

Structure and Properties of Hemp Fabric Treated with Chitosan and Dyed with Mixed Epoxy-Modified Silicone Oil

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ABSTRACT: To enhance the color yield and improve the soft handle, hemp fabrics were treated with chitosan of molecular weight 4200 and degree of deacetylation 0.90, and then dyed using Remazol Brilliant Blue R with mixed epoxy-modified silicone oil in different volume ratios. The structural changes in hemp fibers were investigated by means of scanning electron microscope, FTIR, TG, DSC, and XRD. The properties of tensile, bending, dyeing, and color fastness for hemp fabric were also studied. The results showed that when compared with the untreated hemp fiber, the thermal performance of chitosan/silicone oil-modified hemp fiber changed and the percent residual

weight increased in the range of temperature 25–550°C. The crystal grain size decreased and the degree of crystallization increased. For chitosan/silicone oil-treated hemp fabric, the flexural stiffness and tensile properties degraded. The maximum color yield (*K/S* value) was obtained when the volume ratio of dyeing liquor to silicone oil was 2 : 1. The color fastnesses to rubbing and wet scrubbing were also improved. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 114: 1377–1383, 2009

Key words: hemp fiber; chitosan; epoxy-modified silicone oil; dyeing

INTRODUCTION

With the advantages of ecological concerns, environmental safety, future resource balance, and so on for clothing, hemp has recently been undergoing a revival worldwide as a fast-growing natural source of fibers. Textile products made from hemp fibers are targeted to be used for high value-added applications. Because of the presence of some noncellulosic substances in raw hemp bast, the softness and color fastness of hemp fibers are not satisfactory for producing hemp fabric with comfort and good appearance.^{1,2} Research efforts are mainly concerned with the technological issues to remove noncellulosic substances in the bast without damage to the fiber cellulose.^{3,4} Although the chemical and mechanical processing of bast hemp fibers is effective for removing noncellulosic substance, the degradation of hemp fibers cannot be avoided completely. The dyeing property of hemp fibers after chemical processing is another problem need to be solved. At the same time, as hemp fibers are generally longer and stronger when compared with cotton fibers, garments made from hemp fibers are more stiffer and harsher than other clothes for fabric hand.

Chitosan (β -(1,4)-2-amino-2-deoxy-D-glucose) is a natural product obtained by alkaline deacetylation of chitin. It has been demonstrated by many researchers that chitosan has a great potential for a wide range of applications because of its hydrophilicity, biodegradability, biocompatibility, antimicrobial activity, nontoxicity, and versatile chemical and physical properties.⁵ Chitosan can be used for textile wet processing, including dyeing, printing, durable press, antistatic finishing, etc.^{6–14} It can easily absorb anionic dyes such as direct, acid, and reactive dyes by electrostatic attraction because of its cationic nature in an acidic condition. However, chitosan-treated fabrics generally become harder and harsher for wearing. Furthermore, the yellowing effect during chitosan finishing may occur in the treatment using citric acid as crosslinking agent. Hence, silicone oils are employed to treat fabric for improving the repeated laundering and the hydrophilic and antistatic ability.^{15,16} Besides the performances of good water solubility, stability, and compatibility, epoxy-modified silicone oil can endue fabric with durable characteristics of bulk, soft, elasticity, full, good air permeability, and wrinkle recoverability.¹⁷

In this work, to enhance the color yield and improve the handle of hemp fabric, we used chitosan with a molecular weight of 4200 and degree of deacetylation of 0.90 to treat hemp fabric using citric acid as crosslinking agent at first, and then dyed the

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chitosan-treated hemp fabric using Remazol Brilliant Blue R with the admixture of epoxy-modified silicone oil. The structural changes in hemp fibers as a result of chitosan and silicone oil treatments were investigated by means of scanning electron microscope (SEM), TG, DSC, and XRD. The effects of chitosan and silicone oil treatments on the fabric properties including tensile, bending, dyeing, and color fastness were also studied.

EXPERIMENTAL

Materials

The bleached hemp fabric (plain weave, the linear densities of ends and picks are identical, 14 tex, numbers of ends and picks are identical, 220 per 10 cm) was kindly supplied by Zhejiang Lihui Dyeing & Finishing.

All chemicals, citric acid ($C_6H_8O_7$), sodium hypophosphite monohydrate ($NaH_2PO_2 \cdot H_2O$), sodium chloride (NaCl), sodium hydroxide (NaOH), and sodium carbonate (Na_2CO_3) used in this study were of laboratory reagent grade. FR-920 epoxy-modified silicone oil was provided by Xianjin Fengran Chemical (Guangzhou, Guangdong, China). The wetting agent JFC (main chemical composition: fatty alcohol polyoxyethylene ether) was obtained from Xianyang Jihua Xinsanling Printing and Dyeing (Xianyang, Shaanxi, China). Chitosan of molecular weight 234,000 and degree of deacetylation 0.90 was supplied by Qingdao Heppe Biotechnology (Qingdao, Shandong, China). The Remazol Brilliant Blue R vinyl sulfone (C.I. Reactive Blue 19) was used for dyeing hemp fabric in this experiment.

Chitosan treatment of hemp fabric

First, chitosans were dissolved in 5% acetic acid and 4% hydrogen peroxide, and then ultrasonically degraded for 0.5 h using a SY2200-T sonicator (Shanghai Shengyuan Supersonic Instrument Equipment, Shanghai, China) at an energy level of 100 W at 40°C and subsequently was kept overnight at room temperature. The degraded solutions were neutralized with 0.1M sodium hydroxide to precipitate the degraded chitosans. They were collected and washed with distilled water until neutralized and then dried at 40°C in a DZ-2BC vacuum drying oven (Taisite Instrument, Tianjing, China), thereby yielding chitosans with molecular weight of 4200. Second, 1.4% of degraded chitosan was dissolved in 14% of citric acid solution at room temperature, followed by the addition of 7% of sodium hypophosphite monohydrate and 0.2% of wetting agent, which was mixed to make the chitosan processing solution. Third, hemp fabric was impregnated in chitosan processing solution at 85°C for 30 min at a liq-

uor ratio of 30 : 1, and then padded twice to a wet pickup of 100%. The padded fabric was then dried at 95°C for 2.5 min and cured at 180°C for 1 min. Finally, the sample was washed thoroughly at 80°C for 10 min, neutralized with 0.1M sodium hydroxide, and then again cold washed and dried in air.

Dyeing of hemp fabric with mixed epoxy-modified silicone oil

Chitosan-treated hemp fabric was dyed with Remazol Brilliant Blue R using the following procedure: dye bath was made with dye (o.w.f 1%), sodium chloride (40 g L^{-1}), sodium carbonate (10 g L^{-1}), and wetting agent (2%), keeping the material-to-liquor ratio at 1 : 50. Chitosan-treated fabric sample was dipped into the dye bath and kept for 10 min with stirring at 60°C. After this treatment, 20 g L^{-1} of sodium chloride was added in the same bath every 10 min and kept for 10 min under the same condition. The dye bath was heated to 90°C at a speed of 2°C min^{-1} , followed by adding 10 g L^{-1} of sodium carbonate. The samples were kept for 35 min, and 30 g L^{-1} of transparent organic silicone microemulsion was added and kept for 5 min at the material-to-liquor ratio of 1 : 30. The dyed sample was then dried at 100°C for 3 min and cured at 150°C for 3 min. Thereafter, the sample was washed with cold water, soaped with soap (2 g L^{-1}), sodium carbonate (2 g L^{-1}) at a liquor-to-goods ratio of 30 : 1 for 15 min at boil followed by usual washing, and then dried in air.

Characterization techniques

Scanning electron microscopy

Dry samples of hemp fibers before and after treatment were mounted on SEM stubs and sputter coated with platinum before examination in a JSM-6700F field SEM (JEOL, Japan). Photographs of the samples with surface characteristics were taken randomly.

FTIR spectrometry

The hemp fiber samples were examined in KBr pellets obtained by pressing the pulverized materials at 1% concentration. The FTIR spectrophotometer used was Nicolet Nexus870 model (USA), and the spectra were collected with the aid of OMNIC software. The wavenumbers was in the range of $400\text{--}4000 \text{ cm}^{-1}$.

TG and DSC testing

Thermogravimetric analyses were determined on the samples before and after treatment using a TGA/SDTA851e thermal gravimetric and differential scanning calorimetry analysis (TG-DTA) instrument (Mettler Toledo, Switzerland). Percentage weight

change versus temperature was evaluated at heating rates of $10^{\circ}\text{C min}^{-1}$ with a nitrogen flush rate of 30 mL min^{-1} over the range of $25\text{--}550^{\circ}\text{C}$. The onset and endset decomposing temperatures and the percent residual weight were obtained according to ASTM E 474-80. DSC analyses were performed in a Sapphire apparatus (Perkin Elmer, USA) equipped with a DSC 20 cell purged with nitrogen of 30 mL min^{-1} . The temperature program was set in the range from 25 to 550°C at a heating rate of $10^{\circ}\text{C min}^{-1}$.

XRD testing

X-ray diffraction patterns were collected by a Shimadzu (Japan) diffractometer (Model XRD-7000S, Cu $K\alpha$ radiation, $\lambda = 0.1540562\text{ nm}$, 40 kV, 50 mA) in the 2θ ranging from 5° to 40° with a step width of 0.1° . The apparent crystallite size of the sample was determined using the Scherrer formula. The degree of crystallinity was calculated using Jade 5.0 software.

Fabric tensile testing

Tensile properties of the samples were carried out on an Instron 5565 electromechanical test system (Instron Japan) according to GB/T 3923.1-1997. The initial gauge length was 10 cm, and the width was 5 cm. The testing rate was 50 mm min^{-1} , and the pretension was 4.9 N. Ten samples per treatment set were tested and the breaking load and tensile strain averaged.

Fabric bending testing

The flexural rigidities of hemp fabric before and after treatment were evaluated using the LLY-01B electrical fabric stiffness tester (Laizhou, Shandong, China) according to GB/T 18318-2001. The length of the sample was 15 cm and the width was 2 cm. The bending angle was set on 45° . Values reported were averages of at least five measurements.

Color yield measurement

The color yield K/S values of dyed samples at the maximum light absorption wavelength 650 nm were measured under D65/10° illuminant by using a Datacolor SF300 Plus colorimeter using a measuring area with a diameter of 9 mm. The colorimeter was calibrated before testing against a standard white board. Samples were measured three times in each of five sites, and the average of the measurements was recorded as the K/S value.

Color fastness of the fabrics

The fastnesses to rubbing and wet scrubbing of untreated, chitosan-treated and chitosan/silicone oil-

treated samples were evaluated using the YG571 color fastness tester to assess the color fastness to rubbing and wet scrubbing according to GB/T 3920-

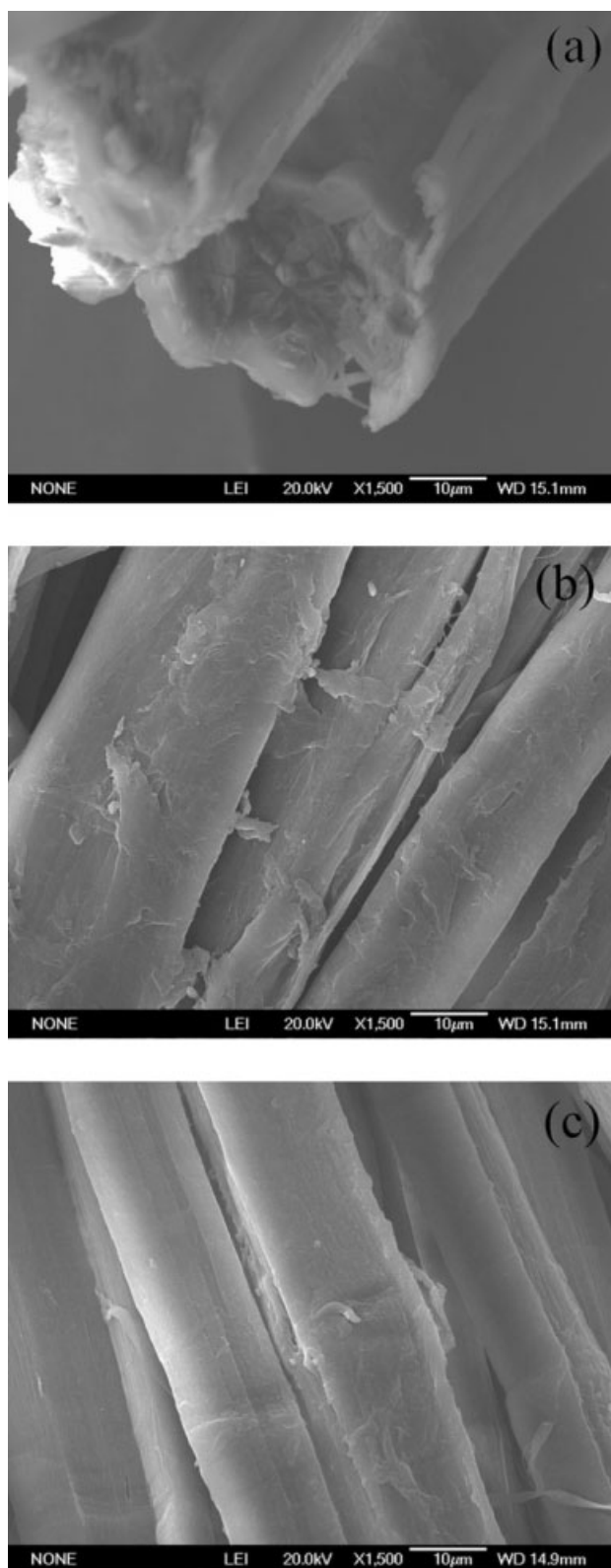


Figure 1 Electron photomicrographs of untreated (a, $\times 1600$; b, $\times 1500$) and chitosan treated and dyed with mixed epoxy-modified silicone oil (c, $\times 1500$) hemp fibers.

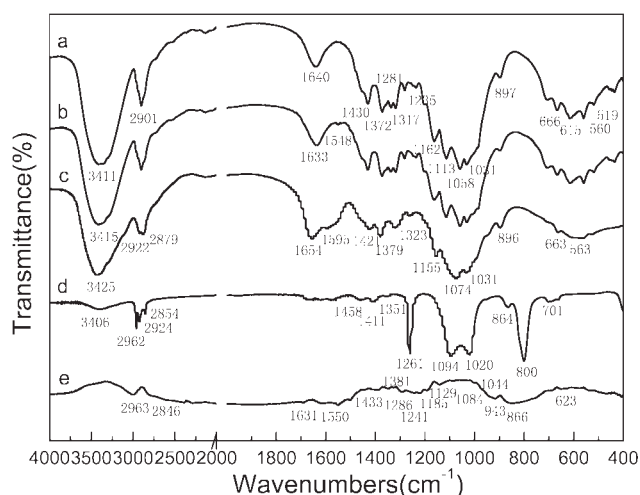


Figure 2 FTIR spectra of the samples: (a) untreated hemp fiber, (b) hemp fiber treated with chitosan and dyed with mixed epoxy-modified silicone oil, (c) chitosan, (d) epoxy-modified silicone oil, and (e) subtract spectrum of b and a.

1997 and GB/T 420-1990, respectively. The color fastnesses were assessed and sorted into five grades, marked 1, 2, 3, 4, 5, according to the ISO/BS standard gray card. Grade 1 means that the color change is evident and Grade 5 is not noticed. All experiments were carried out at least in triplicate.

RESULTS AND DISCUSSION

Fiber surface morphology

The surface of untreated hemp fiber observed with SEM was smooth and clean when compared with the chitosan/silicone oil-treated hemp fiber. Fiber bundles were arranged in the axis of fiber, in which a number of tiny linear grooves were dispersed on its surface. The loose structure and tiny holes could be found on the transverse section and inner surface of hemp fibers [see Fig. 1(a)]. The profiles of the fibers were irregular and most of them had the ellipsoidal or polygonal shape. Besides many small crevices and holes distributed along the warp fibers, a few debris and other nonfibrous impurities could be seen as the polypectate was not degummed completely [see Fig. 1(b)]. After being treated with chitosan and dyed with mixed epoxy-modified silicone oil, the fiber surface was coated with a thin membrane. Some small particles and silky substances were adhered to the fiber surface [Fig. 1(c)].

FTIR analysis

The spectra of hemp fibers before and after treatments are illustrated in Figure 2. The spectra of chitosan and epoxy-modified silicone oil were also given. It could be seen that an analogous pattern was shown by the untreated hemp sample [line (a)],

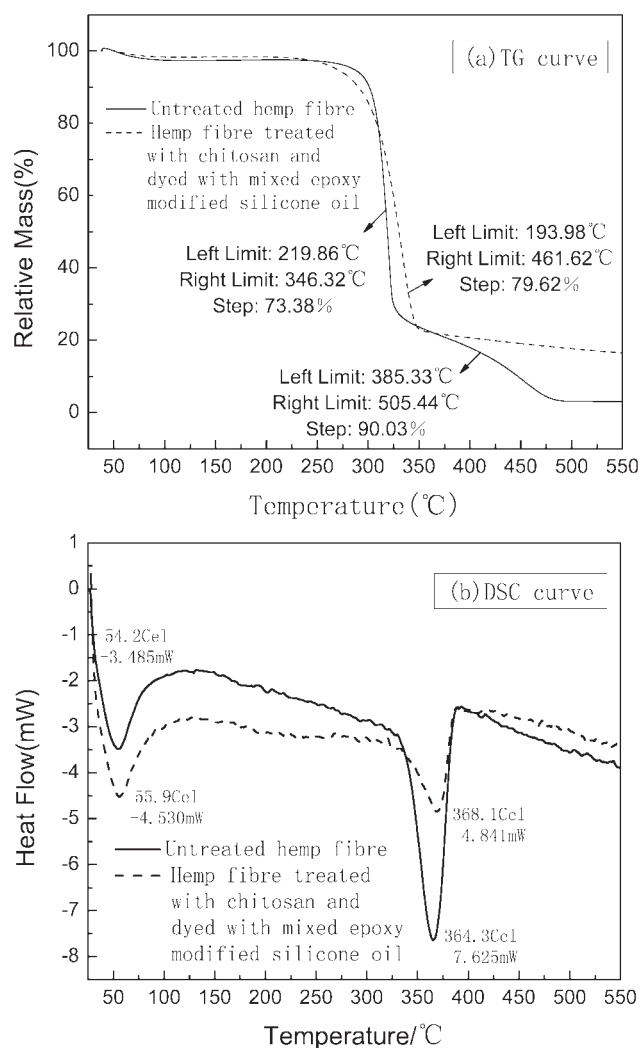


Figure 3 TG and DSC curves of the samples.

whereas in the spectrum of the chitosan/silicone oil-treated sample [line (b)], the peaks did not indicate any significant change. So, the subtraction of (b) and

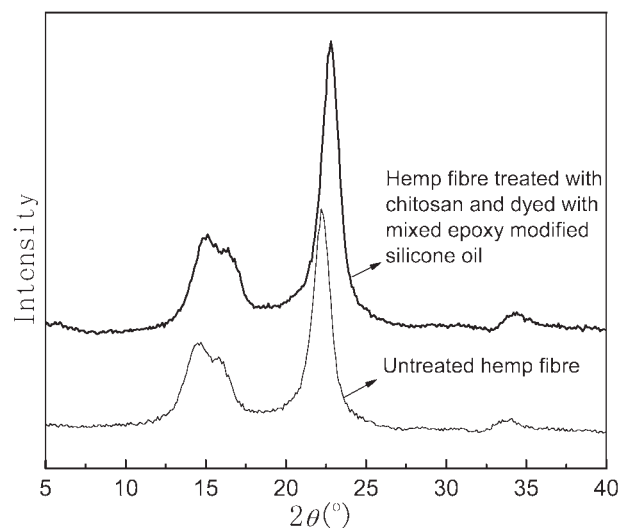


Figure 4 X-ray diffractions of the samples.

TABLE I
X-ray Diffraction of the Samples

Sample	Characteristic peaks 2θ ($^{\circ}$)	Interplanar spacing (nm)	FWHM ($^{\circ}$)	Size of crystal grain (nm)	Crystallinity (%)
Untreated hemp fiber	14.50	0.610	1.897	4.221	62.9
	16.10	0.550	1.414	5.694	
	22.21	0.400	1.384	5.872	
	33.81	0.265	1.779	4.670	
Hemp fiber treated with chitosan and dyed with mixed epoxy-modified silicone oil	15.10	0.586	2.129	3.759	75.8
	16.70	0.530	1.406	5.733	
	22.74	0.391	1.406	5.784	
	34.40	0.261	1.683	4.948	

(a) [line (e)] was analyzed. Compared with the spectrum of chitosan [line (c)], the peak at 1654 cm^{-1} , attributed to amide I band, appeared to be shifted to 1631 cm^{-1} . The amide II band at 1595 cm^{-1} was absent. Instead, the absorption band at 1550 cm^{-1} could be observed. The peak at 1379 cm^{-1} (C—H bending and CH_2 symmetrical deformation) shifted to 1381 cm^{-1} . The peaks at 1421 cm^{-1} (CH_2 bending and CH_3 deformation) for chitosan and 1411 cm^{-1} (Si—CH_3 out-of-plane bending) for silicone oil overlapped with a sharp peak at 1433 cm^{-1} . When compared with the spectrum of silicone oil [line (d)], the peaks at 1261 cm^{-1} (Si—CH_3 in-plane bending), 1094 cm^{-1} , and 1020 cm^{-1} (Si—O—Si stretching) shifted to 1286 , 1084 , and 1026 cm^{-1} , respectively. The peaks at 864 cm^{-1} (Si—OH band), 800 cm^{-1} (Si—C stretching and CH_3 rocking) for silicone oil, and 896 cm^{-1} ($\beta\text{-D-glucopyranoside}$ band) for chitosan overlapped in the range $900\text{--}700\text{ cm}^{-1}$ with a broad peak at around 866 cm^{-1} . At the same time, the new absorption bands at 1241 , 1185 , 1129 cm^{-1} and 941 cm^{-1} were identified, which could be assigned to asymmetrical and symmetric stretching vibrations of —COO^- anion group. So, this suggested that the chemical modification induced in hemp fibers by chitosan/silicone oil treatment was confirmed.

TG and DSC analyses

The effect of chitosan/silicone oil treatment was distinctly observed in the thermograms of hemp fibers

TABLE II
Tensile Properties of the Samples

Test targets	Untreated hemp fabric		Hemp fabric treated with chitosan and dyed with mixed epoxy-modified silicone oil	
	Warp	Weft	Warp	Weft
Breaking load (N)	1008.31	1075.34	790.11	600.02
Tensile strain (%)	18.48	23.10	15.00	14.00

run at a heating rate of $10^{\circ}\text{C min}^{-1}$, as depicted in Figure 3. It was clear from TG curves [Fig. 3(a)] that 26.6% and 10.0% residual weight occurred in the range of $219.8\text{--}346.3^{\circ}\text{C}$ and $385.3\text{--}505.4^{\circ}\text{C}$ for the untreated fiber sample. On treating with chitosan/silicone oil onto hemp fibers, 20.4% residual weight occurred in the range of $194.0\text{--}461.5^{\circ}\text{C}$. Differences in ash left in the crucible at the end of the TG runs had shown that the inorganic content of treated fiber was larger than that of untreated fiber. It was obvious from DSC curves [Fig. 3(b)] that the small endothermal peaks at 54.2°C for the untreated sample and 55.9°C for the chitosan/silicone oil-treated sample were attributed to the dehydration of absorbed water. The maximum endothermal temperature was shifted from 364.3°C to 368.1°C when the hemp fabric was treated with chitosan/silicone oil. This was due to the chemical reactions between hemp fiber, chitosan, and silicone oil.

XRD analysis

The diffraction patterns of hemp fiber before and after treatment are presented in Figure 4. Table I shows the corresponding fine structures and the degree of crystallization. The results indicated that the cellulose I and II crystal forms of hemp fiber before and after treatment did not change. From the XRD pattern of untreated hemp fiber, the four diffraction peaks at $2\theta = 14.50^{\circ}$, 16.10° , 22.21° , and 33.81° were characteristic of hemp fiber and correspond to (101), $(10\bar{1})$, (002), and (040) reflections, respectively. The crystal size was about 5.872 nm at $2\theta = 22.21^{\circ}$ as determined by the XRD line-broadening technique. For the chitosan/silicone oil-treated sample, the corresponding diffraction peaks 2θ increased, but the interplanar spacings decreased. The crystal size decreased to 5.784 nm at $2\theta = 22.74^{\circ}$. Calculation of the percentages of the crystalline fraction showed that the degree of crystallization increased from 62.9% to 75.8% when hemp fibers were treated with chitosan and dyed with mixed epoxy-modified silicone oil. This indicated

TABLE III
Bending Properties of the Samples

Sample	Bending length (cm)		Bending rigidity (mgf cm)			Bending modulus (kgf cm ⁻²)	
	Warp	Weft	Warp	Weft	Total	Warp	Weft
Untreated hemp fabric	3.59	3.30	842.48	654.34	742.48	281.32	218.50
Hemp fabric treated with chitosan and dyed with mixed epoxy-modified silicone oil	3.04	3.09	511.56	537.22	524.23	65.17	68.44

that the macromolecules and elementary fibrils in amorphous fractions arranged compactly as a result of chemical crosslinkage induced by chitosan and silicone oil.

Tensile properties analyses

To study the effect of chitosan/silicone oil treatment on the tensile properties, treated hemp fabric sample was prepared under shrinkage-free conditions. The results are reported in Table II. It was evident that the breaking load and tensile strain decreased when hemp fabric was treated with chitosan/silicone oil. Citric acid presented in chitosan processing liquor itself acted as the hydrolyzation catalyst, which could catalyze the hydrolyzation of cellulose and chitosan. The nonfibrous materials, i.e., lignin, pectin, hemicellulose, etc., remaining in the sample were probably degraded and dissolved by baking and dyeing. The chemical crosslinkage mostly occurred in the amorphous region or in the surface of crystal region. The interfacial forces between the crystal region and the amorphous region were changed. Under the nonhomogeneous grafting condition, the distribution of branched chains was non-uniform, and the lengths of the chains were also different. Therefore, the movements of the macromolecular chains were restricted, which resulted in the stress concentration phenomenon. So, the breaking

load and the tensile strain decreased for the treated hemp fabric.

Bending properties analyses

Table III shows the changes in bending length, bending rigidity, and bending modulus, which related to the softness of hemp fabric. It was clear that when compared with the untreated hemp sample, the bending rigidity and bending modulus of the chitosan/silicone oil-treated hemp sample decreased distinctly. This was mainly due to the lubricant membrane coated onto the fibers, which resulted in the decrease of the resistance to bending.

Color yield analysis

Table IV shows that the chitosan-treated hemp sample produced the highest *K/S* value when compared with other samples, indicating that chitosan could enhance the dyeing depth of hemp fiber dyed using reactive dyes. To optimize the volume ratio of dyeing liquor to silicone oil, different amounts of silicone oil were added to the dyeing liquors. The relationships between the *K/S* values and the volume ratios are shown in Figure 5. It was evident that the *K/S* value increased with increasing the volume ratio of dyeing liquor to silicone oil at first, and then reached the maximum (4.35) when the volume ratio was 2 : 1. Afterward, the *K/S* value began to

TABLE IV
***K/S* Values and Color Fastnesses of the Samples**

Sample	<i>K/S</i> value	Color fastness to rubbing (grade)		Color fastness to wet scrubbing (grade)
		Dry	Wet	
Untreated hemp fabric	3.01	5	3	1–2
Chitosan-treated hemp fabric	5.29	5	4	2–3
Hemp fabric treated with chitosan and dyed with mixed epoxy-modified silicone oil	4.35	5	4–5	3

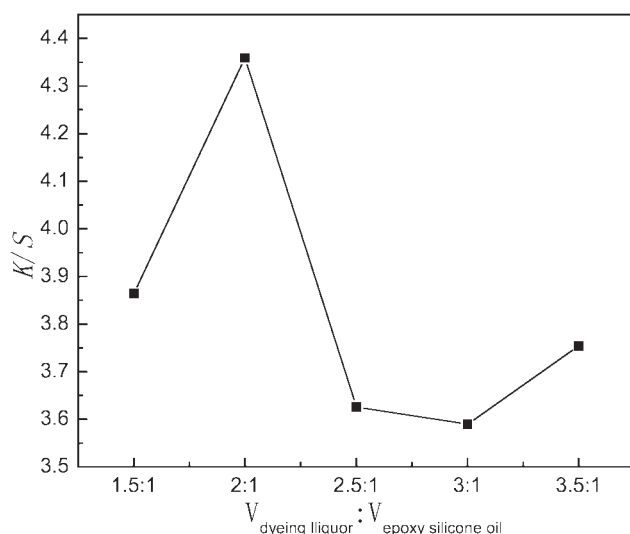


Figure 5 Effect of the volume ratio of dyeing liquor to epoxy-modified silicone oil on the K/S value.

decrease and then increased not more than 3.8 beyond 2 : 1. Epoxy-modified silicone oil reacted with reactive dye remaining inside the fiber and in the bath up to the optimized volume ratio. The higher volume ratio, than the optimized one, acted on the dyestuff in addition to its action with hemp fiber, thereby reducing the K/S value.

Color fastness analysis

The color fastnesses to rubbing and wet scrubbing of untreated, chitosan-treated and chitosan/silicone oil-treated hemp samples ($V_{\text{dyeing liquor}} : V_{\text{epoxy silicone oil}} = 2 : 1$) are given in Table IV. It could be found that the color fastness to dry rubbing was Grade 5 in all cases. The color fastnesses to wet rubbing for three samples were of Grade 3, 4, and 4–5, respectively. The corresponding color fastnesses to wet scrubbing were Grade 1–2, 2–3, and 3. The results indicated that the color fastnesses to rubbing and wet scrubbing could be improved by modifying hemp fibers with chitosan. By adding epoxy-modified silicone oil to the dyeing liquor, the color fastnesses to rubbing and wet scrubbing could be enhanced, although the K/S value decreased when compared with the chitosan-treated hemp fabric.

CONCLUSIONS

In this work, the correlations between the structure and properties of hemp fabric treated with chitosan and dyed with mixed epoxy-modified silicone oil were investigated. The treatment affected the surface configuration, the crystalline structure, and the thermal behavior of hemp fiber. The properties of tensile, bending, dyeing, and color fastness were also changed. SEM showed that hemp fiber was coated with a layer of membrane. TG and DSC showed that the thermal behavior of hemp fiber changed when hemp fabric was treated with chitosan/silicone oil. X-ray diffraction showed that the cellulose I and II did not change. The crystal size decreased but the degree of crystallinity increased. Changes in the crystalline structure and surface configuration led to degraded tensile property. The flexural rigidity decreased owing to the epoxy-modified silicone oil. The K/S value and color fastnesses to rubbing and wet scrubbing were also improved when the optimized volume ratio of dyeing liquor to silicone oil was 2 : 1.

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