



# Application of a fiber-reactive chitosan derivative to cotton fabric as an antimicrobial textile finish

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## Abstract

A fiber-reactive chitosan derivative, *O*-acrylamidomethyl-*N*-[(2-hydroxy-3-trimethylammonium)propyl] chitosan chloride (NMA-HTCC), was applied to cotton fabrics by a cold pad-batch method in the presence of an alkaline catalyst to evaluate its use as a durable antimicrobial textile finish. The antimicrobial activities of the NMA-HTCC treated cotton fabrics were evaluated quantitatively against *Staphylococcus aureus*. The cotton treated with NMA-HTCC at a concentration of 1% on weight of fabric showed 100% of bacterial reduction. The activity was maintained over 99% even after being exposed to 50 consecutive home laundering condition. The effect of an anionic surfactant on the antimicrobial activity of the NMA-HTCC treated fabric was discussed.

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**Keywords:** Antimicrobial textile finish; Chitosan; Cotton; Fiber-reactive chitosan derivative

## 1. Introduction

Textile goods, especially those made from natural fibers, provide an excellent environment for microorganisms to grow, because of their large surface area and ability to retain moisture. A number of chemicals have been employed to impart antimicrobial activity to textile goods. Those chemicals include inorganic salts, organometallics, iodophors (substances that slowly release iodine), phenols and thiophenols, onium salts, antibiotics, heterocyclics with anionic groups, nitro compounds, ureas and related compounds, formaldehyde derivatives, and amines (Vigo, 1983). Many of these chemicals, however, are toxic to humans and do not easily degrade in the environment.

Chitosan, a natural biopolymer, has a combination of many unique properties such as biodegradability, nontoxicity, cationic nature, and antimicrobial activity (Muzzarelli, 1977; Roberts, 1992). The textile industry continues to look for eco-friendly processes that substitute for toxic textile chemicals. In this point of view, chitosan is an excellent candidate for an eco-friendly textile chemical. The various uses of chitosan for textile dyeing and finishing were reviewed in a recent paper (Lim & Hudson, 2003). However,

the major problems of chitosan as an antimicrobial agent are its loss of the antimicrobial activity under alkaline conditions due to its loss of the cationic nature (Sudardshan, Hoover, & Knorr, 1992; Tsai & Su, 1999) and its poor durability on textile fabrics due to its lack of strong bonding with fabrics (Shin & Min, 1996).

We synthesized a novel water-soluble chitosan derivative with a fiber-reactive group, *O*-acrylamidomethyl-*N*-[(2-hydroxy-3-trimethylammonium)propyl] chitosan chloride (referred to here as NMA-HTCC) (Fig. 1) (Lim & Hudson, 2004). The NMA-HTCC has quaternary ammonium salt groups on chitosan backbone, which enhance the antimicrobial activity as well as the water solubility of chitosan over the entire pH range. The acrylamidomethyl group is a fiber-reactive group that can form covalent bonds with cellulose under an alkaline condition. In this paper, we evaluated the optimal application condition of the NMA-HTCC to cotton fabric and the antimicrobial properties of the treated fabrics.

## 2. Experimental

### 2.1. Materials

Chitosan was purchased from Korea Chitosan Co., Ltd. and the NMA-HTCC was prepared using the chitosan as

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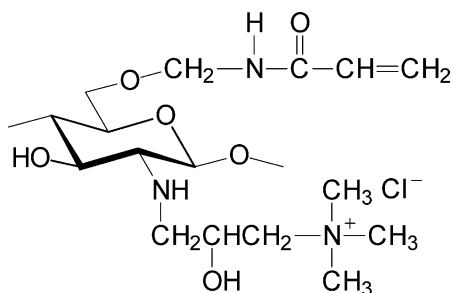


Fig. 1. Structure of NMA-HTCC.

described in our recent paper (Lim & Hudson, 2004). As a test fabric, 100% cotton print cloth (Style # 400M, 107 g/m<sup>2</sup>), which was bleached, desized, and mercerized, was purchased from Testfabrics, Inc., West Pittston, PA. The fabric was further purified by washing in warm water using a home laundering machine (Kenmore). Non-ionic detergent (Kierlon NB-MFB, BASF) was added (1 g/l) for the washing. After washing, the fabric was rinsed with warm water three times and dried using a tumble dryer at 70 °C for 10 min.

C.I. Acid Orange 7 was purchased from Aldrich Chemical Co. with a purity of 87% and further purified by recrystallization using 80% aq ethanol. All other reagents and solvents were purchased from either Fisher Scientific or Aldrich Chemical Co. and were used without further purification. *Staphylococcus aureus* (ATCC 6538) was obtained from American Type Culture Collection (ATCC), Manassas, VA. Bacto™ nutrient broth and Difco™ nutrient agar were purchased from Becton, Dickinson and Company.

## 2.2. Fabric treatment

### 2.2.1. Pad-dry-cure method

Cotton samples were padded with aqueous NMA-HTCC solutions containing an alkaline catalyst at 80% wet pick-up (WPU) using a laboratory padder (Werner Mathis AG). The padded samples were dried at 70 °C for 4 min and then cured in a laboratory oven (Werner Mathis AG) for 5 min at three different temperatures (110, 130, and 150 °C). The treated fabrics were washed with tap water until neutral to pH paper and further washed in warm water using a home laundering machine (normal cycle: washing(6 min)-rinsing-spinning) to remove unfixed NMA-HTCC. The fabric was air-dried at room temperature.

### 2.2.2. Cold pad-batch method

Cotton samples were padded with each aqueous NMA-HTCC solution containing an alkaline catalyst at 100% WPU, placed in plastic sample bags, and tightly sealed to prevent air penetration into the bags. All samples were kept at room temperature for 24 h, washed, and air-dried with the same method used for samples by the pad-dry-cure method.

### 2.2.3. Chitosan treatment

Chitosan, which was a starting material for the NMA-HTCC, was applied to cotton fabric by a pad-dry-cure method. Chitosan was dissolved in 2% acetic acid at a concentration of 1% on weight of bath (owb). Cotton fabric was padded with the chitosan solution at 100% WPU, dried at 100 °C for 3 min, and cured at 150 °C for 3 min. The treated fabric was rinsed thoroughly in warm tap water and air-dried.

## 2.3. Whiteness measurement

The CIE Whiteness Index (WI) was measured on each fabric using a Datacolor Spectraflash® SF 300 spectrophotometer. The spectrophotometer settings were as follows: illuminant D65, large area view, specular component included, reflectance mode, and CIE 1964 Supplemental Standard Observer (10 ° observer). Each fabric was folded twice and the whiteness was measured four times at different portions of the fabric surface. The average value was recorded.

### 2.4. Quantitative analysis of the amount of NMA-HTCC on fabrics

The amount of NMA-HTCC was measured by a stoichiometric dye adsorption method (Roberts, 1997; Shin, Tokino, Ueda, & Suzuki, 1998) using C.I. Acid Orange 7 as a dye indicator. Each NMA-HTCC treated cotton fabric swatch (0.4 g) was dyed in 80 ml aq. dye (C.I. Acid Orange 7) solution (0.5 g/l) at 50 °C for 24 h. The dyed fabric swatches were thoroughly washed in tap water until the wash water did not show any color and dried at 60 °C for 2 h.

Each dyed sample was cut to a small swatch (~0.025 g) and transferred into a vial (23 × 85 mm) containing 10 ml of 25% aq. pyridine. All vials were tightly sealed with screw caps, placed in a shaker incubator, and stirred at 60 °C and 150 rpm for 3 h. The absorbance of the extracted dye solution was measured at  $\lambda_{\text{max}}$  (= 490 nm) using a Varian Cary 3 UV-VIS spectrophotometer. Based on the absorbance, the concentration of the solution was calculated using the Beer-Lambert Law. The calculated concentration was converted to the amount of dye (mmol/kg fiber) adsorbed on the NMA-HTCC treated fabric.

### 2.5. Durability test

To evaluate the durability of NMA-HTCC on fabrics against repeated launderings, treated fabrics were washed according to AATCC Test Method 61(2A)-1996 (American Association of Textile Chemists and Colorists, 2000) using an ATLAS Launder-Ometer. One cycle of laundering by this method is considered equivalent to five home machine launderings at medium or warm setting at the temperature range of 38 ± 3 °C. The fabric size used was 10 × 15 cm

instead of  $5 \times 15$  cm, which was specified in the test method, because at least 1 g of fabric is required for the antimicrobial test. As a detergent, 1993 AATCC Standard Reference Detergent WOB was used. All NMA-HTCC treated fabrics were subject to 10 consecutive launderings either with or without the detergent. At the end of the 10th cycle, all fabrics were rinsed with warm water using a home laundering machine and air-dried.

## 2.6. Antimicrobial test for NMA-HTCC treated fabrics

The antimicrobial properties of the NMA-HTCC treated fabrics were evaluated by ASTM E2149-01 (American Society for Testing and Materials, 2001), which is a quantitative antimicrobial test method performed under dynamic contact conditions. *S. aureus* (ATCC 6538) was used as a test organism. The incubated test culture in a nutrient broth was diluted with a sterilized 0.3 mM phosphate buffer (pH 7.2) to give a final concentration of  $1.5\text{--}3.0 \times 10^5$  colony forming unit (CFU)/ml. This solution was used as a working bacterial dilution. Each fabric (1.00 g) was cut into small pieces ( $1 \times 1$  cm) and transferred to a 250 ml Erlenmeyer flask containing 50 ml of the working bacterial dilution. All flasks were capped loosely, placed on the incubator, and shaken for 1 h at  $37^\circ\text{C}$  and 120 rpm using an incubator shaker (New Brunswick Scientific, NJ, USA). After a series of dilutions of the bacterial solutions using the buffer solution, 1 ml of the dilution was plated in nutrient agar. The inoculated plates were incubated at  $37^\circ\text{C}$  for 2 days and surviving cells were counted. The average values of the duplicates were converted to CFU/ml in the flasks by multiplying by the dilution factor.

The antimicrobial activity was expressed in terms of % reduction of the organism after contact with the test specimen compared to the number of bacterial cells surviving after contact with the control. The percentage

reduction was calculated using the following equation,

$$\% \text{ Reduction} = \frac{B - A}{B} \times 100,$$

where *A* and *B* are the surviving cells (CFU/ml) for the flasks containing test samples (NMA-HTCC treated cotton) and the control (blank cotton), respectively, after 1 h contact time.

## 3. Results and discussion

### 3.1. Application of NMA-HTCC to cotton fabric

The NMA-HTCC can react with cellulose under alkaline conditions as shown in Fig. 2(a). Two application methods, a pad-dry-cure and a cold pad-batch method, were employed to apply the NMA-HTCC to cotton fabric and were compared in terms of whiteness of the treated cotton fabrics, efficiency, and even distribution of the NMA-HTCC on the fabrics.

Sodium bicarbonate ( $\text{NaHCO}_3$ ) was used as an alkaline catalyst for a pad-dry-cure method because it is a latent alkaline catalyst, which is a mild alkali at room temperature but is converted to  $\text{Na}_2\text{CO}_3$  during the curing (Gagliardi, 1951). It is expected that the mild alkalinity of  $\text{NaHCO}_3$  can minimize the hydrolysis (Fig. 2(b)) of the NMA-HTCC in a pad solution before the application.

For the cold pad-batch application, two alkaline catalysts,  $\text{NaOH}$  and  $\text{Na}_2\text{CO}_3$ , were chosen because they are commonly used for a cold pad-batch method. Before the application of the NMA-HTCC, a preliminary experiment was performed, in which two cotton samples were padded with 1% owb NMA-HTCC solutions containing 2% owb of  $\text{NaOH}$  and  $\text{Na}_2\text{CO}_3$ , respectively. The two treated samples were placed in sealed plastic bags at room temperature for 24 h and washed. The fixation of the NMA-HTCC on cotton fabrics was verified by dyeing the fabrics with an anionic

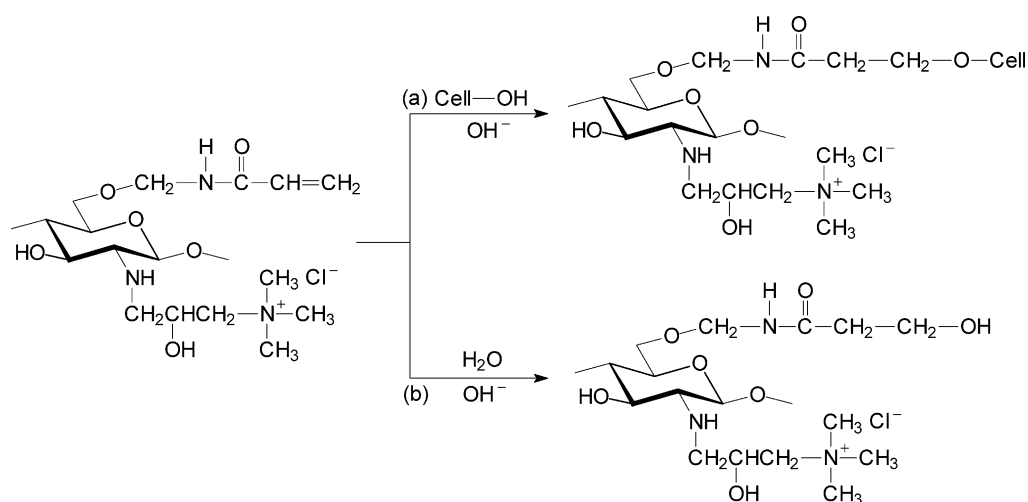


Fig. 2. Reaction of NMA-HTCC with cellulose (a) and hydrolysis of NMA-HTCC (b).

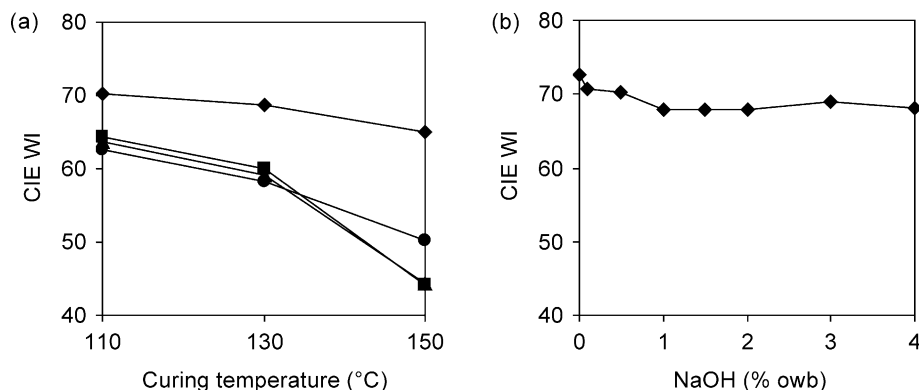


Fig. 3. CIE WI for the fabrics treated with NMA-HTCC (1% owb) by a pad-dry-cure method (a) (amount of alkali ( $\text{NaHCO}_3$ ): ◆ no alkali, ● 1% owb, ▲ 3% owb, and ■ 5% owb) and by a cold pad-batch method (b), (CIE WI of the untreated fabric: 72.86).

dye because the NMA-HTCC contains quaternary ammonium salt groups. Therefore, two samples and an untreated sample were dyed with C.I. Direct Blue 78 (without adding salt) in a beaker for 30 min at boiling. It was observed that the shade of the cotton treated in the presence of NaOH was much deeper than that of cotton treated in the presence of  $\text{Na}_2\text{CO}_3$ , which was slightly deeper than that of the untreated cotton. The preliminary experiment revealed that  $\text{Na}_2\text{CO}_3$  is not effective as an alkaline catalyst. Therefore, NaOH was used as an alkaline catalyst for the cold pad-batch method.

### 3.2. CIE WI of NMA-HTCC treated fabrics

Cellulose is degraded by heat and the degradation involves oxidation and chain scission. The presence of aldehyde groups in oxidized cellulose makes it unstable and causes yellowing. The yellowing is further enhanced by the presence of alkali (Nevell, 1986). The two application methods employed here involve both heat and alkali, or alkali for the pad-dry-cure or cold pad-batch method, respectively. Therefore, the whiteness of the treated fabrics was evaluated.

Fig. 3(a) shows the effect of the curing temperature and amount of alkali on the whiteness of the fabrics treated by a pad-dry-cure method. For the fabric treated without an

alkaline catalyst, the whiteness decreased slightly as temperature increased. However, the fabrics treated with an alkaline catalyst showed significant reduction in whiteness. At lower temperatures (110 and 130 °C), the whiteness decreased similarly regardless of the amount of alkali. Whereas, at a higher temperature (150 °C), the higher amounts (3 and 5% owb) of alkali decreased the whiteness of fabrics compared to that of the fabric treated with a lower amount (1% owb) of alkali. The general trend was that the whiteness decreased by the presence of alkali and the increase of the curing temperature.

As shown in Fig. 3(b), the whiteness of the fabrics treated by a cold pad-batch method decreased very slightly compared to that of fabrics treated by a pad-dry-cure method. The whiteness decreased slightly as the amount of alkali (NaOH) increased up to 1% owb. At the concentration of NaOH over 1% owb, there was no further decrease in whiteness.

### 3.3. Quantitative analysis of the amount of NMA-HTCC on fabrics

The NMA-HTCC treated fabrics were dyed with excess amount of C.I. Acid Orange 7, which is an anionic dye and makes an ionic interaction with the quaternary ammonium salt groups on the NMA-HTCC. The amount of dye

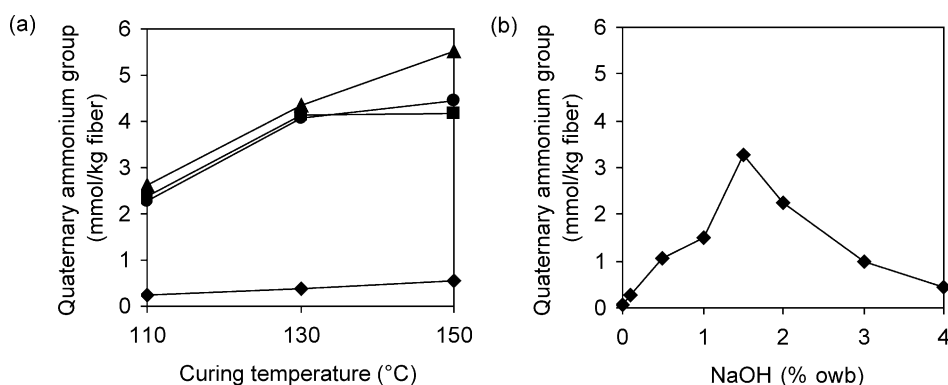


Fig. 4. Quaternary ammonium group content on the fabrics treated with NMA-HTCC (1% owb) by the pad-dry-cure method (a) (amount of alkali ( $\text{NaHCO}_3$ ): ◆ no alkali, ● 1% owb, ▲ 3% owb, and ■ 5% owb) and by the cold pad-batch method (b).

extracted from the dyed fabric was used as a measure of the amount of quaternary ammonium groups on the NMA-HTCC treated fabric. The amount of quaternary ammonium group for each sample was corrected by subtraction of that of a control (0.024 mmol/kg), which is a cotton not treated with the NMA-HTCC.

Fig. 4(a) shows the amount of quaternary ammonium groups on the fabrics treated by the pad-dry-cure method. The general trend was that the amount of quaternary ammonium groups increased as the curing temperature and the amount of alkaline catalyst ( $\text{NaHCO}_3$ ) increased. From the plot, it seems that 3% owb  $\text{NaHCO}_3$  is the most effective because the quaternary ammonium content increased linearly as the temperature increased. It should be noted that there is some fixation of NMA-HTCC on cotton without an alkaline catalyst. The fixation increased linearly as the curing temperature increased. The fixation should be from hydrogen bonds and van der Waals' forces between NMA-HTCC and cellulose due to the presence of hydroxyl groups on cellulose and NMA-HTCC, and the similar chemical structure between cellulose and chitosan backbone, respectively.

Fig. 4(b) shows the amount of quaternary ammonium groups on the fabrics treated by the cold pad-batch method. The amount increased as the amount of alkaline catalyst ( $\text{NaOH}$ ) increased up to 1.5% owb. However, it decreased as the amount of  $\text{NaOH}$  increased over 1.5% owb. The reaction between NMA-HTCC and cotton is a competitive reaction with the alkaline hydrolysis of the NMA-HTCC (Fig. 2). It seems that the increase in the amount of alkali over a certain point (1.5% owb here) increases the hydrolysis of NMA-HTCC rather than the reaction with cotton.

The quaternary ammonium content on the cotton treated with NMA-HTCC without addition of the alkaline catalyst was 0.065 mmol/kg fiber, which is 3.8–8.6 times less than those of fabrics treated by a pad-dry-cure method without alkaline catalyst. This indicates that the heat is an important factor that induces the hydrogen bonding or van der Waals' forces between cotton and MMA-HTCC.

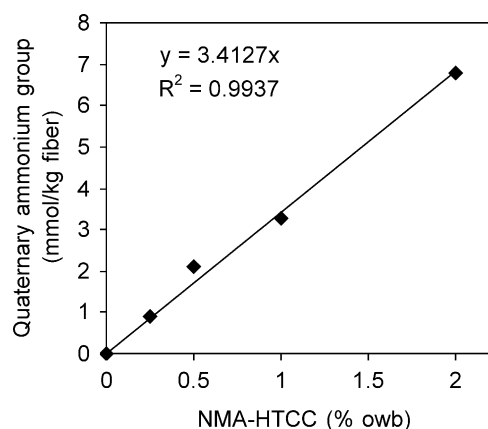


Fig. 5. Quaternary ammonium content on the cotton fabrics treated at different concentrations of the NMA-HTCC. (The amount of an alkaline catalyst ( $\text{NaOH}$ ) was fixed at 1.5% owb).

Table 1

Antimicrobial properties of the treated fabrics against *S. aureus* before launderings

Concentration of NMA-HTCC applied (% owb)	Surviving cells (CFU/ml)	% Reduction
0 (control)	$2.24 \times 10^5$	
0.25	15	99.99
0.5	1	100
1.0	0	100

### 3.4. Optimal application condition

Two application methods of the NMA-HTCC to cotton fabrics, pad-dry-cure and cold pad-batch methods, were compared to choose an optimal application condition. Several factors were considered, such as whiteness of the treated fabrics, amount of fixation of NMA-HTCC on the fabric, and even distribution of NMA-HTCC.

In terms of the fixation, the pad-dry-cure method was more effective than the cold pad-batch method by 1.68 times at their maximal fixation conditions. However, severe yellowing was observed on the fabric treated by the pad-dry-cure method. From the comparison of the dyed fabrics with C.I. Acid Orange 7, all samples treated by the pad-dry-cure method in the presence of an alkaline catalyst showed very uneven distribution of dye on the fabrics and different depth of shade between front and back of the fabrics due to migration. As a result, the cold pad-batch method is desirable for the application of the NMA-HTCC to cotton fabrics and the optimal concentration of the alkaline catalyst obtained was 1.5% owb when 1.0% owb NMA-HTCC was used.

### 3.5. Concentration of NMA-HTCC vs. amount of quaternary ammonium group

Cotton fabrics were treated by the cold pad-batch method to evaluate the effect of the concentration of the NMA-HTCC on the amount of fixation of the NMA-HTCC on cotton fabrics. The effect of concentration of the NMA-HTCC on the quaternary ammonium content was plotted in Fig. 5. The fixation of the NMA-HTCC on the cotton fabrics

Table 2

Antimicrobial properties of the treated fabrics against *S. aureus* after 10 launderings<sup>a</sup> (without AATCC detergent)

Concentration of NMA-HTCC applied (% owb)	Surviving cells (CFU/ml)	% Reduction
0 (control)	$2.32 \times 10^5$	
0.25	268	99.88
0.5	29	99.99
1.0	0	100

<sup>a</sup> 10 launderings by AATCC Test Method 61(2A) are equivalent to 50 home machine launderings.



Table 3

Antimicrobial activity of the 1% NMA-HTCC cotton fabric prepared without an alkaline catalyst against *S. aureus*

Number of launderings <sup>a</sup>	Surviving cells <sup>b</sup> (CFU/ml)	% Reduction
0	$1.09 \times 10^4$	94.76
1	$1.86 \times 10^5$	10.58

<sup>a</sup> One laundering by AATCC TM 61(2A) is equivalent to 5 home machine launderings. AATCC detergent was used for the test.

<sup>b</sup> Surviving cells of control (blank cotton) was  $2.08 \times 10^5$  CFU/ml.

increased linearly as the amount of the NMA-HTCC applied increased.

### 3.6. Antimicrobial properties of NMA-HTCC treated fabrics

The antimicrobial activities of the NMA-HTCC treated cotton fabrics are listed in Table 1. All NMA-HTCC treated fabrics showed very high activities with almost 100% reduction. The activity increased as the amount of NMA-HTCC applied increased and complete 100% reduction was obtained at the concentration of 1.0% owb.

### 3.7. Durability of NMA-HTCC treated fabrics

The antimicrobial properties of the fabrics after launderings are listed in Tables 2 and 3. As shown in Table 2, the antimicrobial activities of the fabrics after 10 launderings without the detergent were almost intact compared to those of the fabrics before launderings (see Table 1). At lower concentrations (0.25 and 0.5% owb) of the NMA-HTCC applied, there were very slight decreases in the activities. This can be attributed to the removal of the NMA-HTCC loosely fixed on the fabric by van der Waals' forces or hydrogen bonding. However, at a higher concentration (1.0% owb), complete reduction of bacteria was maintained.

The NMA-HTCC is a water-soluble chitosan derivative. Therefore, it should be almost completely removed from the fabric after a severe agitation of 10 launderings, if there were no strong bonds with fabrics. The test results show that the NMA-HTCC was fixed on the cotton fabrics covalently.

For the further confirmation, cotton fabric was treated with 1% owb NMA-HTCC solution without an alkaline

Table 4

Antimicrobial test results against *S. aureus* after 10 launderings<sup>a</sup> (with AATCC detergent)

Concentration of NMA- HTCC applied (% owb)	Surviving cells (CFU/ml)	% Reduction
0 (control)	$2.32 \times 10^5$	
0.25	$1.62 \times 10^4$	93.02
0.5	$2.91 \times 10^3$	98.75
1.0	$1.77 \times 10^3$	99.24

<sup>a</sup> 10 launderings by AATCC Test Method 61(2A) are equivalent to 50 home machine launderings.

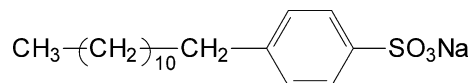


Fig. 6. Structure of sodium dodecylbenzenesulfonate.

catalyst (NaOH) by the cold pad-batch method. The antimicrobial property of the fabric was evaluated before and after one laundering. As shown in Table 3, the fabric lost its antimicrobial activity almost completely after one laundering. This shows that the NMA-HTCC forms covalent bonds with cotton fabric in the presence of the alkaline catalyst (Fig. 2(a)) and the bonds are durable against repeated launderings.

Table 4 shows the antimicrobial activities of the treated fabrics in the presence of the detergent. Further decreases in the activities were observed compared to those tested without the detergent. However, still over 99% of bacterial reduction was maintained at the concentration of 1.0% owb. From the results, it can be assumed that the detergent has some interaction with NMA-HTCC.

### 3.8. Effect of detergent on antimicrobial activity

Detergents are very complex formulations containing several different types of substances. The 1993 AATCC Standard Reference Detergent WOB has a typical composition of commercial laundry product except a few differences (American Association of Textile Chemists and Colorists, 2000) and the main component of the detergent is linear alkylbenzene sulfonate (LAS) (wfk Testgewebe GmbH, 2003), which is an anionic surfactant. The LAS has sulfonate groups and there is a possibility of them to interact with the quaternary ammonium groups of the NMA-HTCC.

To elucidate the effect of the LAS on the antimicrobial activity of the NMA-HTCC treated fabrics, the 1% NMA-HTCC fabrics were treated with sodium dodecylbenzenesulfonate (Fig. 6) under simulated AATCC laundering condition.

Table 5 shows the antimicrobial activities of the 1% NMA-HTCC cotton fabrics treated with LAS at different concentrations. The fabrics treated with LAS showed a slight decrease in antimicrobial activity and showed similar % reduction to the 1% NMA-HTCC fabric (99.24% reduction) subjected to the AATCC TM 61(2A)

Table 5

Antimicrobial test results of 1% NMA-HTCC fabrics contacted with LAS

Amount (g) of LAS	Surviving cells <sup>a</sup> (CFU/mL)	% Reduction
0	0	100
0.041 <sup>b</sup>	$1.73 \times 10^3$	99.12
0.41	$2.37 \times 10^3$	98.79

<sup>a</sup> Surviving cells after contact with the control (blank cotton) was  $1.96 \times 10^5$  CFU/ml.

<sup>b</sup> The amount of 0.041 g is equivalent to that of LAS in the AATCC detergent used for one laundering by AATCC TM 61(2A).

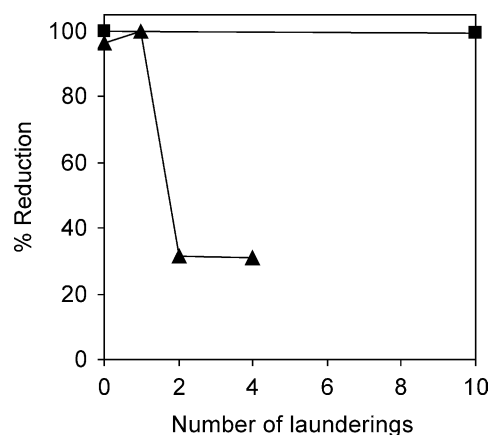


Fig. 7. Antimicrobial activities of chitosan and NMA-HTCC treated fabrics (■ 1% NMA-HTCC treated cotton, ▲ 1% chitosan treated cotton).

laundering test (see Table 4). However, the NMA-HTCC fabric, which was not contacted with LAS, maintained 100% of bacterial reduction. From the result, it was confirmed that LAS affects the antimicrobial activity of the NMA-HTCC treated fabric.

The antimicrobial activity of NMA-HTCC treated fabric comes from its quaternary ammonium groups on the fabric, which interact with the negatively charged bacterial cell surface. It can be thought that the anionic groups of LAS interact ionically with the cationic groups of NMA-HTCC and reduce the chance of NMA-HTCC to interact with bacterial cells.

When it comes to the concentrations of LAS, it seems that the concentration is not an important factor to affect the antimicrobial activity because the difference of the antimicrobial activities of the fabrics treated at low and high concentrations are not so much different. This can be explained in that only a certain amount of LAS remains on the fabrics after rinsing, which removes the excess of LAS.

### 3.9. Antimicrobial activity of chitosan treated cotton fabric

Chitosan, which was the starting material for the NMA-HTCC, was applied to cotton fabric and its antimicrobial activity was compared to that of the NMA-HTCC treated one. The antimicrobial activity of the chitosan treated fabrics against *S. aureus* after different numbers of launderings was plotted in Fig. 7. When the antimicrobial activity of chitosan treated fabric is compared to that of the NMA-HTCC treated one, the chitosan treated fabric lost much of its antimicrobial activity after only two launderings, whereas the NMA-HTCC treated one kept over 99% bacterial reduction even after 10 launderings. The poor durability of chitosan treated fabric is due to the lack of strong binding between chitosan and cotton fabrics. It can be concluded that the chitosan treatment on cotton fabric cannot be used as a durable antimicrobial finishing, because the fabric needs at least 80% bacterial reduction against

*S. aureus* to be considered as an antimicrobial textile product (Monticello, 2002).

One interesting observation was that the activity increased after one laundering compared to that before laundering. This can be attributed to the relatively high amount of chitosan on the fabric before laundering, which may reduce the antimicrobial activity as proposed by Sudarshan et al. in which a large amount of protonated chitosan may coat the bacterial cell surface and prevent the leakage of intracellular components (Sudarshan et al., 1992). The similar behavior of the NMA-HTCC against *S. aureus* was previously observed and reported in our recent paper (Lim and Hudson, 2004).

## 4. Conclusions

The NMA-HTCC, a fiber-reactive chitosan derivative, was applied to 100% cotton fabric in the presence of alkaline catalyst by a pad-dry-cure and a cold pad-batch method. Although the pad-dry-cure method produced higher fixation (up to 1.68 time at each maximal condition) of the NMA-HTCC on cotton, it caused severe yellowing of the fabric and uneven distribution of the NMA-HTCC on the cotton fabric. Therefore, the cold pad-batch method was desirable as an application method. For the cold pad-batch application, the optimal amount of an alkaline catalyst (NaOH) was required to get the highest fixation because a high amount of the catalyst over a certain point decreased the fixation by hydrolyzing the NMA-HTCC. The optimal concentration of the catalyst obtained was 1.5% owb when 1% owb NMA-HTCC was used.

The 1% NMA-HTCC cotton showed 100% of bacterial reduction against *S. aureus* and the activity was maintained over 99% even after exposure of 50 home laundering condition. The slight decrease in the activity after launderings resulted from the interaction of the NMA-HTCC with anionic surfactant in the detergent rather than the removal of the NMA-HTCC from cotton. In contrast, 1% chitosan treated cotton showed about 30% bacterial reduction after 10 home laundering condition due to lack of a strong bonding between chitosan and cotton. From the result, it was concluded that the NMA-HTCC is a very efficient antimicrobial textile finish that overcame the poor laundering durability of chitosan.

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