



# A simple cost-effective and eco-friendly wet chemical process for the fabrication of superhydrophobic cotton fabrics



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## ABSTRACT

Superhydrophobic surfaces were created on hydrophilic cotton fabrics by a simple wet chemical process. The fabric was immersed in a colloidal suspension of zinc hydroxide followed by subsequent hydrophobization with stearic acid. The wettability of the modified cotton fabric sample was studied by water contact angle (WCA) and water shedding angle (WSA) measurements. The modified cotton fabrics exhibited superhydrophobicity with a WCA of  $151^\circ$  for  $8\mu\text{L}$  water droplet and a WSA of  $5\text{--}10^\circ$  for  $40\mu\text{L}$  water droplet. The superhydrophobic cotton sample was also characterized by field emission scanning electron microscopy (FESEM) and energy dispersive X-ray spectroscopy (EDX). The method is simple, eco-friendly and cost-effective and can be applied to large area of cotton fabric materials. It was shown that superhydrophobicity of the fabric was due to the combined effect of surface roughness imparted by zinc hydroxide and the low surface energy of stearic acid.

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## 1. Introduction

Superhydrophobic surfaces with water contact angles (WCA) higher than  $150^\circ$  are inspired by natural surfaces such as the lotus leaf, on which water drops remain almost spherical and easily roll off, removing dirt particles in their path [1,2]. Such surfaces have great potential for numerous scientific and industrial applications. Studies have shown that superhydrophobicity arises due to the combined effect of hierarchical micro- and nanostructures of the surfaces as well as from the low surface energy of the materials [3]. Therefore, SH surfaces can be prepared either by creating roughness on the surface of low surface energy materials or by lowering the surface energy of a rough material. Waterproofing of textiles may be considered as one of the potential applications for the superhydrophobic effect [4–16,18–21,24]. Superhydrophobic textiles would be useful for any kind of application where textile surfaces are exposed to the environment.

Even though there are several publications referring to possible textile applications, there are only few reports that actually pertain to superhydrophobic textiles. It has been reported that application of polysiloxanes by certain methods can provide nanostructures on fabrics with microstructures resulting in superhydrophobicity [4,5]. Zimmermann et al. have prepared superhydrophobic textile fabrics by a gas phase coating procedure by which a layer

of polymethylsilsesquioxane nanofilaments with special surface geometry was grown onto different types of textile fibers [5]. However, most of the other reported methods are based on using silica nanoparticles for creating roughness and low surface energy materials such as polysiloxanes [6,7], fluorochemicals [8,9], or long chain alkyl silanes [10–12] for hydrophobic modification. Water repellent property can be obtained by using polysiloxanes for hydrophobization. However, fluorochemicals have to be incorporated for achieving fabrics with both water and oil-repellent property [7–9]. Superhydrophobic water-repellent cotton fabrics have been prepared by treating cotton with silica nanoparticles and a cost-effective commercial water-repellent agent [12]. Layers of two silica nanoparticles with different shapes and sizes were synthesized via a sol–gel process and used to create micro nano roughness required for achieving superhydrophobicity [12,13]. Superoleophobic textiles were obtained by incorporating perfluoroalkyl groups onto the surface of cotton modified with two layers of silica microparticles and nanoparticles covalently bonded to the fiber. However, fluorochemicals have certain disadvantages such as high cost, potential risk for human health as well as environmental concern. Another important requirement for fabric processing is that the technique should be simple and inexpensive and the resulting coatings should be nontoxic and have good chemical and environmental stability.

Superhydrophobic antibacterial fabrics have been fabricated by embedding silver or copper particles and modification of the particle-containing cotton textiles with alkylsilanes [14,15]. Very recently, superhydrophobic surface on cotton substrates has been prepared by dip-coating in Al sol, and modifying with sodium

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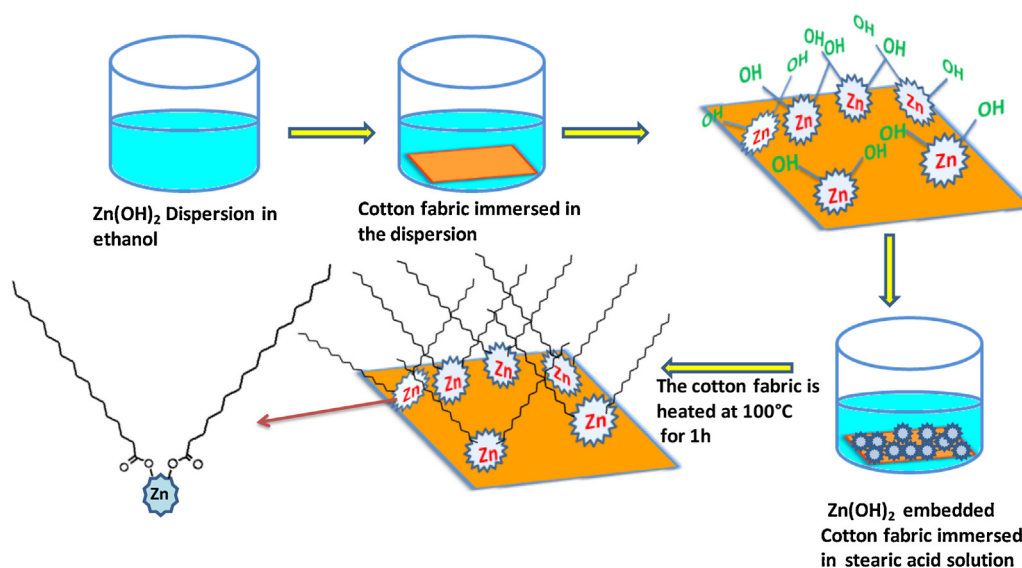


Fig. 1. Schematic of the preparation of superhydrophobic cotton fabric by immersion in ethanolic  $\text{Zn}(\text{OH})_2$  followed by immersion in stearic acid solution.

stearate [16]. However, zinc oxide (ZnO) is well-known for its antibacterial properties and is generally regarded as a safe material for humans; further it is easy to prepare and cost-effective compared to silver and copper nanoparticles. ZnO nanoparticles have been immobilized on fabrics via ultrasound irradiation to render them with anti-bacterial property [17]. Superhydrophobic surfaces on cotton substrate have also been created using ZnO nanorod arrays and subsequent hydrophobic modification with a long chain alkyl silane like n-dodecyl trimethoxysilane [18,19]. Xu et al. have prepared superhydrophobic cotton fabrics by fabricating rough surfaces using silica nanoparticles and ZnO nanorod arrays together with subsequent modification with long chain alkyl silane [19]. Cotton fabrics have also been functionalized with ZnO thin films by pulsed laser deposition to change their wettability [20]. Recently, superhydrophobic and ultraviolet-blocking cotton textiles have been fabricated by a tedious treatment involving various complex processes steps such as ZnO seeding, hydrothermal growth of ZnO nanorods, layer-by-layer deposition of a silica shell on the surface of ZnO nanorods, and hydrophobic modification with octadecyltrimethoxysilane [21].

In the present work, we have employed a facile wet chemical process to render superhydrophobic property to cotton fabric. The cotton fabric is immersed in a dispersion of zinc hydroxide in ethanol and hydrophobically modified by immersing in solution of stearic acid. Zinc hydroxide was synthesized by a simple precipitation method using low cost and non-toxic materials. The treated cotton samples were characterized by water shedding angle measurements, field emission scanning electron microscopy (FESEM) and energy dispersive X-ray spectroscopy (EDX).

## 2. Experimental

### 2.1. Materials

Zinc sulfate was purchased from SD Lab Chem Industry. Anhydrous sodium carbonate was purchased from Sarabhai M Chemicals, Baroda. Sodium hydroxide flakes and ethanol were procured from Merck. Stearic acid (n-octadecanoic acid) was purchased from S.D. Fine-Chem Ltd. Stearic acid solution (0.05 M) was prepared by dissolving 0.70 g in 50 mL ethanol.

### 2.2. Preparation of treated cotton fabric samples

Zinc hydroxide was prepared by a modified precipitation method reported earlier by us and described elsewhere [22]. In brief, 50 mL of 0.10 M aqueous zinc sulfate solution was mixed with 50 mL 0.10 M sodium carbonate and 50 mL 0.10 M sodium hydroxide in a 250 mL two-necked round bottomed flask kept at 70 °C in a water bath while stirring vigorously. The stirring was continued for further 30 min while maintaining the temperature constant at 70 °C. The precipitated zinc hydroxide was separated by centrifugation and repeatedly washed with distilled water and ethanol. The white residue of zinc hydroxide was dried under ambient conditions for several hours till a powder of constant weight was obtained.

0.10 g of zinc hydroxide was dispersed in 10 mL ethanol and ultrasonicated for 30 min to get a colloidal suspension. Clean and dry cotton fabric samples (2 in. × 2 in.) were immersed in the suspension and padded with a glass rod for 10 min on each side, the cotton fabric was then dried at 60 °C for 1 h. Hydrophobic modification of the zinc hydroxide-treated cotton fabric was carried out by immersing it in 10 mL of stearic acid solution for 30 min. The modified cotton fabric was then dried under ambient conditions for 4 h and heat treated at 100 °C for 1 h in an air oven. Fig. 1 shows the schematic representation of the steps involved in the preparation of superhydrophobic cotton fabric.

For comparison, cotton fabric sample treated only with zinc hydroxide was prepared by immersing in zinc hydroxide suspension. Another cotton fabric sample was treated with zinc stearate by immersing in zinc stearate suspension prepared by stirring 0.10 g each of zinc hydroxide and 0.14 g stearic acid in 10 mL ethanol. A third cotton fabric sample was immersed in stearic acid solution. Finally, all three treated fabrics were dried at 100 °C for 1 h. Similarly, samples of zinc hydroxide, zinc stearate and stearic acid coatings on glass slides were also prepared.

### 2.3. Characterization

The crystalline structure of zinc hydroxide applied on the fabric before and after stearic acid treatment after heating at 100 °C was examined by X-ray diffraction (XRD) technique, model Rigaku D/max 2200 powder diffractometer, using Cu K $\alpha$  radiation. The diffraction patterns were scanned between 10° and 80° in steps

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