




## Article

# Surface Modification of Polyester/Viscose Fabric with Silica Hydrosol and Amino-Functionalized Polydimethylsiloxane for the Preparation of a Fluorine-Free Superhydrophobic and Breathable Textile

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**Abstract:** This work attempted to fabricate superhydrophobic fabric via a simple immersion technique. Textile fabrics were coated with silica nanoparticles prepared from tetraethoxysilane (TEOS) to obtain sufficient roughness with hydrophobic surface chemistry. Then, the coated fabrics were treated with polydimethylsiloxane (PDMS) and aminopropyltriethoxysilane (APTES) to reduce the surface energy. The effects of the PDMS concentration on the surface morphology and superhydrophobicity of as-prepared fabric were investigated. The morphology and the composition of superhydrophobic fabric were characterized by scanning electron microscopy (SEM), energy dispersive X-ray (EDS), and Fourier transform infrared (FTIR) spectroscopy. The results revealed the formation of spherical silica nanoparticles with an average particle size of 250 nm throughout the fabric surface. The possible interactions between silica nanoparticles and APTES, as well as the fabrics, were elucidated. Investigating the hydrophobicity of fabrics via water contact angle (WCA) measurement showed that the treated fabric exhibits excellent water repellency with a water contact angle as high as 151° and a very low water sliding angle. It was also found that the treated fabric maintained most of its hydrophobicity against repeated washing, as the WCA of superhydrophobic fabrics decreased to 141° after 25 repeated washing cycles. The comfort properties of the obtained superhydrophobic fabrics in terms of air permeability and bending length did not reveal any significant changes.

**Keywords:** sol–gel; hybrid coating; superhydrophobic; textile fabric; polydimethylsiloxane; contact angle



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## 1. Introduction

A surface with a water contact angle (at equilibrium) higher than 150° and contact angle hysteresis lower than 10° is considered a superhydrophobic surface. Nowadays, superhydrophobic coatings have gained increasing attention due to their diverse applications, including electronic devices, water repellent and self-cleaning textiles, anti-icing surfaces suitable for power network equipment, anticorrosion devices, biomedical devices, construction industry, antibacterial fabrics, anti-biofouling surfaces in the marine industry, and oil–water separation [1–3].

A combination of micro–nano roughness and low surface energy can lead to a superhydrophobic surface. So, an intrinsically hydrophobic substrate can be converted to a superhydrophobic structure by creating micro–nano roughness on its surface by utilizing plasma etching, chemical etching, nanoparticle attachment, etc. The hydrophilic surfaces' superhydrophobicity can be obtained by chemically modifying the micro–nano rough surface with low-surface-energy compounds [2,4–7].

The application of different nanoparticles on the surface of textiles and grafting of hydrophobic bio-based and synthetic compounds has been considered as an effective approach for the fabrication of hydrophobic and water-repellent fabrics [8–12]. One of the widely studied strategies for the fabrication of superhydrophobic textiles is based on increasing the surface roughness by coating the surface with inorganic nanoparticles through the sol–gel process and subsequently lowering the surface energy by attachment of a hydrophobic compound on its surface. Additionally, using a sol, modified with an appropriate hydrophobic compound, may lead to a superhydrophobic surface coating by a one-step process [13]. Hoefnagels et al. [14] turned hydrophilic cotton to superhydrophobic with a two-step process including the sol–gel-based in situ growing of silica micro-particles on cotton fibers followed by a hydrophobization step using polydimethylsiloxane (PDMS). Xu et al. [15] examined the coating of the cotton surface with SiO<sub>2</sub> nanoparticles or ZnO nanorods for the creation of nano-roughness and modification of the rough surface with n-dodecyltrimethoxysilane (DTMS) to lower the surface energy and prepared superhydrophobic cotton fabrics. Based on their results, cotton fabrics prepared based on SiO<sub>2</sub> nanoparticles and ZnO nanorods showed static water contact angles (WCAs) of 159° and 153°, respectively. Endiarova et al. obtained superhydrophobicity on coarse calico fabric (cotton) by dipping in a solution of AlCl<sub>3</sub> (25–35 g/L) for 15 min and ironing at 120 °C due to the formation of aluminum oxide on the surface of the cotton fibers [16]. Seth and Jana fabricated a superhydrophobic cotton fabric based on micro and nano structures of nickel stearate, which showed a static water contact angle of 160°. It showed wash-fast antibacterial properties and high efficiency in oil/water separation as well [17]. The vapor phase polymerization of pyrrole on cotton using Fe<sup>3+</sup> ions and stearic acid resulted in superhydrophobic cotton with a high separation efficiency of oils from water [18].

Superhydrophobic PET fabric was obtained by a two-step process, including coating with SiO<sub>2</sub> nanoparticles and PDMS in the first step and subsequent hydrophobization through a sol–gel process using tetraethoxysilane and cetyltrimethoxysilane. The coated textile showed a WCA of 162.5° and was resistant to hydrostatic pressure up to 38.6 KPa [19]. Xue et al. [20] fabricated a self-healing and superhydrophobic PET fabric using polydimethylsiloxane and octadecylamine (ODA) through a dip-coating followed by a curing procedure. The required roughness was provided by the self-roughed property of ODA, and the obtained superhydrophobicity was durable to abrasion (5000 cycles) and washing (120 cycles). Huang et al. prepared superhydrophobic polyester fabrics with a one-pot sol–gel method employing hexadecyltrimethoxysilane (HDTMS), polymethylhydrosiloxane (PMHS), and hydroxyl-terminated polydimethylsiloxane (HTPDMS), which showed high efficiency in oil separation from water [21].

TiO<sub>2</sub> sol modified with poly(hexafluorobutyl methacrylate) was another approach employed by Yang et al. [22] to prepare superhydrophobic cotton fabrics with a WCA of 152.5°. Chauhan et al. reported the preparation of superhydrophobic cotton with self-cleaning and stain-resistant properties by simple coating with hexadecyltrimethoxysilane with the immersion–drying method. A WCA of 157° was obtained. It was concluded that the high contact angle was due to the hierarchical microstructures and the presence of long-chain alkyl groups on the modified cotton surface [23].

Herein, we present a facile and non-fluorinated approach to construct superhydrophobic and breathable fabric based on hydrolysis and condensation of tetraethylorthosilicate (TEOS) followed by crosslinking with amino-modified polydimethylsiloxane (PDMS). The formation of silica nanoparticles, morphology, and structure of treated fabric were investigated by scanning electron microscopy (SEM), energy dispersive X-ray (EDS), and Fourier transform infrared (FTIR) spectroscopy. The treated fabric exhibited excellent water repellency with durable washing fastness.

## 2. Experimental Procedure

### 2.1. Materials

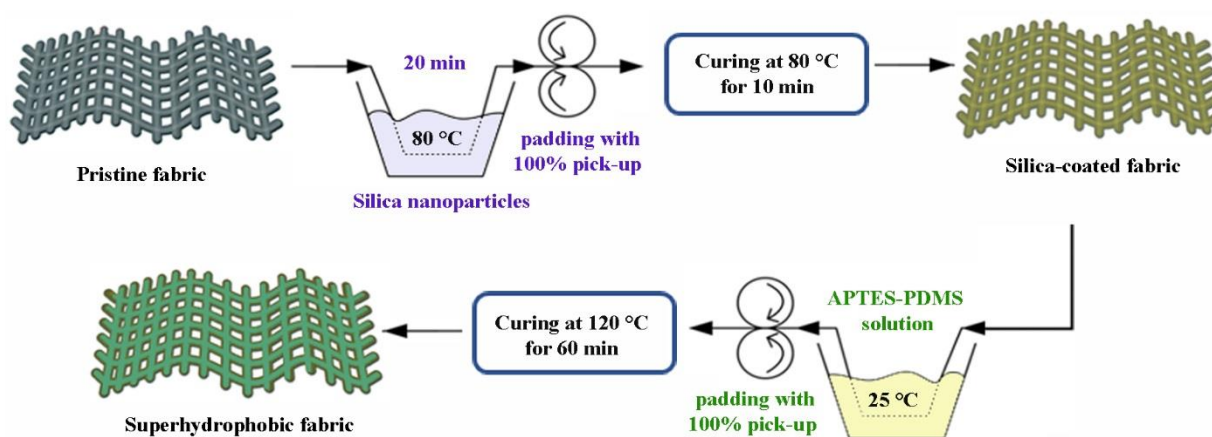
Poly(dimethylsiloxane) mono-glycidyl ether terminated (PDMS) and tetraethylorthosilicate (TEOS, 98%) were purchased from Sigma–Aldrich, (St. Louis, MO, USA). Ammonium hydroxide (25% in water), ethanol ( $C_2H_6O$ ), toluene ( $C_7H_8$ ), and 3-aminopropyltriethoxysilane (APTES) were supplied from Merck Co. (Darmstadt, Germany) and used without additional purification. Commercial polyester-viscose fabrics ( $350\text{ g/m}^2$ , Yazdbaf Co., Yazd, Iran) were used as the substrate. The fabric was scoured with 5 g/L of non-ionic detergent (Triton X-100, Sigma-Aldrich, St. Louis, MO, USA), rinsed with distilled water separately several times, and dried at  $100\text{ }^\circ\text{C}$  for 30 min. Deionized water was used in all the prepared solutions.

### 2.2. Fabrication of Silica-Coated Fabrics

The spherical silica nanoparticles were prepared by the modified Stober method [24]. Briefly, first, a mixture of TEOS (0.39 mol) and ethanol (2.35 mol) was prepared as solution A. Similarly, 2.94 mol distilled water, 2.35 mol ethanol, and  $NH_4OH$  were mixed to form solution B. Then, solutions A and B were mixed at  $40\text{ }^\circ\text{C}$  for 40 min. Finally, the washed fabric was immersed in a solution of silica nanoparticles for 20 min and after dip-coating (100% pick-up), the fabrics were left at room temperature to remove the solvent and cured at  $80\text{ }^\circ\text{C}$  for 10 min.

### 2.3. Fabrication of Superhydrophobic Fabric

In this procedure, different amounts of PDMS were first added into 50 mL toluene to find the optimum value. After stirring about 60 min, 0.5 mL APTES solution was added. Then, the mixture was stirred (500 rpm) for 3 h at room temperature. Finally, the silica-coated fabrics were dip-coated in APTES-PDMS solution for 20 min (100% pick-up) and cured at  $120\text{ }^\circ\text{C}$  for 60 min. The schematic representation of the superhydrophobic coating of fabric is shown in Figure 1.



**Figure 1.** Schematic representation of fabric superhydrophobic coating.

### 2.4. Characterization

The surface morphology of pristine and treated fabrics, as well as the composition of materials, were studied after gold coating of samples by scanning electron microscopy (Hitachi su3500, Tokyo, Japan) and energy-dispersive X-ray spectroscopy (EDS). Fourier transformed infrared (FTIR) spectra of samples were recorded on AVATAR FTIR instrument (Thermo Nicolet, Madison, WI, USA) to study the functional group analysis and possible reactions.

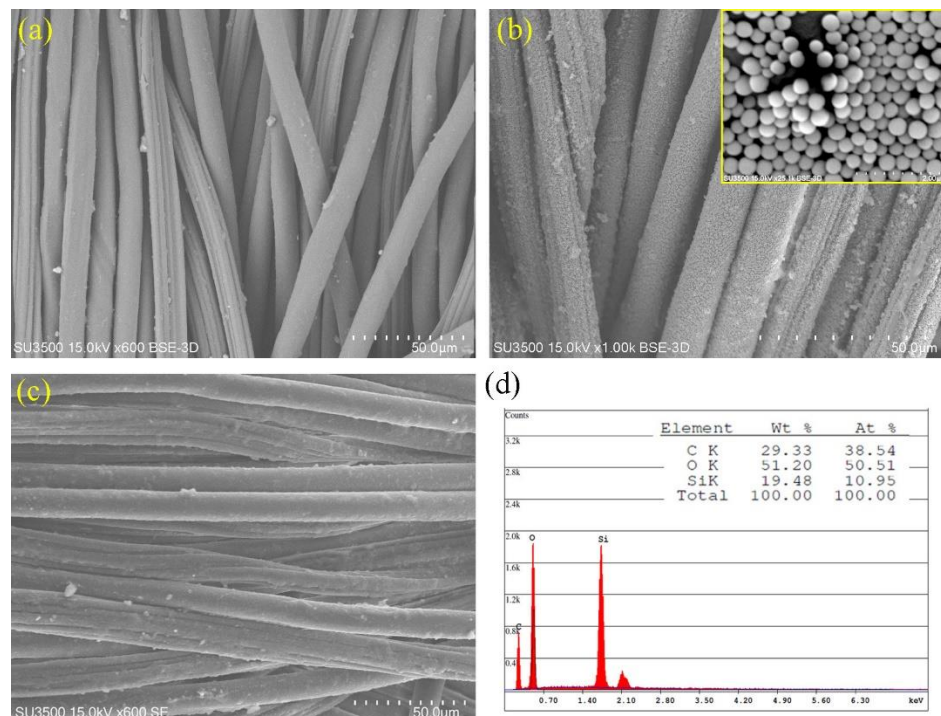
Water contact angle (CA) measurement was carried out at room temperature using homemade instrumentation, including a microscope equipped with a CCD camera and PCTV vision software (Version: 4.1.0.148). A  $5\text{ }\mu\text{L}$  water droplet was dropped to the five different locations of fabric surface, and an average value was reported. Wash fastness of

superhydrophobic fabric, as an important characteristic in terms of users, was evaluated based on AATCC Test Method 61-2A. The treated fabrics were washed with an aqueous solution of an anionic detergent (0.23%) at 45 °C with mild agitation, followed by rinsing with water and drying at 49 °C for 45 min after each washing cycle. According to this standard, each washing cycle is equivalent to five home machine launderings at 38 °C. The washing operation was repeated five times. The air permeability of treated samples was evaluated based on ASTM-D737-75 by using the SDL-Air Permeability device (TESTEX, Guangdong, China). Crease recovery angle was measured based on ASTM D123 standard with Shirley device. The bending test, as a very important factor reflecting the flexibility of the fabrics, was performed according to ASTM-D1388 standard with Shirley Bending length device. Five samples (both warp and weft) were measured, and the average value was reported.

### 3. Results and Discussion

#### 3.1. Morphological Analysis

Figure 2 shows the SEM micrographs of pristine and treated fabrics. The results exhibited a rough surface and the presence of silica nanoparticles on the surface of the fabric. The high-magnification (inset) SEM image (Figure 2b) showed that the uniform silica nanoparticles were deposited throughout the fabric surface with an average particle size of about 290 nm. After modification of silica-coated fabric with the PDMS-APTES solution, the surface morphology becomes inhomogeneous with sticky particles. The change in the morphology of PDMS-APTES treated silica-coated fabric is clearly shown in Figure 2c. The results of EDS spectra (Figure 2d) confirm the presence of Si on the superhydrophobic fabric surface. The peaks of C and O elements were also detected in EDS spectra, which are attributed to the polymer chain.



**Figure 2.** SEM micrographs of fabrics: (a) pristine, (b) silica-coated, (c) PDMS-APTES treated, and (d) EDS pattern of superhydrophobic fabric.

#### 3.2. Structural Analysis

Amino-modification of mono-glycidyl ether terminated PDMS with APTES was carried out, and the possible reactions were investigated by FTIR (Figure 3a). The diminishing characteristic absorption band of the epoxide ring at 913  $\text{cm}^{-1}$  as well as the increasing



hydroxyl and amine groups at  $3420$  and  $1495\text{ cm}^{-1}$ , respectively, confirm the successful amine-modification of PDMS [25]. As can be seen in the spectrum of modified PDMS, the absorption band of the oxirane group was completely disappeared, revealing a complete reaction between APTES and PDMS. The possible chemical reaction process between silica nanoparticles and modified PDMS is represented in Scheme 1. The FTIR spectrum of pristine and treated fabrics is shown in Figure 3b. As can be seen, the Si–O–Si absorption band near  $1100\text{ cm}^{-1}$  becomes sharper and more intense after the treatment of fabric with silica and modified PDMS.

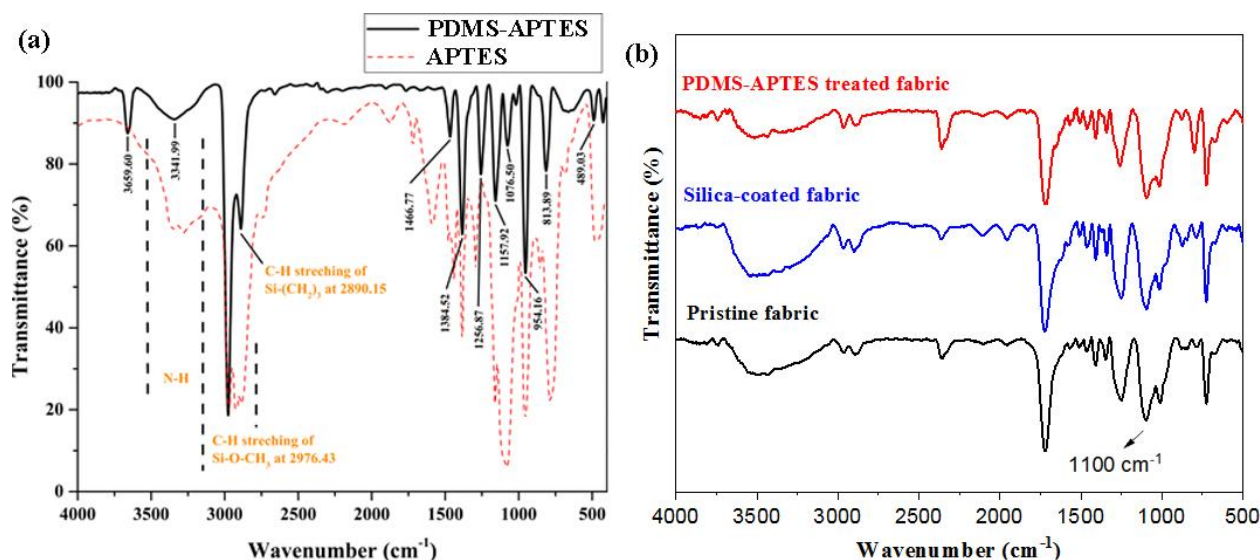
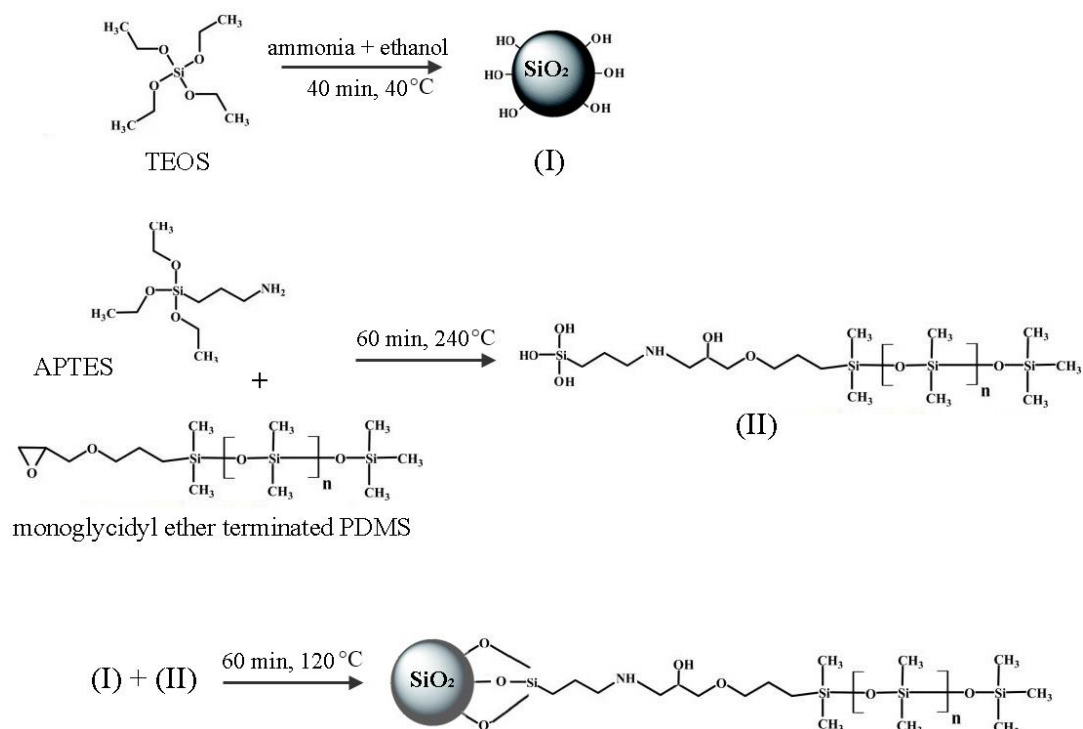


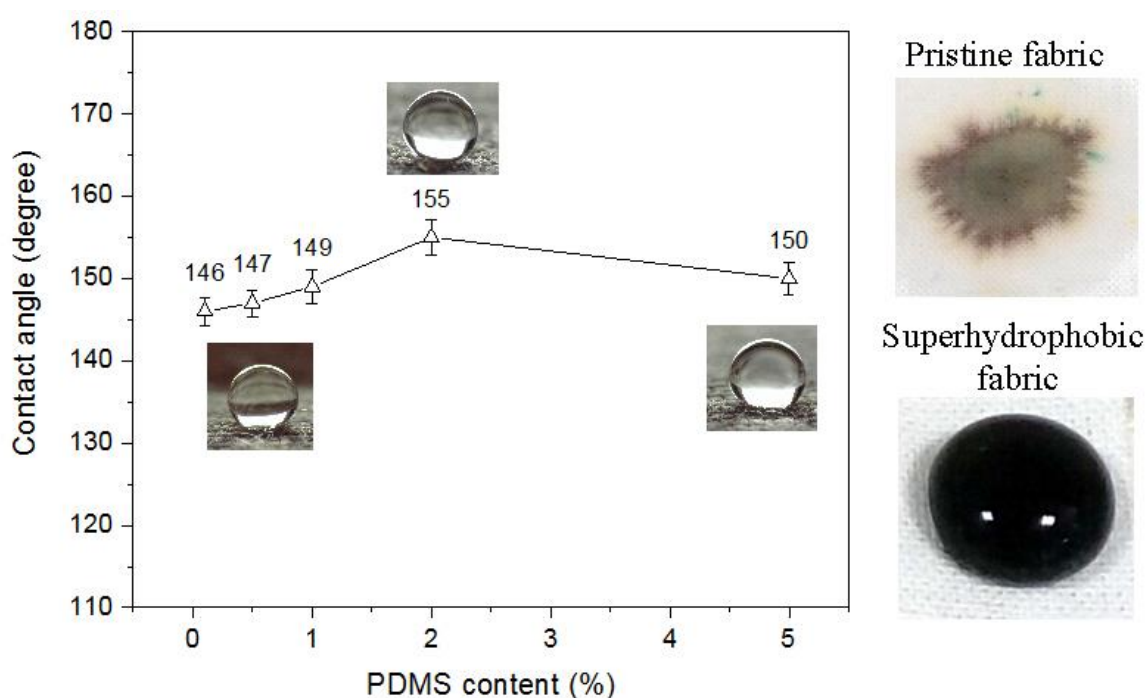
Figure 3. FTIR spectra of (a) amino-modified PDMS solution; (b) pristine and treated fabrics.



Scheme 1. Possible chemical reaction scheme for the synthesis of amino-modified PDMS and superhydrophobic fabric.

### 3.3. Superhydrophobic Property

The hydrophobicity of fabrics was assessed using water contact angle (WCA) measurement. The pristine fabric, due to the presence of hydrophilic hydroxyl and carboxyl groups, was completely wetted by a water droplet. The in situ deposition of silica nanoparticles makes the fabric surface rough, as depicted in the SEM image (Figure 2b), and induces hydrophobicity. After modification of the silica-coated fabric with the APTES-PDMS solution, the fabric was completely turned superhydrophobic (Figure 4) due to the low-surface energy of PDMS. The effect of the PDMS concentration on the hydrophobicity and contact angle of treated fabric was studied at 25 °C by changing its value in the range of 1 to 5%. As can be seen in Figure 4, all the concentrations provided superhydrophobicity, although the maximum contact angle (155°) was achieved at a 2% concentration of PDMS. The high water contact angle obtained here is due to the formation of micro-nano roughness by silica nanoparticles as well as the low surface energy provided by PDMS.



**Figure 4.** The effect of PDMS content on contact angle of treated fabrics.

Table 1 summarizes the hydrophobic properties of different substrates in terms of fabrication approach, contact angle, and washing fastness. As shown in Table 1, with respect to the reported literature, the proposed fluorine-free coating formulation exhibited robust superhydrophobic with a water contact angle higher than 150°.

### 3.4. Physical Properties

The breathability and physical properties of pristine and superhydrophobic fabric were measured in terms of the air permeability, crease recovery angle, and bending length, and the obtained results are summarized in Table 2. The air permeability of treated fabrics, which serves as an indication of their breathability, was investigated. It can be seen that the superhydrophobic treatment of silica-coated fabric with the PDMS-APTES solution moderately decreases the air permeability of fabric by about 36.3%. The crease recovery angle of fabrics (Table 2) indicates that the superhydrophobic fabric exhibits a lower crease recovery angle than the pristine fabric. It can be seen that for pristine fabric, the crease recovery angle in warp and weft directions are 155° and 163°, respectively, and both values gradually decreased by only 10.6% (warp) and 10.4% (weft) after superhydrophobic treatment. The effects of superhydrophobic treatment on the bending rigidity of fabrics in both the warp and weft directions are also shown in Table 2. There were increases in

the bending length of superhydrophobic fabric for both the warp and weft directions. This could be attributed to the formation of hydrogen bonds between the hydroxyl group of fibers and the hydroxyl group of the superhydrophobic coating, making the fiber a bit difficult to bend.

**Table 1.** Comparison of hydrophobicity of different substrates using various approaches.

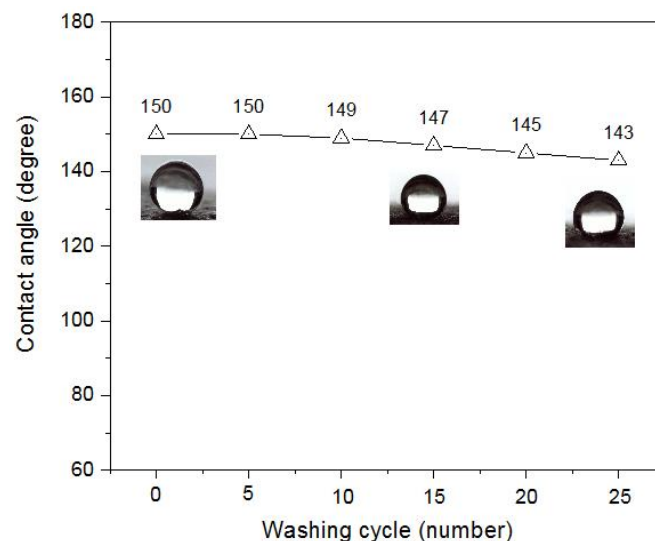
No.	Substrate	Material	Hydrophobic Agent (%)	Method	Contact Angle (°)	Wash Cycle (Number)	Ref.
1	Cotton	HDTMS-GPTMS-TEOS	3	Immersion	141	10	[26]
2	Polyester-cotton	Flour-acrylate monomer	-	Plasma	145	-	[27]
3	Silicon	Si with XeF <sub>2</sub>	-	Etch	156	-	[28]
4	Cotton	TEOS-OTES-AgNO <sub>3</sub>	3	Immersion	151	-	[29]
5	Cotton	MTMS-HDTMS	3	Sol-gel	134	-	[30]
6	Cotton	MTCS	1	Immersion	150	-	[31]
7	Silicon	Modified SiNPs with APTS	1	Immersion	143	-	[32]
8	Silicone	PDMS/SiO <sub>2</sub>	4	Immersion	153	5	[33]
9	Glass	PDMS-nanosilica + FAS	4	Spray	158	-	[34]
10	Glass	Polydimethylsiloxane/silica	0.6	Drop-coating	155	-	[35]
11	Cotton	TEOS-FAS	1.4	Drop-coating	163	100	[36]
12	Glass	PDMS and polystyrene/ SiO <sub>2</sub>	-	Etch	155	4	[37]
13	Glass	PDMS	0.5	Immersion	120	-	[38]
14	Cotton	PDMS-ormosil	2	Immersion	160	5	[39]
15	Polyester	HDTMS-TEOS	4	Immersion	150	30	[13]
16	Polyester-viscose	Modified silica nanoparticles with PDMS	2	Immersion	152	25	This study

**Table 2.** Physical properties of pristine and superhydrophobic fabrics.

Sample	Air Permeability (mL/s. cm <sup>2</sup> )	Crease Recovery Angle (°)		Bending Length (cm)	
		Warp	Weft	Warp	Weft
Pristine Fabric	26.67	155	163	1.69	1.27
Superhydrophobic Fabric	17.00	138.5	146	2.94	1.61

### 3.5. Durability

To study the durability of the treated fabric against laundering, they were subjected to several washing cycles. The CA of treated fabric as a function of washing cycles is shown in Figure 5. The obtained results imply that the CA of superhydrophobic fabric after 25 domestic washing cycles with water and 0.23% detergent solution decreased from 150° to 143°.



**Figure 5.** The contact angle of superhydrophobic fabrics as a function of washing cycles.

#### 4. Conclusions

We successfully fabricated superhydrophobic fabric by modifying the pristine fabric with silica nanoparticles followed by amino-modified PDMS through a dip-coating process. The morphological study of treated fabric exhibits spherical silica nanoparticles distributed throughout the fabric surface, providing the necessary nanoscale roughness for hydrophobicity. The prepared fabric shows outstanding superhydrophobicity with a contact angle of  $151^\circ$  and long-term stability in washing cycles. The employed compounds provided micro-nano roughness as well as the suitable functional groups favoring the low surface energy provided by PDMS. The air permeability, bending length, and crease recovery angle of superhydrophobic fabrics revealed that the treatment did not significantly affect the comfort properties of the fabric. This work provides new insights into the fabrication of robust superhydrophobic and water-repellent fabrics with physical comfortability.

**Author Contributions:** Conceptualization, M.H. and H.S.F.; methodology, M.H. and H.S.F.; validation, M.H. and A.H.; formal analysis, H.S.F.; investigation, H.S.F.; resources, M.H. and G.R.; data curation, A.H.; writing—original draft preparation, H.S.F.; writing—review and editing, M.H. and A.H.; supervision, M.H.; project administration, M.H.; funding acquisition, M.H. and G.R. All authors have read and agreed to the published version of the manuscript.

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