

Water-Soluble Cationic Chitosan Derivative to Improve Pigment-Based Inkjet Printing and Antibacterial Properties for Cellulose Substrates

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Received 1 November 2010; accepted 18 May 2011

DOI 10.1002/app.34916

Published online 17 January 2012 in Wiley Online Library (wileyonlinelibrary.com).

ABSTRACT: The water-soluble *N*-(2-hydroxy)propyl-3-trimethylammonium chitosan chloride (HTCC) was prepared by chitosan and glycidyl trimethyl ammonium chloride. Fourier-transform infrared spectroscopic analysis spectrum confirmed that glycidyl trimethyl ammonium chloride was grafted to the amino groups of chitosan via substitution reaction (Zhao et al., *Int J Pharm* 2010, 393, 268; Montazer and Afjeh, *J Appl Polym Sci* 2007, 103, 178). The obtained chitosan derivative was used to modify cotton fabrics for improving aqueous pigment-based inkjet printing and antibacterial properties. Scanning electron microscope images showed that HTCC was adhered onto the cotton fabrics surface and formed film structure. The K/S value on printed cotton substrates increased from 3.20 to 4.87, which indicated that higher color yield was achieved in this way. Modified samples performed better

crocking fastness and laundering fastness than the control cotton fabrics. The line definition of modified cotton fabrics respectively, improved 16.5% and 12.6% in the warp and weft direction as the specified line width was $0.5 \times 10^3 \mu\text{m}$. Samples also showed good antibacterial potential, the inhibitory rate for *Staphylococcus aureus* and *Escherichia coli* were both more than 95% when the concentration of HTCC used in the pretreatment solution was 0.8%. The antibacterial effect was found to be durable for 20 laundering cycles. However, the hand feeling of modified cotton fabrics presented a slight decrease. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 125: 1674–1680, 2012

Key words: water-soluble chitosan derivative; inkjet printing; outline sharpness; antibacterial activity; hand feeling

INTRODUCTION

Chitosan, a copolymer of glucosamine and *N*-acetylglucosamine units linked by 1-4 glucosidic bonds, is the second abundant natural polymer found in a wide range such as crustaceans, fungi, and insects.^{3–5} The chemistry of chitosan is similar to that of cellulose, but it reveals the fact that the 2-hydroxyl group of the cellulose has been replaced with a primary aliphatic amino group. This polysaccharide has several useful properties, such as nontoxicity, biocompatibility, biodegradability, antimicrobial activity, chemical reactivity, and film forming ability, which makes it an important biopolymer for textile applications.^{6–9} Eom¹⁰ crosslinked the hydroxyl functional groups of positron emission tomography with the amine groups of chitosan using a catalyst and some diacids to produce an antistatic finish. Tseng et al.¹¹ evaluated the properties of nylon textiles grafted with chitosan oligomer or chitosan

polymer after being activated by open air plasma, and the chitosan-grafted nylon textiles showed a good antibacterial potential as well as biocompatibility. However, chitosan is insoluble in water due to its formation of strong hydrogen bonds, which severely limits its application.¹² As an attempt to develop the chitosan water-soluble performance for the purpose of broadening its application, Ma et al.¹³ reported the synthesis of a water-soluble chitosan derivative by Michael reaction. Taking advantage of the known capacity of solubility and ultraviolet absorption, the chitosan derivatives opened new possibilities for use as a sunscreen. Zuniga et al.¹⁴ introduced a propyl chain onto a modified chitosan, which offers the presence of hydrophobic and hydrophilic branches for controlling solubility properties of the new derivative.

Dye-based and pigment-based inks are two commercially kinds of textile inkjet printing inks. Compared with the dye-based inks, pigment-based inks are widely applied to textile inkjet printing technology, because of its simplicity and convenience. The print products can be achieved by simple heat curing without steaming and washing.^{15–18} However, inkjet printing with pigment-based inks also exists some

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defects, mainly performs the ink bleeding, especially with the low-viscosity aqueous pigment-based inks. To enhance inkjet printing quality, especially color depth and outline sharpness of printing samples, Kanik et al.¹⁹ modified the cotton woven fabric with 2,3-epoxypropyltrimethylammonium chloride using a cold pad-batch method and investigated the effect of cationization on inkjet printing quality. Choi et al.²⁰ studied the possibility and effectiveness of chitosan in pretreatment print paste for digital inkjet printing for cotton fabric. And the results showed that chitosan treatment enhanced the outline sharpness of the prints. Supaporn et al.²¹ modified chitosan pretreatment of polyester fabric for printing by inkjet ink. The K/S values, color gamut, tone reproduction, outline sharpness, and the surface appearance of the fabrics were characterized.

The aim of this study is to prepare a water-soluble chitosan derivative and apply it to improve aqueous pigment-based inkjet printing and antibacterial properties of cotton substrates. The structures of chitosan and water-soluble chitosan derivative were characterized by Fourier-transform infrared (FTIR) spectrum. The surface morphology of the control and modified cotton fibers were observed by scanning electron microscope (SEM). The color yield, outline sharpness, print pattern, color fastness, antibacterial activities, and hand feeling of inkjet printing samples were also discussed.

EXPERIMENTAL

Materials

A scoured, bleached cotton fabric (157 g/m²) was used. Acetone was supplied by Sinopharm Chemical Reagent, China. Chitosan (deacetylation 85%) was available by Zhejiang Golden-Shell Biochemical, China. Glycidyl trimethyl ammonium chloride (GTMAC) was obtained from Yantai Sangong Chemical, China.

Synthesis of HTCC

Two grams of chitosan was dispersed in 100 mL water, and 10 g of GTMAC was added to the

solution. The reaction was carried out at 80°C under nitrogen atmosphere with mechanical stirring. After 12 h of reaction, the clear viscous solution was poured into an excess amount of acetone. The precipitates were filtered, washed with acetone several times, and dried under vacuum at room temperature. The reaction mechanism between chitosan and GTMAC was characterized as Figure 1.

Fabric pretreatment

The cotton fabric was treated with various amounts of *N*-(2-hydroxy)propyl-3-trimethylammonium chitosan chloride (HTCC; 0-1.2 wt %) at a liquor ratio of 30 : 1 by the immersion method. pH values were then adjusted to 7, and the temperature was raised to 80°C over various times (5-30 min). The treated cotton sample was dried by baking at 90°C for 15 min.

Inkjet printing

Samples were printed by an Epson R-20 inkjet printer with bidirection six passes and variable 720 dpi in a low speed at 25°C and 65% humidity condition. Six self-made aqueous pigment-based inks with black, magenta, yellow, cyan, light cyan, and light magenta were used. The printed samples were dried at 70°C for 5 min and fixed at 140°C for 5 min by the curing fixation method.

CHARACTERIZATION AND TESTING

FTIR

FTIR spectrum was performed with a Bruker IFS 66/S spectrophotometer, equipped with silver gate Attenuated total reflectance (ATR) cell (45° angle incidence) employing a cylindrical Ge crystal ($n = 4.0$) with a diameter of 7 mm and single reflection. The samples were tightly pressed against a Ge crystal. The spectrum was recorded over the range 4000–400 cm⁻¹ with a resolution of 4 cm⁻¹.

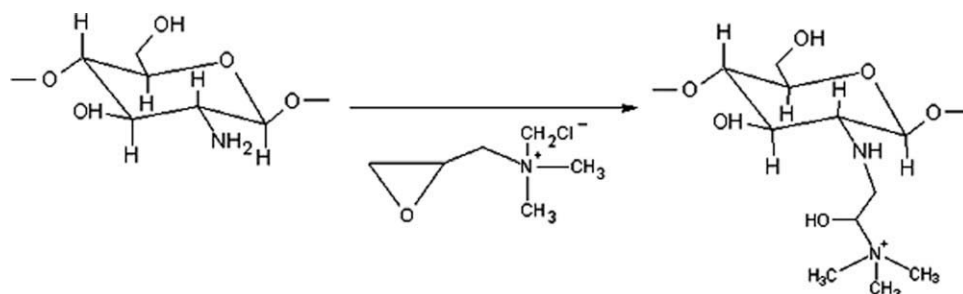


Figure 1 Reaction mechanism between chitosan and GTMAC.

SEM

Morphological characterization of the control and modified cotton fiber samples were performed on images acquired using a SU1510 scanning electron microscope at 5.0 KV accelerated voltage and 10–15mm working distance. The samples were coated with platinum of 10 nm thicknesses to make the samples conductive.

Color yield measurement

The color yield, expressed in K/S value, was determined by an X-Rite 8400 spectrophotometer (X-rite, The United States X-Rite Co., Ltd., US). Samples printed with magenta color pigment ink were measured in the system of CIE Lab, using the middle aperture (9 mm) by illuminant D65 and visual angle 10°. The K/S value was calculated with the Kubelka-Munk eq. (1). For measurement, the fabric samples were folded four layers and an average of three times per sample taken. The higher K/S value, the better the color yield will be, and *vice versa*.

$$K/S = (1 - R^2)/2R \quad (1)$$

Where K is the absorption coefficient, depending on the concentration of colorant; S is the scattering coefficient, caused by the colored substrate; R is the reflectance of printed fabric at the wavelength of the maximum absorption.

Outline sharpness analysis

To compare the outline sharpness of the prints, the line widths of the printed lines in both warp and weft directions were specified as $0.5 \times 10^3 \mu\text{m}$, $1.0 \times 10^3 \mu\text{m}$, and $1.5 \times 10^3 \mu\text{m}$. The practical widths were photographed by DZ3-Ultra High Magnification Zoom Microscope (Union Optical, Japan) and Pinnacle Studio Version 8.0 Software with a magnification of 75 multiple.

Color fastness properties

The color fastness properties of inkjet printed cotton fabrics to crocking fastness and laundering fastness were assessed by ISO 105-X12:1993 and ISO 105-C03:1989, respectively.

Antibacterial activity evaluation

The antibacterial activities were evaluated for the Gram-positive bacteria *Staphylococcus aureus* and the Gram-negative bacteria *Escherichia coli* according to the AATCC 100-2004 standard method. A liquid culture was prepared by mixing 5 g peptone and 3 g

beef extract in 1000 mL water. Cotton samples (2 cm \times 2 cm) were put onto agar plates and covered with agar seeded with 10^6 CFU/mL from microbe culture. Then the plates were incubated at 37°C for 24 h.²² The colonies of the bacterium on the agar plate were counted, and the reduction in the number of bacteria was calculated with the following eq. (2):

$$R = (B - A)/B \quad (2)$$

Where R is the reduction of bacteria; A is the number of bacteria recovered from the inoculated treated test specimen swatches in the jar incubated over the desired contact period; B is the number of bacteria recovered from the inoculated treated test specimen swatches in the jar immediately after inoculation.

Hand feeling

The hand feeling of samples, which was described by bending rigidity B and bending hysteresis HB , was measured by a KES FB2 AUTO-A automatic pure bending tester (KES Kato Tech, The United States X-Rite Co., Ltd., US). The values of bending rigidity and bending hysteresis are associated with softness and elasticity of the samples, respectively.

RESULTS AND DISCUSSION

FTIR spectrum

FTIR spectrums of both chitosan and chitosan derivative were presented in Figure 2. The FTIR spectrum of chitosan (A) showed that the main characteristic peaks at 3458 cm^{-1} which could be attributed to the stretching vibration of OH group, 2920 cm^{-1} and 2880 cm^{-1} assigned to CH stretching. The bands at 1650 cm^{-1} and 893 cm^{-1} showed the evidence for the presence of primary amino groups. The absorptions at 1160 cm^{-1} and 1090 cm^{-1} could be due to the result of bridge O and CO stretching, respectively. For the spectrum of chitosan derivative (B), the absorption at 1477 cm^{-1} was related to deformation and swing vibration of CH_3 in the trimethylammonium. The existence of $\text{CH}_2\text{CH}_2\text{NH}_2$ group was revealed by the peaks at 1448 cm^{-1} and 1409 cm^{-1} that represented the deformation and swing vibration of CH_2 in CH_2NH_2 , respectively. The peak at 937 cm^{-1} represented the deformation and swing vibration of OH in GTMAC. These results suggested that substitution reaction have occurred at amino groups.

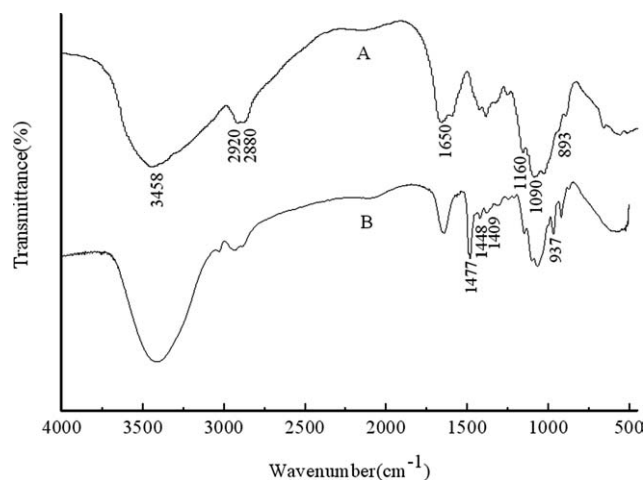


Figure 2 FTIR spectrums of chitosan (A) and chitosan derivative HTCC (B).

SEM image

The SEM images were used to observe the surface morphology of the control and modified cotton fibers, as shown in Figure 3. The control cotton fiber exhibited twisted shape and had a large number of grooves. However, the surface morphology of the modified cotton fiber was smooth and these grooves decreased significantly. This phenomenon showed that HTCC was adhered onto the cotton fabrics surface due to the existence of van der Waals force, hydrogen bonding, and electrostatic attractions, making cotton fibers with positive charges.

HTCC concentration

Different amounts of HTCC in pretreatment solution could impart different positive charges on the cotton surface, affecting the K/S value. Figure 4 shows the color yields of the digital inkjet printed fabrics under various amounts of HTCC used in the pretreatment solution.

It can be seen from Figure 4 that K/S value of printed fabrics increased along with the HTCC concentration increased, and K/S value achieved the maximum value, reached 4.87 as the HTCC concentration

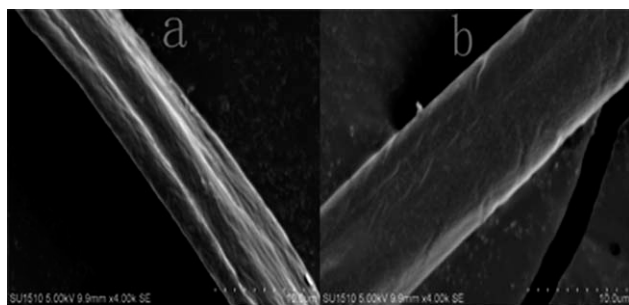


Figure 3 SEM images of the control (a) and modified (b) cotton fibers.

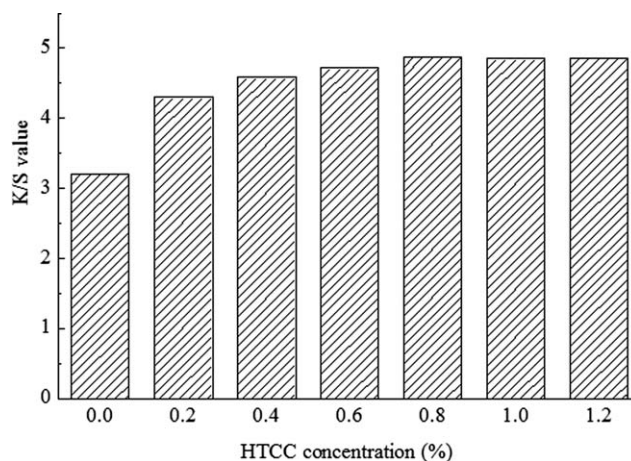


Figure 4 Effect of HTCC concentration on K/S value.

increased up to 0.8%. After that the K/S value had a little decrease with the increasing amount of HTCC. The color depth of modified samples was much deeper than that of control sample. The reason for K/S value increase is that ionic attraction occurred between the anionic pigment and the cationic charges on the modified cotton surface. When HTCC was applied on the cotton substrates, it was probably that the quaternary ammonium salt groups being electropositive in nature could facilitate the attraction of the negatively charge pigment anion onto the cotton fabric surface. The amount of the pigment particles per unit area in the printed areas increased. Moreover, HTCC was adhered to the cotton fabrics surface, which probably resulted in the reduction of the wicking effect. Few ink droplets would penetrate into the internal fabric. As the HTCC concentration increased up to 0.8%, the amount of the pigment particles per unit area with no further fluctuation, the K/S value presented a slight change and seemed to achieve a saturation value.

Modification time

Figure 5 showed the color yields of the digital inkjet printed fabrics under various times when the HTCC concentration used in the pretreatment solution was 0.8%. K/S value of printed fabrics increased along with modification time increased, and K/S value reached 4.87 as modification time was 20 min. After that K/S value presented a slight change. The reason for the phenomenon could be that the absorption of HTCC on cotton fabrics was also up to a saturation value when modification time was over 20 min. As the amount of the pigment particles per unit area achieved a saturation value, the K/S value almost unchanged.

Outline sharpness

To determine the effect of HTCC on print outline sharpness, Table I has listed the practical printed

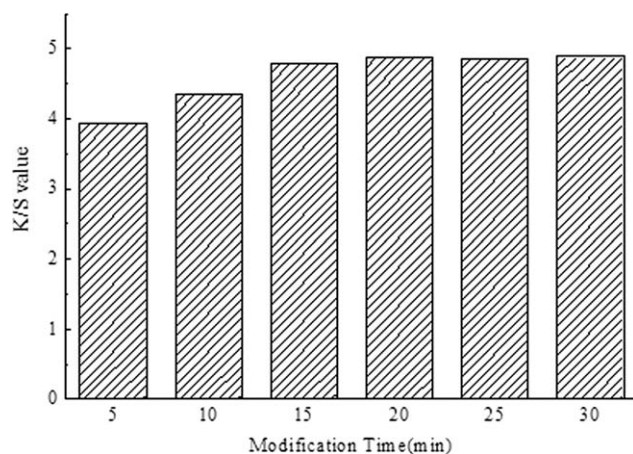


Figure 5 Effect of modification time on K/S value.

line widths in both warp and weft direction which were specified as $0.5 \times 10^3 \mu\text{m}$, $1.0 \times 10^3 \mu\text{m}$, and $1.5 \times 10^3 \mu\text{m}$. The concentration of HTCC used in the pretreatment solution was 0.8%.

As shown in Table I, the printed lines on the modified cotton were narrower than those on the control samples both in the warp and weft direction. When the specified width was $0.5 \times 10^3 \mu\text{m}$, the practical widths of modified cotton in the warp and weft direction were $0.7386 \times 10^3 \mu\text{m}$ and $0.6757 \times 10^3 \mu\text{m}$. The line definition improved 16.5% and 12.6%, respectively. This can be attributed to reduce spreading of the pigment-based ink droplets due to the strong ionic attraction between the HTCC-modified cotton and anionic pigment-based inks. It can also be seen from Table I that the inkjet printed lines in the warp direction were thicker than those in the weft direction, both in the control and modified samples. This was due to the differential wicking effect caused by the warp and weft yarns.²³

Color fastness properties

The color fastness grading of the inkjet printed cotton substrates was summarized in Table II. It can be seen that HTCC modification improved the color fastness properties. The crocking fastness and laundering fastness of modified cotton substrates were

TABLE I
Outline Sharpness of Inkjet Printed Fabrics

Direction	Specified widths ($\times 10^3/\mu\text{m}$)	Control sample ($\times 10^3/\mu\text{m}$)	Modified sample ($\times 10^3/\mu\text{m}$)	Growth rate (%)
Warp	0.5	0.8847	0.7386	16.5
	1.0	1.4795	1.2540	15.2
	1.5	1.9239	1.8294	4.9
Weft	0.5	0.7730	0.6757	12.6
	1.0	1.3828	1.2323	10.9
	1.5	1.7350	1.6637	4.1

TABLE II
Color Fastness Properties

Sample	Crocking fastness		Laundering fastness	
	Dry	Wet	Staining	Alteration
Modified sample	4	3-4	3-4	4
Control sample	3-4	3	3	3

better than those of the control samples with an improvement of at least half a grade. This was due to ionic attraction occurred between the anionic pigment and the cationic charges on the modified cotton surface. The positively charged sites tightly electrostatic adsorbed the anionic pigment. As a result, the pigment particles were difficult to be rubbed or washed off from cotton substrates.

Printed patterns

The printed patterns on the cotton fabrics without and with modification were presented in Figure 6. The concentration of HTCC used in the pretreatment solution was 0.8%. As shown in Figure 6, the printed pattern on modified cotton substrates was clearer and more colorful, in comparison to the pattern on the control sample. The definition and color yield of the control fabrics were poorer. The veins of flower petals were blurred and could not be easily recognized. On the contrast, the pattern on modified cotton substrates had excellent edge acuity and the color yield was also deeper. In addition, the printed pattern on the modified sample presented better sense of layer and three-dimensional effect. The results were due to the ionic bonds formed between modified cotton and anionic pigment-based inks, prevented the proliferation of pigment-based inks and improved the clarity of the modified samples. The other reason might be that HTCC was adhered onto the cotton fabrics surface, the surface morphology of fiber was smooth and grooves decreased significantly, which reduced capillary effect among the

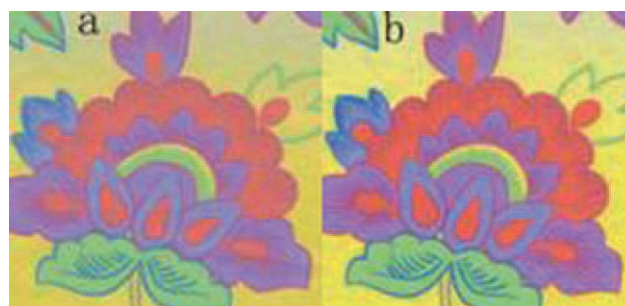


Figure 6 The inkjet printed patterns on the cotton fabrics without (a) and with (b) modification. [Color figure can be viewed in the online issue, which is available at www.onlinelibrary.wiley.com]

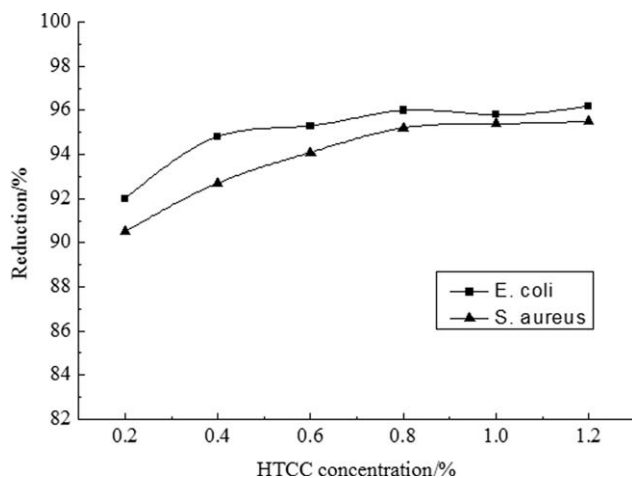


Figure 7 Effect of HTCC concentrations on antibacterial activities of modified cotton substrates.

fiber, making more pigment particles adhered in the printed area.

Antibacterial activities

The antibacterial activities of HTCC-modified cotton substrates were tested against *E. coli* and *S. aureus* by using a shaking flask method. Figure 7 showed results of the quantitative antibacterial activity evaluation for the samples with different HTCC concentrations.

As appeared in Figure 7, the modified cotton fabrics prepared with different HTCC concentrations showed high antibacterial activities with about 90%–95% reduction against *E. coli* and *S. aureus*, which can be due to the high antibacterial activity of HTCC. HTCC-modified cotton fabrics surface was active moieties against microorganisms by interaction with the cell membrane, which induced protein inactivation. In addition, HTCC affected the DNA molecules of bacteria and caused losses in their abilities to replicate and transfer.^{24,25} As a result, the modified samples performed excellent antibacterial effect. Moreover, it was shown from Table III that modified samples showed good durable antibacterial activity against *E. coli* and *S. aureus* at 0.8% concentration of HTCC. After 20 laundering cycles, antibacterial activity had only around 20% reduction against *E. coli* and *S. aureus*. Compare with Figure 7,

TABLE III
Antibacterial Activity of Modified Cotton Samples after Laundering

Reduction(%)	Laundering cycles				
	0	5	10	15	20
<i>E. coli</i>	95.62	89.31	88.93	83.60	77.76
<i>S. aureus</i>	94.86	88.76	87.42	82.91	74.35

TABLE IV
Hand Feeling of Cotton Fabrics

Sample	B (N cm/cm ²)		HB (N cm/cm ²)	
	Warp	Weft	Warp	Weft
Control sample	0.1574	0.0613	0.1769	0.0604
Modified sample	0.1711	0.0652	0.1864	0.0626
Growth rate(%)	8.7	6.3	5.3	3.6

the antibacterial activity of 0.8% HTCC-modified cotton substrate decreased to 89% for both bacteria groups after five laundering cycles, which indicated that 75% of the HTCC coating was removed from the surfaces. The reason for the phenomenon could be that part of HTCC was washed off from cotton substrates due to the decline of electrostatic attraction. Also, modified samples exhibited good durable antibacterial activity because free amino groups on cotton substrates surface had powerful antibacterial properties.²⁶

Hand feeling

Fabric hand feeling is an essential property of textile fabrics, which affects the sensorial perception of humans when they touch a fabric.^{27,28} To evaluate the cotton fabrics hand feeling, bending rigidity B and bending hysteresis HB were measured as shown in Table IV. The bending rigidity and bending hysteresis associated with softness and elasticity of the samples, respectively. The bigger bending rigidity and bending hysteresis are the worse fabric softness will be.

As listed in Table IV, modified cotton fabrics showed a slight increase in bending rigidity and bending hysteresis compared with the control sample. The growth rates of bending rigidity in the warp and weft direction were 8.7% and 6.3%, respectively. The bending hysteresis increased only 5.3% and 3.6%, respectively. The reason could be that HTCC was electrostatic adhered to the cotton fabric surface, which enhanced friction among fibers and limited their bending motions, making the hand feeling of modified cotton fabrics a slight decline.

CONCLUSIONS

The water-soluble chitosan derivative was prepared using chitosan and GTMAC via substitution reaction. The FTIR of HTCC indicated that substitution reaction occurred at amino groups of chitosan. SEM images showed that HTCC was adhered onto the cotton fabrics surface and formed film structure. The surface morphology of the modified cotton fiber was smooth and grooves decreased significantly. Cotton was modified with HTCC by the immersion method and printed with aqueous pigment-based inks. The color depth of the modified samples was deeper

along with HTCC concentration increased. As the HTCC concentration increased up to 0.8%, the K/S value reached 4.87. Outline sharpness and definition of printed patterns were improved markedly because of electrostatic attraction between modified cotton substrates and anionic pigment-based inks. Color fastness of modified samples was also better than those of the control cotton with an improvement of at least half a grade. Modified cotton fabrics exhibited satisfactory antibacterial activities, the inhibitory rate for *S. aureus* and *E. coli* were both more than 95% when the concentration of HTCC used in the pretreatment solution was 0.8%. The antibacterial effect was found to be durable for 20 laundering cycles and the hand feeling of modified sample showed a slight decline.

We are grateful to the financial support of the National Natural Science Foundation of China (No. 20674031 and No. 21174055) and the Business Doctoral Innovation Project of Jiangsu Province in China (BK2009672).

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