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Research Article

A Novel Two-Step Method for Fabricating Silver Plating Cotton Fabrics

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A novel two-step method was presented for fabricating silver plating cotton fabrics (SPCFs) with high electrical conductivity and excellent washing fastness. First, polydopamine (PDA) film was coated on the surface of cotton fabrics by *in situ* polymerization of dopamine, the silver ions in silver nitrate solution were reduced by the catechol groups of polydopamine, and silver nanoparticles were combined with polydopamine by covalent bond on the surface of cotton fabrics. Second, silver ions were reduced by glucose, and silver plating was coated on the surface. Subsequently, the properties of SPCFs were characterized by field emission scanning electron microscopy (FESEM), X-ray photoelectron spectroscopy (XPS) and thermogravimetric analysis (TGA), and so forth. With the increasing of silver-ammonia solution concentration or dopamine concentration, the surface resistivity of SPCFs decreases and gradually stabilized. The surface resistivity of the SPCFs can reach $0.12 \pm 0.02\ \Omega$, and electromagnetic shielding effectiveness (ESE) of the SPCFs can reach 58.5 ± 4.5 dB. Conductive fabrics have wide application prospect in many of fields, such as antibacterial, intelligent textiles, smart garments, electromagnetic shielding, and flexible sensors.

1. Introduction

Conductive fabrics have attracted a great deal of researchers' attention due to the rapid development and huge market potential of wearable electronics and smart textiles [1, 2]. Conductive fabrics can be applied to many fields, such as textile electrode [3], electromagnetic shielding [4–6], and heating fabrics [7]. Conductive fabrics can be produced by coating metals, metallic salts films, and conducting polymers on the surface of fabrics [1]. A lot of methods have been applied to development of conductive fabrics, such as vacuum deposition [8], chemical plating [9], and composite plating by *in situ* polymerization [10]. The chemical plating method has better industrial application prospect for fabricating conductive textiles due to operation simplification and high efficiency. In recent years, electroless silver plating has been utilized for surface metalizing of many of electrical insulating materials due to superior electrochemical properties and good electric conductivity of silver nanoparticles. Gout et al. [11] obtained silver patterns onto flexible polymer surfaces at the micron and submicron scale using electroless metallization. Yu et al. [12] prepared silver plating wool fabric using electroless silver plating. Lu et al. [13] fabricated silver plating polyethylene terephthalate (PET) fabric by ultrasonic-assisted electroless silver plating. However, silver plating fabrics have low washing fastness when silver plating is coated on surface of fabrics by electroless plating method. Hence, surface modification of substrate is of great importance for improvement of the adhesive force between the metal and the substrate [14]. Lee et al. [15, 16] reported that polydopamine films could be formed on the surface of materials by self-polymerization of dopamine and have a wide range surface-adherent for all types of inorganic and organic materials, including noble metals, oxides, polymers, semiconductors, and ceramics. Further studies found that the self-polymerization of dopamine has secondary reaction and weak reducibility [15–20]. The process of polydopamine preparation is simple and environment-friendly. Lee et al. [15] dissolved dopamine into the Tris buffer solution (pH = 8.5) and then formed a layer of polydopamine film under aerobic

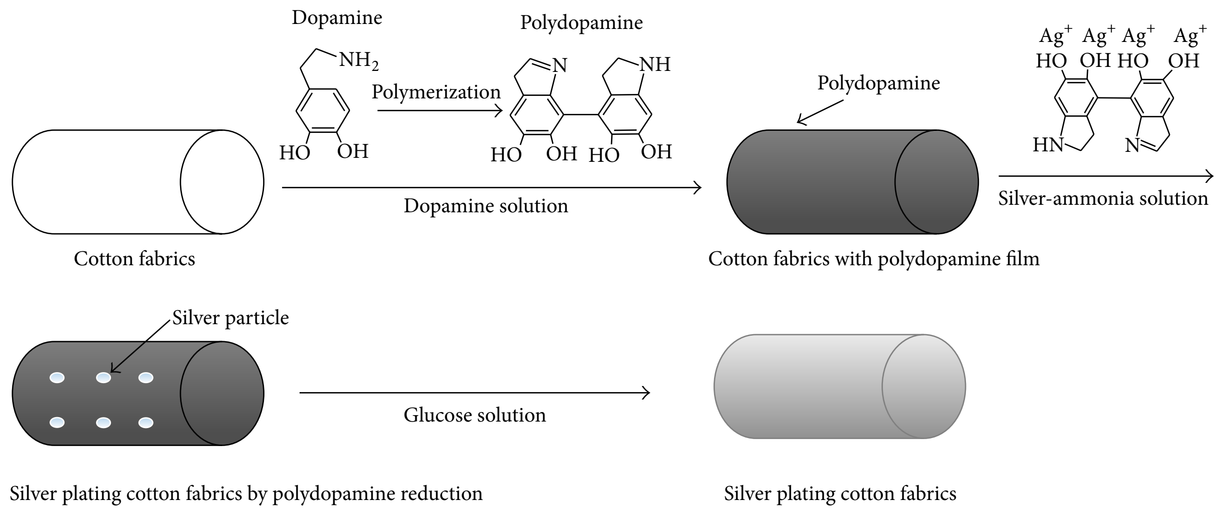


Figure 1: Process of silver plating cotton fabrics (SPCFs) by electroless silver plating.

condition. In general, adhesion mechanism of polydopamine film shows that catechol and amino functional groups in polydopamine have the covalent and noncovalent bonds combination with the inorganic or organic materials [14–17]. Xu et al. [18] fabricated silver plating glass fibers by dopamine functionalization and electroless plating. Wang et al. [19] prepared surface silverized meta-aramid fibers by bioinspired functional polydopamine.

In this study, silver plating cotton fabrics (SPCFs) will be fabricated through polydopamine reduction and glucose reduction reaction. First, the silver nanoparticles will be reduced from silver nitrate by *in situ* reduction of polydopamine (PDA). Second, silver ions will be reduced by glucose, and silver nanoparticles were coated on the surface. The whole process is shown in Figure 1. Subsequently, some tests will be performed to obtain the properties of SPCFs by using field emission scanning electron microscopy (FESEM), X-ray photoelectron spectroscopy (XPS) and thermogravimetric analysis (TGA), and so forth. Silver plating fabrics can be applied in these fields such as antibacterial, intelligent textiles, smart garments, electromagnetic shielding, and flexible sensors.

2. Experimental

2.1. Materials. The dopamine (3,4-dihydroxyphenylalanine) was purchased from Sigma-Aldrich Co., LLC, USA. Tris (hydroxymethyl) aminomethane (Tris) and other chemicals were all obtained from Tianjin recovery technology Co. Ltd., China, and used without further purification. The cotton fabrics (150 g/m^2) were provided by Changxing Jiahong Co. Ltd. in China. Commercial silver plating fabrics were from Qingdao Hengtong X-Silver speciality textile Co., LTD, in China.

2.2. Preparation Methods

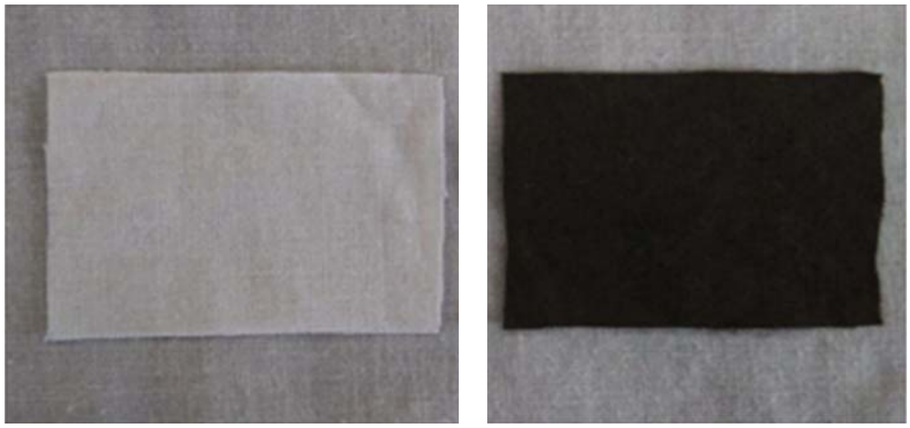
2.2.1. Pretreatment of Cotton Fabrics. Cotton fabrics with 10 cm × 10 cm size were immersed in a diethyl ether solution for 30 minutes at room temperature and dried at 30°C, and they were kept in the dry state until next treatment.

Table 1: Composition of dopamine solution.

Tris (g)	Ethanol (mL)	Deionized water (mL)	Dopamine (g/L)
0.420	150	350	1~6

2.2.2. Modification of Cotton Fabrics by In Situ Polymerization of Dopamine. The composition of dopamine solution was shown in Table 1. pH value of tris buffer solution was regulated to 8.5, the dopamine was dissolved in the tris buffer solution, and the dopamine solution was obtained by mixing the tris buffer solution, ethanol, and deionized water according to proportion as shown in Table 1. The pretreated cotton fabrics were, respectively, immersed into dopamine solution with the dopamine content from 1 to 6 g/L interval 1 g/L, the bath ratio being 1 : 50, and stirred for 24 hours at room temperature. Polydopamine film was coated on surface of cotton fabrics by *in situ* polymerization. Subsequently, the cotton fabrics with polydopamine film (CFPF) were washed using deionized water and dried at room temperature.

2.2.3. Preparation of Conductive Cotton Fabrics. First, silver-ammonia ($[Ag(NH_3)_2]OH$) solution consisted of silver nitrate (99%), sodium hydroxide (0.1 mol/L), and ammonia (200 mL/L). Silver-ammonia solution with silver nitrate from 0.1 to 0.25 mol/L interval 0.05 mol/L was, respectively, poured into different beakers. To perform the polydopamine reduction reaction, these cotton fabrics were immersed in corresponding beakers and were heated in a water bath (40°C) for 8 hours. Subsequently, these beakers were taken out and cooled in air. Second, different concentration (as shown in Table 2) glucose solution (100 mL) was prepared and poured into corresponding beaker. The mixed solution was stirred for 1 hour to perform the glucose reduction reaction; subsequently, the cotton fabrics were taken out, rinsed using deionized water, and dried in the air.



(a) (b)

Figure 2: Photos of (a) pristine cotton fabric and (b) silver plating cotton fabrics by polydopamine reduction.

Table 2: Technological parameters of glucose reduction reaction.

Composition	Concentration (mol/L)			
Silver-ammonia (mol/L)	0.1	0.15	0.2	0.25
Glucose (mol/L)	0.2	0.3	0.4	0.5

2.3. Characterizations. Surface morphology of the samples was characterized by a field emission scanning electron microscope (FESEM, S4800, Hitachi). The surface chemical compositions of cotton fabrics and polydopamine films were measured by X-ray photoelectron spectroscopy (XPS, K-Aepna, ThermoFisher). XPS curves can be utilized for analyzing the surface element content of samples according to the peak area ratio, and the measurement errors are less than 5%. The surface resistance of SPCFs was measured by U3402A digital multimeters (Agilent Technologies, USA), and surface resistivity of SPCFs can be calculated by

$$\rho_s = \frac{d}{L} R_s, \quad (1)$$

where ρ_s is the surface resistivity, in Ω, R_s is the surface resistance between two electrodes on the surface of samples, in Ω, L is width of the two electrodes, in cm, and d is the distance between two electrodes, in cm.

Thermal gravimetric analysis (TGA) of pristine cotton fabrics and the SPCFs were performed by simultaneous thermal analyzer STA409PC (NETZSCH, Germany) in nitrogen environment from room temperature to 600°C at a rate of 10°C/min.

The washing fastness test of the SPCFs was performed according to the standard of China (FZ/T60014-93). The liquid ratio was 40 : 1 and the temperature of liquid was controlled in 40 ± 2°C. The SPCFs were washed in a washing-machine with water including 1 g/L detergent for 10 minutes and dried in ventilating oven at 40°C. To evaluate the stability of silver plating, the resistance of the SPCFs was measured after each washing.

Electromagnetic shielding test of the SPCFs was performed by vect-netw analyzer ZNB40 (ROHDE&SCHWARZ, Germany). Measurement results are located usually in the corresponding interval with a probability of approximately 95% (coverage factor $k = 2$).

3. Results and Discussions

Figures 2(a) and 2(b) show the photos of pristine cotton fabric and silver plating cotton fabric by polydopamine reduction (SPCF-PR), respectively. One can observe that the pristine cotton fabric is white, but the first-step silver plating cotton fabric is dark brown; obviously, the reason is that polydopamine film was coated on the surface of cotton fibers.

The theoretical nitrogen to carbon signal ratio (N/C) of the dopamine is 0.125 [15, 20]. Table 3 shows the N/C of cotton fabrics and cotton fabrics with polydopamine film (CFPF) is, respectively, 0.015 and 0.10; obviously, the N/C of the CFPF approximates to theoretical value of polydopamine. Hence, we can confirm the polydopamine film has been coated on surface of cotton fabrics. Table 3 also shows 2.6% silver content is coated on the surface of the CFPF after performing polydopamine reduction reaction.

XPS is performed to explore that the polydopamine layer was coated on the surface of pristine cotton fabrics. Figure 3 shows the XPS curves of the pristine cotton fabrics and the CFPF. With process of the dopamine oxidative self-polymerization, the color of solution quickly turned to pink as the catechol was oxidized to benzoquinone. Subsequently, the pink solution turned slowly to deep brown; the reason is that the polymerization was followed by a part of melanin formation [6, 10]. The C 1s core-level spectrum of the pristine cotton fabrics in Figures 3(a) and 3(b) could be fitted into three peak components at binding energies (BEs) of about 285.0 eV, 287.9 eV, and 286.6 eV; these binding energies are corresponding with C-H, C=O, and C-O species, respectively. The C 1s core-level spectrum of the CFPF in Figure 3(b) has three same peak components as that of the pristine cotton fabric. However, an additional peak component of

Table 3: Element content of pristine cotton fabrics and CFPF and SPCF-PR.

	Element content			
	Pristine cotton fabrics	Polydopamine (theoretic)	CFPF	SPCF-PR
C 1s (at%)	65	72.7	72	72.05
N 1s (at%)	1	9.1	7	6.95
O 1s (at%)	34	18.2	21	18.39
Ag 3d (at%)	0	0	0	2.61
N/C	0.015	0.125	0.10	\

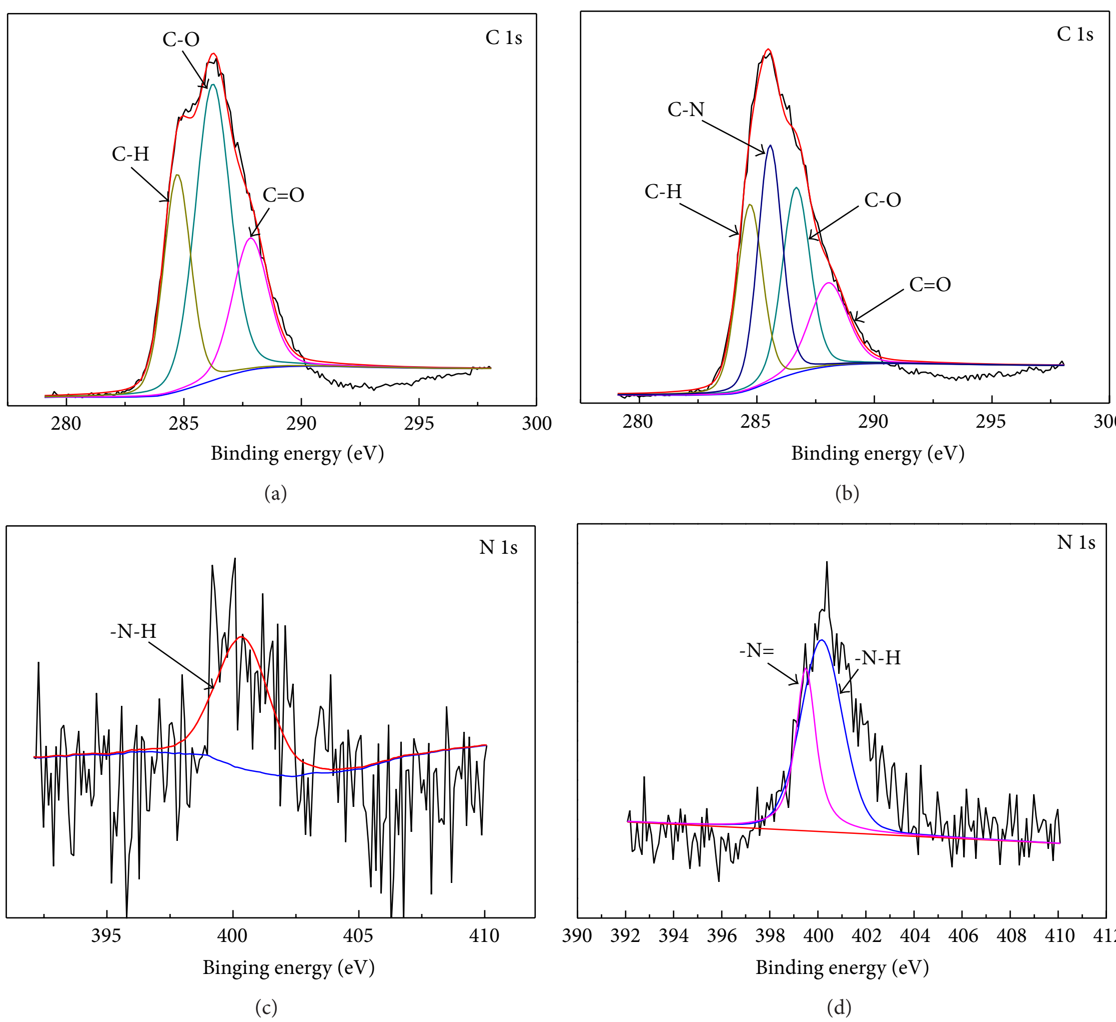


Figure 3: XPS C 1s core-level spectra of (a) pristine cotton fabric and (b) CFPF and XPS N 1s core-level spectra of (c) pristine cotton fabric and (d) CFPF.

C-N (285.6 eV) which is from polydopamine is observed in Figure 3(b). As shown in Figure 3(c), the N 1s core-level spectrum of the pristine cotton fabrics has one peak component at the BEs of about 399.5 eV which is attributable to the -N-H species. The N 1s core-level spectrum of the CFPF in Figure 3(d) can be fitted with two peak components which is attributable to the amine (-N-H) species at a binding energy (BE) of 399.5 eV and the imine (-N=) species at the BE of 398.5 eV. The -N-H species is derived from the amine group of the polydopamine. The -N= species is formed through structure evolution during the self-polymerization process of dopamine [21–25]. The characteristic peaks in XPS curve exhibit polydopamine was successfully coated on the surface of the cotton fabrics.

Figure 4 is the FESEM images of fibers of pristine cotton fabrics, cotton fabrics with polydopamine film, cotton fabrics with silver nanoparticles reduced by polydopamine, and cotton fabrics with silver layer reduced by glucose in magnification of 1000 times and 10000 times, respectively. The surface of pristine cotton fibers in Figures 4(a) and

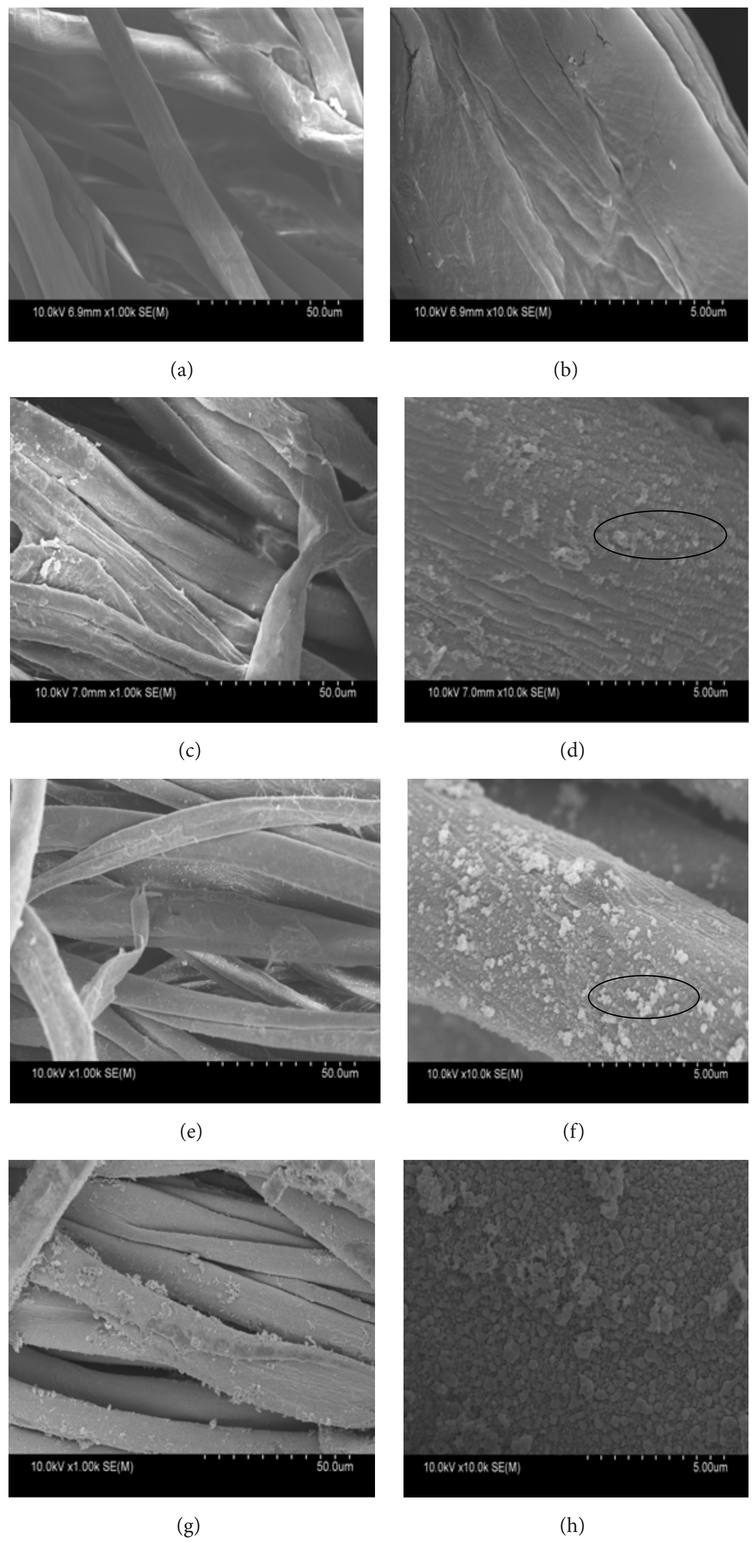


Figure 4: FESEM images of (a, b) the pristine cotton fibers, (c, d) CFPF, (e, f) SPCF-PR, and (g, h) SPCFs.

4(b) is smooth. One can observe from Figure 2(b) that the pristine cotton fabrics are coated with a layer of dark brown polydopamine film; Figures 4(c) and 4(d) show that some polydopamine pieces in black circle are coated with polydopamine film after 24 h self-polymerization. Figure 4(f) shows some sparse silver particles in black circle are coated on polydopamine film of cotton fibers that were immersed into the silver-ammonia solution for *in situ* reduction reaction for

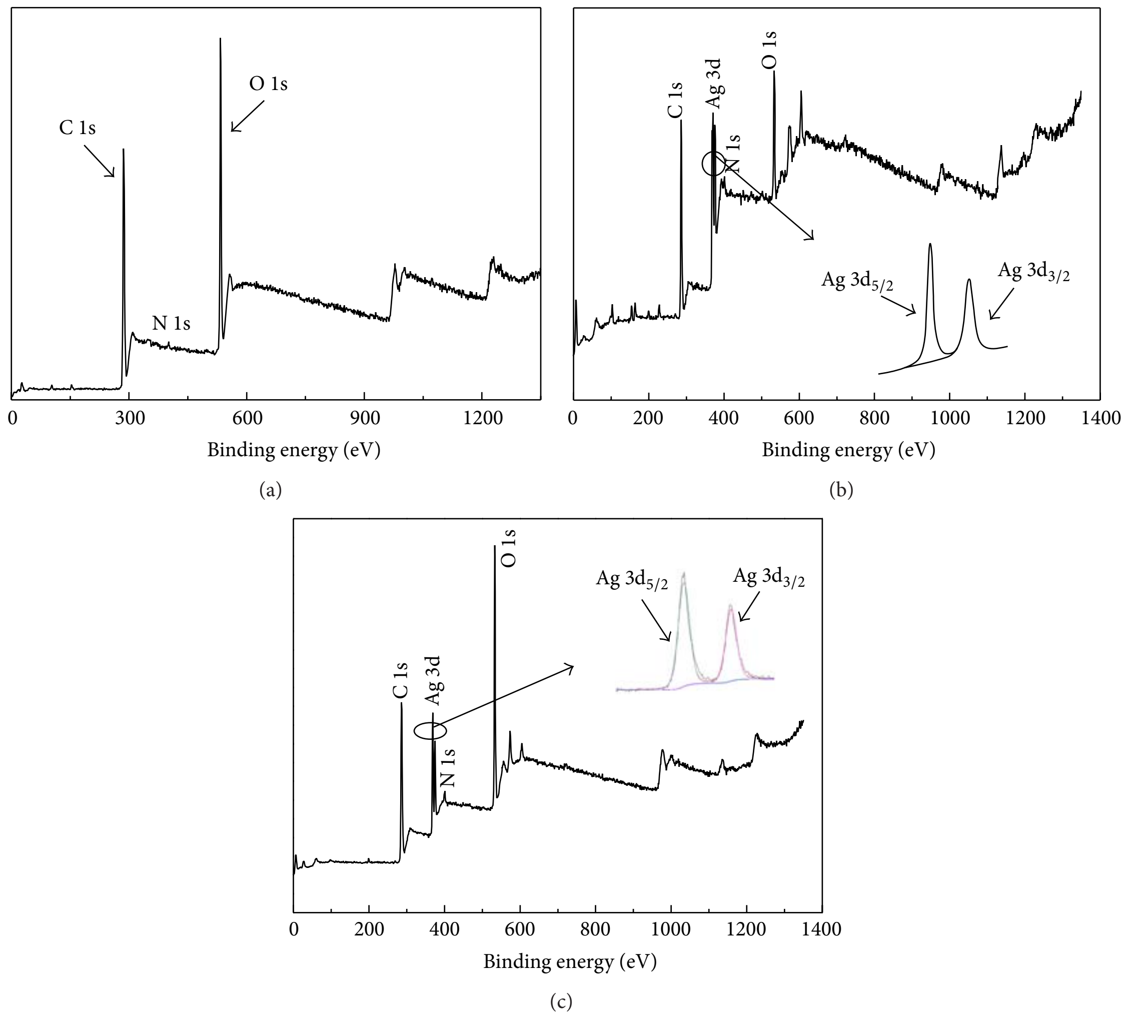


FIGURE 5: XPS wide scan spectrum of (a) pristine cotton fabrics and (b) SPCF-PR (c) SPCFs.

8 hours. Figure 4(g) shows some silver pieces are adhered on the surface of cotton fibers; however, Figure 4(h) further shows compact silver particles are coated evenly on the surface of cotton fibers.

XPS is performed to explore the chemical state of silver on the surface of SPCF-PR and SPCFs. Figures 5(a), 5(b), and 5(c) show the XPS wide scan spectrum of the pristine cotton fabrics, SPCF-PR and SPCFs. Figures 5(b) and 5(c) show the essential marker of silver 3d (Ag 3d) peaks of SPCF-PR and SPCFs; however, no peaks corresponding to silver were observed from XPS wide scan spectrum for the pristine cotton fabrics (Figure 5(a)), which means the silver particles have been coated on the surface of polydopamine on the cotton fabrics. To further explore the chemical state of the silver particles, a detailed deconvolution of the Ag 3d peak is exhibited. In Figures 5(b) and 5(c), the Ag 3d core-level spectrum consists of two peak components at BE of 368.48 eV (Ag $3d_{5/2}$) and 374.48 eV (Ag $3d_{3/2}$), which indicates the silver particles are metallic silver particles.

Low surface resistivity is very important for conductive fabrics which can be used as electromagnetic shielding or electrode materials. Figure 6 shows surface resistivity of SPCFs decreases with increasing of concentrations of silver-ammonia ($[Ag(NH_3)_2]OH$) solution from 0.1 to 0.25 mol/L interval 0.05 mol/L when concentration of dopamine is a constant value, and the surface resistivity of SPCFs has slight decreasing or increasing when the concentration of silver-ammonia varies from 0.2 to 0.25 mol/L and the concentration of dopamine varies from 1 to 6 g/L. Meanwhile, the surface resistivity of SPCFs decreases with increasing of concentration (from 3 to 6 g/L) of dopamine. The surface resistivity of SPCFs is in the range of $0.12 \pm 0.02\,\Omega$, when the concentration of silver-ammonia solution is 0.2 mol/L, and the concentration of dopamine of is 6 g/L. The surface resistivities of commercial silver plating woven fabrics and commercial silver plating knitted fabric are, respectively, $0.136 \pm 0.02\,\Omega$ and $0.22 \pm 0.03\,\Omega$; the conductivity of SPCFs is approximate to that of the commercial conductive fabrics. Table 4 shows the surface resistivity of silver plating cotton fabrics by glucose reduction and silver plating cotton fabrics by polydopamine reduction. The surface resistivity of silver plating cotton fabrics by polydopamine reduction is $12.53\,\Omega$

Table 4: Surface resistivity of (A) the SPCF-PR and (B) silver plating cotton fabrics by glucose reduction before and after washing.

Samples/washing times	0 times	5 times	10 times	15 times
A (Ω)	12.53 ± 2.3	13.68 ± 2.3	16.27 ± 3.5	17.68 ± 2.3
B (Ω)	0.24 ± 0.03	1200 ± 56	\	\

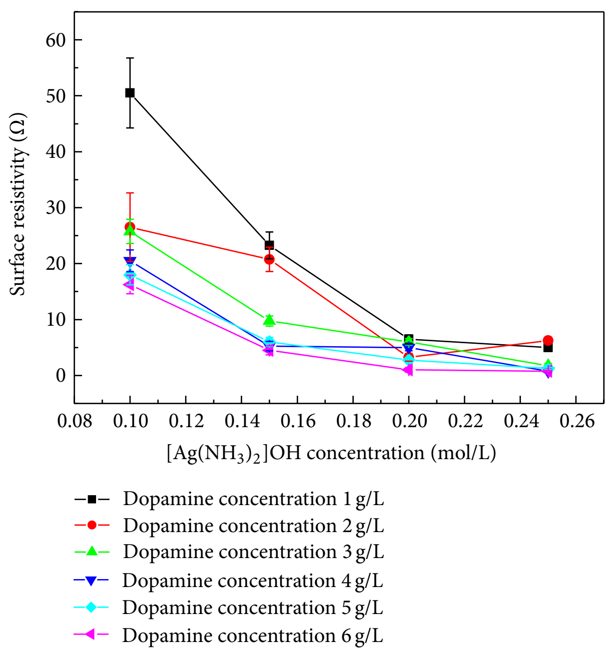


Figure 6: The surface resistivity of SPCFs in condition of different technological parameters.

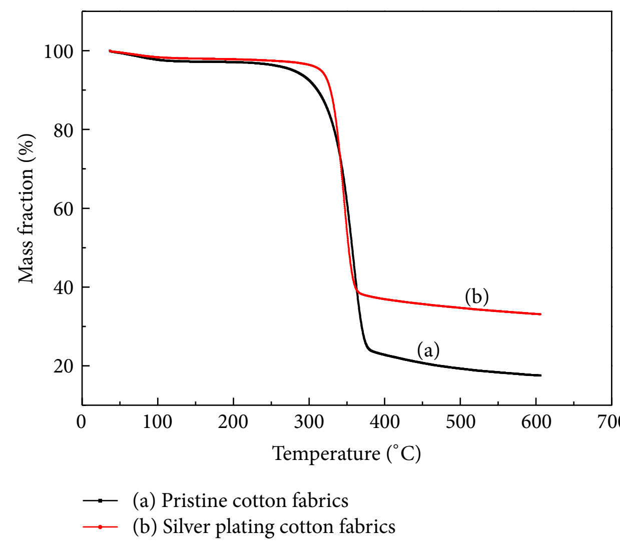


Figure 7: Thermogravimetric analysis diagram of (a) pristine cotton fabrics and (b) SPCFs.

before washing and is 17.68 Ω after 15 times washing. But the surface resistivity of silver plating fabrics by glucose reduction is 0.24 Ω before washing and is 1200 Ω after 5 times washing. Obviously, the washing fastness of silver plating cotton fabrics by polydopamine reduction is better than that of silver plating cotton fabrics by glucose reduction.

TG analysis was performed to acquire the thermal stability of pristine cotton fabrics and SPCFs. The SPCFs were fabricated according to the following parameters: 0.2 mol/L silver-ammonia solution and 6 g/L dopamine. The curves in Figure 7 indicate the decomposition temperature of the pristine cotton fabrics and the SPCFs were, respectively, 330.44°C and 331.12°C. Obviously, silver plating hardly affects the decomposition temperature of cotton fabrics. However, when the decomposition temperature is more than 600°C, the residual content of the two samples keeps at 17.57% and 33.15%, respectively. Because the decomposition temperature of silver is far more than 600°C, hence, we can confirm the silver content in SPCFs sample is 15.58%.

Figures 8(a), 8(b), 8(c), and 8(d) show, respectively, the FESEM images of SPCFs after 0, 5, 10, and 15 times washing. An even and smooth silver layer was coated on the surface of SPCFs before washing. However, some silver pieces which were stripped from the surface of SPCFs after washing were enclosed in black circle in Figures 8(b), 8(c), and 8(d). With increasing of washing times, the abscission of silver plating is severer.

Figure 9 shows the surface resistivity increases with increasing of washing times. The SPCFs were fabricated in our laboratory by using 0.2 mol/L silver-ammonia solution and 6 g/L dopamine; the surface resistivity quickly increases for the first washing; subsequently, the surface resistivity of SPCFs slowly increases and reaches 0.8 ± 0.03 Ω; however, the surface resistivity of commercial silver plating fabrics smoothly increases with the increasing of washing times. The washing fastness of silver plating on cotton fibers is not as good as that of the commercial silvered fabrics. The reason is possible that the cotton fibers (a natural fiber) have more complex chemical bonds than nylon fibers in commercial silver plating fabric. However, the conductive cotton fabrics have many advantages such as excellent wearability, air permeability, and moisture absorption. Hence, they can be utilized for biopotential dry electrode, electromagnetic shielding garments, or other smart textiles.

Figure 10 shows the photos of SPCFs and commercial silver plating fabrics before washing and after 15 times washing and in air for 30 days. After performing 15 times washing, the color of SPCFs fabricated in our laboratory has no obvious change as shown in Figures 10(a) and 10(b), but the commercial silver plating fabrics become darker and fade partially in color due to the presence of silver oxide and the stripping of the surface silver layer as shown in Figures 10(d) and 10(f). The surface resistivities of SPCFs, commercial silver plating woven fabrics, and commercial silver plating

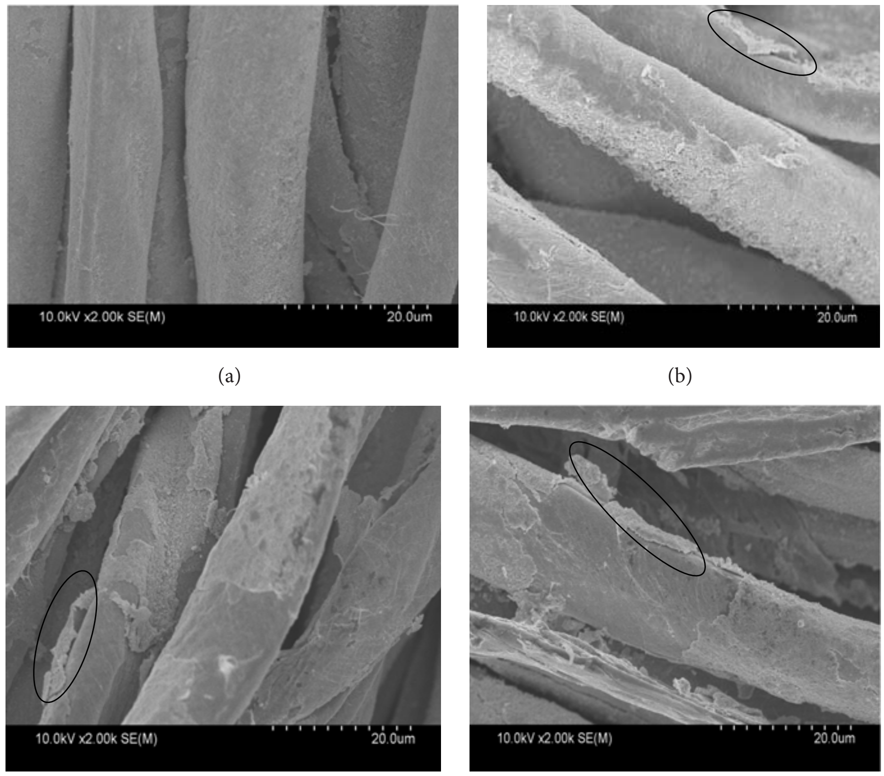


Figure 8: FESEM images of SPCFs after (a) 0 times, (b) 5 times, (c) 10 times, and (d) 15 times washing.

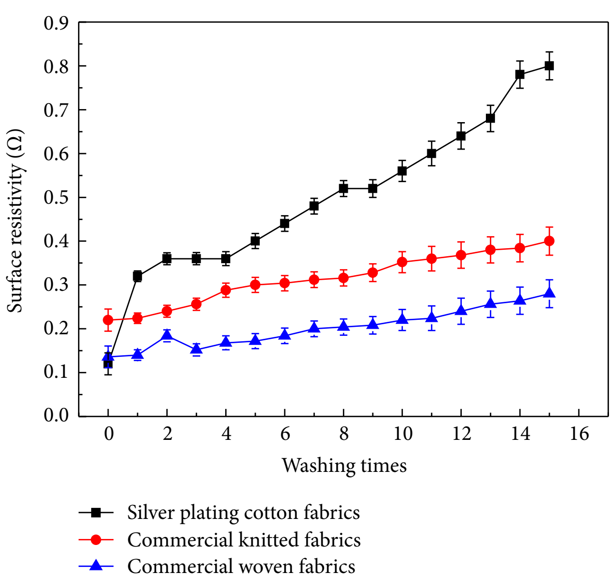


Figure 9: Surface resistivity versus washing times curve of silver plating fabrics.

knitted fabrics are, respectively, 0.8 ± 0.03 Ω, 0.28 ± 0.04 Ω, and 0.4 ± 0.03 Ω. The increasing of surface resistivity of SPCFs is due to separation of partial silver nanoparticles after 15 times washing, but the change amplitude of surface resistivity is acceptable and has not remarkable influence for electrical signal transmission in smart garments or intelligent textiles. Obviously, separation of silver plating is not the main reason of color change. The change in SPCFs in color is inconspicuous due to good color fastness and dark brown surface of polydopamine film; however, light coloured commercial silver plating fabrics fade easily.

Figure 11 shows the ESE of SPCFs fabricated in different technological parameters under electromagnetic radiation ranging from 10 MHz to 3 GHz. When the concentration of dopamine and silver-ammonia solution is, respectively, 6 g/L and 0.25 mol/L, the surface resistivity of SPCF is the smallest (0.12 ± 0.02 Ω), and the ESE is the best (in the range of 58.5 ± 4.5 dB). Table 5 shows the electromagnetic shielding effectiveness of SPCFs is in negative correlation with their surface resistivity when the structure and material of fabrics are identical.

4. Conclusion

This paper presented a novel two-step method for fabricating the SPCF with high conductivity and excellent washing fastness. Polydopamine (PDA) film was coated on the surface of cotton fabrics by self-polymerization of dopamine, the silver ions in silver nitrate solution were reduced by the catechol groups of polydopamine, and silver nanoparticles were combined with polydopamine by covalent bond on surface of cotton fabrics. Subsequently, silver plating was

Figure 10: Images of the (a) SPCFs before, (c) the commercial conductive nylon woven fabric, (e) the commercial conductive nylon knitted fabric, and (b) SPCFs and (d, f) commercial fabrics were treated after washing 15 times and exposed in air for 30 days.

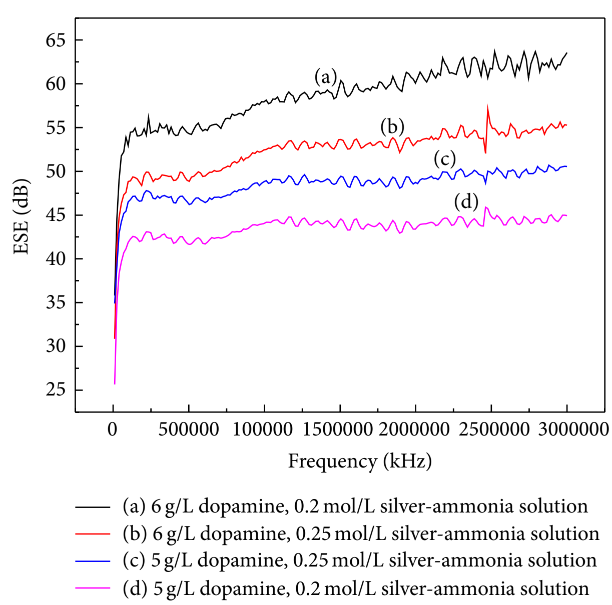


Figure 11: Electromagnetic shielding effectiveness (ESE) of the SPCFs.

Table 5: ESE (100 M~3 GHz) and surface resistivity of SPCFs fabricated by different technological parameters.

Sample number	1	2	3	4
Concentration of dopamine (g/L)	6	6	5	5
Concentration of silver-ammonia solution (mol/L)	0.2	0.25	0.2	0.25
ESE (dB)	58.5 ± 4.5	51.1 ± 3.2	48.6 ± 1.9	43.6 ± 1.4
Surface resistivity (Ω)	0.12 ± 0.02	0.15 ± 0.01	0.75 ± 0.2	1.25 ± 0.2

coated on the surface by glucose reduction reaction. With the increasing of silver-ammonia solution concentration or dopamine concentration, the surface resistivity of SPCFs all decreases and gradually stabilized. With the increasing of washing times, the surface resistivity increased. After 1st washing and 15th washing, the surface resistivity increased to $0.32 \pm 0.01\,\Omega$ and $0.8 \pm 0.03\,\Omega$, respectively. The electromagnetic shielding effectiveness of SPCFs is in negative correlation with their surface resistivity when the structure and material of fabrics are identical. When the concentration of dopamine and silver-ammonia solution is, respectively, 6 g/L and 0.25 mol/L, the surface resistivity of SPCF is the smallest ($0.12 \pm 0.02\,\Omega$), and the ESE is the best (in the range of 58.5 ± 4.5 dB). Silver plating fabrics can be widely applied in these fields, such as antibacterial, intelligent textiles, smart garments, electromagnetic shielding, and flexible sensors.

Competing Interests

The authors confirm having no competing interests related to this paper.

Acknowledgments

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