



Ambient-curable superhydrophobic fabric coating prepared by water-based non-fluorinated formulation



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ABSTRACT

Superhydrophobic coatings can be applied to fabrics to provide water repellent and easy clean properties. Hence, there is an intensive commercial demand for environmentally friendly, cost effective, and scalable technologies of superhydrophobic fabric coatings. Herein, we report a water-based and non-fluorinated sol-gel formulation for ambient curable superhydrophobic fabric coatings. The coating material was prepared by surface modification of silica nanoparticles with (3-aminopropyl) triethoxysilane (APTES) and hexadecyltrimethoxysilane (HDTMS). In this formulation, APTES stabilizes the dispersion of silica nanoparticles in water, and also enhances the adhesion of modified nanoparticles on fabric surfaces. The silica nanoparticles and HDTMS respectively contribute to the superhydrophobic performance by enhancing the surface roughness and reducing the surface free energy. The aqueous coating solution can be applied to fabric surfaces by one-step spraying process, and the coating is ambient curable at room temperature. This facilitates scale-up applications with simplified treatment processes. Coatings have been applied on various fabrics including polyester, cotton and mixed cotton/polyester. Superhydrophobic properties have been verified for coated cotton and cotton/polyester. Static water contact angles above 150° with hysteresis around 10° for 2 μL water droplets have been achieved. The durability of the coating was tested by washing/drying cycles and abrasion cycles.

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1. Introduction

There is an intensive industrial demand for superhydrophobic fabric coatings to provide water repellent and easy clean applications [1]. Meanwhile, in the area of fibre reinforced polymer composites, there is also large demand of hydrophobic coating techniques for natural fibres [2,3]. Some advanced technologies are based on gas phase coating procedures by which a layer of silicone nanofilaments is grown onto the individual fibres [4,5]. Durable hydrophobic performance can be achieved on a wide range of textile surfaces. However, such dry coating process requires an enclosed reaction chamber with controlled atmosphere, which restricts the implementation for practical applications.

Comparably, superhydrophobic coating techniques based on wet chemical processes have attracted more interest due to the capability of simple implementation [6]. Most wet chemical processes are based on solvent-borne treatments with alkyl- [7–11] or partially fluorinated alkyl-components [12–14]. However, such solvent-borne treatments are environmentally undesirable, and at the same time restrict some industrial manufacturers from adopting

the technologies due to various controls or regulatory limitations for specific solvent-borne processing. Besides, for practical applications, fluorinated compounds are not desirable due to the potential risks of the degradation byproducts to human health and the environment [15,16].

Therefore, water-borne coating processes, especially those based on non-fluorinated compositions, are of higher technological interest due to the environmental and health advantages. Nevertheless, the existing water-based non-fluorinated hydrophobic coating technologies usually involve multiple coating steps [17–19] or mandatory thermal curing [20–22]. The requirement of multiple coating steps results in long production time, while mandatory thermal curing makes the technology unsuitable for some specific fabric materials which cannot sustain high temperature treatment.

Herein, we report a formulation method based on water-borne non-fluorinated sol-gel for superhydrophobic fabric coatings. The as-prepared sol-gel formulation can be applied onto fabrics by simple one-step spraying. Besides, durable coatings can be formed by natural curing at room temperature. Therefore, compared with the prior art technologies, the method reported here is more environmentally friendly since the coating material is water-based and non-fluorinated. At the same time, this method is scalable and can be easily adopted due to the capability of one-step spraying and ambient curing at room temperature.

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2. Experiment work

The involved silica nanoparticle was Aeroxide LE2 from Evonik Degussa. (3-Aminopropyl) triethoxysilane (APTES, 99%) and hexadecyltrimethoxysilane (HDTMS, >85%) were purchased from Sigma-Aldrich. All chemicals were directly applied without any further purification. 3 types of fabric substrates were kindly contributed by MEISEI & Co., Ltd., including 100% cotton, 100% polyester, and mixed 35% cotton/65% polyester. The 3 types of substrates are all twill fabrics with weight/area of 128.57 g/m², 143.75 g/m², and 112.50 g/m², respectively. All the pristine fabrics can be immediately wetted by water droplets.

In a typical formulation process, 0.15 g of LE2 nanoparticles was dispersed in 4 g of APTES followed by continuous stirring for 1 h. Then, the mixture was added dropwise into 36 g of distilled water while stirring. After stirring for 2 h, 0.5 g HDTMS was added dropwise into the water solution. The solution was stirred for another 2 h before being applied to fabric substrates. The prepared sol was sprayed onto the fabrics until the fabrics were totally wet. Subsequently, the coated fabrics were naturally dried at room temperature overnight.

Water contact angles were measured by VCA Optima (VCA-2500XE AST products, Inc.) machine with 2 μ L water droplets deposited on the fabrics. The advancing and receding angles were respectively obtained by adding and retrieving water from the droplet with 0.2 μ L interval. Each value of contact angle was calculated based on measurements at 5 positions along the sample surface. The surface morphologies of the coated fabric samples were characterized by scanning electron microscopy (SEM) (Ultra Plus FESEM, operating at 0.8 kV) and Atomic Force Microscopy (AFM) (Dimension Icon Scanning Probe Microscope, operating at Peak Force QNM mode). The sizes of the clusters were examined by Zetasizer Nano ZS Particle Size and Zeta Potential Analyzer (Zetasizer Nano ZS, Malvern).

For each washing/drying test cycle, the fabric sample was firstly hand washed in water solution of 1 wt.% detergent. Then, the fabric surface was thoroughly rinsed with water and dried naturally at room temperature. For the abrasion test, the fabric sample was firstly soaked in water solution of 1 wt.% detergent for 10 min. Then, the fabric surface was brushed for a defined number of cycles at a rate of 30 cycles/min (ASTM D2486, Elcometer 1720 Abrasion Tester), followed by rinsing with water and drying naturally at room temperature.

3. Results and discussion

The coating solution was prepared by the procedures as described in the section [Experiment work](#). The wettability of the coated polyester, cotton, and cotton/polyester samples was characterized by contact

angle measurements. [Fig. 1\(a\)](#) summarizes the static contact angles, advancing angles and receding angles of 2 μ L water droplets deposited on the three types of coated fabrics. [Fig. 1\(b–d\)](#) shows the images of 2 μ L water droplets deposited on the coated fabrics. The average static contact angles on coated polyester, cotton and cotton/polyester were respectively measured to be 146.5°, 153.2° and 155.3°. Besides, according to the measured advancing angles and receding angles, the hysteresis for the three types of coated fabrics were all around 10°. Since a superhydrophobic surface is defined to be a surface with water contact angle larger than 150° and with hysteresis less than 10°, the superhydrophobicity of the coated cotton and cotton/polyester can be confirmed.

The principle of superhydrophobicity is usually attributed to the low surface free energy and the surface roughness. Young's equation explains the wettability of a flat surface, which concludes that larger water contact angle can be achieved by lowering the surface free energy of the flat surface [23]. On the other hand, the Wenzel model [24] and the Cassie–Baxter model [25] have predicted the hydrophobicity of a rough surface resulting from the presence of micro- or nanoscale asperities. Both models predict that higher hydrophobicity can be achieved by lower surface free energy of the solid material or increased roughness of the given surface.

In this work, the superhydrophobic coating material was achieved by surface modification of LE2 nanoparticles with APTES and HDTMS. According to the datasheet provided by the supplier, LE2 nanoparticles are modified silica nanoparticles with hydrophobic surface property due to the presence of methyl groups along the surface. The chemical bonds presenting over the surface of LE2 nanoparticle are Si–CH₃ and Si–OH. Due to the hydrophobic surface property, LE2 nanoparticles cannot be stably dispersed in water. To generate water dispersion of LE2 nanoparticles, APTES is firstly applied to mix with LE2 nanoparticles, followed by dispersing the mixture into water. The nanoparticles will be aggregated into clusters due to the reaction between the hydrolysed APTES and the Si–OH groups along the nanoparticle surface [26–28]. The amine groups (–NH₂) along the surface of each cluster stabilize the dispersion of the individual cluster in water. Subsequently, HDTMS is added into the water dispersion. The hydrolysed HDTMS molecules are bonded to the cluster surface by the formation of Si–O–Si bonds [20]. As a result, long-chain organosilane with low surface free energy is introduced onto each cluster surface, producing an aqueous coating solution for superhydrophobic fabric coatings.

By applying the as-prepared aqueous coating solution onto fabrics, the Si–OH groups along the cluster surfaces bond with the fabric surfaces, and the –NH₂ groups further enhance the adhesion of clusters onto the fabrics. Therefore, the clusters of LE2 nanoparticles can be effectively attached onto the fabrics, enhancing the roughness of the

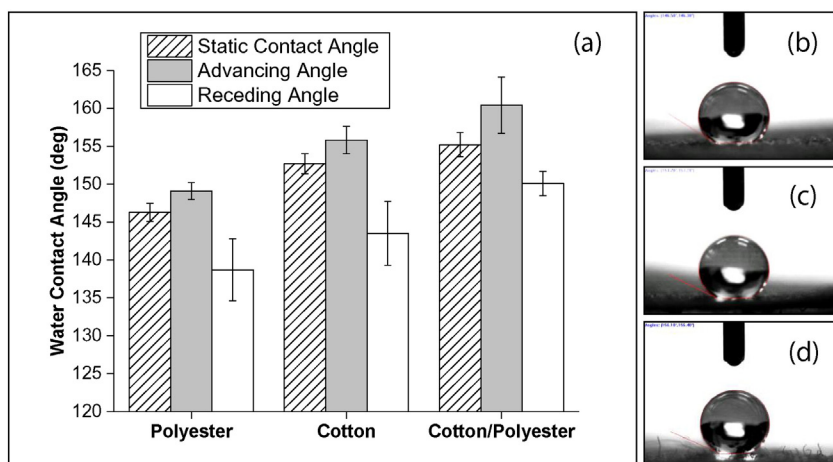


Fig. 1. (a) Measured contact angles on different types of coated fabrics; images of 2 μ L water droplets on coated (b) polyester, (c) cotton, and (d) cotton/polyester.

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