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Multifunctional Finishing of Cotton Fabric based on *In Situ* Fabrication of Polymer-Hybrid Nanoparticles

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ABSTRACT: This article describes a new strategy of fabricating multifunctional textile through *in situ* synthesis of polymer-hybrid nanoparticles in the fabric by a one-step microwave irradiation. The amino-terminated hyperbranched polymer (HBP-NH_2) was employed as the smart polymer, which not only can entrap silver ions and reduce them for the *in situ* generation of Ag nanoparticles, but also react with zinc ions to produce ZnO nanoparticles. Therefore, the two kinds of nanoparticles could be *in situ* fabricated in textile simultaneously under the control of HBP-NH_2. Cotton fabric was chosen for this study. The mechanism of the multifunctional finishing process of cotton fabric was discussed in detail. The treated cotton fabrics were characterized, and the ultraviolet (UV) protective properties and antibacterial activities of them were investigated in comparison with the cotton fabric treated with only one kind of nanoparticle. The results indicated that the nanoparticles were assembled on cotton fibers with size range from 20 to 100 nm. The multifunctional finishing could offer cotton fabric with excellent UV protective property and antibacterial activity simultaneously.
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INTRODUCTION

Over the past few decades, modification of natural fibers and functional finishing of textiles have attracted tremendous attention because of increasing awareness of human beings toward environmental protection and healthy, safe, and comfortable life.[1–4] They can impart desired functional properties to the fibers and textiles for their applications, such as ultraviolet (UV) resistance, antibacterial resistance, flame retardancy, antistatic, conductivity, hydrophobic, and hydrophilic surface.[5] The auxiliaries of treatment usually include organic compounds and inorganic nanomaterials. Compared with organic compounds, the inorganic nanomaterials have the advantages of nontoxic and chemically stable under high temperature and sunlight.[6] In order to assemble the nanomaterials on the fibers or textiles, many methods have been developed. Recently, *in situ* synthesis technique has inspired a great deal of interest because of its facile, efficient, and eco-friendly process as well as the uniform distribution and stability of the nanomaterials.[7–9]

Babu et al.[10] reported *in situ* synthesis of polypyrrole silver nanocomposite on cotton fabrics by a one-step redox reaction between pyrrole and silver nitrate. When the silver ions were reduced for generation of Ag nanoparticles, the pyrrole monomers were oxidized and polymerized to form polypyrrole which could fix the Ag nanoparticles on cotton. The treated cotton fabrics showed improved conductivity and excellent antibacterial activity. Li et al.[11] *in situ* fabricated nano-ZnO assembled cotton fabrics by microwave assistant using zinc nitrate and sodium hydroxide as the precursors. The ZnO nanoparticles with about 30–40 nm in diameter could be *in situ* generated in the lumen as well as in mesoporous structure of cotton fibers which provided excellent UV protective property and water-wash durability to the cotton fabrics. Peng et al.[12] presented a method of *in situ* growth of TiO_2 nanoflowers on cellulose-grafted polyethylene terephthalate (PET) nonwoven fabric by hydrolysis of $TiCl_4$ in aqueous solution at low temperature. The prepared PET fabrics exhibited excellent self-cleaning performance. However, the *in situ* synthesis method usually assembled only one kind of nanomaterials in textile providing a certain functional property. In order to realize multifunctional finishing of textile with a variety of nanomaterials, the textile should be treated with precursors of nanomaterials by several processes.[13]

Hyperbranched polymers, characterized by a three-dimensional structure and a large number of terminal groups, have been

applied as the templates for the control synthesis of nanoparticles, such as Ag, Au, and ZnO nanoparticles.[14–17] The synthesized nanoparticles exhibited good stability with small size and narrow size distribution. In our previous study, an amino-terminated hyperbranched polymer (HBP-NH_2) was synthesized.[18] Because of its special structure and amino functional groups, it was able to capture silver ions and reduce them to generate Ag nanoparticles without any other reducer and stabilizer in aqueous solution.[19] Otherwise, the numerous amino groups of HBP-NH_2 could release OH^- in aqueous solution. Therefore, HBP-NH_2 reacted with zinc salt directly for the fabrication of ZnO nanoparticles.[20]

The purpose of present work is to *in situ* fabricate multifunctional textiles with Ag nanoparticles and ZnO nanoparticles by a one-step treatment. To this end, the smart polymer HBP-NH_2 was utilized to react with silver salt and zinc salt at the same time. Otherwise, microwave-assisted synthetic technology has been extensively used for the preparation of nanomaterials owing to numerous advantages offered by microwave irradiation heating process, including volumetric heating, selectivity, fast kinetics, homogeneity, energy saving, compactness of equipments, and pollution-free environment.[21,22] In this regard, the Ag and ZnO nanoparticles can be *in situ* synthesized in textile by microwave irradiation based on the smart polymer. In this research, cotton fabric was chosen as the substrate. The mechanism of multifunctional finishing of cotton fabric by HBP-NH_2 was proposed. The treated cotton fabrics were characterized and UV protective properties and antibacterial activities of them were investigated. The treated cotton fabrics were found with excellent UV protective property and antibacterial activity simultaneously.

EXPERIMENTAL

Materials

Zinc nitrate hexahydrate, silver nitrate, and nitric acid (65%) were purchased from Sinopharm Chemical Reagent Co. Ltd., Shanghai, China and were used as received without any further purification or treatment. The HBP-NH_2 ($M_n = 2684$; $M_w = 7759$) was prepared as described in our previous paper.[18] Woven, bleached, and scoured cotton fabric (58 × 30 number of yarns per cm^2, 120 $g{\cdot}m^{-2}$) was obtained from the Huafang Group, Jiangsu, China. *Staphylococcus aureus* (*S. aureus*, ATCC 6538) and *Escherichia coli* (*E. coli*, ATCC 8099) were obtained from College of Life Science, Soochow University, Jiangsu, China. Nutrient broth and nutrient agar were purchased from SCAS Ecoscience Technology Co. Ltd., Jiangsu, China. Deionized water (18 MΩ.cm) was used in the experiments for preparation of all samples.

In situ Fabricating Polymer-Hybrid Nanoparticles in Cotton Fabric

HBP-NH_2, $Zn(NO_3)_2{\cdot}6H_2O$, and $AgNO_3$ were dissolved in deionized water to prepare stock solutions at concentrations of 100 g/L, 1 *M*, and 0.1 *M*, respectively. The HBP-NH_2 stock solution was added into deionized water, and then $Zn(NO_3)_2$ and $AgNO_3$ solutions were added in a dropwise manner into the solution successively and stirred constantly at room temperature. The concentrations of three precursors were varied to obtain optimal conditions for the functional finishing. The dried cotton fabrics were immersed in the hybrid solutions with liquor-to-fabric ratio of 50:1 (v/w) for 30 min with constant stirring. Then the cotton fabrics were taken out and placed in a household microwave oven (Galanz, P70D20P-TD model). The cotton fabrics were treated for 4 min under a power of 700 W. Finally, the treated samples were rinsed with tap water and dried at 80°C in a laboratory oven for 2 h.

Characterization of Treated Cotton Fabrics

Ultraviolet protection factor (UPF) and transmittance curve of treated cotton fabrics were measured by a UV-1000F Labsphere Transmittance Analyzer, North Sutton, NH using EN 13758-1:2001 standard at 25°C. The samples were tested with single layer. Each sample was measured six times. The whiteness of them was determined by an Ultrascan XE spectrophotometer (HunterLab Co. Ltd., Reston, VA). A Hitachi S-4800 scanning electron microscope (SEM) was employed to observe morphologies of control and treated cotton fabrics after coating with a gold layer to provide proper surface conduction. The ZnO and Ag contents of treated cotton fabrics were determined by a Vista MPX Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) (VARIAN Corp., Palo Alto, CA). The procedure is as follows: a sample (100 mg) was cut into small pieces and immersed in a 10 mL solution of HNO_3 (65%). After it dissolved, 90 mL deionized water was used to dilute it. Then 10 mL of the diluted solution was drawn to measure the concentrations of Zn^{2+} and Ag^+, and the ZnO and Ag contents in cotton fabric were calculated according to the results.

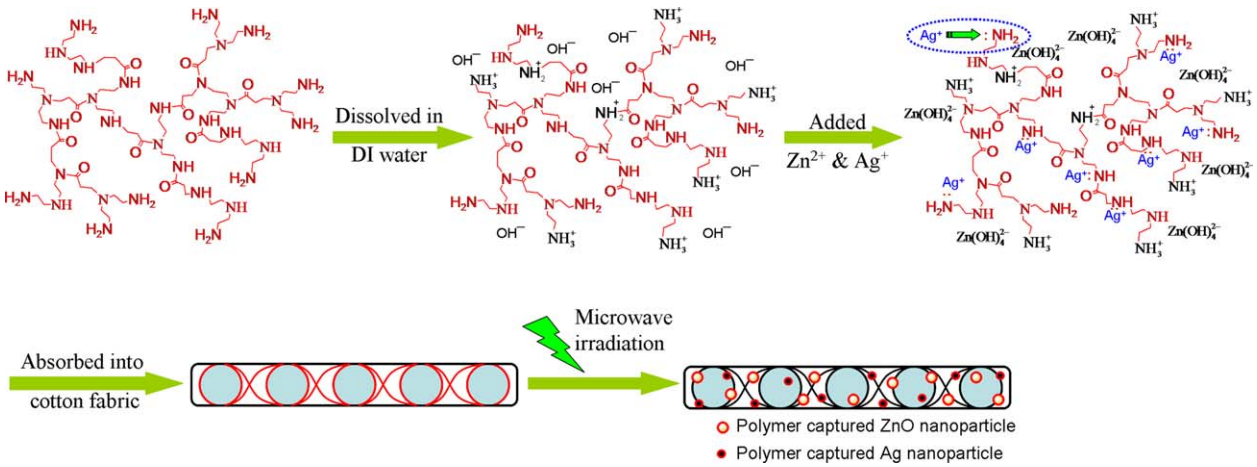
Antibacterial Testing

The antibacterial activity of treated cotton fabrics was evaluated using two categories of bacterial tests, qualitative and quantitative, against *E. coli* and *S. aureus*. For the qualitative bacterial test, an agar-diffusion plate method was applied. The agar plate was prepared by pouring the hot nutrient agar onto sterile petri dishes until it solidified. One milliliter of microbial culture ($1 \times 10^6 - 5 \times 10^6$ CFU/mL) was distributed uniformly on each plate. The treated cotton fabric disks as well as a control cotton fabric disk (7 mm diameter) were placed on the plates simultaneously. After 24 h incubation at 37°C, the dimension of inhibition zone was determined to evaluate the antibacterial property of samples.

The quantitative antibacterial test was carried out by a shake flask method according to GB/T 20944.3–2008 (China). The test procedure was performed as follows: a 0.75 g sample fabric was cut into small pieces of dimensions around 0.5 × 0.5 cm and dipped into a flask containing 70 mL of phosphate buffered saline (PBS; pH ≈ 7.2) and 5 mL of bacterial culture which had a cell concentration of $3 \times 10^5 - 4 \times 10^5$ CFU/mL. The flask was placed on a rotary shaker at 150 rpm for 18 h at 24°C. Solution (1 mL) was drawn from each sample well, diluted, and distributed into an agar plate. All plates were incubated at 37°C for 24–48 h and the colonies were counted. The percentage reduction (*R*; %) was determined by the following equation:

$$R(\%) = \frac{C - A}{C} \times 100 \quad (1)$$

where, *C* and *A* are the bacterial colonies of the control and treated cotton fabrics, respectively.



Scheme 1. Illustration of multifunctional finishing of cotton fabric based on *in situ* fabrication of polymer-hybrid nanoparticles by microwave irradiation. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

RESULTS AND DISCUSSION

Mechanism of Multifunctional Finishing of Cotton Fabric by HBP-NH_2

The textile finished with nanomaterials will impart corresponding properties of the nanomaterials to the textile. For instance, Ag nanoparticles treated textile shows excellent antibacterial activity. ZnO nanoparticles can provide effective UV protective property to the textile and impart a certain antibacterial property. In order to provide textile with excellent antibacterial activity and UV protective property at the same time, in this paper, multifunctional cotton fabrics treated with Ag and ZnO nanoparticles were fabricated by a one-step microwave irradiation based on the smart hyperbranched polymer, which could act as reagent, template, and binder in the finishing process. The mechanism of this process was described in Scheme 1. HBP-NH_2 has a three-dimensional architecture with numerous imino groups and terminal primary amino groups. When dissolved in deionized water, it could release OH^- because of protonation of amino groups. Therefore, the pH value of HBP-NH_2 aqueous solution was 10.81 at concentration of 3 g/L. Concurrently, HBP-NH_2 generated numerous positive charges in its structure. The zeta potential of HBP-NH_2 in aqueous solution was 23.5 ± 2.23 mV. After adding zinc ions and silver ions into the polymer aqueous solution, the zinc ions converted into $Zn(OH)_4^{2-}$ in the alkaline solution as reported by Wu et al. and Jiang et al.[23,24] The pH value of the solution decreased to 8.48 correspondingly because of consumption of OH^-. Meanwhile, silver ions were captured by HBP-NH_2 because of the complexation of its imino and amino groups. The zeta potential of the polymer hybrid increased to 45.62 ± 4.37 mV owning to the decrease of pH value. During the immersion process, the positively charged polymer with silver ions could take initiative to absorb into cotton fabric by electrostatic interaction owing to negative zeta potential of cotton fabric at neutral and alkaline conditions.[25] The $Zn(OH)_4^{2-}$ followed HBP-NH_2 was also absorbed into cotton fabric because of positive charge in the polymer. Subsequently, the cotton fabric was treated with microwave irradiation. During the microwave treatment process, the captured silver ions could be reduced by HBP-NH_2, which could provide electron source for the redox reaction and generated Ag nanoparticles under the control of polymer.[26,27] The formed $Zn(OH)_4^{2-}$ also transformed into ZnO nuclei and produced ZnO nanoparticles with the $Zn(OH)_4^{2-}$ as growth unit. The *in situ*-generated Ag and ZnO nanoparticles will be assembled and fixed in cotton fabric by HBP-NH_2 which can interact with cotton fibers through hydrogen bond.[28]

Ag and ZnO Nanoparticle-Treated Cotton Fabrics

In order to investigate the advantages of Ag–ZnO nanoparticles treated cotton fabrics by this method, whiteness, UV protective property, and antibacterial activity of them were studied in comparison with only one kind of nanoparticle-treated cotton fabric. Table I shows the content of nanoparticles, whiteness, UPF values, and transmittances of part A of UV radiation (UVA; wavelength from 315 to 400 nm) and part B of UV radiation (UVB; wavelength from 290 to 315 nm) of treated cotton fabrics. The concentrations of $AgNO_3$, $Zn(NO_3)_2$, and HBP-NH_2 for treatments were 0.2 m*M*, 4 m*M*, and 3 g/L, respectively. Compared with control cotton fabric, the UPF value of fabric treated with ZnO nanoparticles increased from 4.79 to

Table I. Comparison of Cotton Fabrics Treated via Different Strategies

Sample	Content of ZnO (wt %)	Content of Ag (mg/g)	Whiteness	UPF	T_{UVA} (%)	T_{UVB} (%)
Control	-	-	91.88	4.79 ± 0.10	27.79	19.10
Ag	-	0.403	79.26	5.03 ± 0.12	27.15	18.84
ZnO	0.76	-	93.57	58.10 ± 2.52	10.37	1.07
Ag–ZnO	0.72	0.389	82.13	54.91 ± 5.96	11.03	1.36

Table II. Different Concentrations of $AgNO_3$-Treated Cotton Fabrics

Sample	Concentration of $AgNO_3$ (m*M*)	Content of ZnO (wt %)	Content of Ag (mg/g)	UPF	Whiteness
Control	–	–	–	4.79 ± 0.10	91.88
a	0.1	0.69	0.190	58.31 ± 2.25	86.88
b	0.2	0.73	0.391	56.77 ± 4.67	81.40
c	0.4	0.77	0.908	62.38 ± 3.82	74.54
d	1	0.62	2.239	57.44 ± 4.91	66.83
e	2	0.75	4.585	60.84 ± 3.67	61.09

around 58. Generally, the UV protective properties of fabrics were evaluated as good when the UPF value reached above 30.[29] This treated cotton fabric with good UV protective property is because of the sufficient ZnO nanoparticles fabricated *in situ* in cotton fabric, which can absorb UV radiation. Therefore, the transmittances of UVA and UVB decreased correspondingly. Because of the localized surface plasmon resonance property of Ag nanoparticles, the whiteness of cotton fabric treated with them decreased from 91.88 to 79.26 compared with control cotton fabric.[30] However, the white ZnO nanoparticle enhanced the whiteness of its treated cotton fabric. Therefore, the multifunctional finishing with both nanoparticles could reduce the yellowness index of cotton fabric loaded with Ag nanoparticles. The whiteness enhanced from 79.26 to 82.13.

The content of Ag and ZnO nanoparticles in treated cotton fabrics could be adjusted through if treated with different concentrations of the precursors. Table II exhibits the content of nanoparticles, whiteness, and UPF values of cotton fabric treated with different concentrations of $AgNO_3$ when the concentration of $Zn(NO_3)_2$ and HBP-NH_2 were 4 m*M* and 3 g/L, respectively. With the increasing concentration of $AgNO_3$, the silver content in cotton fabrics increased, which will provide higher antibacterial activity to treated fabric. Correspondingly, the whiteness of them decreased gradually. Because of the same concentration of $Zn(NO_3)_2$, ZnO content and UPF values of treated cotton fabric in Table II are similar. When the concentration of $AgNO_3$ was set as a constant (0.2 m*M*) with variation of concentration of $Zn(NO_3)_2$ and HBP-NH_2, the results are displayed in detail in Table III. Because the dosage ratio between $Zn(NO_3)_2$ and HBP-NH_2 was crucial for generation of ZnO nanoparticles, concentrations of the two reagents were changed simultaneously at the same ratio. With the increasing of concentrations of the two reagents, ZnO contents and UPF values of treated cotton fabrics were both enhanced. Meanwhile, the whiteness of them increased owing to the increasing of content of white ZnO nanoparticles. When the concentration of $Zn(NO_3)_2$ and HBP-NH_2 were 8 m*M* and 6 g/L, respectively, the UPF value of treated cotton fabric has exceeded 100.

Characterization of Treated Cotton Fabric

The surface morphology of cotton fiber suffered multifunctional finishing with polymer Ag–ZnO nanoparticles hybrid via microwave irradiation was observed through SEM. Figure 1 shows the SEM images of control and treated cotton fibers. There is an obvious difference between them in terms of the surface roughness of the fiber. The control cotton fibers [Figure 1(a)] demonstrate smooth texture, whereas many particles can be found dispersed on the surface of the treated ones [Figure 1(b,c)]. From the SEM image of treated cotton fiber at high magnification [Figure 1(c)], it is obvious that the particles dispersed on the surface of cotton fiber are sphere- and rice-like in shape. The size of the particles is about from 20 to 100 nm as determined by SEM. These nanoparticles include Ag and ZnO nanoparticles according to the mechanism of the functional finishing. Therefore, the element composition of the surface of treated cotton fabric was also investigated by SEM equipment. Figure 2 depicts the energy dispersive X-ray (EDX) spectrum of the treated cotton fabric. It exhibits strong carbon and oxygen peaks. The peak of carbon was attributed to the cotton substrate. The oxygen may come from both the cotton fabric and ZnO. The appearance of zinc and silver peaks in the spectrum is as expected which are associated with the ZnO and Ag nanoparticles on the cotton fabric as shown in Figure 1. It should be noted that the peak related to gold comes from the coating of sample for SEM measurement. The peak of nitrogen owes to

Table III. Different Concentrations of $Zn(NO_3)_2$- and HBP-NH_2-Treated Cotton Fabrics

Sample	Concentration of $Zn(NO_3)_2$ (m*M*)	Concentration of HBP-NH_2 (g/L)	Content of ZnO (wt %)	UPF	Content of Ag (mg/g)	Whiteness
Control	–	–	–	4.79 ± 0.10	–	91.88
1	2	1.5	0.37	26.35 ± 2.73	0.423	80.51
2	4	3	0.72	54.91 ± 5.96	0.389	82.13
3	8	6	1.18	101.97 ± 6.26	0.427	83.42
4	12	9	1.57	118.02 ± 6.29	0.379	84.16

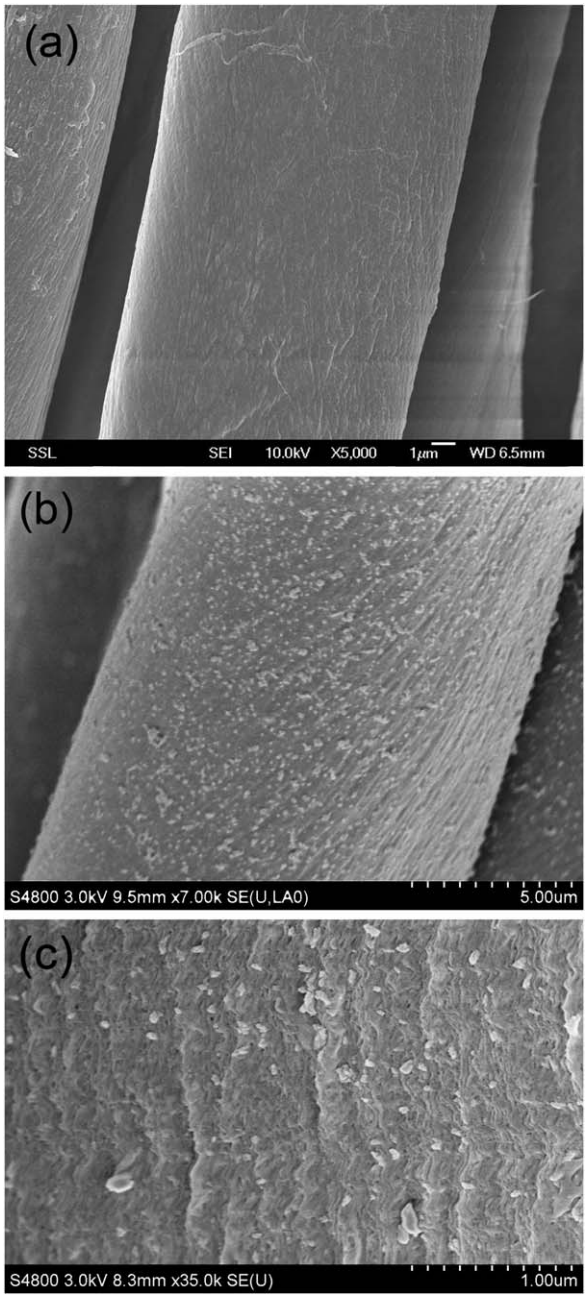


Figure 1. SEM images of (a) control cotton fibers, (b) polymer Ag–ZnO nanoparticles hybrid-treated cotton fibers, and (c) treated cotton fiber at high magnification.

the smart hyperbranched polymer adsorbed on the surface of nanoparticles and cotton fibers, supplying the binding between them.

UV Protective Property of Treated Cotton Fabrics

The treated cotton fabrics show excellent UV protective property because of the ZnO nanoparticles assembled in cotton fabric by HBP-NH_2. Therefore, the UV protective property of treated cotton fabric is related to the ZnO content of them. As shown in Table III, with increasing concentration of the precursors for generation of ZnO nanoparticles, the UPF values of treated cotton fabrics were enhanced. The corresponding UV

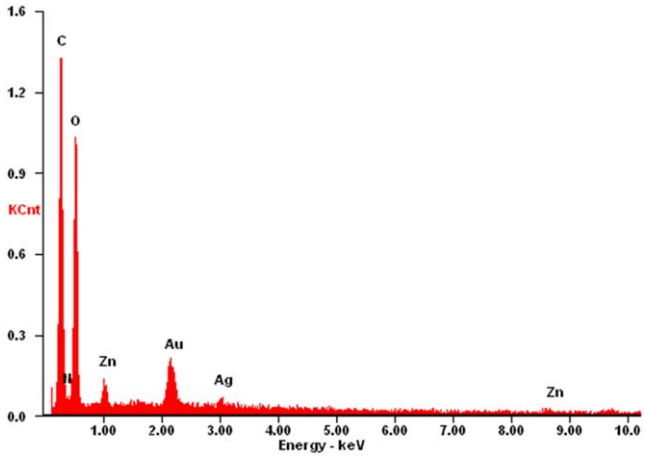


Figure 2. EDX spectrum of polymer Ag–ZnO nanoparticles hybrid-treated cotton fabric. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

transmission spectra of treated samples listed in Table III were described in Figure 3. It shows that the control cotton fabric had a high UV transmittance. After treated with ZnO nanoparticles, the UV transmittance of cotton fabric decreased rapidly. With increasing content of ZnO in cotton fabrics, the UV transmittance of them decreased constantly. When the ZnO content was 1.57%, the UPF value of treated cotton fabric reached 118 and the transmittances of UVA and UVB were 5.79% and 0.56%, respectively.

Antibacterial Activity of Treated Cotton Fabrics

The antibacterial activity tests of treated cotton fabrics were carried out against *S. aureus* and *E. coli*. Figure 4 shows the inhibition zones of control and treated cotton fabrics based on qualitative bacterial test. Sample "a" is control cotton fabric. Sample "b," "c," and "d" are Ag, ZnO, and Ag–ZnO nanoparticle-treated cotton fabrics, respectively, as shown in Table I. There was a dense population of bacterial colonies around the control cotton fabric, which indicated no antibacterial activity. In contrast, a clear inhibition zone could be distinctly observed around treated ones. The inhibition zones

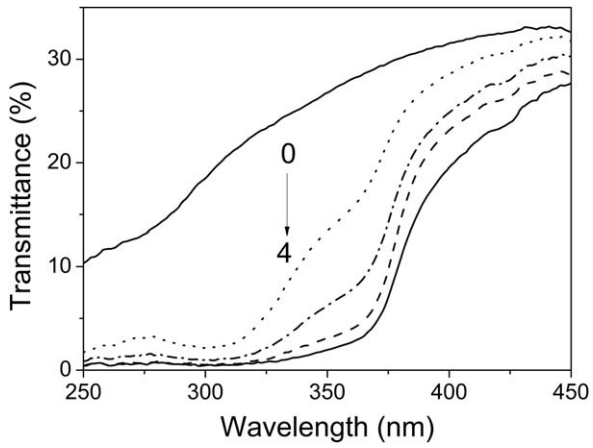


Figure 3. UV transmission spectra of control and treated cotton fabrics.

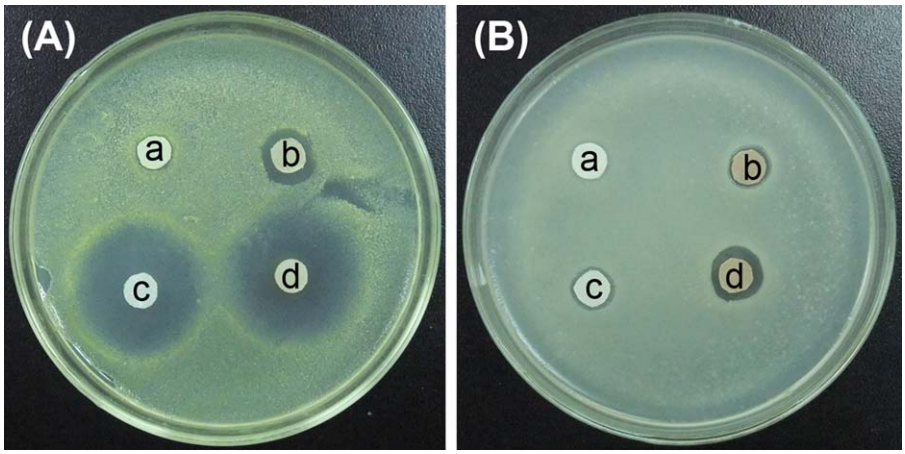


Figure 4. Photographs of the inhibition zone for the control and treated cotton fabrics against (A) *S. aureus* and (B) *E. coli.* [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

of three treated samples against *S. aureus* were larger than that against *E. coli*, suggesting that the treated cotton fabrics have more effective antibacterial activity against *S. aureus*. In addition, the photograph of inhibition zones against *E. coli* clearly shows that the inhibition zone of Ag–ZnO-treated cotton fabric was larger than that of cotton fabric treated with only one kind of nanoparticle, which demonstrated that the multifunctional finishing could enhance the antibacterial activity of textile.

Subsequently, the quantitative antibacterial tests were carried out and the results were listed in Table IV. The Ag nanoparticle-treated cotton fabric exhibited excellent antibacterial activity because of the exceptional antibacterial efficiency and broad antibacterial spectrum of Ag nanoparticles.[31] The bacterial reduction rates both exceeded 99% against *S. aureus* and *E. coli*. The bacterial reduction rate of ZnO nanoparticle-treated cotton fabric against *S. aureus* was 99.64%. However, its bacterial reduction rate against *E. coli* was only 72.62%, which indicated the insufficient antibacterial activity against some bacteria. This result is agreeable with the literatures, which reported that the minimum concentration of ZnO for efficient killing of bacteria is 2 wt % in textiles.[8,32] Therefore, the multifunctional finishing with Ag and ZnO nanoparticles could make up for this deficiency of ZnO-treated fabric and provide the treated substrate with excellent UV protective property and antibacterial activity simultaneously. The bacterial reduction rates of Ag–ZnO-treated cotton fabric against *S. aureus* and *E. coli* both reached 99.99%.

Table IV. Antibacterial Activity of Ag-Treated, ZnO-Treated, and Ag–ZnO-Treated Cotton Fabrics

	S. aureus		*E. coli*	
Samples	Surviving cells (CFU/mL)	Reduction (%)	Surviving cells (CFU/mL)	Reduction (%)
Control	1.37×10^6	-	8.4×10^6	-
Ag	8.20×10^2	99.94	1.09×10^4	99.87
ZnO	5.00×10^3	99.64	2.30×10^6	72.62
Ag–ZnO	1.37×10^2	99.99	8.40×10^2	99.99

CONCLUSIONS

In this research, multifunctional cotton fabric assembled with Ag and ZnO nanoparticles polymer hybrid was fabricated by a one-step *in situ* synthesis method via microwave irradiation. The smart hyperbranched polymer HBP-NH_2 not only can capture silver ions and reduce them to generate Ag nanoparticles, but can also release OH^- for the preparation of ZnO nanoparticles. Not only that, it exhibited multiple roles as reagent, template, and binder in the fabrication process. The treated cotton fabric with ZnO nanoparticles shows excellent UV protective property and a certain antibacterial activity. Because of the high antibacterial efficiency of Ag nanoparticles, the multifunctional finishing could provide the cotton fabric with excellent UV protective property and antibacterial activity simultaneously. The Ag content and ZnO content of cotton fabric could be adjusted by treating with different concentration of precursors, providing them with different UV protective property and antibacterial activity. When the concentration of silver nitrate, zinc nitrate, and HBP-NH_2 were 0.2 m*M*, 4 m*M*, and 3 g/L, respectively, the UPF value of treated cotton fabric was 55 and its bacterial reduction rate against *S. aureus* and *E. coli* both reached 99.99%.

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