INFLUENCES OF CROSSLINKER AND MOLECULAR WEIGHT OF CHITOSAN ON PHYSICO-CHEMICAL PROPERTIES OF ANTIBACTERIAL TREATED COTTON FABRICS

THO LUU THI,* HONG KHANH VU THI,** TUAN ANH NGUYEN*** and THU NGUYEN THI KIM****

Received May 16, 2024

This study investigated the influences of crosslinking agent and molecular weight on the surface and comfort properties of cotton fabrics treated by chitosan. Two types of chitosan, with molecular weight (2.6 kDa and 187 kDa), with deacetylation degree (DD) of 75%, were used, along with two types of crosslinking agents: citric acid (CA) and dimethylol dihydroxyethyleneurea (DMDHEU). These agents were applied to cotton fabrics for antibacterial treatments. The treated cotton fabrics were evaluated using several quality indicators related to physico-chemical properties, including whiteness (according to ISO 105 J02), breathability (according to ASTM D737:2004), moisture (according to ASTM D 2495-87), and thermal and moisture resistance under steady-state conditions (according to ISO 11092). Moreover, surface features of treated samples were observed through SEM images. The results showed that the antibacterial treatment of fabrics with lower molecular weight (Mw) chitosan was more favorable for the finishing processes, although the whiteness of the treated samples was quite low. Additionally, cotton fabrics treated with the CA agent exhibited better hygroscopicity and vapor transmission, but tended to have more pronounced yellow color, compared to those treated with the DMDHEU agent. These physico-chemical findings clarified the bonding mechanism of cellulose–crosslinker–chitosan in antibacterial treated cotton fabrics.

Keywords: molecular weight (Mw), physico-chemical properties, chitosan (CTS), citric acid (CA), dimethylol dihydroxyethyleneurea (DMDHEU)

INTRODUCTION

Chitosan is a deacetylated derivative of chitin that has many unique properties, such as nontoxicity, biocompatibility, and biodegradation.^{1.4} It has garnered significant interest from scientists and industries across various fields, such as biotechnology, pharmaceuticals, medicine, chemistry, wastewater treatment, cosmetics, agriculture, food technology, textiles, pulp and paper industry, winemaking, dentistry, and photography.^{2,5,6} The legal regulations on the safety of chitosan application vary among countries and as a function of its intended uses, such as food, dietary supplement and medical devices.

In recent years, the application of chitosan in the textile industry has drawn much research interest. Despite cotton fabric being highly user-friendly, it lacks resistance to microorganisms because of its natural composition and structural characteristics.⁷ Cotton is made of cellulose, a polysaccharide that provides a food source for microorganisms. It has high moisture absorbency, which promotes the growth of microorganisms.⁸ Its porous structure allows microorganisms to

penetrate and colonize, and it is often exposed to environments where microorganisms are present. Therefore, utilizing chitosan for antibacterial finishing treatments on cotton fabric has been proven as a promising approach, offering textiles that are harmless to the human skin.

The antibacterial activity of chitosan depends on its molecular weight, however, the bactericidal ability of treated fabric has shown conflicting results. Chitosan exhibits high antibacterial properties by several possible mechanisms: (1) by interacting with negatively charged bacterial cell membranes, (2) by increasing osmotic pressure outside bacterial cells, causing dehydration and death, (3) by penetrating cells and binding to DNA, inhibiting RNA and protein synthesis, and (4) by forming a protective film on bacterial surfaces, leading to inhibition or cell death.^{9,10} Shin et al. and Zhang et al. suggested that increasing molecular weight enhances the bactericidal ability of treated fabrics, whereas Khaled et al. found contrary results.¹¹⁻¹³ It was explained that the antibacterial effectiveness of chitosan varies depending on its solubility and viscosity, which are generated by the molecular weight or length of chitosan.^{9,14} It is easier for low molecular weight chitosan to penetrate the cell walls of bacteria, while high molecular weight chitosan is more effective at forming a protective film on the surface of bacterial cells to inhibit nutrient uptake and gas exchange, leading to bacterial cell death.

To enhance the antibacterial action on cotton fabric, many researchers have employed crosslinkers to bind chitosan to cellulose molecules, such as citric acid (CA), 1,2,3,4butanetetracarboxylic acid (BTCA).^{11,15-18} The crosslinkers are supposed to be safe in medical textiles when used with the correct procedure and dosage. Obviously, the crosslinkers play an important role in not only ensuring the antibacterial fastness (especially wash durability), but also enhancing winkle resistance and physical properties for antibacterial chitosan-treated cotton fabrics.

Consequently, antibacterial treatments with chitosan often alter properties such as softness, dyeability, absorbance and moisture retention, tensile strength, crease recovery and surface characteristic.^{4,11,19-22} Our previous works have shown that the antibacterial ability of treated cotton fabric gradually increases with higher molecular weight of chitosan (2.6 kDa, 50 kDa, 187 kDa), as well as in the presence of various crosslinkers.²³ Furthermore, these studies indicated that the molecular weight of chitosan and the types of crosslinkers also affect properties such as viscosity and the pH value of treated fabrics.

In this research, chitosan of different molecular weight and two types of crosslinkers (CA and DMDHEU) were used for antibacterial treatment on cotton woven fabrics to assess their impacts on the mechanical properties of treated samples, such as whiteness, breathability, moisture content, heat and moisture transmission and surface. These properties are crucial for the comfort and aesthetics of finished fabrics used in protective and civilian clothing.

EXPERIMENTAL

Materials

Cotton woven fabrics (twill 1/3, weft/warp count 16/34 (Ne), 175 and 410 threads/cm in the crosswise and lengthwise, areal density of 230 g/m²) were purchased from Nam Dinh Textile Garment JSC. All fabrics were desized, scoured, and bleached before performing the experiments. All specifications of the fabrics are shown in Table 1.

Two types of chitosan (DD 75%), with molecular weights (Mw) of 2.6 kDa and 187 kDa, were purchased from Vietnam Chitosan Co. Ltd. Chitosan derived from shrimp shells was irradiated to obtain modified chitosan with different molecular weights, which were determined through their viscosity.

Two types of commercial crosslinkers, including citric acid (CA) agent and dimethylol dihydroxyethyleneurea (DMDHEU) agent were purchased from Huntsman Ltd Co.

 Table 1

 Basic specifications of experimental fabrics

Fiber content	Construction (weave)	C (warp	ount Ne) weft	Dens (threads/ warp	sity (10 cm) weft	Specific weight (g/m ²)
100% cotton	Twill	34	16	410	175	230

Ĩ	degree	of deacetyl	ation (DD)	of chitosan	U X	,
Irradiation dose (kGy)	η (dL/g)	DD (%)	Mw (kDa)	η (dL/g)	Mw (kDa)	DD (%)
0	1.68	73.57	69.0	3.56	187.0	72.21
25	1.12	77.64	40.0	2.09	93.0	75.32
50	0.73	77.02	23.0	1.43	56.0	75.25
75	0.47	77.86	13.0	1.05	37.0	75.83
100	0.37	78.04	9.0	0.81	26.0	76.41
200	0.28	78.03	6.0	0.56	16.0	77.23
500	0.17	78.89	2.6	0.36	9.0	77.03

 $Table \ 2 \\ Relationship \ between \ radiation \ dose, \ viscosity \ (\eta), \ molecular \ weight \ (Mw) \ and \\ degree \ of \ deacetylation \ (DD) \ of \ chitosan \\ \end{array}$

In our previous work, the effect of crosslinking agent on the antibacterial ability of cotton fabrics treated with chitosan has been clarified.²³ The relationship between radiation dose, viscosity, and molecular weight is reported in Table 2.

Methods

All cotton fabric samples (35 cm x 35 cm) were washed under running water to remove all residual chemicals in non-ionic water (pH = 7.0), then dried and relaxed at room temperature. Next, the samples were impregnated in the given finishing solution and padded with two dips at 80% wet pick-up value. The padded samples were dried at 100 °C for 3 minutes and cured at 160 °C for 2 minutes. The fixed recipe of the aqueous chitosan solution was prepared as follows: chitosan (2.6 kDa or 187 kDa), CA (0.3% owf), owf, NaPO₂H₂.H₂O (7% 1:1 mole ratio), demineralization (Hostapal MRN from Clariant, 0.1% owf); DMDHEU (100 g/L); catalyst NKC (30 g/L) and CH₃COOH (2 g/L).

The whiteness of cotton fabrics was measured according to ISO 105 J02: 97, using a spectrophotometer (Gretag Macbeth Color Eye -2180UV). The breathability of untreated and treated cotton fabrics was determined according to ASTM D737:2004, using an air permeability tester (M021A, Switzerland). The moisture of the experimental samples was evaluated according to ASTM D 2495-87 (1993) using an oven in combination with an electronic balance. The heat resistance and moisture transfer properties of treated samples were examined according to ISO 11092: 2014 using a sweating guarded hotplate thermal controller (USA). The heat resistance and moisture resistance of the samples were determined under standard conditions (20±0.1 °C, 25±3% RH) and specific condition conditions (35±0.1 °C, 40±3% RH), respectively.

FTIR spectra (Nicolet 6700) and SEM images (JSM 7600) were used to evaluate the structural changes between untreated and treated samples. The antibacterial activity of the samples treated with chitosan was evaluated according to the ASTM E2149-01 test method under dynamic contact conditions.

RESULTS AND DISCUSSION

In our previous publication, as shown in Table 3, the results indicated that the crosslinking agent (CA, DMDHEU), molecular weight (CTS1 2.6 kDa, CTS2 187 kDa), exposure time (2 min, 60 min) and washing condition (0 cycles and 20 cycles) caused significant changes in the antibacterial ability of cotton fabrics treated with CTS1/CTS2.²³

Many previous studies have shown excellent antibacterial properties of chitosan. However, these works have not evaluated the effects of compounds (CTS1/CTS2, CA/DMDHEU) on other chemical and physical characteristics of treated cotton fabrics. Therefore, it is essential to examine the physico-chemical properties to ensure the effectiveness and quality of antibacterial fabrics treated with chitosan. To demonstrate the structural difference between CTS1 and CTS2, FTIR spectra were recorded, as shown in Figure 1. It can be confirmed that a moderate irradiation dose (approximately 500 kGy) did not alter the chemical structure of chitosan. However, the broadened regions around the peaks at 3312.8 cm⁻¹ and at 1639.7 cm⁻¹ indicate changes in the number of hydroxyl groups and amine groups, respectively, which are consistent with the difference in chain length between CTS1 and CTS2.

The mechanism of bonding between chitosan, citric acid, and cellulose is determined by the presence of citric acid, a polycarboxylic acid, which reacts with the hydroxyl groups of cellulose through esterification. At elevated temperatures, citric acid undergoes dehydration to form an anhydride intermediate, which can then react with the amino groups of chitosan to create amide bonds, or with additional hydroxyl groups on cellulose to form ester bonds. Meanwhile, dimethylol dihydroxyethyleneurea, which contains methylol and urea groups, undergoes condensation reactions to generate highly reactive intermediates. These intermediates can subsequently form covalent bonds: ether linkages with the hydroxyl groups of cellulose and amide linkages with the amino groups of chitosan.

*** 1 *	Bacterial reduction (%)					
Washing	CA	agent	DMDHEU agent			
cycle	2 min	60 min	2 min	60 min		
0	56.0	100.0	48.0	80.4		
20	6.2	57.6	3.7	35.0		
0	60.0	100.0	56.0	84.1		
20	30.4	63.0	10.3	40.2		
Lausemittan 110 - 100 - 00 100 - 00 80 - 70 - 60 - 50 - 60 - 50 - 60 - 50 - 60 - 50 - 60 - 50 - 60 - 50 - 60 - 50 - 60 - 50 - 5	Treated with CTS1	(2.6 kDa) with CTS2 (187 kDa)	1,2221 1,2221 1,2221 1,2221 1,2221 1,222 1,22 1,222 1,			

 Table 3

 Bacterial reduction of antibacterial treated fabrics with CTS1/CTS2 and CA/DMDHEU after 0 and 20 washing cycles

Figure 1: FTIR spectra of cotton fabrics treated with CTS1/CTS2 and CA/DMDHEU



Figure 2: Whiteness of cotton samples before and after antibacterial treatments with CTS1/CTS2 and CA/DMDHEU

Table 4
Results of wash fastness for cotton fabrics treated with CTS1-CA, CTS2-CA, CTS1-DMDHEU
and CTS2-DMDHEU

Washing souls	K/S value					
wasning cycle	CTS1-CA	CTS2-CA	CTS1-DMDHEU	CTS2-DMDHEU		
0	0.86	0.90	1.44	1.56		
5	0.82	0.84	1.42	1.50		
10	0.81	0.84	1.41	1.46		
15	0.77	0.82	1.31	1.40		
20	0.76	0.80	1.25	1.30		

Whiteness of samples treated with CTS1/CTS2 and CA/DMDHEU

The whiteness of cotton fabrics treated with 0.3% owf CTS1/CTS2 and CA/DMDHEU was evaluated in comparison with that of untreated samples. The results presented in Figure 2 indicate that the whiteness of all the antibacterial cotton samples treated with chitosan was reduced, compared to untreated samples. Among sample group where the same crosslinking agent was used, those treated with lower molecular weight chitosan (CTS1) exhibited lower whiteness than those treated with higher molecular weight chitosan (CTS2). This difference can also attributed to the origin of chitosan rather than to its molecular weight, as CTS1, derived from slicing the gamma-ray band of CTS2, inherently has a darker color. Comparing treatments with the same molecular weight chitosan, the samples treated with DMDHEU presented higher whiteness than those with CA. The whiteness of samples treated with CA was notably lower, a limitation also reported in previous studies.^{15,24} These results suggest that DMDHEU may be a superior crosslinker to CA due to its ability to mitigate yellowing and achieve higher whiteness.

Table 4 indicates the effect of washing on the K/S values of cotton fabrics treated with CTS1-



CA, CTS2-CA, CTS1-DMDHEU, and CTS2-DMDHEU. It can be seen that, at the same number of washing cycles, the K/S values of CTS1-CA and CTS2-CA are significantly lower than those of CTS1-DMDHEU and CTS2-DMDHEU, respectively, indicating that crosslinking with the DMDHEU agent results in darker fabrics compared to the CA agent. Additionally, the K/S values of all the samples slightly decreased with an increasing number of washing cycles, with a more pronounced increase observed in the samples treated with DMDHEU.

Surface features of treated cotton fabrics through SEM images

In Figure 3, SEM images reveal several grooves, spirals and gaps on the surface of untreated cotton fibers. In contrast, the fibers in all chitosan-treated fabric samples are tightly pressed together, without noticeable gaps. This phenomenon is attributed to the high viscosity of the chitosan solution, which causes the fibers to adhere tightly during the padding process under pressure.

Upon closer inspection of the samples treated with the same crosslinking agent, there was no significant difference in fiber surface between the untreated samples and those treated with CTS1.





Figure 3: SEM images of untreated sample (a) and samples treated with CTS1-CA (b), CTS2-CA (c), CTS1-DMDHEU (d), and CTS2-DMDHEU (e)

 Table 5

 Air permeability (P) of untreated and treated cotton fabrics with CTS1/CTS2 and CA/DMDHEU

Sample	P (1/m ² /s)	Average	σ (Standard deviation)	Reduction (%)
	85.20			
Untreated	85.43	85.43 85.42		-
	85.64			
	69.51			
Treated with CTS1-CA	69.58	69.50	0.080	18.64
	69.42			
Treated with CTS1	69.16			
DMDHEI	69.32	69.31	0.140	18.86
DMDHEO	69.44			
	69.63			
Treated with CTS2-CA	69.56	69.63	0.075	18.49
	69.71			
Transferd suith CTS2	69.66			
DMDUEL	69.45	69.61	0.146	18.51
DMDREU	69.73			



Figure 4: Air permeability of cotton samples treated with CTS1/CTS2 and CA/DMDHEU

However, a noticeable difference was observed between untreated samples and those treated with CTS2, despite the absence of visible grooves and spiral lines. These findings are aligned with claims regarding the antibacterial and other properties of treated fabrics.

Effects of molecular weight of chitosan and various crosslinkers on breathability of treated samples

The air permeability of untreated samples and cotton fabric treated with two different types of chitosan (2.6 kDa, 187 kDa) and two different crosslinkers (CA and DMDHEU) was evaluated according to ASTM D737:2004, and the results are shown in Table 5 and Figure 4.

The results indicate a significant reduction in breathability of all treated samples, compared to untreated ones (of approximately 20%). This decrease in breathability may be attributed to the formation of antibacterial chitosan films on the surface of cotton fibers, which restrict air exchange between the sides of the fabric. Interestingly, air permeability among all treated fabric samples did not vary significantly. Therefore, it can be concluded that the molecular weight of chitosan and the types of crosslinkers had minimal impact on the breathability of woven fabrics. In practice, treating cotton fabrics with 0.3% chitosan reduced air permeability by nearly 20%, which could significantly affect the comfort of garments.

Changes in moisture content of samples treated with CTS1/CTS2 and CA/DMDHEU

The moisture contents of the untreated sample and samples treated with CTS1/CTS2 and CA/DMDHEU were determined according to ASTM D2495-87 (1993) and the results were shown in Table 6.

The results indicate that cotton samples treated with 0.3% of CTS1/CTS2 crosslinked with CA/DMDHEU agent exhibited a slight decrease in hygroscopicity, compared to the untreated cotton samples. Interestingly, the moisture content of the samples treated with CTS2 and CA agents increased. It can be concluded that these chemicals did not significantly affect the water absorption of the treated samples.

Table 6
Moisture of untreated and treated cotton fabric with CTS1/CTS2 and CA/DMDHEU

Maaaaa	Linturated	Moisture (%)			
Measurement	sample -	CA		DMDHEU	
order		CTS1	CTS2	CTS1	CTS2
1	8.44	8.47	8.83	7.74	7.83
2	8.33	8.44	8.65	7.58	7.66
3	8.36	8.35	8.71	7.63	7.71
Average	8.38	8.35	8.73	7.65	7.73
CV (%)	0.32	0.35	0.49	0.50	0.53



Figure 5: Thermal resistance (a) and moisture resistance (b) of cotton fabrics treated with CTS1/CTS2 and CA/DMDHEU agents

Thermal resistance (Rt) and moisture resistance (Rm) of antibacterial samples treated with CTS1/CTS2 and CA/DMDHEU

As shown in Figure 5 (a), the thermal resistance (Rt) of antibacterial cotton fabrics treated with CTS1/CTS2 and CA/DMDHEU agents was determined according to ISO 11092:2014. This treatment caused an insignificant increase in heat resistance (i.e., thermal insulation) for all treated fabrics, having little effect on wearing comfort or thermophysiological comfort. Especially, the Rt values of the samples were about 0.43 to 0.45 m²C/W, excluding the samples treated with CTS2-CA. This is consistent with the obtained results of breathability, which was reduced as discussed in the previous section. It should be evidenced further that the amount of coated chitosan generated a thin film on the surface of cotton fibers, leading to a negligible reduction in heat transmission, as well as breathability. The average Rm value for untreated cotton fabrics was measured as 7.39 m²Pa/W, which will be compared to the treated samples. As depicted in Figure 5 (b), the Rm of cotton fabrics increased when treated with CTS1/CTS2 and crosslinked with CA/DMDHEU. Especially, the Rm of CTS1-CA sample saw a negligible increase to 7.40 $m^{2}Pa/W$ (close to 0%), while that of the CTS1-DMDHE sample slightly rose to 7.60 m²Pa/W (approximately 2.84%). However, the Rm of CTS2-CA increased to 7.90 m²Pa/W (about 6.90%), and that of CTS2-DMDHE significantly increased to $8.20 \text{ m}^2\text{Pa/W}$ (about 10.96%). Clearly, the Rm of cotton samples treated with higher molecular weight chitosan (CTS2) was greater than that of cotton samples treated with lower molecular weight chitosan (CTS1). Additionally, the fabrics treated with DMDHEU exhibited higher Rm, compared to those treated with CA. In other words, both moisture transmission and air permeability decreased with increasing molecular weight and were further reduced when crosslinked by the DMDHEU agent. This suggests that the enhanced Rm observed with the DMDHEU-treated samples was due to a reduced moisture absorption capacity.

CONCLUSION

This study examined the impact of chitosan with varying molecular weights and two types of crosslinkers (CA and DMDHEU) on the physicochemical properties of cotton fabrics, specifically focusing on antibacterial effectiveness, whiteness, breathability, moisture content, heat resistance, surface characteristics. The findings and demonstrate that chitosan, regardless of molecular weight, significantly improves the antibacterial properties of cotton fabrics. Higher molecular weight chitosan (CTS2) was more effective in antibacterial treatment and retaining whiteness, compared to lower molecular weight chitosan (CTS1). In addition, DMDHEU proved to be a superior crosslinker over CA, enhancing fabric whiteness and maintaining better wash durability. However, all antibacterial treatments reduced the breathability by approximately 20%. Moisture content and thermal resistance changes were minimal, indicating that the treatments did not significantly impair fabric performance.

ACKNOWLEDGEMENTS: The authors would like to express their thanks for the support from Hanoi University of Industry, Industrial University of Ho Chi Minh City, Hanoi University of Technology and Science, and Ho Chi Minh City University of Technology and Education.

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