Journal of Applied Polymer Science

Microwave-Assisted Synthesis of Silver Nanoparticles on Cotton Fabric Modified with 3-Aminopropyltrimethoxysilane

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ABSTRACT: In this study, silver nanoparticles were synthesized on cotton fabric modified with 3-aminopropyltrimethoxysilane (APTMS) using sodium citrate as a reducing/stabilizing agent by microwave-assisted process. The presence of a highly oriented amino-terminated self-assembled monolayer and formation of APTMS was demonstrated by an X-ray photoelectron spectroscopy (XPS) analysis. The silver-coated cotton fabrics were examined by scanning electron microscopy (SEM) and energy dispersive X-ray (EDX). UV protection, antistatic, and hydrophobic properties were also evaluated. The results show that silver-coated fabric modified with APTMS possesses excellent antistatic, UV protection with ultraviolet protection factor (UPF) of 396.5 and superhydrophobic properties with contact angle of 153.2°. APTMS pretreatment improves the adhesive strength between silver coatings and cotton fabric. © 2013 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 130: 3862–3868, 2013

KEYWORDS: coatings; cellulose and other wood products; textiles

Received 28 February 2013; accepted 4 June 2013; Published online 26 June 2013

DOI: 10.1002/app.39636

INTRODUCTION

In recent years, metal nanoparticles have been widely synthesized and applied to textiles because of their size dependent optical and electronic properties.[1,2] Among them, silver nanoparticles have attracted considerable attention because of its potential applications in various fields such as photonics,[3] microelectronics,[4] photocatalysis,[5] lithography,[6] and biosensor material.[7] Silver nanoparticle deposited on textile substrates has been used to produce multifunctional materials with properties of antibacterial and antistatic, electrical conductivity, EMI shielding, ultraviolet (UV) protection, and water repellency.[8–11]

Numerous efforts have been made to synthesize silver on textiles such as chemical reduction, electrochemical coating, spraying, plasma treatment, laser, vapor deposition, sputtering, and layer-by-layer methods.[12–16] Among them, chemical reduction method by using a reducing agent such as sodium borohydrate, formaldehyde, sodium citrate, glucose, hydrazine, or ascorbic acid in a silver salt solution is most common.[17–21] Although this kind of method have been successfully used to prepare silver nanoparticle-coated textiles, it is still desirable to develop fast and efficient routes for the chemical reduction coating. Currently, microwave heating provides a promising method for the preparation of silver nanoparticles because of its characteristics of rapid volumetric heating, high reaction rate, short reaction time, enhanced reaction selectivity, and energy saving.[22,23] The use of microwave irradiation instead of conventional heating in various chemical reactions has been receiving increased interest in the past decades.[24] To the best of our knowledge, few studies have reported on the microwave-assisted synthesis of silver nanoparticle on textiles.

In addition, the silver coating formed on textile surface has poor adhesion to the substrate because of the absence of chemical conjunction between silver coating and fibers. Therefore, it is necessary to explore a proper surface modification technique for textiles prior to the coating. Recently, there has been growing use of 3-aminopropyltrimethoxysilane (APTMS) molecules to improve the adhesion between metallic nanoparticles and textile surfaces.[25,26] In this study, a microwave-assisted synthesis of silver nanoparticles on cotton fabric modified with APTMS from an aqueous solution including silver nitrate and sodium citrate was developed. The composition and chemical structure of cotton fabric modified with APTMS were investigated. Surface morphology and chemical composition of the silver nanoparticle-coated cotton fabrics were characterized. UV protection, hydrophobicity, antistatic property, and adhesive strength of silver-coated cotton fabrics were evaluated.

EXPERIMENTAL

Synthesis of Silver Nanoparticles on Cotton Fabric

Plain weave 100% cotton fabric (58 × 40 counts/cm^2; 136 g/m^2) with an area of 10 cm × 10 cm was used as the substrate. Silver

nitrate (99.8%), trisodium citrate (99%), acetone (99.5%), and ethanol (99.7%) were purchased from Chengdu Kelong Chemical Co. Ltd. China. APTMS (97%) was obtained from Aladdin Chemical Co. Ltd, China. All chemicals were of analytic grade and used without further purification.

Prior to the deposition of silver nanoparticles, the cotton fabrics were initially cleaned in ultrasonic baths with acetone, ethanol, and deionized water for 15 min, respectively. The samples were then immersed into a 1% APTMS acetone solution at room temperature for 24 h to form a self-assemble monolayer of APTMS on the surface of the fabric. After silanization, the fabrics were baked in an air oven at 70°C for 30 min. Subsequently, the fabrics were washed in deionized water to remove excess silane molecules. Silver nanoparticles were synthesized on the surface of the cotton fabrics by direct reduction of Ag^+ using sodium citrate as a reducing agent and stabilizing agent under microwave irradiation. In a typical experiment, 5 g of $AgNO_3$ was dissolved in 100 mL of deionized water, 5 g sodium citrate was then added under magnetic stirring, and further stirred until the chemicals were dissolved. The cotton fabrics were then immersed in the mixture solution. The reaction mixture was exposed to high intense microwave irradiation (800W) in a domestic microwave oven operating in a cycling mode (on 10 s, off 5 s) to prevent boiling of solvent. For comparison, silver nanoparticles were coated on cotton fabrics without modification of APTMS. In this method, the cotton fabric was immersed into 100 mL aqueous solution that contains 5 g of $AgNO_3$ and 5 g sodium citrate, and treated under microwave irradiation. Finally, the silver nanoparticle-coated cotton fabrics were washed thoroughly with deionized water and then dried at 60°C in an oven for characterization.

Characterization

X-ray photoelectron spectroscopy (XPS) was used for the surface analysis of APTMS-modified cotton fabric. XPS (PHI 5600, Physical Electronics Inc., Chanhassen, MN) was performed by using an Al Kα source (14 kV and 350 W). The binding energy scale was calibrated to 285.0 eV for the main C (1 s) peak. The surface morphology of the silver-coated cotton fibers was carried out by field emission scanning electron microscopy (FESEM) (JSM-6335F). The elemental compositions of the silver nanoparticle-coated cotton fabrics were determined by using an energy-dispersive X-ray (EDX) analyzer that was attached to the scanning electron microscope (SEM).

The UV protective characteristics of the coated fabrics were determined in accordance with the Australian/New Zealand Standard AS/NZS 4399:1996 by using UV–visible spectrophotometer (Varian, CARY 300 Conc, LabX, Ontario, Canada) over wavelengths ranging from 280 to 400 nm. The ultraviolet protection factor (UPF) (average of eight scans) was computed using the following formula[15]:

$$\mathrm{UPF} = \frac{\sum_{280\mathrm{nm}}^{400\mathrm{nm}} E_\lambda S_\lambda \Delta\lambda}{\sum_{280\mathrm{nm}}^{400\mathrm{nm}} E_\lambda S_\lambda T \Delta\lambda} \quad (1)$$

where S_λ is the spectral irradiation of the skin in UV region (280–400 nm); E_λ is relative erythemal spectral effectiveness,

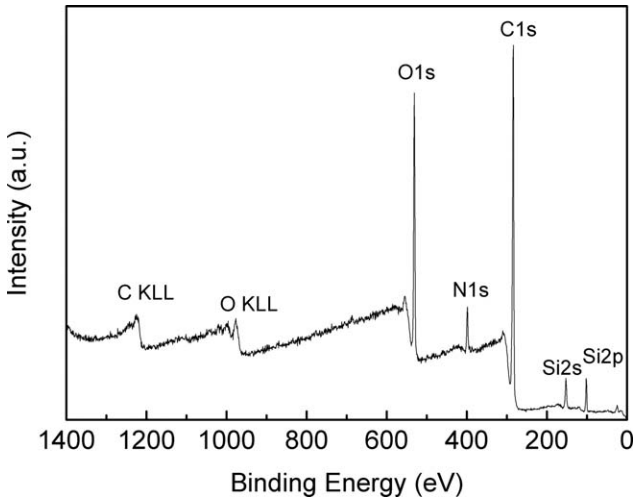

Figure 1. XPS wide spectra of APTMS-treated cotton fabric.

T_λ is spectral transmittance of the fabric; $\Delta\lambda$ is increment relating to wavelength, and λ is wavelength in nanometer.

According to the Australian classification scheme, fabric can be rated as providing good protection, very good protection, and excellent protection if their UPF values are 15–24, 25–39, and >40, respectively. In no event is a fabric assigned a UPF rating greater than 50.[15]

The hydrophobicity of silver nanoparticle-coated cotton fabrics was characterized by the water contact angles. The contact angles of the fabrics were measured with 5 μL deionized water at ambient temperature on a video optical contact angle instrument (OCA 20, DataPhysics, Germany). Each contact angle presented was the average value of those measured at five different locations of each fabric.

The antistatic property of the cotton fabrics was determined using resistance measurement with a static voltmeter R-1020 (Rothschild, Switzerland). All the fabric specimens with the size of 1 × 10 cm were tested in both warp and weft directions. Each specimen was fixed between two fixation screws. The insulated terminal was charged from the built-in DC source. The antistatic property was evaluated by the elapsing time, t (s), which was defined as the time required to discharging half of the charge in the specimen. The resistance [R (Ω)] of the specimen was calculated according to eq. (2).[11]

$$R = 1 \times 10^{11} \times t \quad (2)$$

Weight change of silver-coated cotton fabric after laundering was used for assessing the adhesive strength between the silver coating and the cotton fabric in accordance with standard method AATCC 61–2003. After washing for 40 min, weight loss of silver nanoparticle coating before and after laundering was calculated.

RESULTS AND DISCUSSION

XPS Analysis

An XPS analysis of the APTMS-modified cotton fabric was carried out in order to quantify the orientation distribution of the

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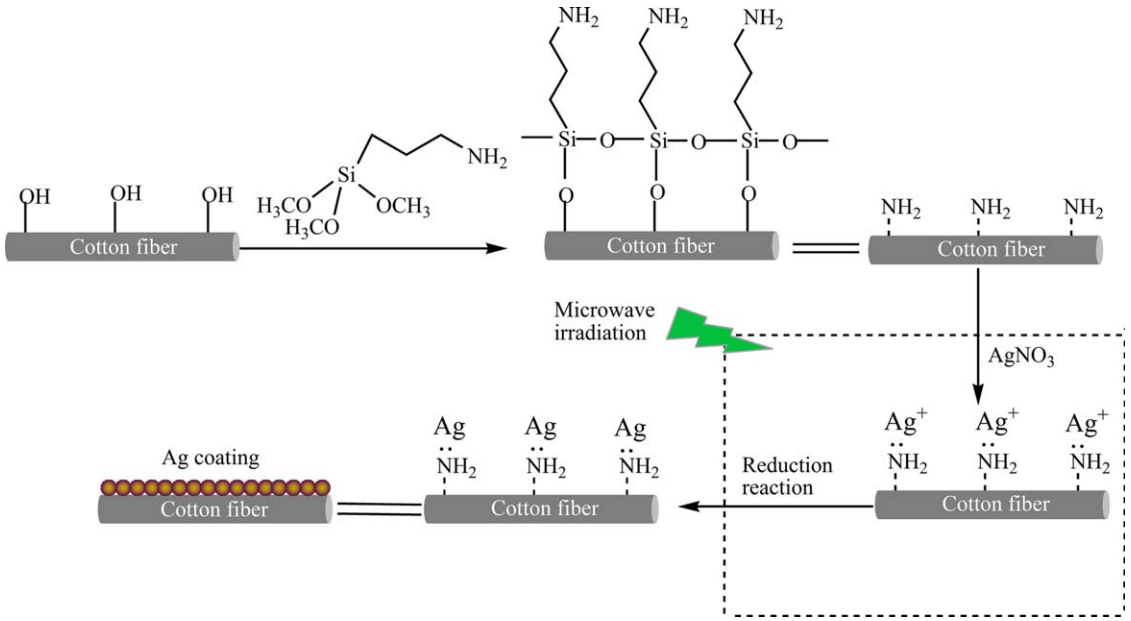

Figure 2. Mechanism for growing silver particles on cotton fabric using sodium citrate as reducing agent under microwave radiation. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

–NH_2 groups on the cotton fiber. The wide scan XPS spectrum of the cotton fabric after APTMS treatment is shown in Figure 1. C and O are the most detected species and they occur at 285.00 and 532.25 eV, respectively. The presence of silicon on the surface is detected from their characteristic emission peaks at 102.75 and 158.02 eV. The binding energy value of the single N1s line at 399.36 eV corresponds to the amino group on the surface of cotton fabric.[19] The findings indicate the formation of a self-assembled monolayer on the cotton surface. The elements of C, O, N, and Si were detected from the APTMS-modified cotton fabric. N and Si are attributed to APTMS, while C and O are both from the APTMS, the cotton fabric, and air. The APTMS easily reacts with the hydroxyl groups on the cotton fabric to form a self-assembled monolayer. It is confirmed that the APTMS is formed on the cotton fiber.

The mechanism of silver nanoparticle deposition on the APTMS-modified cotton fabric can be described in Figure 2. The APTMS molecule has an amino group and an ethoxy group at each end. The ethoxy group forms a silanol group by hydrolysis reaction with a water molecule. Then, the silanol group of the APTMS forms an Si–O–Si bond with an –OH group on the cotton fiber surface. After all –OH groups on the cotton fiber surface are bonded with the silanol groups of the APTMS, no further APTMS molecules are deposited, and the cotton fiber surface is covered by the APTMS monolayer. As is well known, the –NH_2 group is a strong electron donor and possesses great ligand capability to transition metal ions because of the lone pair electrons of nitrogen atom. However, Ag(I), as a soft metal ion whose outermost electron is configured as $4d^{10}5s^0$, has an empty lower energy orbit that can accept electrons.[21] Its coordination chemistry is satisfactory for forming stronger bonds with N. As cotton fabric modified with APTMS is placed into the reaction solution containing $AgNO_3$ and sodium citrate, the

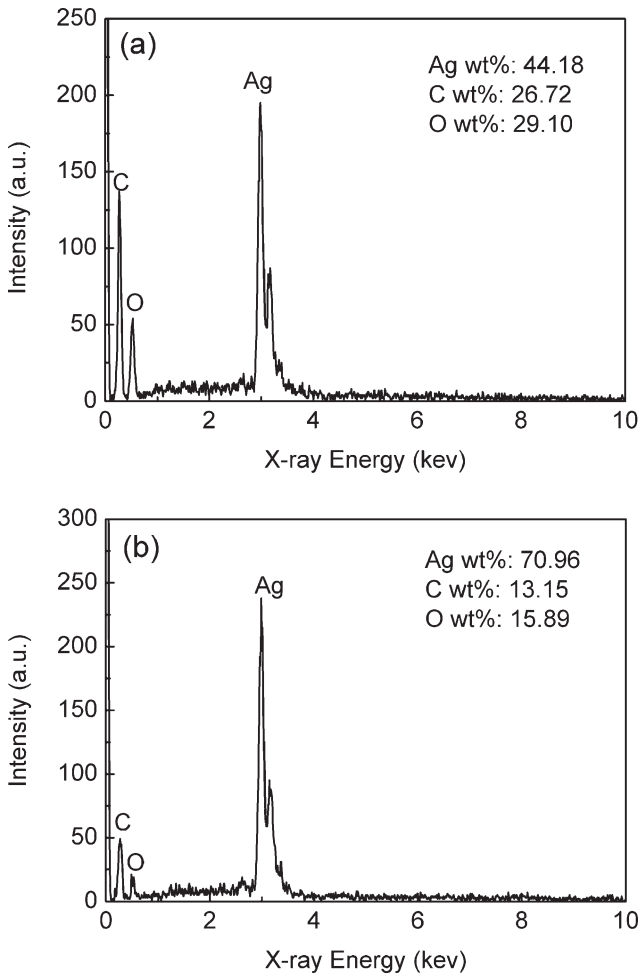

Figure 3. EDX analysis of the silver-coated cotton fabric (a) without and (b) with modification of APTMS.

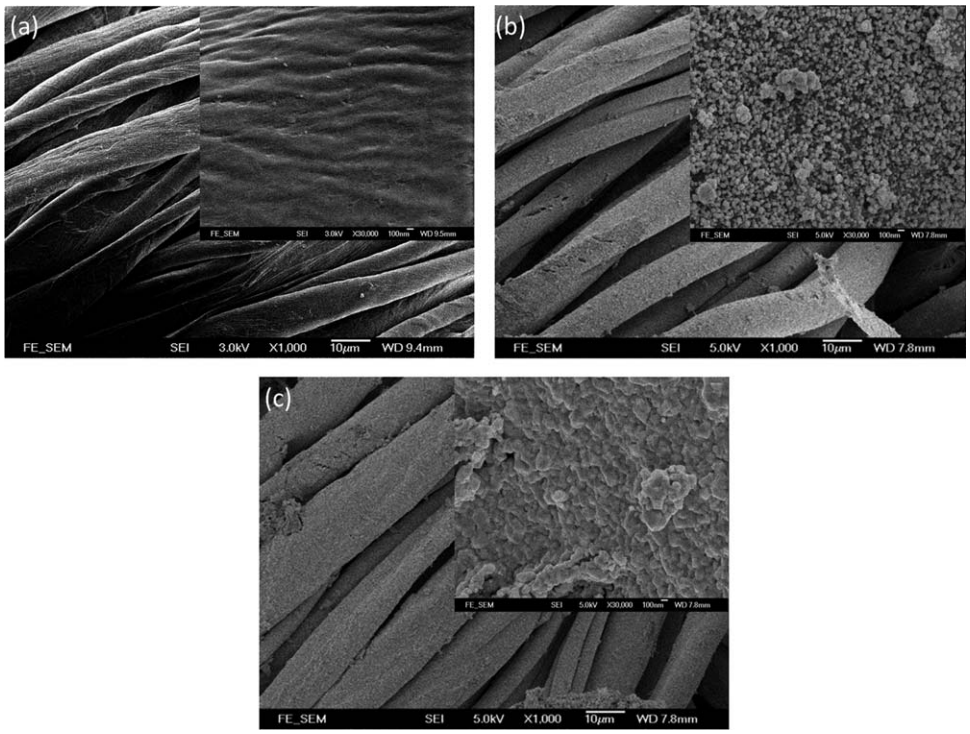

Figure 4. SEM micrographs of (a) original cotton fibers and silver-coated cotton fibers (b) without and (c) with modification of APTMS.

silver ions are attracted by NH_2 group and form a strong coordination bond between APTMS chain and silver ions. The attached silver ions are reduced by sodium citrate in the solution under microwave irradiation and serve as the seed for the continuous silver coating. As the reaction is going on, Ag undergoes further aggregation to larger clusters. These silver particles are stabilized by the terminal amino groups on the surface of the cotton fiber via chemical bond.[27] The bonding energy between the cotton fiber and the silver nanoparticles through APTMS chain is much larger than van der Waals force. Therefore, the silver nanoparticles are firmly attached on the surface of the cotton fabric.

The overall reaction can be described by the following equation[28]:

$$C_6H_5O_7^{3-} + 2Ag^+ \rightarrow C_5H_4O_5^{2-} + H^+ + CO_2 + 2Ag^0 \quad (3)$$

Deposit Composition

EDX was employed to establish the chemical identity of the observed particles on silver-coated cotton fabrics. Figure 3 shows the EDX spectra of the silver-coated cotton fabrics without and with APTMS. Ag, C, and O elements are observed. The Ag peaks are attributed to the coating. No other impurities are observed in the silver coating, within the resolution limit of EDX, which indicates that high purity silver coating was obtained. It should be noted that APTMS is not detected after the silver coating, which indicates that the silver coating is thick and dense. The composition of silver on the cotton fabric modified with APTMS is higher than that without modification of APTMS because of the increase of deposit thickness. Indeed, silver is known to have a high mobility on cotton surfaces because of the relatively weak interactions between silver and cotton substrate. Therefore, silver nanoparticles on the cotton fabric are less. For APTMS-modified cotton fabric having available electron donor atoms N, silver atoms are probably less mobile on the surface. These donor atoms can act as privileged nucleation sites and accelerate deposition of silver. Therefore, thickness of the silver coating on the APTMS-modified cotton is higher than that on cotton without APTMS treatment.

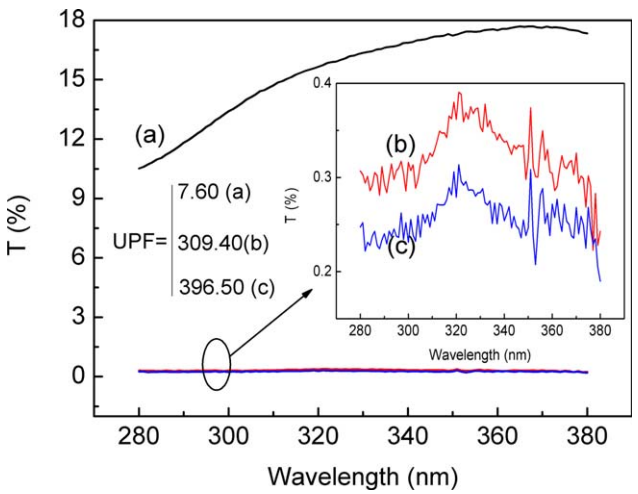

Figure 5. UV blocking characterization of (a) untreated cotton fibers and silver-coated cotton fabric (b) without and (c) with modification of APTMS. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

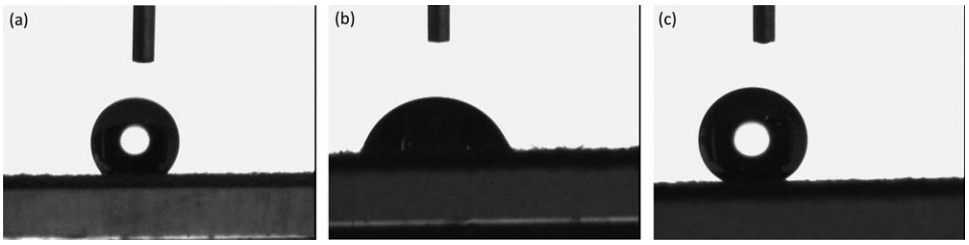

Figure 6. Images of a water droplet (5 μL) on (a) APTMS-modified cotton fabric, and silver-coated cotton fabrics (b) without and (c) with modification of APTMS.

Surface Morphology

SEM images of both untreated and silver nanoparticle-coated cotton fabrics are presented in Figure 4. It can be seen from Figure 4(a) that the cotton fibers show typical longitudinal fibril structure with clean and smooth surface. After silver coating, the cotton fiber surfaces show a compact and uniform covering of silver nanoparticles, as shown in Figure 4(b,c). Silver nanoparticles on the fabric without APTMS [Figure 4(b)] are spheroidal with particle size ranging from 30 to 80 nm. The shapes of the silver nanoparticles coating with APTMS [Figure 4(c)] are irregular with particle size ranging from 100 to 200 nm. The cotton modified with APTMS can efficiently absorb silver atoms, which would result in fast crystal nucleation and growth. The migration and aggregation of silver particles are probably driven largely by the instability of silver atoms because of their high surface-free energy. Their aggregation would produce thermodynamically stable particles with bigger sizes. In addition, the silver nanoparticles on the fabric with APTMS have more well-defined edges, corners, and sharper surface features because a rapid reaction weakens the optimal directional growth which leads to the formation of irregular nanoparticles.

Ultraviolet Radiation Penetration

UPF can directly evaluate the UV shielding activity of fabric. The UPF results were calculated using the methods described in the Australian/New Zealand Standard AS/NZS 4399:1996. UV-blocking properties of the original and the silver-coated cotton fabrics are illustrated in Figure 5.

The calculated UPF increases from 7.60 for untreated cotton fabric to 309.40 and 396.50 for the silver-coated cotton without and with APTMS, respectively. The results confirm that silver-coated cotton offers excellent protection from UV radiation as indicated by a UPF rating of 50+. During the silver-coating process, the silver particles are deposited not only on the fiber surface but also on the spaces between the yarns in the cotton fabric. The coverage of the spaces can prevent the penetration of the UV radiation through the fabric and reach the skin. Therefore, the silver-coated cotton can improve protection from UV radiation with excellent results. However, the mean UPF values increase from 309.40 for the silver-coated cotton without APTMS pretreatment to 396.50 with APTMS pretreatment. It is obvious that the UPF values of the silver-coated cotton fabric modified with APTMS is greater than that without APTMS. Because APTMS can accelerate deposition of silver nanoparticles on the cotton fabric, more silver nanoparticles are deposited on the cotton fabric. In addition, the size of the nanoparticles on APTMS-modified cotton fabric is larger than that without APTMS as shown in Figure 4 and give more coverage on the fabric with effective UV radiation protection. Therefore, APTMS pretreatment could provide a better protection of UV radiation.

Contact Angle

It is generally known that the wettability of a solid surface depends on both the chemical composition of surface and its geometrical structure. Wettability was evaluated by the water contact angle measurement of silver-coated cotton fabrics. Figure 6 shows the optical photograph of water droplets on the APTMS-modified cotton fabric and the silver-coated cotton fabrics.

Cotton is an intrinsically hydrophilic fiber, which can be completely wetted by water because of the abundant hydroxyl groups in its structure. Therefore, it has a quicker absorption time of less than 1 s and the contact angle of 0°. After the cotton is treated with APTMS, contact angle of cotton is increased to 138° [Figure 6(a)]. Once the self-assembled APTMS layer is deposited, the surface of cotton fabric is functionalized by an amine group, which leads to the lower surface energy compared with a hydroxyl-terminated cotton fabric. The surface energy of the silver-coated cotton fabric, which is in contact with silver film, which predominantly determines the contact angle of the silver-coated fabric. When the cotton without APTMS is coated with silver nanoparticles, it is partly hydrophilic with a contact angle of about 70.1° [Figure 6(b)]. The result indicates that the

Table I. Antistatic Properties and Adhesive Strength of Silver Coating on Cotton Fabric

	Average static half-life (s)		Weight loss after washing (%)
	Warp	Weft	
Original	20	21	
Silver-coated cotton fabric without APTMS pretreatment	19	18	3.0
Silver-coated cotton fabric with APTMS pretreatment	<0.5	<0.5	2.1

APTMS, 3-aminopropyltrimethoxysilane.

deposition of silver in the spaces between yarns partly reduces the amount of void area in the fabric and decreases water penetration silver-coated fabric.

However, cotton fabric modified with APTMS was turned superhydrophobic after silver coating, having a contact angle of 153.2° for a 5-μL water droplet, as shown in Figure 6(c). The results confirm that APTMS pretreatment has a profound effect on hydrophobic properties. The increase of the contact angle on the surface of cotton fabrics after pretreatment of APTMS is because of the increase in its surface roughness.

The air that is trapped in the rough surface of protuberances and cavities on cotton fabric can significantly enhance the surface hydrophobilicity, and hence, increase the contact angle. The contact angle is usually described by Cassie's equation as follows[15]:

$$\cos\theta^* = -1 + \varphi_s(1+\cos\theta) \quad (4)$$

where $\theta^\star$ is the contact angle on the rough surface; θ is the contact angle on a flat surface; and φ_s is the fraction of the solid surface in contact with the water droplet.

The difference in the hydrophobilicity of the surface is because of the difference in surface roughness. In Cassie's equation, $\cos\theta^\star$ is always less than $\cos\theta$ because φ_s is less than or equal to 1; that is, $\cos\theta^\star$ is less than or equal to $\cos\theta$, regardless of θ.

From SEM images (Figure 4), the surface of silver-coated cotton modified with APTMS is rougher than that without APTMS. In addition, the silane treatment could create the secondary nanoscale roughness by the combination of hydrolysis and condensing reactions both in solution as well as on the cotton surface. Therefore, silver-coated fabric modified with APTMS has a larger contact angle.

Antistatic Property

The half-elapsing times of the original cotton fabric and the silver-coated cotton fabrics without and with APTMS treatment are shown in Table I. It can be seen that the average static half-life values of the silver-coated cotton fabrics without and with pretreatment of APTMS at both the warp direction and the weft direction are lower than that of the original cotton fabrics. Especially, the average static half-life value of the silver-coated cotton fabrics with APTMS is less than 0.5 s, which is greatly lower than that without APTMS. The smaller static half-life value implies better antistatic property.[11] The results indicate that the APTMS pretreatment can provide a better antistatic property than that without APTMS pretreatment because of the fact that more silver particles are deposited on the cotton fabric after being modified with APTMS.

Adhesive Strength

The adhesion of silver to cotton fabric is one of the most important concerns to the silver coating on the cotton fabric. A washing fastness test was employed to evaluate the adhesion of the silver coating on the cotton fabric without and with APTMS, and it was repeated three times on each specimen. The weight loss of the silver-coated fabrics with and without APTMS modification after five cycles of home washing are shown in Table I. A difference in the adhesive strength of the coatings was detected in the washing measurement. It can be noticed that less weight loss of the coating is observed from the silver-coated fabric modified with APTMS than that without APTMS. Moreover, weight losses of silver-coated fabric modified with APTMS pretreatment are less than that without APTMS pretreatment after 5 and 10 cycles of washing. The adhesive strength between silver coatings to the APTMS-modified cotton surface remarkably improves because of the formation of covalent bonds between the silver coating and the cotton fiber. The binding force of the covalent bond between silver nanoparticles and cotton fiber is much stronger than the van der Waals force. Thus, the effective contribution of APTMS modification is ascertained to improve the adhesion of silver onto cotton fiber surface.

CONCLUSIONS

In this study, a microwave-assisted synthesis of silver nanoparticles on cotton fabric modified with APTMS by the reduction of silver nitrate with sodium citrate was investigated. Silver nanoparticles synthesized on cotton fabric modified with APTMS are irregular with large particle size. The mean UPF and contact angle of cotton fabric after silver coating with APTMS pretreatment increase to 396.50° and 153.2°, respectively. The result indicates that the APTMS pretreatment could provide cotton fabric with an excellent protection of UV radiation, superhydrophobic, and antistatic properties under microwave irradiation. The APTMS pretreatment significantly improves adhesive strength between the silver coating and the cotton fabric. In summary, there is a potential application of APTMS pretreatment in microwave-assisted synthesis for the preparation of silver nanoparticles on cotton fabric to improve functional properties and adhesive stability.

ACKNOWLEDGMENTS

This work was financially supported by The National Natural Science Foundation of China (Grant No. 51203099) and the Science Foundation for Youth Scholars of Sichuan University, People's Republic of China (Grant No.YJ2011020).

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