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A self-roughened and biodegradable superhydrophobic coating with UV shielding, solar-induced self-healing and versatile oil-water separation ability

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Traditional superhydrophobic coatings prepared from non-degradable materials tend to do harm to the environment throughout the fabrication process as well as after being discarded. Great efforts have been devoted to exploring more environmental friendly approaches and materials to settle this problem. Here we report an eco-friendly strategy based on aqueous systems to construct the superhydrophobic coating onto various fabrics. The fabrics were firstly coated with polydopamine (PDA) and then modified by the stearic acid emulsion to introduce the desired surface morphology and energy. The as-prepared fabrics achieved robust superhydrophobicity with a contact angle (CA) about 162.0° and sliding angle (SA) about 7.8°. Moreover, due to the UV-absorbing and the photo-thermal ability of the PDA, the modified fabrics exhibited excellent UV shielding and solar-induced self-healing property. The as-prepared fabrics also possessed a high efficiency oil-water separation. Without the usage of harmful organic solvents and the addition of micro/nano-particles, this biodegradable superhydrophobic fabric exhibited has a clear advantage of being environmentally friendly over the conventional coatings. Furthermore, the facile and low-cost fabricating process makes it easy for large-scale production.

Introduction

Superhydrophobic surfaces with the water contact angle (CA) more than 150° and the sliding angle (SA) less than 10° have exhibited excellent application potential in many fields, such as oil-water separation,¹⁻³ self-cleaning,⁴⁻⁵ water collection,⁶⁻⁸ liquid transport,⁹⁻¹⁰ anti-corrosion,¹¹⁻¹² anti-icing¹³⁻¹⁴ and drag reduction.¹⁵⁻¹⁷ It is well-known that the representative construction strategy of superhydrophobic coatings mainly includes two crucial factors: micro/nano structures and low surface energy components. The common construction approaches include dip-coating,¹⁸⁻¹⁹ spray-coating,²⁰⁻²¹ etching,²²⁻²³ chemical vapor deposition (CVD),²⁴⁻²⁵ electrophoretic deposition²⁶⁻²⁷ and so on. Many recent researches have used inorganic nanoparticles (e.g. ZnO, TiO₂, SiO₂) in superhydrophobic coatings to create surface roughness.^{5, 28-29} However, the wide use of inorganic nanoparticles, if not handled properly, may pose dangerous consequences to both human health and environment.³⁰ This is because that these nanoparticles are not biodegradable and could be nocuous due

to the small length scales. Moreover, it will increase the production cost to some extent.

Many manufacturing processes of superhydrophobic fabrics involve extensive chemical processing, which often causes damage to the environment because of the use of organic solvents as well as fluorine-based polymer. To tackle the problems, we have constructed self-roughened superhydrophobic surfaces based on a fluorine-free and water-rich system by taking polydimethylsiloxane (PDMS) as bonder, tetrahydrofuran (THF) as solvent and water as non-solvent.³¹ Meanwhile, by using THF and n-hexane as co-solvent, we demonstrated that roughness could be induced on PDMS coatings to achieve superhydrophobic coatings.³² Recently, Chen and co-workers fabricated biodegradable polylactic acid nonwoven fabric as superhydrophobic and superoleophilic material consisting of hydrophobic polystyrene microspheres and SiO₂ nano-particles deposited on the polydopamine-modified polylactic fabric.³³ However, these fabrication processes still involved the organic solvent or addition of the nano-particles. Therefore, it is urgent to find an eco-friendly self-roughened and biodegradable superhydrophobic coating based on aqueous systems.³⁴⁻³⁶

The oxidized self-polymerization of dopamine (DA) in an alkaline environment can be utilized to fabricate mussel-inspired polydopamine coatings on various surfaces because of the strongly interfacial adhesive forces.³⁷⁻³⁸ In this work, we proposed a biodegradable superhydrophobic cotton fabrics coated with polydopamine as an adhesive and stearic acid (STA) as low surface energy component.^{29, 39} Herein, the

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polydopamine coatings not only act as an adhesive but also construct micro/nano structure roughness on the material surfaces during the process of oxidative polymerization. Stearic acid, also called octadecanoic acid, can significantly reduce the surface energy due to its long alkyl chain. Compared with fluorinated organic compounds, raw materials including polydopamine and stearic acid are environmentally-friendly, nontoxic and biodegradable.^{33, 40} The modified cotton fabrics coated with polydopamine and stearic acid exhibited remarkable superhydrophobicity with a CA of 162.0° and SA of 7.8°. Moreover, due to the UV-absorption and the photo-thermal response of the PDA,⁴¹⁻⁴² the modified fabrics exhibited excellent UV shielding and solar-induced self-healing property. The as-prepared fabrics also possessed an excellent separation ability for different types of oil-water mixtures, such as heavy oil-water, light oil-water mixtures and even corrosive mixtures of oil and acid/alkali solutions. With no reliance on organic solvents and addition of nano-particles, this as-prepared superhydrophobic fabric coating is biodegradable and environmentally friendly. This facile and rapid fabrication technology could be applied on various substrates such as silk, wool, acrylic, polyester, nylon, viscose etc, and is expected to be easy to achieve large-scale production.

Experimental

Materials

Various substrates including cotton, silk, nylon, polyester, viscose, ramie, wool and acrylic fabrics were purchased from Shanghai Textile Industry. Dopamine hydrochloride (98%, AR) and tris (hydroxymethyl) aminomethane were obtained from Aladdin Industrial Corporation. Stearic acid and copper(II) sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, AR) were purchased from Sinopharm Chemical Reagent (China). Hydrogen peroxide (30%, AR) was acquired from Shanghai Lingfeng Chemical Reagent Co., Ltd.

Polymerization and deposition of DA triggered by $\text{CuSO}_4/\text{H}_2\text{O}_2$

Dopamine (2 mg/mL), $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (5 mM), and H_2O_2 (19.6 mM) were dissolved in tris buffer (50 mM) in sequence to prepare the deposition solution. Then the fabrics were immersed in the solution and with mechanical stirring at 25 °C for 4 h. Subsequently, the samples were washed by deionized water for several times, and dried in a drying oven at 75 °C for 2 h. The above steps were repeated for three times.

Preparation of superhydrophobic coating

The molten stearic acid was added into the hot water at 75 °C (higher than the melting point of stearic acid) on a magnetic thermostatic agitator, and the solution was stirred at 1500 rpm for 1 h to prepare stearic acid emulsion. The concentration of stearic acid was 0.1 wt. %. The fabrics were then immersed into the prepared stearic acid emulsion for 1 min and dried in a 75 °C oven for 2 h.

Characterization

The microstructures of sample were examined with a field emission scanning electron microscope (FESEM, Hitachi S-4800).

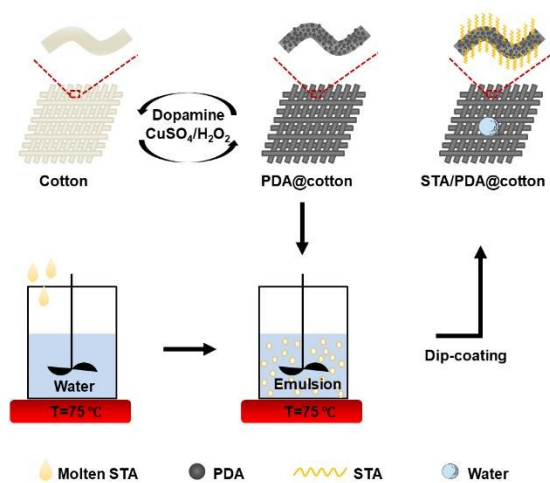
The chemical composition of fabrics was analyzed via an energy-dispersive X-ray spectrometer (EDS) as well as Kratos Axis Ultra HAS X-ray photoelectron spectrometer (XPS). Fourier Transform infrared (FTIR) spectroscopy was provided by Nicolet 5700. The surface roughness was measured using an atomic force microscope (AFM, Dimension Loon, Bruker) with a scan area of $2 \mu\text{m} \times 2 \mu\text{m}$. The wetting properties of water on the fabrics were measured using a Krüss DSA 100 (Krüss, Germany) apparatus. The water contact angle was acquired by measuring five different positions of the same sample. The tensile breaking strength and elongation at break was obtained via a universal testing machine INSTRON-3365. Moreover, the washing durability was conducted according to 2A condition following the standard AATCC 61-2006 method. For the abrasion test, the superhydrophobic fabric was rubbed against a pristine cotton fabric as the abrasion surface. In addition, air permeability was tested by a digital air permeability instrument (YG461E-11), and flexural rigidity was characterized by a tester (YGB022D).

Results and discussion

Scheme 1 illustrates the fabrication procedure of superhydrophobic STA/PDA@cotton fabrics via an environmentally friendly two-step approach. Two eco-friendly and biodegradable materials were involved here. On one hand, PDA was coated onto cotton fabrics through the polymerization and deposition of dopamine triggered by $\text{CuSO}_4/\text{H}_2\text{O}_2$.^{35, 43} To make the PDA coating rougher on the surface of cotton fibres, the process was repeated for 3 times. Furthermore, the use of $\text{CuSO}_4/\text{H}_2\text{O}_2$ as a trigger can create reactive oxygen species, which enormously enhances the deposition rate as well as the stability and uniformity of polydopamine coatings.⁴³ On the other hand, stearic acid was used to modify the PDA@cotton fabrics to lower the surface energy. In briefly, the magnetic thermostatic agitator was set at 75 °C above the stearic-acid's melting point. Then the molten stearic acid was added into hot water and constantly stirred to form a uniform and stable emulsion. It should be noticed that no surfactant or organic solvent was introduced during the whole process, which makes the preparation procedures more environmentally friendly. The CA and adhesion force of superhydrophobic cotton fabric with different concentration of stearic acid was shown in Fig. S1 (supporting information). Low concentration of stearic acid emulsion is not only eco-friendly, but also inexpensive and time-saving. The PDA@cotton fabric was immersed into the hot STA emulsion for a short time, and stearic acid can be easily and effectively bonded with it because of the excellent adhesive ability of dopamine.

AFM images of the cotton fabrics coated with polydopamine for different times and STA were shown in the Figure 1. The root mean square (RMS) surface roughness of pristine cotton and modified cotton was quite different. Apparently, the surface roughness of cotton coated with PDA for three times was higher than the ones coated for once or twice. The pristine cotton fabric is very smooth with RMS value of about 5 nm. While the surface roughness of modified cotton coated with

different coating cycles of PDA increased from 23.9 nm to 47.5 nm.



Scheme 1. Schematic illustration of the procedure to construct the superhydrophobic STA/PDA@cotton fabric in a waterborne and fluorine-free system via a two-step environmentally-friendly approach.

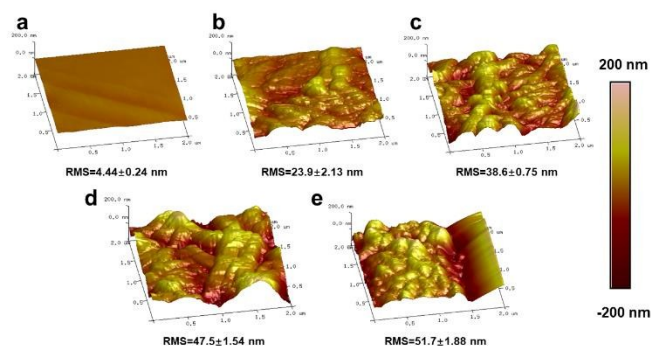


Figure 1. AFM images and corresponding RMS value of surfaces of (a) pristine cotton fabric, (b-d) cotton fabrics coated with PDA once, twice or three times, (e) STA/PDA@cotton fabric (2 $\mu\text{m} \times 2 \mu\text{m}$).

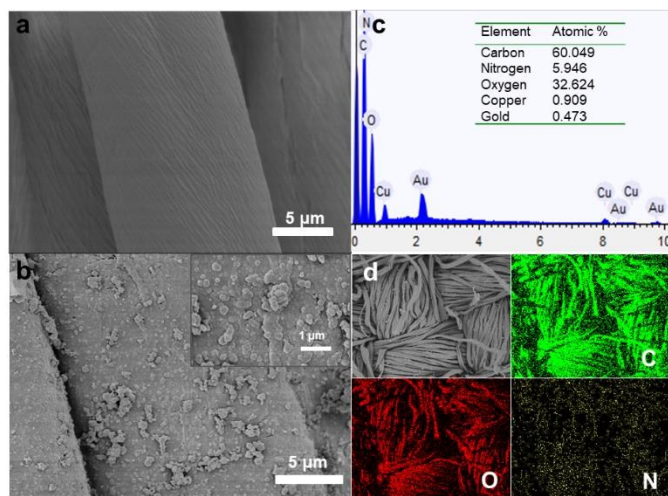


Figure 2. SEM images of (a) pristine cotton fabric, (b) STA/PDA@cotton fabric, (c, d) the elemental mapping for C, O and N and the EDS spectrum of STA/PDA@cotton fabric with a scan area of 600 $\mu\text{m} \times 600 \mu\text{m}$.

The final RMS roughness of superhydrophobic STA/PDA@cotton fabric enhanced to 51.7 nm. As shown in Fig. 1, the PDA particles aggregated and constructed micro/nano surface roughness on the fibers during the polymerization and deposition process. SEM images shown in Fig. 2a also confirmed that the surface of pristine cotton was relatively flat and smooth. While the surface of modified cotton coated with PDA and STA was much rougher due to the polymerization of dopamine (Fig. 2b). Carbon, oxygen and nitrogen mapping images and element analysis confirmed that PDA and STA adhered onto the cotton fabric uniformly (Fig. 2c, d).

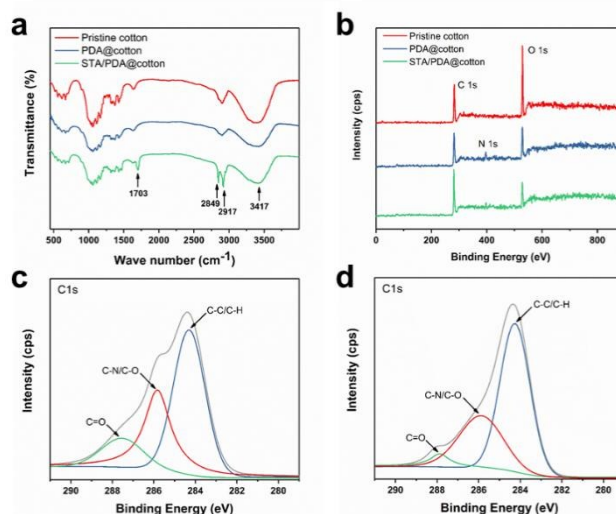


Figure 3. (a) FTIR and (b) wide XPS spectra of pristine cotton, PDA@cotton and STA/PDA@cotton fabric, (c, d) high-resolution C 1s spectrum of PDA@cotton and STA/PDA@cotton fabric.

FTIR and XPS were investigated to further analyze the information of surface chemical bonding and composition. As displayed in Fig. 3a, the absorption peaks of pristine cotton and PDA@cotton didn't show significant difference, the stretching vibrations absorption peaks of -OH and stretching vibrations of -CH₂ appeared at 3417 cm^{-1} and 2900 cm^{-1} were present.³⁴ While after modified by stearic acid, the absorption peaks at 2849 cm^{-1} and 2917 cm^{-1} correspond to the C-H symmetric and asymmetric stretching vibrations of -CH₂ groups in stearic acid, and the absorption peak at 1703 cm^{-1} is ascribed to the stretching vibration of -COO due to stearic acid.³⁹ As exhibited in Fig. 3b, compared with the pristine cotton, a new peaks appeared at 398.4 eV (N 1s), which is ascribed to the polydopamine coating. In addition, the high resolution C 1s spectrum displayed three distinct peaks at 284.2 eV for C-C/C-H bonds, 285.9 eV for C-N/C-O bonds and 287.8 eV for C=O bonds.⁴³ The ratio of -CH_x (C-C and -CH₃) increases for the long chain alkyl in stearic acid.

Compared to the pristine cotton which is entirely wetted by water (Fig. 4a, b), the modified cotton fabrics coated with polydopamine and stearic acid exhibited a great

superdophobicity with a CA about 162.0° and an SA about 7.8° (Fig. 4c and Fig. 4d). To further confirm the dynamic anti-wetting behavior, the adhesive force between water droplet and as-prepared fabric surface was measured, showing a lower adhesion of $54.3 \mu\text{N}$ (Fig. S1). The stability of STA emulsion was shown in Fig. 4e, compared with the newly prepared STA emulsion, a small amount of white precipitate appears in the STA emulsion that prepared 10 weeks ago. After ultrasonic agitation at 40 kHz at 50°C for 5 min, and STA emulsion showed no noticeable effect on the superdophobicity of STA/PDA@cotton (Fig. S2).

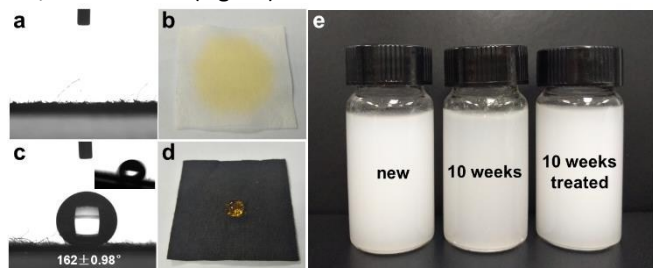


Figure 4. (a, c) The static behaviour of water droplet on the STA/PDA@cotton fabric, the inset picture shows sliding of water droplet, (b, d) the photo of the droplets ($40 \mu\text{L}$ dyed by methyl orange) on pristine cotton fabric and modified cotton fabric ($3 \text{ cm} \times 3 \text{ cm}$), (e) images of the STA emulsions, from left to right, the newly prepared, stored (10 weeks), and stored with ultrasonic agitation.

The CAs of the STA/PDA@cotton fabric surface decreased gradually with the increasing of abrasion cycles (Fig. 5a). The modified cotton fabric still kept good superdophobic property with a CA above 150° even after 1000 abrasion cycles. The accelerated laundering durability was evaluated according to the AATCC 61-2006 standard method under 2A condition. The superdophobicity could also be maintained even after 5 accelerated laundering cycles (Fig. 5b). One accelerated laundering cycle is equivalent to five cycles of domestic or commercial laundering. This enhanced mechanical stability and laundering durability are ascribed to the strong binding by the dopamine between the substrate and stearic acid. What's more, the superdophobic modified cotton fabric could float on the water surface even with a water droplet on it (Fig. 6b). In addition, the modified cotton was submerged into water to evaluate the ability resist high hydraulic pressure. A mirror-like surface was shown on the superdophobic cotton fabric since air was trapped on the surfaces (Fig. 6c), while the pristine cotton was wetted as soon as it contacted water.

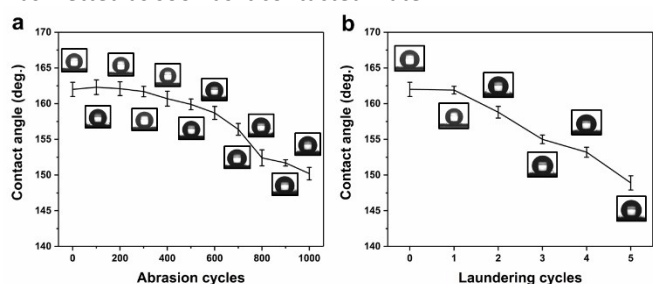


Figure 5. The effect of (a) abrasion and (b) laundering cycles on water contact angle of superhydrophobic cotton fabric, the insets were the corresponding droplet images under different conditions.

Chemical stability of this superhydrophobic coating was also evaluated by dipping the modified fabric into salt solution (NaCl , $\text{pH} = 7$), acid solutions (HCl , $\text{pH} = 1$ and $\text{pH} = 2$) and alkali solution (NaOH , $\text{pH} = 12$) for 24 h. As exhibited in Fig. 6a, there were no significant change on CAs of the modified fabrics after these treatments. However, the superhydrophobic property declined after immersed into alkaline solution, because stearic acid can react with bases and the ester bond would hydrolyze under alkaline condition.

Then the effect on physical and mechanical properties of as-modified cotton fabric was investigated and summarized in Table 1. The mechanical properties of this modified fabric such as flexural rigidity, tensile strength and air permeability did not show sharp change because of the mild modification condition.

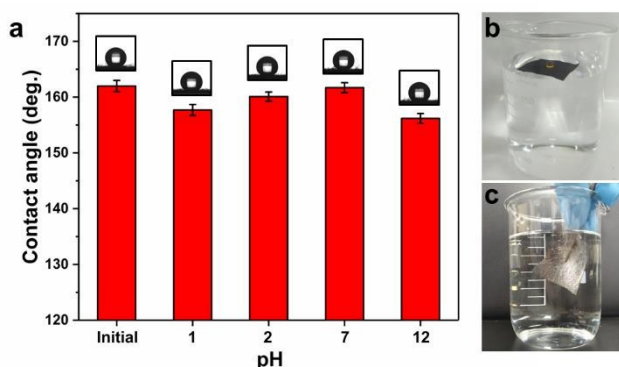


Figure 6. (a) Contact angle of superhydrophobic cotton fabric with corresponding droplet images inset before and after immersed into acid solution (HCl , $\text{pH} = 1$ and $\text{pH} = 2$), alkali solution (NaOH , $\text{pH} = 12$), and salt solution (NaCl , $\text{pH} = 7$) for 24 hours, (b, c) images of the superhydrophobic cotton fabric ($3 \text{ cm} \times 3 \text{ cm}$) floated on the surface of water with a droplet ($40 \mu\text{L}$ dyed by methyl orange) and submerged in water.

Table 1. The physical properties of pristine cotton fabric and as modified cotton fabric.

Property	Pristine cotton	Modified cotton
Tensile strength (N cm^{-1})	500.55 ± 11.85	466.28 ± 8.99
	(warp)	(warp)
Elongation at break (%)	410.23 ± 15.91	357.44 ± 7.85
	(weft)	(weft)
Flexural rigidity ($\text{mg} \times \text{mm}$)	7.07 ± 0.19	7.24 ± 0.22
	(warp)	(warp)
Air permeability (mm s^{-1})	19.28 ± 0.46	25.79 ± 0.10
	(weft)	(weft)
Air permeability (mm s^{-1})	1256.1 (warp)	753.3 (warp)
	438.84 (weft)	342.9 (weft)
Air permeability (mm s^{-1})	742.45 (total)	539.8 (total)
	420.5 ± 4.88	298.6 ± 3.56

The cotton fabrics coated with polydopamine and stearic acid possess excellent UV shielding property as well. The UV transmittance of the modified cotton fabrics is below 1% in the range of 250 nm to 400 nm (Fig. 7a). The ultraviolet protection factor (UPF) of cotton fabrics before and after coating was given in Fig. 7b. It can be found that the UPF value has a sharp increase from 4.22 to 161.94 after coated with PDA (the key role) and STA, which has far exceeded the EN 13758-1:2001 standard requirement of UPF = 50+ of. PDA has many attractive properties of natural eumelanin due to the structure similarity to eumelanin. It can absorb ultraviolet light from the sun and protect skin against ultraviolet injury.^{42, 44}

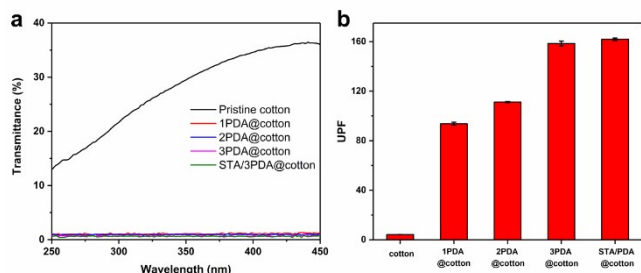


Figure 7. (a) UV-transmission and (b) UPF value of pristine cotton, cotton fabrics coated with PDA once, twice or three times and STA/PDA@ cotton fabric.

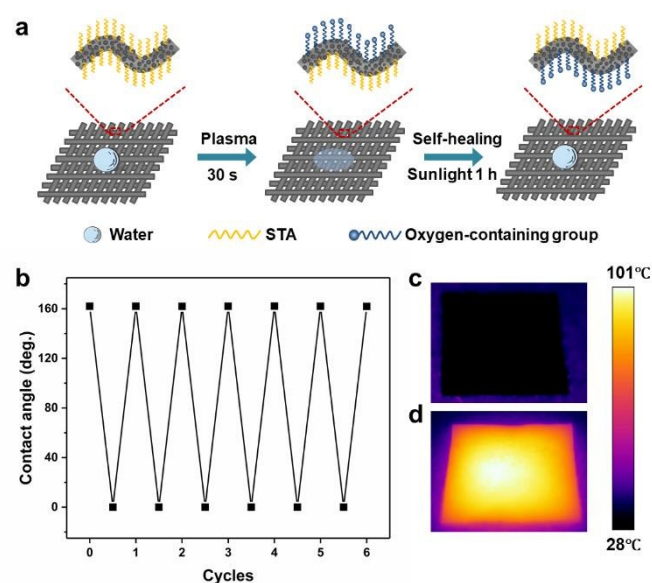


Figure 8. (a) Schematic illustrations of the plasma/self-healing mechanism of STA/PDA@ cotton fabric, (b) the water contact angle of the superhydrophobic cotton fabric after plasma etching and solar heating treatment for 6 cycles, (c, d) IR images of the modified cotton fabric (3 cm×3 cm) before and after solar heating treatment for 2 min.

We have also evaluated the self-healing property of the superhydrophobic cotton fabric. The mechanism of the plasma/self-healing process was shown in Fig. 8a. Firstly, the fabric was treated by plasma to make it superhydrophilic. Then the self-healing process was carried under light irradiation at

simulated sunlight intensity of 100 mW/cm². The whole process of plasma treatment and light irradiation was repeated for 6 times. As exhibited in Fig. 8b, the treated fabric recovered its superhydrophobicity after the light treatment in all 6 cycles. For the first three cycles, the recovery time was less than 1 h, while it would take more than 9 h if there was no irradiation. After the first three self-healing cycles, a longer time was required to recover. The reason is that the total amount of the hydrophobic groups (stearic acid) gradually decreases because of the plasma-induced damage.⁴⁵ The as-prepared cotton fabric can also recover its superhydrophobicity after abrasion. The contact angle was below 150° after 1200 and 1400 abrasion cycles. And it can self-heal to superhydrophobic again with the contact angle about 162.0° under about 10 hours of light irradiation (Figure S3). It is interesting to be able to switch from superhydrophilic to superhydrophobic only with light irradiation, without external heat treatment or electric consuming. As known to all, PDA possesses many prominent properties of natural eumelanin and can absorb nearly 99% incident photon energy over a wide solar spectrum and convert it into heat rapidly within tens of pico-seconds.^{41-42, 44} The demonstrated self-healing property of this modified fabric may be attributed to the photo-thermal ability of PDA. As shown in the IR images of Fig. 8c, d, the plasma-treated cotton fabric was only 28.7 °C (the room temperature). However, the temperature could reach 100 °C after solar heating treatment in just 2 min (Movie S1). Accordingly, the whole healing process can be explained. After the plasma treatment, the STA on the shallow surface is decomposed and some polar groups, such as oxygen-containing groups, are introduced.⁴⁶ With light irradiation, the carbon chain of STA migrates out quickly to the surface and the polar groups hide inside the coating layer due to the highly temperature. The high stabilized temperature and high heating rate are necessary for the treated cotton fabric to rapidly recover in such a speed as high as more than 9 times than in dark condition.^{30, 47}

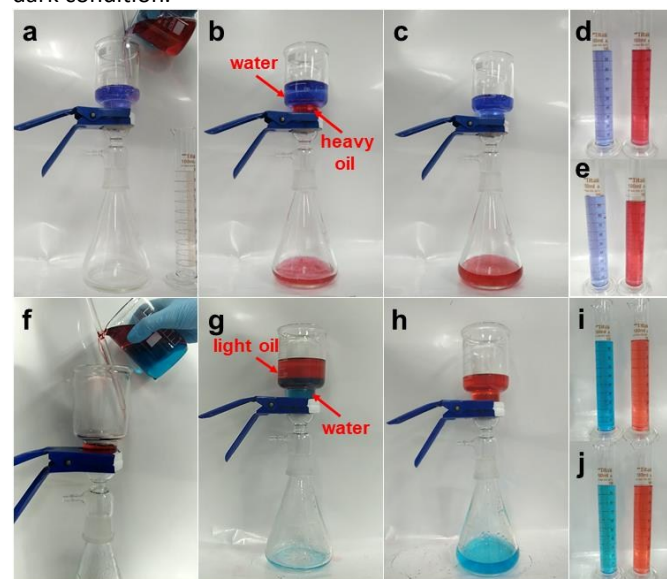


Figure 9. Sequence of the heavy oil-water separation process with modified cotton fabric (a-c), water was dyed by methylene blue and heavy oil (trichloromethane) dyed by oil red O before

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(d) and after (e) separation, sequence of the light oil-water separation process with modified cotton fabric (f-h), water was dyed by methylene blue and light oil (cyclohexane) was dyed by oil red O before (i) and after (j) separation.

The as-prepared STA/PDA@cotton fabric could be used for separation of different types of oil-water mixtures. Due to the excellent superhydrophobicity and superoleophilicity of this modified cotton fabric, it could be utilized as a selective filter membrane to realize heavy oil-water separation. In the current study, this superhydrophobic cotton fabric was directly used as a filter as shown in Fig. 9a-c. A 200 mL of mixture including a 100 mL of water dyed by methylene blue (MB) and a 100 mL of heavy oil (trichloromethane) dyed by oil red O was poured into the container through the filter. Then the mixture was separated rapidly due to the immiscibility of water and oil. The superhydrophobic fabric could only let the heavy oil permeate through by its gravity force and the water was blocked due to the superhydrophobicity of as-applied fabric (Movie S2). The separation efficiency calculated according to the volume of collected water before and after separation, is shown in Fig. 9d, e. The volume of water was almost unchanged after the separation, which means the separation efficiency of this modified cotton fabric was nearly 100%. Moreover, the same separation process was repeated for 5 times with hardly little volume loss of water (Fig. S4).

For light oil-mixture, the superhydrophobic STA/PDA@cotton fabric was treated by plasma to make it superhydrophilic (Movie S3). The separation procedure was similar as the one for heavy oil separation. The superhydrophilic fabric was wetted by the water in advance and then fixed between two glass vessels A 200 mL of oil/water mixture was poured into bottom glass vessel (Fig. 9f-h). The separation efficiency was nearly 100% after counting the volume of collected water before and after separation (Fig. 9d, e). It was obvious in Fig. S5 that there was almost no loss of water after 5 times of oil-water separation. Furthermore, the as-modified cotton fabric can separate heavy oil-water mixtures again after the solar-induced self-healing process as mentioned above. These results indicated that this as-prepared cotton fabric possesses an excellent and switchable separation capability.

Chemical durability is important for the superhydrophobic surfaces to practical oil/water separation in various harsh environments.⁴⁸ Figure S6 displays the separating ability of the superhydrophobic fabric for the acid solution (HCl, pH=1) or alkali solution (NaOH, pH=12) and oil mixtures. Due to its chemical durability, it is also efficient to separate the corrosive mixtures of oil and acid/alkali solutions.

Furthermore, this modification technology can be applied on other substrates due to the widely applicability of dopamine. Herein we chose another six types of fabrics silk, wool, polyester, acrylic, viscose and nylon to investigate their anti-wetting performance. It was noted that all substrates exhibited excellent superhydrophobic property, and the water droplets displayed spherical shape on all these surfaces (Fig. 10). Therefore, the demonstrated technique in this study has a much broader application potential on different substrates.

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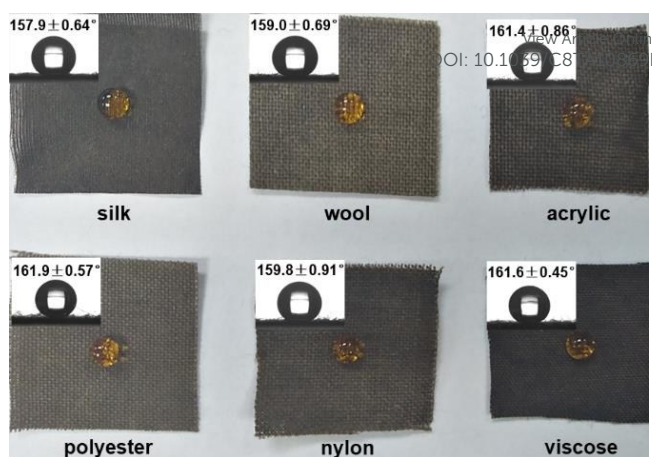


Figure 10. Superhydrophobic property was displayed after modification including different types of fabrics, the inset pictures was the CA of water droplet (40 μ L), and the water droplet was dyed by methyl orange.

Conclusions

In summary, we have successfully constructed eco-friendly and biodegradable superhydrophobic fabrics coated with polydopamine and stearic acid. The coating showed an excellent superhydrophobicity with a CA larger than 160° and a SA lower than 10° . This modified cotton fabric exhibited excellent mechanical stability, laundering durability and chemical stability. In addition, due to the high UV absorption and the photo-thermal conversion of PDA, the modified fabric exhibited excellent UV shielding and solar-induced rapid self-healing property. These remarkable properties made it a good candidate for a range of outdoor textile products for use in conditions with harsh mechanical, chemical, and UV exposure. Besides, the prepared fabric could be utilized as a selective filter membrane for separation of different types of oil-water mixtures. Without using any organic solvents and addition of nano-particles, this biodegradable superhydrophobic fabric is environmentally-friendly. Meanwhile, this facile and inexpensive fabrication process makes it potential for large-scale production of superhydrophobic fabrics.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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