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Abstract: The facile, simple, highly efficient, and fluorine-free fabrication of superhydrophobic surfaces on fabrics with high durability has attracted considerable attention because of its urgent practical application. The simple dip-coating method was adopted to make a stable and durable polydimethyl-siloxane/stearic acid/silica (PDMS/STA/SiO₂) superhydrophobic fabric. The fabric's surface morphology, roughness, and composition were analyzed by scanning electron microscopy, atomic force microscopy, and Fourier transform infrared spectroscopy, respectively. The PDMS/STA/SiO₂-coated fabric: demonstrated strong superhydrophobicity (a water contact angle (WCA) of around 163°), efficiently repelled different liquids (milk, coffee, orange juice, Coca-Cola, and 1 M of HCl and NaOH) with a contact angle above 155°, had excellent self-cleaning performance, and retained superhydrophobicity with a WCA greater than 150° after 72 h of ultraviolet irradiation and 700 cycles of mechanical abrasion. The PDMS/STA/SiO₂ coating had few influences on the color fastness of the fabric. Superhydrophobic coatings are expected to be practically applied in the textile industry.

Keywords: superhydrophobicity; robustness; resistance; polyster fabric; color fastness

1. Introduction

Polyester fabrics can be extensively applied in daily life and industries by virtue of their advantages such as comfortableness, excellent elasticity, wrinkle resistance, and breathability [1–3]. However, their further application is limited by the drawbacks of not being waterproof and being easily contaminable. Showing special wettability with a water contact angle (WCA) of over 150° and a sliding angle (SA) of below 10°, superhydrophobic materials have developed rapidly since the 1990s [4–7] and have drawn widespread attention because of their practical application value, including self-cleaning [8–11], oilwater separation [12–14], anti-corrosion [15–18], etc. A superhydrophobic property can be realized by using materials with a low surface energy and by constructing appropriate surface roughness with hierarchical micro/nano-structures [19–21]. Fabrics are a suitable candidate to obtain a superhydrophobic surface because of their textured structure, which renders a micro-scale roughness [22] whose superhydrophobic property can help to extend their application.

Consequently, many research efforts have been devoted to the field of superhydrophobic fabrics in recent years. A superhydrophobic fabric surface was fabricated with many methods, including chemical vapor deposition [23,24], plasma etching [25,26], solgel [27–29], electrospinning [30], etc. Xu and his co-researchers [31] were the first to use SiO₂ to immobilize a fabric surface and then cover it with a diblock copolymer of poly((methyl methacrylate)-b-(lauryl methacrylate)) (PMMA-b-PLMA), finding that the obtained fabric sample had a WCA of 156° and that the WCA was still above 150° even after 80 laundering



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). cycles or 1600 abrasion cycles. In a two-step process, Fu et al. [32] used Fe_3O_4 , polydopamine (PDA), and hexadecyltrimethoxysilane as coating materials to prepare a durable superhydrophobic fabric exhibiting a WCA of 156°. Coating has enough durability to withstand at least 50 home laundering cycles and 500 abrasion cycles without losing its superhydrophobicity. These studies have made some progress in the preparation of a stable superhydrophobic fabric surface, but most of these fabrication procedures are sophisticated and time-consuming, limiting practical application. Iqbal et al. [33] embedded candle ash nanoparticles into polydimethylsiloxane (PDMS) to prepare a stable superhydrophobic surface. Despite its cost effectiveness, expediency, and environmental friendliness, this method is inappropriate for fabrics as fabrics may be destroyed by the flame of candles. Milionis et al. [34] applied spray coating to prepare a superhydrophobic rubber composite surface. With good elasticity, rubber is capable of absorbing friction energy. Therefore, superhydrophobic surfaces on elastic rubber demonstrate good mechanical stability and strong abrasive resistance. Nevertheless, the method is hard to apply in fabrics because of its requirement of a high temperature (240 °C). Lu et al. [35] made use of commercial adhesives to prepare a superhydrophobic surface with robust mechanical stability but adopted fluorine reagents in their work. Fluorine reagents are costly and generally cause harm to humans and the environment. In practical applications, it remains highly challenging to fabricate a superhydrophobic fabric with good mechanical durability in a simple, highly efficient, and fluorine-free way.

In this study, the dip-coating method was adopted to prepare a durable polydimethylsiloxane/stearic acid/silica (PDMS/STA/SiO₂) superhydrophobic fabric. PDMS, STA, and nano-SiO₂ were mixed in an ethyl acetate solution (EA), where PDMS played the role of a hydrophobic agent and adhesive layer, the long alkyl chains from STA provided low surface energy, and nano-SiO₂ offered nanoscale roughness. The above-mentioned materials, used in the preparation process without complex procedures, are common, cheap, and green. Exhibiting resistance to a variety of liquids, good waterproof property, excellent self-cleaning performance, and ultraviolet (UV) resistance, the PDMS/STA/SiO₂-coated fabric remained superhydrophobic with a WCA above 150° after 700 cycles of mechanical abrasion. Furthermore, that PDMS/STA/SiO₂ coating had few influences on the color fastness of the fabric.

2. Materials and Methods

2.1. Materials

The nano-SiO₂ (an average diameter of 7–40 nm) was purchased from Shanghai Zibo Biotechnology Company. STA (AR) and EA (\geq 99.7%) were obtained from Tianjin Fengchuan. The PDMS prepolymer (Sylgard 184A) and the curing agent (Sylgard 184B) were bought from Dow Corning Corporation (Midland, MI, USA) A commercial polyester fabric (190T, 75D, 96F × 96F) was acquired from a local supermarket and used before being cleaned with distilled water and acetone. All these reagents were used as received.

2.2. Fabrication of Different Hydrophobic Fabrics

Scheme 1 illustrates the process of fabricating the superhydrophobic PDMS/STA/SiO₂ coated fabric. The PDMS (1 g), the curing agent (0.01 g), and the STA (0.5 g) were dissolved in the EA (100 g) and stirred at 300 rpm for 10 min. Then, nano-SiO₂ (0.5 g) was further dispersed in the above solution. Next, the mixed solution was treated with a cell crusher (SCIENTZ-IID) at an amplitude of 60% for 10 min, in which the washed fabric was soaked and subsequently dried at 100 °C for 2 h to obtain the PDMS/STA/SiO₂-coated fabric whose mass loading was ~0.5 mg/cm². For comparison, two other hydrophobic fabrics were prepared by soaking the fabrics in the above mixed solution but without STA and nano-SiO₂ or nano-SiO₂ and denoted as PDMS- and PDMS/STA-coated fabrics whose mass loading was about ~0.45 and 0.47 mg/cm², respectively.



Scheme 1. Fabrication scheme of the superhydrophobic coating on the fabric.

2.3. Tests on Different Liquid Resistance and Waterproof Properties

Different liquids including water, milk, coffee, orange juice, Coca-Cola, and 1 M of HCl (red color) and NaOH (yellow color)—red and yellow colors are due to the methyl orange indicator—were dropped on the fabrics and tested for contact angles (CAs). The waterproof performance test was performed by using two identical plastic bottles (with a diameter of 6 cm) and cutting off the bottoms, which were wrapped with pristine and PDMS/STA/SiO₂-coated fabrics. Then, 200 mL of deionized water (the calculated water column pressure was 700 Pa) was injected into the bottle from the top to observe the waterproof properties of pristine and PDMS/STA/SiO₂-coated fabrics.

2.4. Self-Cleaning Property

The self-cleaning property was measured by injecting water droplets to rinse off the dirt sprinkled on the PDMS/STA/SiO₂-coated fabric.

2.5. Abrasion Analysis and UV Resistance Test

Abrasion analysis was carried out according to national standard ISO 7784-2:1997 by a wear-resistant instrument (Taber JM-IV) mounted on a turntable by two rubber wheels (MSM PU 690450-15), each of which had a load of 500 g. The speed of counterclockwise rotation was 100 circles/min and the WCA was measured every 100 grinding turns. According to national standard ISO 4892-3:2006, the PDMS/STA/SiO₂-coated fabric was placed in a WS-UV-1 UV aging box to test its UV resistance under a 365 nm UV lamp at 60 °C.

2.6. Effect of Coating on the Color Fastness of the Fabric

According to national standard ISO 105-X12-2001, the dry and wet friction resistance fastness of pristine and PDMS/STA/SiO₂-coated fabrics was measured by a rubbing fastness tester (YuanMao Y517). In the test on dry rubbing fastness, the fabrics (pristine and PDMS/STA/SiO₂) were fixed on the testbed and then rubbed by the standard dry white cloth, which was fixed on the rubbing head. With a weight of 9 N, the grinding head had a running range of 10 cm at a reciprocating speed of 60 times per minute. After a standard friction period (20 times), a comparison was made between the colors of the standard dry white cloth (D1 and D2) rubbed by pristine and PDMS/STA/SiO₂-coated fabrics, respectively. In the test on wet rubbing fastness, the standard dry white cloth was moistened with distilled water and then subjected to the same test as above. After a standard friction period (20 times), a comparison was made between the colors of the standard wet white cloth (W1 and W2) rubbed by pristine and PDMS/STA/SiO₂-coated fabrics, respectively.

2.7. Characterization and Instruments

The CA and the SA were measured by a CA analyzer (DSTA 30, KRÜSS, Germany) using 10 μ L liquid drops. Laplace–Young's model was used for the fitting method. For the same sample, the CA and the SA were calculated from five points of dispersion on its surface and were averaged. Fourier transform infrared spectroscopy (FT-IR, Nicolet iS10 FT-IR Spectrometer, Thermo Fisher Scientific, USA) was utilized to confirm the functional groups of the fabrics. Field-emission scanning electron microscopy (SEM, Nova Nano-SEM 450, FEI, USA) was applied to characterize the surface morphologies of the samples. Atomic force microscopy (AFM, MultiMode 8, Bruker, Germany) in a tapping mode was adopted to evaluate the three-dimensional (3D) images of surface roughness.

3. Results and Discussion

3.1. Fabrication and Characterization of Hydrophobic Fabrics

The superhydrophobic property can be realized by using materials with low surface energies and by constructing an appropriate surface roughness with hierarchical micro/nano-structures. Scheme 1 presents the process of preparing the superhydrophobic PDMS/STA/SiO₂-coated fabric. The washed fabric was used as a self-supported substrate to gain an evenly distributed PDMS/STA/SiO₂-coated fabric in a facile dipping coating process using the EA solution mixed with PDMS, STA and nano-SiO₂, among which the PDMS served as a hydrophobic agent and an adhesive layer, the long alkyl chains from the STA provided low surface energy, and the nano-SiO₂ offered nanoscale roughness.

The FTIR spectra of pristine, PDMS-coated, PDMS/STA-coated, and PDMS/STA/SiO₂coated fabrics are shown in Figure 1. Absorption peaks at 1717, 1260, and 726 cm^{-1} are characterized as the polyester fabric [36] and appeared in pristine (Figure 1a) and other fabric samples (Figure 1b–d). It is worth noting that some new peaks appeared in the spectrum of the PDMS/STA/SiO₂-coated fabric (Figure 1d). The new absorption peak at 2963 cm⁻¹ belonged to the bending vibration of Si–CH₃ and those at 1096 and 1019 cm⁻¹ were related to the stretching vibration of Si–O–Si from PDMS [37], which also appeared in the PDMS-coated fabric (Figure 1b). The results indicate that PDMS was successfully coated on the surface of the fabric. Peaks at 2912 and 2850 cm⁻¹ were ascribed to the $-CH_2$ and $-CH_3$ groups of STA, and the peak at 722 cm⁻¹ appertained to characteristic absorbance from the long chains of $-(CH_2)n-(n \ge 4)$ in the long alkyl chain of STA [38,39], which is consistent with the PDMS/STA-coated fabric (Figure 1c) and indicates that the surface of the fabric was coated with STA successfully. The newly appeared absorption band and bending vibration absorption peak at 3450 cm⁻¹ and 474 cm⁻¹ belonged to the Si–OH bond and the Si–O–Si bond in SiO_2 , respectively [40,41], which proves that the PDMS/STA/SiO₂-coated fabric was obtained.

SEM was used to characterize the surface morphologies of pristine, PDMS-coated, PDMS/STA-coated, and PDMS/STA/SiO₂-coated fabric samples (Figure 2). Clearly, the diameter of a single fiber was about $10-20 \,\mu\text{m}$ and the pristine fabric showed a relatively smooth surface with no micro/nano-structures (Figure 2a). The WCA is 0°. Figure 2b shows the surface of the fabric were covered with a layer of PDMS. The PDMS-coated fabric exhibited a WCA of 130° and increased hydrophobicity compared with the pristine fabric because the PDMS provides the methyl group with a low surface energy as a hydrophobic agent [42]. The PDMS/STA-coated fabric appeared to be thicker because of some tiny bulges (Figure 2c). The PDMS/STA-coated fabric's WCA was increased to 151° because the long alkyl chains from the STA further provided some roughness and low surface energy [37]. The surface morphology of the PDMS/STA/SiO₂ fabric sample is presented in Figure 2d. It can be seen clearly that nanoparticles nearly covered the whole fiber structure and formed a micro/nano structure that beneficially complemented the microscale roughness inherently existing in the fabric weave, directing appropriate roughness to the realization of superhydrophobicity. As expected, the WCA of the PDMS/STA/SiO₂ fabric reached 163°, which was the largest among the samples.



Figure 1. Fourier transform infrared spectroscopy (FTIR) spectra of fabrics: (**a**) pristine fabric, (**b**) PDMS-coated fabric, (**c**) PDMS/STA-coated fabric, and (**d**) PDMS/STA/SiO₂-coated fabric.



Figure 2. Scanning electron microscopy (SEM) images of the fabrics: (**a**) pristine fabric, (**b**) PDMS-coated fabric, (**c**) PDMS/STA-coated fabric, and (**d**) PDMS/STA/SiO₂-coated fabric. Insets are differently magnified SEM images and WCAs of corresponding fabrics.

The surface topographies and the root-mean-square roughness (RMS) of the pristine, PDMS-coated, PDMS/STA-coated, and PDMS/STA/SiO₂-coated fabric samples were analyzed by AFM (Figure 3). AFM is an effective tool to characterize roughness and the RMS value represents more significant roughness. It can be observed that the fiber of the pristine fabric was relatively smooth, and the RMS was only 4.6 nm (Figure 3a). After only being treated with the PDMS, the RMS was 7.7 nm (Figure 3b). With the addition of STA, the fluctuation of the fabric surface increased slightly and the RMS increased to 13.9 nm (Figure 3c). Figure 3d shows the AFM of the PDMS/STA/SiO₂-coated fabric samples. With the addition of nano-SiO₂, the RMS increased to 43.6 nm. This result indicates that immobilized nano-SiO₂ on the fabric surface can greatly improve the surface roughness. The surface topography information obtained from the AFM images is consistent with the SEM characterization.



Figure 3. Atomic force microscopy (AFM) images of the fabrics: (**a**) pristine fabric, (**b**) PDMS-coated fabric, (**c**) PDMS/STA-coated fabric, and (**d**) PDMS/STA/SiO₂-coated fabric.

3.2. Superhydrophobicity of the PDMS/STA/SiO₂-Coated Fabric

Fabrics are a kind of widely used materials in daily life. After being treated with PDMS/STA/SiO₂, the fabric exhibited superhydrophobicity to resist different liquids. Figure 4a shows the shapes of water, milk, coffee, orange juice, Coca-Cola, and 1 M of HCl and NaOH droplets on the PDMS/STA/SiO₂-coated fabric, which had a CA of 163°, 160°, 162°, 156°, 160°, 156°, and 156° and a SA of 7°, 9°, 9°, 8°, 7°, 8°, and 9°, respectively (Figure 4b). Resistant to corrosive liquids, the PDMS/STA/SiO₂-coated fabric can protect human skin from damage, which is difficult for pristine fabrics to achieve because of permeation. To further manifest the excellent waterproof performance of the PDMS/STA/SiO₂-coated fabric, the bottoms of plastic bottles were wrapped with pristine and PDMS/STA/SiO₂-coated fabrics and filled with 200 mL of deionized water (Figure 4c). The pristine fabric was quickly infiltrated because of the capillary effect of its fibrous structure. As expected, the PDMS/STA/SiO₂-coated fabric remained unsoaked after 30 min, and a typical bright silver mirror-like surface was noticed because of the total reflectance of light trapped on the surface in the air layer, indicating the stable Cassie-Baxter state of the water drops and the presence of a stable air cushion beneath them. This exceptional superhydrophobicity was attributed to the synergistic effect of rich roughness



and the low energy surface of the PDMS/STA/SiO₂-coated fabric, contributing to the capture of more air and the enhancement of superhydrophobicity.

Figure 4. (a) Images of different kinds of liquids dropped on the PDMS/STA/SiO₂-coated fabric. (b) contact angles (CAs) and sliding angles (SAs) of different liquids dropped on the PDMS/STA/SiO₂-coated fabric. (c) Waterproof performance of pristine and coated fabrics and a bright silver mirror-like surface observed on the waterlogged side of the PDMS/STA/SiO₂-coated fabric.

3.3. Self-Cleaning Property of the PDMS/STA/SiO₂-Coated Fabric

The pristine fabric has a commonly known weakness of easy contamination, whereas the PDMS/STA/SiO₂-coated fabric possesses good self-cleaning performance for the easy removal of dust, as demonstrated in Figure 5. Water drops were rolled to completely clean up the dust scattered on the surface of the PDMS/STA/SiO₂-coated fabric, which finally became clean (Supplementary Materials Video S1), demonstrating that the PDMS/STA/SiO₂-coated fabric has an excellent self-cleaning property.

3.4. UV Resistance of the PDMS/STA/SiO₂-Coated Fabric

Because of frequent exposure to UV irradiation outdoors, fabrics have possess the property of UV resistance. The UV aging box was applied to perform the UV resistance of the superhydrophobic fabric. WCAs and SAs at different UV irradiation times are shown in Figure 6. After UV illumination for 72 h, the WCAs of the coated fabric barely decreased, but all surpassed 150°, and SAs increased slightly with UV irradiation time. It can be seen that the water droplet still exhibited a sphere shape on the surface of the PDMS/STA/SiO₂-coated fabric, which can be ascribed to a large number of Si–O bonds that cannot be broken by UV light [43]. Considering that the intensity of UV light is stronger than that of sunlight, the results clearly show that the PDMS/STA/SiO₂-coated fabric has excellent UV resistance.



Figure 5. The self-cleaning property of the PDMS/STA/SiO₂-coated fabric.



Figure 6. Changes in the WCAs and SAs of the PDMS/STA/SiO₂-coated fabric with UV irradiation time. Insets are photographs of a water droplet on the PDMS/STA/SiO₂-coated fabric before and after 72 h of UV irradiation.

3.5. Effect of PDMS/STA/SiO₂ Coating on the Color Fastness of the Fabric

Color fastness refers to the fading degree of dyed fabrics under the action of external factors (such as squeezing, friction, washing, and perspiration). Color fastness to rubbing is the most basic assessment standard for the color fastness of textiles and includes two types: dry and wet rubbing. In this study, a comparison was made between the dry and wet color fastness of pristine and PDMS/STA/SiO₂-coated fabrics, respectively. As illustrated in Figure 7, no significant color difference was shown between the standard rubbing

Color fastness to dry rubbing

D1

D2

Color fastness to wet rubbing

W1

W2

white cloth rubbed by pristine and PDMS/STA/SiO₂-coated fabrics, indicating that the PDMS/STA/SiO₂ coating has few influences on the color fastness of the fabric.

Figure 7. Comparison of the dry and wet color fastness of pristine and PDMS/STA/SiO₂-coated fabrics.

3.6. Mechanical Stability of the PDMS/STA/SiO₂-Coated Fabric

Fabrics are widely used in the textile industry, whose wear resistance is essential, but no unified test method and standard has been developed for wear resistance. Most literature reports the use of sandpaper or grinding wheels to test the wear resistance of superhydrophobic fabrics. Considering the rough surfaces of superhydrophobic materials, sandpaper and grinding wheels can even increase the roughness of originally rough surfaces and can break down fibers; it is difficult to estimate the true abrasion resistance of coated fabrics. In this study, a rubber wheel with an elastic surface was used for the test of abrasion resistance [18]. Figure 8a shows the changes in the WCAs and SAs of the PDMS/STA/SiO₂-coated fabric with wear condition. The coated fabric had a WCA of 163° and 151° and a SA of 7° and 18° before abrasion and after 700 wear cycles, respectively (Supplementary Materials Video S2). SEM images of the PDMS/STA/SiO₂-coated fabric with and without the abrasion area are shown in Figure 8b. It can be seen that the surface roughness of the PDMS/STA/SiO₂-coated fabric with and without the abrasion area are shown in Figure 8b. It can be seen that the surface roughness of the PDMS/STA/SiO₂-coated fabric with and without the abrasion area cycles.





Figure 8. (a) Changes in the WCAs and SAs of the PDMS/STA/SiO₂-coated fabric with wear condition. (The insets are an SEM image after 700 wear cycles and a photograph of the rubber wheel wear instrument). (b) Shapes of water droplets on the PDMS/STA/SiO₂-coated fabric with and without the abrasion area.

4. Conclusions

a

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In summary, the PDMS/STA/SiO₂-coated fabric was successfully fabricated with the simple and facile dipping coating method, demonstrated good superhydrophobicity with a WCA of about 163°, efficiently repelled different liquids (milk, coffee, orange juice, Coca-Cola, and 1 M of HCl and NaOH) with a CA of above 155°, exhibited excellent self-cleaning performance and UV resistance (72 h), and still preserved a WCA of 151° even after 700 standard abrasion cycles. Moreover, the PDMS/STA/SiO₂ coating had few influences on the color fastness of the fabric. These findings provide a facile avenue for the effective construction of durable superhydrophobic fabrics and the scale-up preparation of multifunctional textiles.

Supplementary Materials: The following are available online at https://www.mdpi.com/2079-641 2/11/3/326/s1. Video S1: self-cleaning property of the PDMS/STA/SiO₂-coated fabric. Video S2: mechanical stability of the PDMS/STA/SiO₂-coated fabric.

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