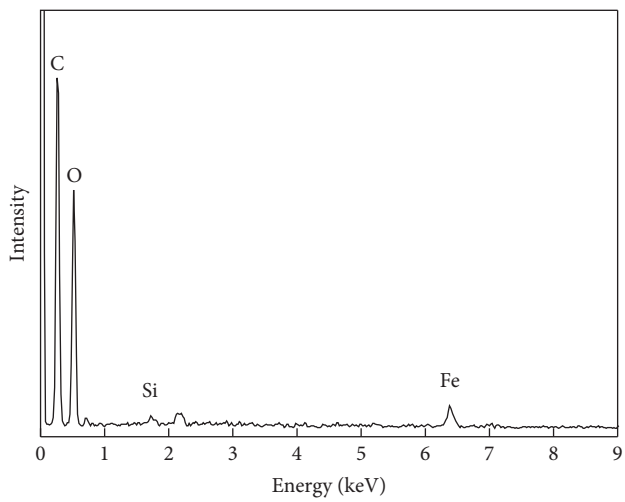


Figure 4: EDS spectrum of the superhydrophobic cotton fabric.

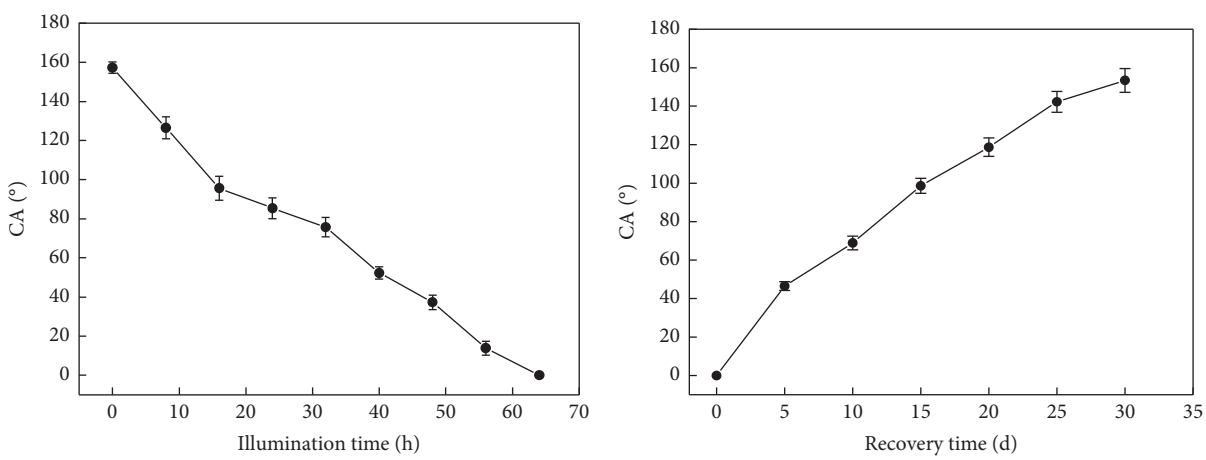


Figure 5: The effect of UV illumination time on the contact angles of the superhydrophobic cotton fabric (a), and the effect of recovery time on the contact angles of the superhydrophilic cotton fabric (b).

illumination time, CAs decreased gradually and then reduced to 0° after 64 h illumination. This result demonstrated that the as-prepared cotton fabric surface could be converted from superhydrophobicity to superhydrophilicity, indicating its wettability switchable performance.

Wettability recovery capability is also a crucial performance of the wettability switchable materials. To check the recovery of superhydrophobicity, the superhydrophilic cotton fabric after UV illumination was stored in the dark, and the CAs were recorded every 5 d. The effect of recovery

time on the CAs of the fabric is shown in Figure 5(b). It was observed that CAs increased gradually and then reached to $153.6 \pm 5.2^{\circ}$ after 30 d storage in the dark. This result proved that the as-prepared superhydrophobic cotton fabric possessed wettability recovery ability. Up to now, many materials were applied to prepare photoinduced reversible wettability switchable surfaces. However, the research of iron oxide in this area has not been previously reported.

Photoinduced reversible wettability switching of semiconductor metal oxides has been explored by many researchers, and the switching mechanism was also studied. At present, researchers still have two different views on the mechanism. One is the removal of organic substances from the superhydrophobic surface by a photocatalytic decomposition process [20–24]. The other is that hydroxyl groups were adsorbed on the photogenerated surface oxygen vacancies [25, 26].

In order to explore the photoinduced wettability switching mechanism, the cotton fabrics before and after UV illumination were analyzed by means of FTIR and XPS.

As shown in Figure 6, the FTIR spectra were applied to obtain the functional group details of pristine cotton fabric, the cotton fabrics before and after UV illumination. In Figure 6 (a), the FTIR spectrum of pristine cotton fabric exhibited the characteristic peaks of O-H stretching vibration at 3340 cm^{-1}, C-H stretching vibration at 2917 cm^{-1}, and glucosidic ring stretching vibration at 898 cm^{-1} [27]. In Figure 6 (b), besides the characteristic peaks of cotton, the peaks at 1165 and 1729 cm^{-1} were assigned to C-O-C and C=O stretching vibration of ester group on BA structure unit, respectively [28]. The peaks at 1280 and 1074 cm^{-1} were attributed to Si-CH_3 and Si-O-Si of SR, respectively [29]. Moreover, the peak at 545 cm^{-1} was assigned to Fe-O stretching vibration of α-Fe_2O_3 [30]. These results further confirmed that the hydrophobic BA-SR copolymer and α-Fe_2O_3 nanoparticles were covered on the superhydrophilic cotton fabric. In Figure 6 (c), the characteristic peaks of BA structure unit and SR could still be observed in the FTIR spectrum of the cotton fabric after UV illumination. The analysis results of the cotton fabrics demonstrated that the low-surface energy materials still remained on the fabric surface. So it could be inferred that the wettability switching of the superhydrophobic cotton fabric should not be caused by the decomposition of low-surface energy materials.

In order to further investigate their surface composition, XPS spectra of cotton fabrics before and after UV illumination were also recorded. As shown in Figure 7 (a) and (b), the characteristic signals of iron (Fe 2p), oxygen (O 1s), carbon (C 1s), and silicon (Si 2s and Si 2p) at 711.5, 530.4, 284.8, 154.5, and 103.0 eV were detected, respectively. In addition, the atomic concentrations of the above four elements on the surface of the two cotton fabrics were also determined by XPS and tabulated in Table 1. By contrasting the surface atomic concentrations of the two fabrics, it can be found that Si and C concentrations did not decrease significantly after UV illumination, which indicated that low-surface energy materials were still located on the fabric surface. Hence, based on the results of FTIR and XPS analysis, it could be concluded that the wettability switching mechanism was not the removal of low-surface energy materials from the superhydrophobic surface by the photocatalytic decomposition of α-Fe_2O_3 nanoparticles.

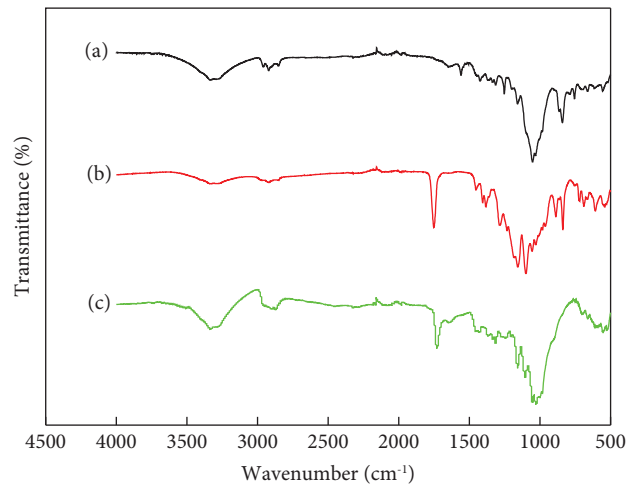


Figure 6: FTIR spectra of pristine cotton fabric (a), cotton fabrics before (b) and after (c) UV illumination.

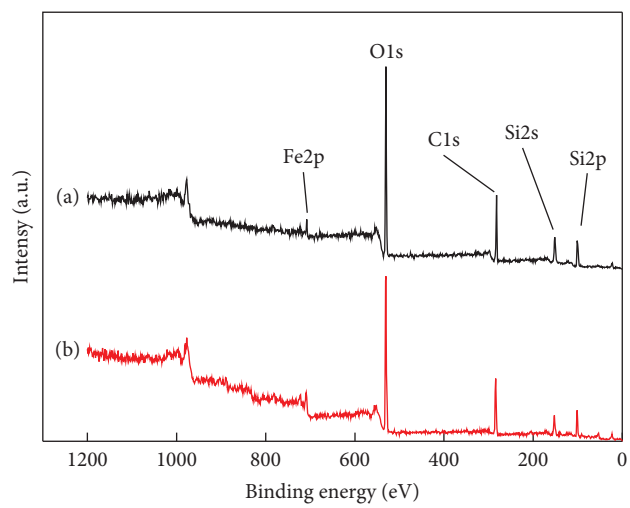


Figure 7: XPS survey spectra of cotton fabrics before (a) and after (b) UV illumination.

Table 1: Atomic concentration of elements on the surface of cotton fabrics before (a) and after (b) UV illumination.

Atomic concentration (%)	Fe	O	C	Si
Cotton fabric before UV illumination	4.59	37.38	38.25	19.58
Cotton fabric after UV illumination	6.67	41.54	34.03	17.76

To further explore the wettability switching mechanism, the hydroxyl groups on the surfaces of fabrics before and after UV illumination were determined by XPS. The O 1s peaks of cotton fabrics were curve-fitted into four peaks positioned at 532.2, 530.9, 529.9, and 527.5 eV, respectively. The first peak could be ascribed to the oxygen in surface water (denoted as H_2O). The second one was attributed to surface adsorbed hydroxyl groups (Fe-OH_{ad}). The third peak

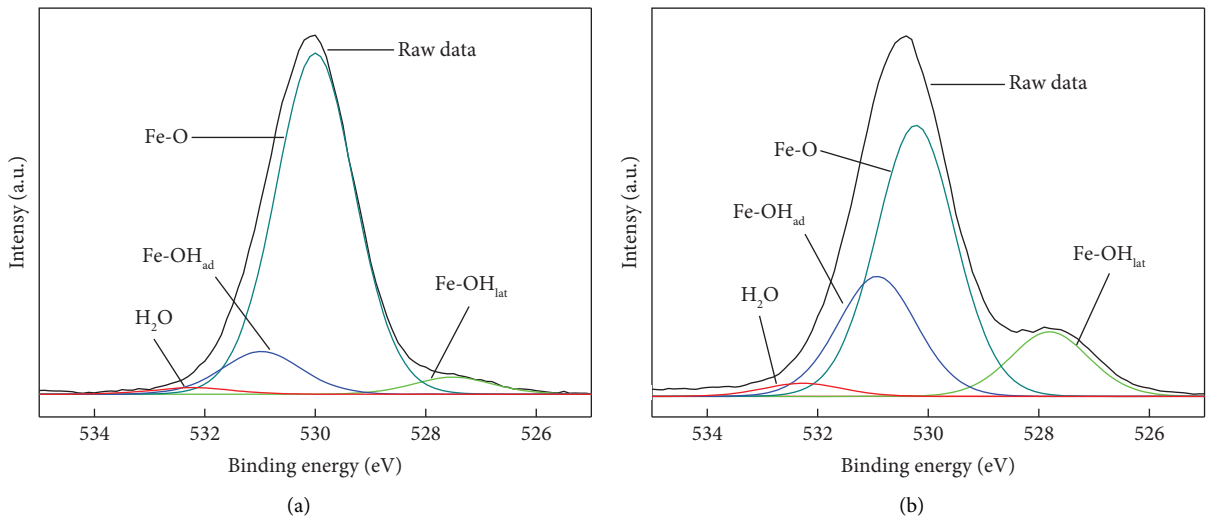


Figure 8: XPS O 1s spectra of the cotton fabrics before (a) and after (b) UV illumination.

was ascribed to the lattice oxygen binding with Fe (Fe-O). The fourth one was related to surface lattice hydroxyl groups (Fe-OH$_{lat}$) [31]. As shown in Figure 8, the amount of oxygen in the adsorbed hydroxyl groups increased after UV illumination. Accordingly, the adsorbed hydroxyl group content of the fabric surface increased from 3.90% to 10.63%.

As an important photocatalytic material, α-Fe_2O_3 is applied to degrade organic pollutants in air and aqueous media. After UV illumination, electron and hole pairs would generate on the surface of α-Fe_2O_3 [32], and could be expressed as

$$Fe_2O_3 + h\nu \longrightarrow Fe_2O_3\left(e^-, h^+\right). \quad (1)$$

Accordingly, based on the wettability switching mechanism of ZnO and TiO_2, the possible reason for the wettability switching of the α-Fe_2O_3 coated cotton fabric surface under UV illumination could be explained as follows:

The photogenerated holes diffused to the surface of α-Fe_2O_3 and reacted with the lattice oxygen atoms to form oxygen vacancies. Oxygen vacancies dissociated the water adsorbed on the α-Fe_2O_3 surface, and then the adsorbed hydroxyl group content increased. As a result, the high surface energy of the hydroxyl groups results in hydrophilic surface properties [15, 33]. However, the hydroxyl groups adsorbed on the α-Fe_2O_3 surface are thermodynamically unstable. Stored in the dark, the hydroxyl group would be replaced by O_2 in the air and the hydroxyl group contents decreased. Consequently, the surface reverted back to the initial superhydropbobic state [34, 35]. The simplified summary of the overall process could be represented as

$$-Fe-O-Fe-+H_2O \underset{\text{dark}}{\overset{h\nu}{\rightleftarrows}} 2HO-Fe-. \quad (2)$$

To investigate its wettability switchable performance under sunlight, the superhydrophobic cotton fabric was illuminated by simulated sunlight, which was provided by a xenon lamp (500 W) at a working distance of 50 mm. The effect of illumination time on the CAs was studied. As shown in Figure 9, with the increase of illumination time, CAs decreased gradually and then reduced to 0° after 120 h illumination. This result demonstrated that the surface wettability of the as-prepared superhydrophobic cotton fabric could also be converted by sunlight. Nowadays, most of the photosensitive oxide semiconductors, such as TiO_2 and ZnO could only convert surface wettability by UV. As a semiconductor material, α-Fe_2O_3 with a band gap energy of 2.2 eV can adsorb light up to 600 nm [31]. Sunlight is the most abundant, clean, and reproducible light source; therefore, α-Fe_2O_3 is a promising material in the area of photoinduced wettability switchable surfaces.

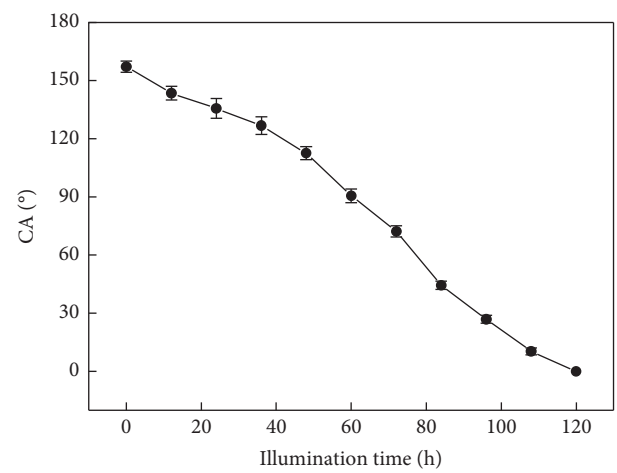


Figure 9: The effect of sunlight illumination time on the contact angles of the superhydrophobic cotton fabric.

4. Conclusion

In summary, a superhydrophobic cotton fabric with switchable wettability was prepared by coating of the hydrophobic BA-SR copolymer and α-Fe_2O_3 nanoparticles.

It exhibited the excellent superhydrophobic property with the CA of $157.3 \pm 2.9^{\circ}$. The as-prepared fabric surface became superhydrophilic after UV illumination for 64 h and reverted back to the initial superhydrophobic state after being stored in the dark for 30 d. Based on the XPS and FTIR analyses, the possible mechanism was discussed, and the switchable wettability was caused by the content change of the hydroxyl groups on the α-Fe_2O_3 surface. Moreover, the superhydrophobic cotton fabric also became superhydrophilic after sunlight illumination for 120 h. These results demonstrated that α-Fe_2O_3 is a promising material in the area of photoinduced wettability switchable surfaces.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

Acknowledgments

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