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Superhydrophobic conductive textiles with antibacterial property by coating fibers with silver nanoparticles

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ABSTRACT

Silver nanoparticles (Ag NPs) were produced on cotton fibers by reduction of $[Ag(NH_3)_2]^+$ complex with glucose. Further modification of the fibers coated by Ag NPs with hexadecyltrimethoxysilane led to superhydrophobic cotton textiles. Scanning electron microscopy images of the textiles showed that the treated fibers were covered with uniform Ag NPs, which generate a dual-size roughness on the textiles favouring the formation of superhydrophobic surfaces, and the Ag NPs formed dense coating around the fibers rendering the intrinsic insulating cotton textiles conductive. Antibacterial test showed that the as-fabricated textiles had high antibacterial activity against the gram-negative bacteria, *Escherichia coli*. These multifunctional textiles might find applications in biomedical electronic devices.

1. Introduction

Materials capable of performing multiple functions simultaneously or sequentially in time are of significance to improve performance of products. Textiles with enhanced functionalities, such as antibacterial [1–4], antistatic [5,6], stain resistant [7], conductive [8], and UV protection [9–12], are greatly appreciated by a more discerning and demanding consumer market for high-value-added products [13]. Multifunctional textiles could be fabricated through combined treatments using several materials with specific property on fibers [14]. However, multifunctionalization of textiles by a single material with intrinsic multifunctionalities, or by a simple treatment which induces mutifunctions simultaneously is preferable.

Silver has been known to be a bactericide since ancient times [15]. Recently, nanosized silver nanoparticles (Ag NPs) have been reported to exhibit antimicrobial properties [16–22]. The incorporation of Ag NPs into various matrices has been intensively investigated to extend their utility in materials and biomedical applications [20,23].

Dubas et al. [24] reported antimicrobial Ag NPs immobilized on nylon and silk fibers by following a layer-by-layer deposition method. The sequential dipping of nylon or silk fibers in dilute solutions of poly(diallyldimethylammonium chloride) and Ag NPs capped with poly(methacrylic acid) led to the formation of a colored thin film possessing antimicrobial properties. Lee et al. [25] reported coating of stable Ag NPs onto cotton fabric by soaking in ethanolic solutions of $AgNO_3$ and butylamine. The Ag NP coated cotton fabric exhibited excellent bactericidal effects on pathogenic bacteria without causing direct skin irritation. Zhao et al. [26] produced nanostructured silver coating layers on polyimide films by treatment with aqueous KOH and $AgNO_3$, followed by thermal treatment at 200 °C or higher temperatures. Further modification of the gold-coated silver layers with n-dodecanethiol led to hydrophobic surfaces.

Khalil-Abad and Yazdanshenas [27] fabricated superhydrophobic antibacterial surfaces on cotton textiles by introducing microsized silver particles to the woven fiber network. They achieved these results through treatment with aqueous KOH and $AgNO_3$, followed by surface hydrophobization. The dual-size surface structure and low surface energy of octyltriethoxysilane modification led to remarkable superhydrophobic surfaces with contact angle as high as 151°. The modified cotton textiles were capable of killing both Gram-negative and Gram-positive bacteria on surfaces.

It is worth noting that, in addition to the biomedical applications, superhydrophobic textiles decorated with silver nanostructures might also be imparted with other properties, such as electrical conductivity [28], antistatic properties [28,29],

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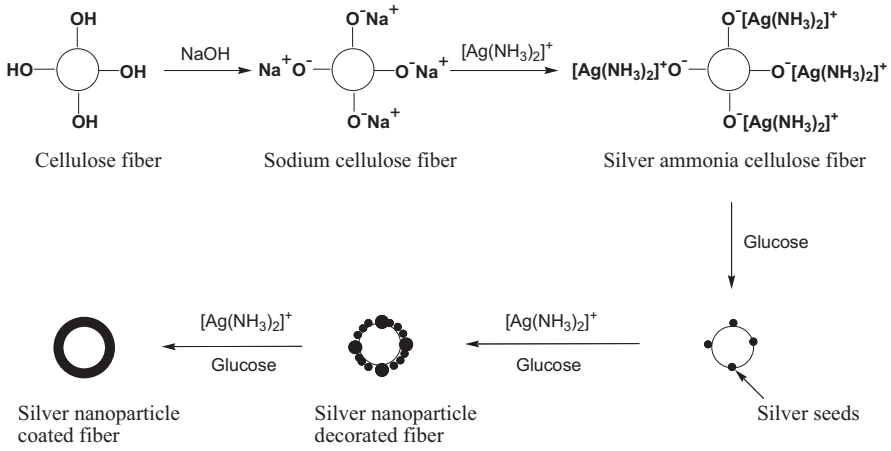

Scheme 1. Illustration of the processes involved in the coating of cotton fibers with silver nanoparticle.

UV-protection [23], etc. Since Ag NPs are easy to prepare with size and shape controllable, multifunctional textiles could be obtained by decorating fibers with size controlled Ag NPs [27,30].

In this contribution, Ag NPs were produced on cotton fibers by *in situ* reduction of $[Ag(NH_3)_2]^+$ with glucose, then the treated textiles were modified by alkylsilane with long chain expecting to fabricate functional textiles with combined properties of superhydrophobicity, conductivity, antibacterial, etc.

2. Experimental

2.1. Materials

$AgNO_3$ (>99%), glucose (>99%), aqua ammonia (28 wt.%), and NaOH were purchased from Sinopharm Chemical Reagent Co., Ltd. Hexadecyltrimethoxysilane (HDTMS) was purchased from Hangzhou Silong Material Technology Co., Ltd. All chemicals were used as received. Desized, scoured, and bleached plain woven cotton textiles (60 ends/in. × 60 picks/in., 20 s × 20 s, 143 g/m^3) were cleaned with deionized water and ethanol before drying for use. Deionized water was used throughout all the experiments.

2.2. Alkali treatment of cotton textiles and coating of fibers with silver particles

Cotton textiles were treated with 10 wt.% aqueous NaOH solution at room temperature for 10 min followed by rinsing with copious amount of distilled water. Aqua ammonia (28 wt.%) was added drop by drop into a 0.5 M $AgNO_3$ aqueous solution with stirring until a transparent colorless $[Ag(NH_3)_2]^+$ solution was formed. The alkali treated textiles were dipped into the $[Ag(NH_3)_2]^+$ solution for 1 h, then transferred into a 0.1 M glucose stock solution. After 5 min, the residual $[Ag(NH_3)_2]^+$ solution was also poured into the glucose solution. The reaction was continued for 15 min. At last, the textiles were rinsed with water and dried in air.

2.3. Hydrophobization of textiles with HDTMS

Due to the low surface energy of silane based molecules with long hydrocarbon chain, HDTMS was used to hydrophobize the textiles according to the report by Khalil-Abad and Yazdanshenas [27] with some modification. Textiles were hydrophobized by dipping the textile sample in an HDTMS ethanol solution (3% vol.), and reacted for typically 1 h at room temperature. The sample was then dried at 80 °C for 10 min and cured at 130 °C for 1 h.

2.4. Characterization

The morphologies of the textiles were observed by a SEM (JSM-6700, JEOL, Japan). The samples were coated with gold using a vacuum sputter coater before SEM use. The water contact angles (WCAs) of the samples were measured at ambient temperature on a video optical contact angle system (OCA 20, Dataphysics, Germany). All the contact angles were determined by averaging values measured at 5–6 different points on each sample surface. X-ray diffraction (XRD) patterns were recorded on a D/max 2200PC diffractometer in θ–2θ configuration.

2.5. Antimicrobial test

Antimicrobial tests were conducted by the bacterial inhibition ring method (agar plate diffusion test/CEN/TC 248 WG 13) and the reduction of bacterial growth test (EN ISO 20743:2007 Transfer Method).

The evaluation of the antibacterial activity of textiles against Gram-negative bacteria *Escherichia coli* (*E. coli*) was as follows. A mixture of nutrient broth and nutrient agar in 1 L distilled water at pH 7.2 as well as the empty Petri plates were autoclaved. The agar medium was then cast into the Petri plates and cooled. Approximately 10^5 colony-forming units of each bacterium were inoculated on plates, and then each textile samples was planted onto the agar plates. All the plates were incubated at 37 °C for 24 h, following which the zone of inhibition was measured.

Reduction of bacterial growth on the textiles was estimated for the *E. coli* using the EN ISO 20743:2007 Transfer Method. The agar plates were inoculated with 1 mL of a nutrient broth culture containing $(3–4) \times 10^5$ colony forming units of bacteria. Afterward, a swatch of the test sample was plated on the agar surface and pressed down. The test sample was then transferred from the agar surface into a 100 mL container with the transferred surface face up, and incubated at 37 °C for 24 h in a humidity chamber. After incubation, 20 mL of neutralizing solution was poured on the test sample and shaken vigorously for 1 min. Serial dilutions were made with sterilized water, and the suspensions were plated on nutrient agar and incubated at 37 °C for 24 h. The reduction of bacterial growth (RA) on the silver treated sample in comparison to the untreated one was calculated as

$$\mathrm{RA}(\%) = \frac{(C - A)}{C} \times 100$$

where C is the number of bacteria-forming units on the untreated textile after 24 h of incubation and A

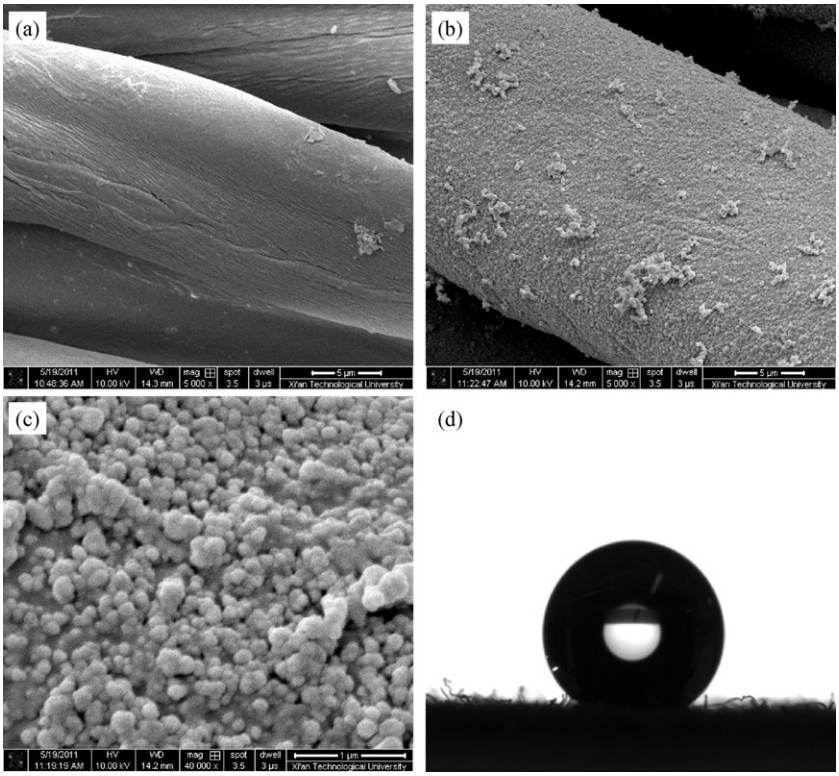

Fig. 1. SEM images of (a) original cotton textiles and (b) cotton fibers treated with silver particles; (c) higher magnification image of (a); (d) image of water droplet on the textile treated with silver particles and HDTMS.

the number of bacteria-forming units on the silver treated textile under the same conditions.

2.6. Resistance measurement

The conductivity of the original, and the modified textiles after hydrophobization was evaluated by randomly measuring the resistance between two points with 1 cm distance on the textiles for the sake of convenience. All the resistances were determined by averaging 6 measured values on each sample surface. Lower resistance means higher conductivity.

3. Results and discussion

Chemical reduction is the most frequently applied method for the preparation of Ag NPs as stable, colloidal dispersions in water or organic solvents [22,31,32]. Commonly used reductants are borohydride, citrate, ascorbate, and elemental hydrogen [22]. Previous studies showed that use of borohydride which is a strong reductant, resulted in small particles that were somewhat monodisperse, but the generation of larger particles was difficult to control [33–35]. Use of citrate which is a weaker reductant resulted in a slower reduction rate [36], but the size distribution was far from narrow [37], and citrate also acts as a capping agent which stabilizes the colloidal particles [38] as dispersion. In this work, it is important to produce silver nanoparticles on fibers instead of in water as dispersion in order to fabricate Ag NP functionalized textiles. Therefore, the cotton fibers were pretreated with NaOH making the fiber surface negatively charged. After dipping the NaOH treated fibers into the silver ammonia complex solution, the complexes of $[Ag(NH_3)_2]^+$ were easily and abundantly absorbed on the fiber surface. And when the silver ammonia complex loaded fibers were transferred in the solution of glucose, $[Ag(NH_3)_2]^+$ was *in situ* reduced into silver seeds on the fiber surface. With the addition of $[Ag(NH_3)_2]^+$ and glucose, more and more silver ions were absorbed onto the fiber surface and reduced into silver, and the silver seeds grew larger into Ag NP, attached to each other forming a compact Ag NP coating on the fibers. These processes were illustrated in Scheme 1.

SEM images of cotton fibers before and after coating with silver particles are shown in Fig. 1. It can be seen from Fig. 1(a) that the pristine cotton fibers show typical longitudinal fibril structure with clean and smooth surface. After coating treatment, the cotton fiber surfaces show a compact and uniform covering of particles, as shown in Fig. 1(b). And the higher magnification image of the treated fiber in Fig. 1(c) shows that the particles of sizes ranging from 100 to 300 nm are clearly visible, making the fiber

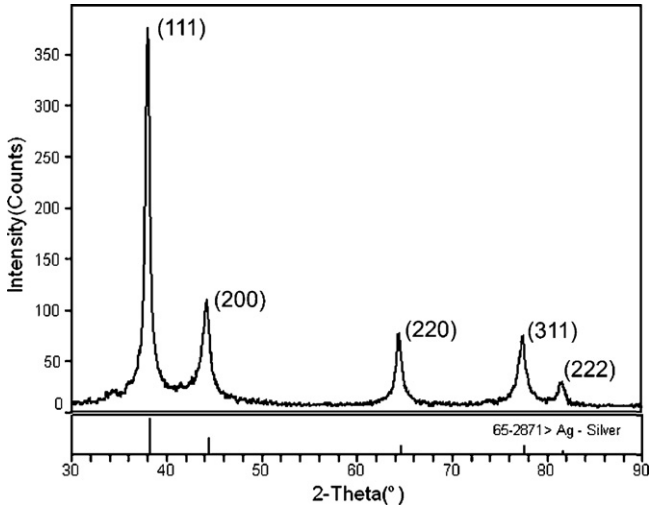

Fig. 2. XRD pattern of the textiles coated with silver particles.

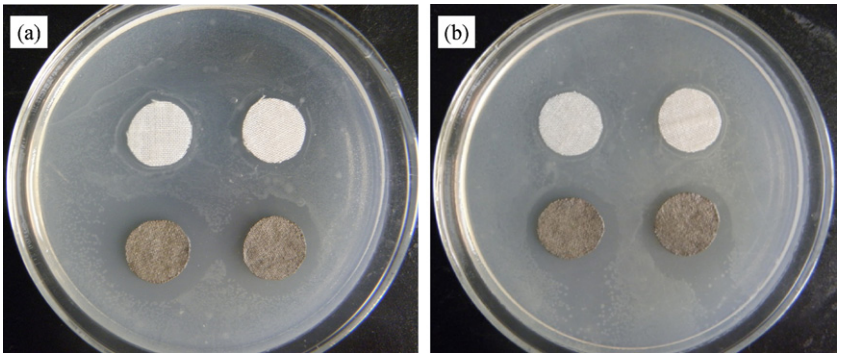

Fig. 3. Antibacterial activity of (a) normal cotton (the upper two) and Ag NP modified cotton (the lower two) textiles, and (b) normal cotton (the upper two) and hydrophobized Ag NP modified cotton (the lower two) textiles.

surface rough, thus generating a dual-size surface structure on the textiles [39]. Therefore, after hydrophobization with HDTMS, the intrinsically hydrophilic cotton textile [39] surface which can be completely wetted by water due to the abundant hydroxyl groups in its structure was turned superhydrophobic, having a water contact angle of $157.3^\circ \pm 1.6^\circ$ for a 5 μL water droplet, as shown in Fig. 1(d). And the water droplets rolls easily on the textile surface when moving the textile sample horizontally. This phenomenon was completely different from that reported by Khalil-Abad and Yazdanshenas [27], in which the water droplet adhered firmly to the textile surfaces modified by silver particles.

Different approaches to coating cotton fibers with silver particles have been reported [25,27,30,40]. However, it is the first time that such dense film coating of silver nanoparticles on fibers as shown in Fig. 1(b) was seen to the best of our knowledge. The weight percentage of Ag added on the sample was calculated to be 34.49% based on the weight of the textile before silver coating. Fig. 2 shows the XRD pattern of the cotton fibers coated with silver particles. Four obvious peaks at 38°, 44°, 64°, and 77° correspond to (1 1 1), (2 0 0), (2 2 0), and (3 1 1) planes of silver crystal (JCPDS cards 4-0783), respectively. The average size of particles was estimated to be 257 nm, using the Scherrer equation. No characteristic peaks were observed for the other impurities such as Ag_2O. Thus, the coating on the fibers is composed of silver crystals only and no other compounds exist, which imparts high conductivity to the textiles with electric resistance as low as $37.0\,\Omega \pm 1.8\,\Omega$ measured using a multimeter. However, in the case of the original textile, the resistance is infinity due to its insulation.

Antibacterial activity of fabric samples was determined in terms of inhibition zone formed on agar medium. Fig. 3 shows that the normal cotton samples, which were used as control, did not show any antibacterial activity. The silver modified cotton textiles placed on the bacteria-inoculated surfaces killed all the bacteria under and around them. A distinct inhibition zone with an average width of 8.78 mm around the cotton samples was observed. Previous work [41] showed that fabrics with coatings to repel water had antibacterial effect. However, after hydrophobization, the average width of the inhibition zone of the silver modified samples was reduced to 6.84 mm. The decrease of the inhibition width might be caused by the prohibition of the diffusion of Ag^+ from the silver particles by hydrophobization. The reduction of bacterial growth on the silver treated samples, whether hydrophobized or not, in comparison to the untreated one was calculated to be 99.99%, showing strong antibacterial property.

In order to investigate the durability of superhydrophobicity, conductivity, and antibacterial property, we used glass stick to keep the textile sample immersed in a water bath and stirred it by a magnetic stirrer for 10 min at room temperature and dried the sample. This process was repeated ten times. It was found that the water contact angle maintained to be $151.5^\circ \pm 1.4^\circ$ for a 5 μL water droplet. The resistance of the textiles did not show significant change maintaining to be $39.2\,\Omega \pm 1.5\,\Omega$. And the reduction of bacterial growth of all the silver modified samples maintained 99.99% after 10 times water washing. All these results indicated that the silver particles are quite strongly attached to the cotton textile surface.

4. Conclusions

Superhydrophobic conductive cotton textiles with antibacterial property were fabricated successfully by *in situ* coating fibers with Ag NPs followed by hydrophobization. The nanoparticle roughening effect of the fiber surface favours the construction of superhydrophobic surfaces, while the compact coating of silver imparts not only the metallic feature to the fibers rendering the textiles conductive, but also the antibacterial property to the textiles. This method to multifunctionalizing conventional textiles with one material is useful in the textile industry, and this strategy is expected to become a powerful platform for the fabrication of multifunctional materials.

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