

Antimicrobial Finishing of Wool Using an Oxidative Pretreatment to Enhance the Exhaustion of Quaternary Ammonium Compounds

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ABSTRACT: Antimicrobial treatments of textiles confer multiple benefits to the textiles and the wearers. We previously demonstrated that a pretreatment of wool with peroxymonosulfate and sulfite uniquely enabled the wool to exhaust large quantities of the cationic biocide poly(hexamethylene biguanide). In this study, we extended our previous work to quaternary ammonium compounds (QACs), using cetylpyridinium chloride (CPC) as an example. QACs are cationic biocides widely used in household and industrial disinfectants. It was found that the pretreatment enabled the otherwise unreceptive wool to exhaust up to 10% of CPC on the weight of wool. The presence of CPC on the wool was confirmed by scanning electron micros-

copy and Fourier transform infrared spectroscopy. The exhaustion proceeded rapidly and efficiently at room temperature and could be slightly enhanced at 40–60°C but was not affected by pH over a broad range. Dyeing with reactive dye had little effect on the exhaustion, but acid dye, premetalized dye, and chrome dye decreased CPC exhaustion by 25–50%. Wool fabrics finished with CPC exhibited a strong antimicrobial ability, deactivating 99.9% of *Escherichia coli* inoculated into the fabrics. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 000: 000–000, 2012

Key words: biofibers; biological applications of polymers; fibers

INTRODUCTION

Antimicrobial treatments of textiles prevent odor formation and the microbe-associated deterioration of the textiles during their use and storage, especially under humid conditions for hygienic clothing and active-wear.^{1–3} Antimicrobial textiles are one of the fastest growing sectors of the textile market, with an increase of 15% per year in developed countries in recent years.⁴

Antimicrobial textiles commonly use biocides, such as silver nanoparticles (or their salts), quaternary ammonium compounds (QACs), poly(hexamethylene biguanide) (PHMB), triclosan, and chitosan, as active agents. These agents are either incorporated into the fibers during extrusion or attached to the fiber surface during finishing.³ QACs are widely used as disinfectants and have been applied to textiles to confer antimicrobial functionality. These compounds carry a positive charge at the N atom in solution and damage cell membranes to cause bacterial death.⁵ The attachment of QACs to a textile substrate is believed to be predominantly accomplished by ionic interactions between the cationic

QACs and the anionic fiber surface.^{6,7} Therefore, for some synthetic fibers, such as Acrilan (Solutia, Decatur, AL) and Orlon (DuPont, Wilmington, DE), which contain carboxylic or sulfonate groups, QACs can be directly exhausted under near-boiling conditions.^{8–10} For synthetic fibers without such groups (e.g., nylon 6.6), dye molecules may serve as bridges to bind QACs to the fiber surface under alkaline conditions.^{6,7,11} The ionic interaction between the dye and the QAC is strong enough to provide a semidurable antimicrobial finish. One commercial antimicrobial textile product that uses QAC as the active agent is Bioguard, which is produced by AEGIS Environments (Midland, MI). The active substance, 3-trimethoxysilylpropyldimethyloctadecyl ammonium chloride, is made into an aqueous solution and applied by padding, spraying, or foam finishing. Upon drying, the nonvolatile silane forms a durable finish on cotton, polyester, and nylon fabrics.¹²

Wool is more difficult to finish with antimicrobial agents because of the presence of a hydrophobic lipid layer on the fiber surface. Nevertheless, wool contains carboxylic groups (e.g., in glutamyl and aspartyl residues) that may provide ionic interaction with QACs. There have been reports that untreated wool could exhaust QACs such as cetylpyridinium chloride (CPC), aminopyridinium salts, and benzyldimethylhexadecylammonium chloride to levels of approximately 5% on

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the weight of wool and exhibit strong antimicrobial activity with durability to 10 launderings.^{13,14}

We have previously shown that an oxidative pretreatment with peroxydisulfate enabled wool to exhaust large quantities of the cationic biocide PHMB.^{15–17} This pretreatment, which was originally developed for wool shrink resistance,^{18,19} partially removes the lipid layer and, more importantly, produces a large amount of Bunte salts on the wool surface.²⁰ The Bunte salts appear to play a critical role in the exhaustion of PHMB through ionic interaction.¹⁵ In this study, we demonstrated that this pretreatment was also effective for QACs and that the treatment imparted strong antimicrobial functionality to the wool.

EXPERIMENTAL

Materials

The woven wool fabrics used in this study were made of untreated and unchlorinated Australian Merino wool (19 μm in diameter on average) and had an area mass of 190 g/m^2 . Potassium peroxydisulfate (PMS; i.e., potassium monopersulfate, trade name Oxone) was purchased from DuPont. CPC ($\text{C}_{21}\text{H}_{38}\text{ClN}$, molecular weight = 339.99), alkyl-dimethylbenzylammonium chloride (i.e., benzalkonium chloride), sodium sulfite, yeast extract, and tryptone were purchased from Sigma-Aldrich.

Methods

PMS/sulfite pretreatment

Wool fabrics (150 \times 150 mm) were pretreated before being used for CPC exhaustion. The conditions used in this study were directly adopted from those of Denning et al.²⁰ Under such conditions, the pretreatment produced significant changes on the wool surface²⁰ and largely maintained the physical properties of the wool.¹⁶ The pretreatment consisted of two steps. The fabrics were first treated with a PMS solution (2 g/L) containing 1 mL/L of the nonionic surfactant Triton X-100 on a shaker at room temperature for 15 min and were then rinsed with water. Subsequently, the fabrics were treated with sodium sulfite (10 g/L , pH adjusted to 8.2–8.5 with 2M sulfuric acid) on a shaker at room temperature for 15 min and were again rinsed with water. The liquor-to-wool ratio for both the PMS and sulfite treatments was 40 : 1 v/w. All fabrics were dried in an oven at 80°C for 45 min and stored at room temperature before further use.

CPC exhaustion

The CPC solutions had a pH of 5.3, and unless stated otherwise, their pH was not adjusted before

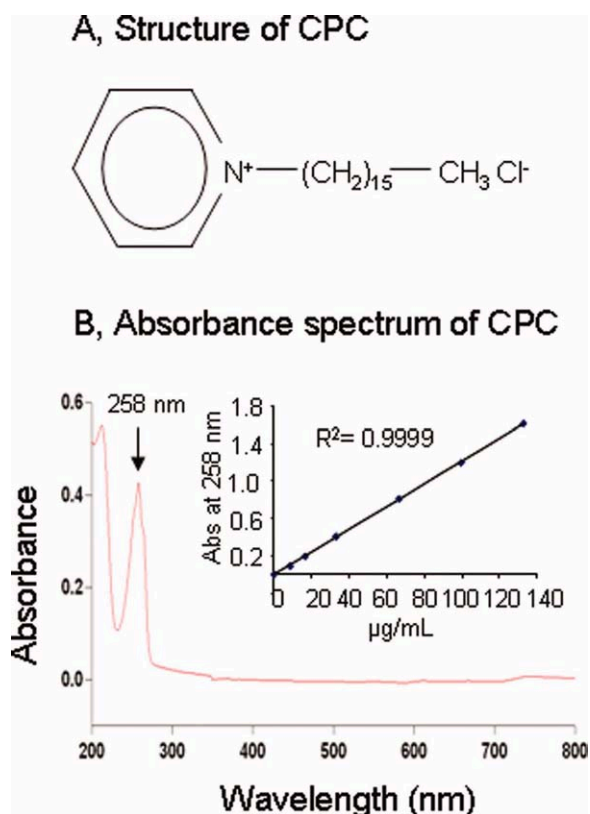


Figure 1 (A) Structure and (B) UV absorbance of CPC. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

exhaustion. Untreated or PMS/sulfite pretreated wool fabrics (0.40 g) were individually immersed with 20-mL solutions containing the stated amounts of CPC with continuous shaking at 20°C (room temperature) or at 40 or 60°C (in a water bath) for 60 min. The liquor-to-wool ratio was 50 : 1 v/w. As CPC is an effective surfactant, no other surfactant was needed to wet out the fabrics in the treatment. At designated times, 50 μL of the exhaustion solution was withdrawn and diluted with 2.95 mL of deionized water. The absorbance of the diluted solutions was read at 258 nm, where CPC had a sharp absorbance peak (see Fig. 1). The exhaustion of CPC was calculated from the difference between the initial absorbance and the absorbance at the designated time. The amounts of CPC present in the bath or exhausted onto the fabrics were expressed as a percentage of fabric weight [i.e., on weight of fabric (owf)]. All exhaustions were performed with at least two duplicate fabrics, and standard deviations were typically around 5% of the mean values.

To examine the effect of pH on CPC exhaustion, the CPC solutions were adjusted to pH values of 4.2 and 5.3 (both with 100 mM citric acid/ Na_2HPO_4 buffer), to pH 8.0 with 100 mM phosphate buffer, or to pH 10.0 with 100 mM sodium bicarbonate buffer.

For Fourier transform infrared (FTIR) analysis, scanning electron microscopy (SEM) and antimicrobial tests, the pretreated fabrics were exhausted with the indicated amounts of CPC at a liquor-to-wool ratio of 50 : 1 v/w at 40°C for 60 min, thoroughly rinsed in deionized water (i.e., vigorous shaking in copious amount of water for 2 h with three water changes), and air-dried before use.

Dyeing procedure

A Pretema (Zurich, Switzerland) Multicolor dyeing machine was employed in the dyeing work. Untreated or PMS/sulfite pretreated wool fabrics (ca. 12 g each) were mounted in the dyeing cylinders. Four types of dyes were used. For the reactive dye (1% Lanazol Red 6G), the dyeing bath was set with 0.5 g/L Albigal FFA, 4% ammonium sulfate, and 1% Albigal B and adjusted to pH 5.5 with 1% acetic acid. The bath was held at 50°C for 10 min before the dye was added and then held at 98°C for 60 min for dyeing. For the premetalized dye (1% Lanaset Red G), the bath was set with 0.5 g/L Albigal FFA, 1.0 g/L sodium acetate, 5% sodium sulfate, and 1% Albigal SET and adjusted to pH 4.5 with 1% acetic acid. The bath was held at 50°C for 10 min before the dye was added and then held at 98°C for 45 min for dyeing. For the acid dye (1.5% Sandolan Red MF-GRL), the bath was set at 50°C in 1 g/L sodium acetate, 1% Lyogen MF liquid, and 5% sodium sulfate and adjusted to pH 4.5 with 10% acetic acid. After the dye was added, the bath was held at 98°C for 45 min. For the chrome dye (1% Eriochrome Red G), the bath was set with 0.5 g/L Albigal FFA and adjusted to pH 5 with 10% acetic acid. The temperature was held at 50°C for 10 min before the dye was added. The bath was held at 98°C for 30 min and cooled to 70–80°C. Formic acid was added to the bath to bring the solution to pH 4, and the temperature was raised back to 98°C and held for another 30 min. The bath was then cooled to 70°C, and potassium dichromate (0.3–0.5%) was added. The bath was finally heated back up to 98°C for a further 45 min before it was cooled and drained. At the end of dyeing, all fabric samples were rinsed thoroughly in warm water, spun, and oven-dried at 50°C. Each dyeing was successful, as indicated by the very good dye exhaustion, color depth, and colorfastness.

FTIR analysis and SEM

A Vertex 7.0 FTIR spectrometer (Bruker optics, Inc., Ettlingen, Germany) with attenuated total reflection was used to examine the surface of the CPC-treated wool fabrics. SEM was performed with a Hitachi S4300 SEM (Tokyo, Japan) instrument at a 1.2-kV accelerating voltage at 2500 and 5000× magnifications on samples that had been sputter-coated with Pt/Pd.

Antimicrobial testing

Antimicrobial tests were performed according to the Assessment of Antibacterial Activity on Textile Materials (AATCC test method 100-1999). *Escherichia coli* (ATCC-8739) was grown overnight in Luria Broth (LB) broth (5 g/L yeast extract, 10 g/L tryptone, and 10 g/L NaCl at pH 7.2) from a frozen stock and diluted to 4×10^7 cells/mL in the nutrient broth. Wool fabrics (4 pieces of 25 × 25 mm stacked together) were inoculated with 250 μL of the diluted inoculum (1×10^7 cells). Care was taken to ensure that the liquid was fully adsorbed into the fabrics. The stacks were then transferred to 250-mL sterile jars, sealed, and incubated at 37°C for 5 h. After the incubation, 100 mL of sterile Milli-Q water was added to each jar, which was then shaken vigorously by hand for 1 min to elute bacteria from the fabrics. The numbers of live bacteria were determined by serial dilution and plating on nutrient agar plates. Time-zero assays were performed by elution of the cells immediately after the inoculation of the untreated fabrics. Antimicrobial activity was expressed as bacterial reduction:

$$\text{Bacterial Reduction (\%)} = 100\% \times (B - A)/B$$

where A is the number of live bacteria after the 5-h incubation from the test samples and B is the number of live bacteria at zero contact time. If $A > B$, there was no reduction (NR).

RESULTS

Monitoring CPC exhaustion by UV absorbance

The structure of CPC used in this study is shown in Figure 1(A). CPC had a pyridinium group in its structure and, therefore, absorbed UV with a single sharp peak at 258 nm [Fig. 1(B)]. The absorbance obeyed the Beer–Lambert Law; that is, it was proportional to the concentration of the substance. The absorbance coefficient was calculated to be 4.1×10^3 M/cm at 258 nm [or 82.8 μg/mL to produce an absorbance of 1; Fig. 1(B) insert]. This property was exploited in this study as a simple and sensitive means of quantifying CPC exhaustion onto fabrics from solutions. All of the absorbance readings of CPC solution on the UV–visible spectrophotometer fell within the linear range of the regression curve in this study.

CPC exhaustion onto the PMS/sulfite pretreated wool

Untreated or PMS/sulfite pretreated wool fabrics were used for CPC exhaustion. Figure 2 shows that the pretreated wool fabrics were able to exhaust CPC at 40°C. The exhaustion proceeded rapidly and

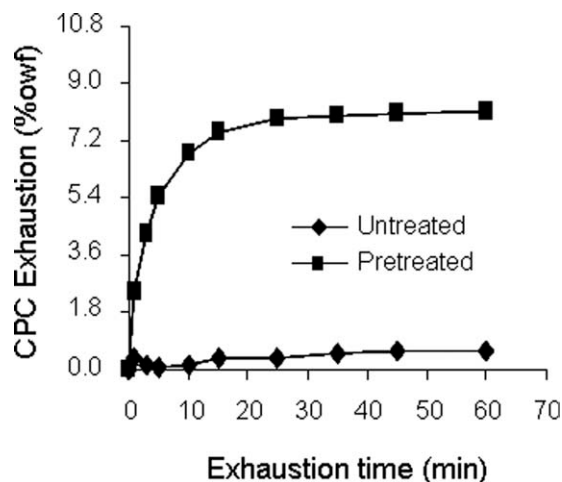


Figure 2 Exhaustion of CPC by untreated and PMS/sulfite pretreated wool fabrics at 40°C. The initial amount of CPC in the exhaustion bath was 9% owf.

efficiently, reaching an almost steady state in the first 15 min and taking up 8.1% owf of the total CPC (9% owf) present in the exhaustion bath in 1 h. On the other hand, untreated wool fabrics exhausted only 0.5% CPC (owf) under the same conditions.

FTIR analysis

FTIR analysis was carried out to confirm the presence of CPC on the PMS/sulfite pretreated wool. Upon pretreatment, two new peaks at 1023 and 1199 cm^{-1} appeared in the wool (Fig. 3). These peaks were attributed to Bunte salts (Wool-SSO_3^-) produced during the pretreatment²⁰ and indicated that the pretreatment was successful. The exhaustion of CPC onto the pretreated wool produced two sharp peaks at 2922 and 2854 cm^{-1} . These two peaks were almost identical to the peaks at 2920 and 2852 cm^{-1} from the CPC solid (due to the alkane C–H bonds in CPC; Fig. 3). These results indicate that CPC was present on the surface of the wool after the exhaustion.

SEM imaging

SEM was used to visualize the presence of CPC on the wool surface after exhaustion. The untreated wool had sharp edges on the cuticle cells. The PMS/sulfite pretreatment produced wrinkles on the surface and softened the scales and their edges. The pretreated wool was then exhausted with 9% CPC (owf) in the bath for 60 min at 40°C. CPC was observed on the entire surface of the wool (Fig. 4). The coverage appeared to be relatively even, without lumps or particles. From the amount of CPC on the wool (e.g., ~ 8% owf) and the diameter of the fiber (e.g., ~ 19 μm), the CPC layer was estimated to be in the vicinity of 0.4 μm in thickness.

Effect of PMS and sulfite in the pretreatment on the CPC uptake

The pretreatment comprised an oxidation (PMS) step followed by a reduction (sulfite) step. To examine which step was critical for CPC exhaustion, the wool fabrics were pretreated with PMS only, sulfite only, or a combination of the two. Figure 5 shows that the untreated and PMS-treated fabrics exhausted a small amount of CPC (i.e., 0.5% owf), whereas the sulfite-treated fabrics exhausted approximately 3.5% owf CPC from a total of 9% CPC in the bath over 1 h at 40°C. On the other hand, the PMS/sulfite-pretreated fabrics exhausted 7.4% under these conditions. It was thus evident that both PMS and sulfite were needed in the pretreatment for efficient CPC exhaustion.

Effect of the CPC concentration on exhaustion

As demonstrated in Figures 2 and 5, the PMS/sulfite-pretreated wool fabrics were capable of taking up about 80–90% of the total 9% owf CPC present in the bath. It was likely that the fabrics were still unsaturated with CPC under those conditions. To examine the maximum amount of CPC that could be exhausted onto the pretreated fabrics, fabrics were incubated with various amounts of CPC in the exhaustion bath at 40°C for 1 h (1–18% owf, liquor/wool ratio = 50 : 1). The exhaustion increased almost linearly with increasing CPC amount up to 9% owf CPC in the bath [Fig. 6(A)] but started to plateau at higher levels. As such, increasing CPC in the exhaustion bath from 9 to 18% owf only increased

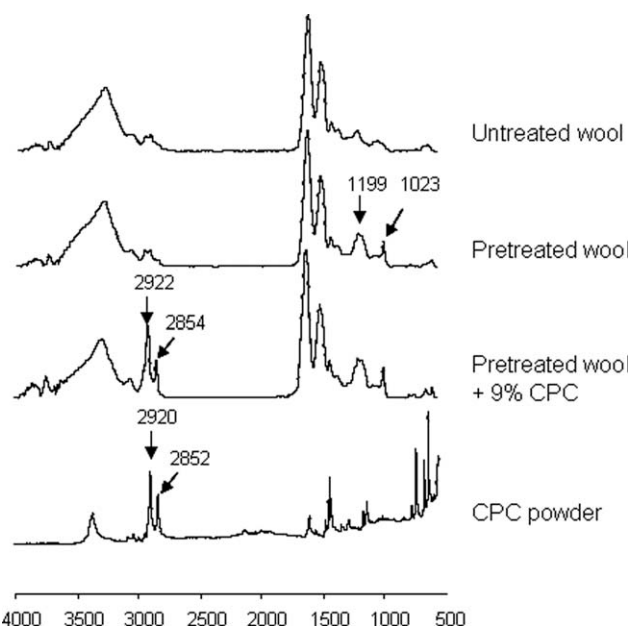


Figure 3 FTIR spectra of the CPC chemical, untreated wool, wool after PMS/sulfite pretreatment, and wool after the pretreatment and CPC exhaustion.

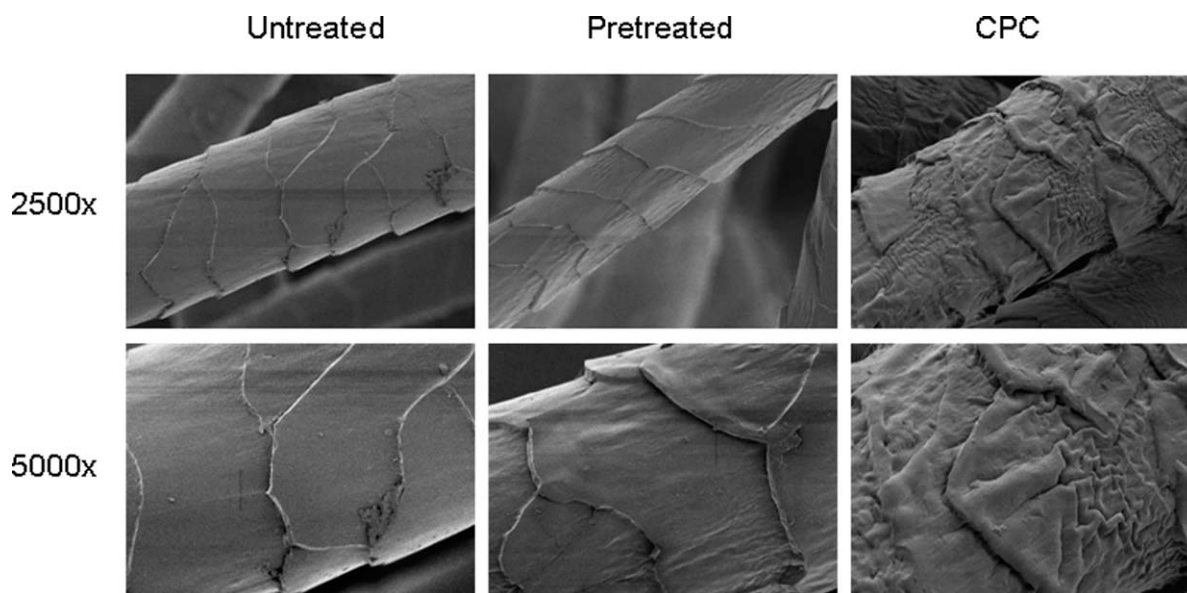


Figure 4 SEM images of the untreated, PMS/sulfite pretreated wool, and wool after pretreatment and further finishing with 9% CPC in the bath for 60 min at 40°C. The magnifications are 2500 and 5000×.

the actual exhaustion from 8.1 to 10.1% owf [Fig. 6(A)]. The fabrics could almost deplete CPC from the bath when less than 9% owf was available but could take up only 56% of the 18% owf CPC in the bath [Fig. 6(B)].

but was able to exhaust 1.8% owf at 60°C. In pretreated wool, the exhaustion was initially fast at all temperatures, reaching 5.5, 6.5, and 7.9% owf at 20, 40, and 60°C, respectively, after 15 min of treatment

Effect of the temperature and pH on the CPC exhaustion

The temperature typically has an effect on the exhaustion of chemicals onto textiles. Figure 7(A) shows that the untreated wool fabrics exhausted approximately 0.5% CPC over 1 h at 20 and 40°C

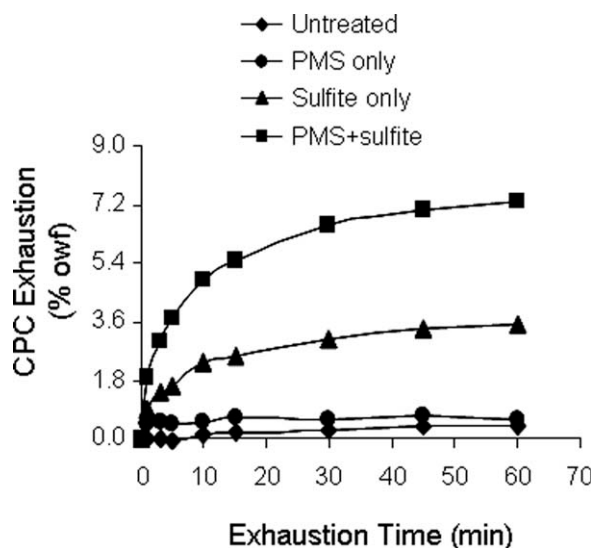


Figure 5 Effect of PMS oxidation, sulfite reduction, and their combination on CPC exhaustion at 40°C. The initial amount of CPC in the exhaustion bath was 9% owf.

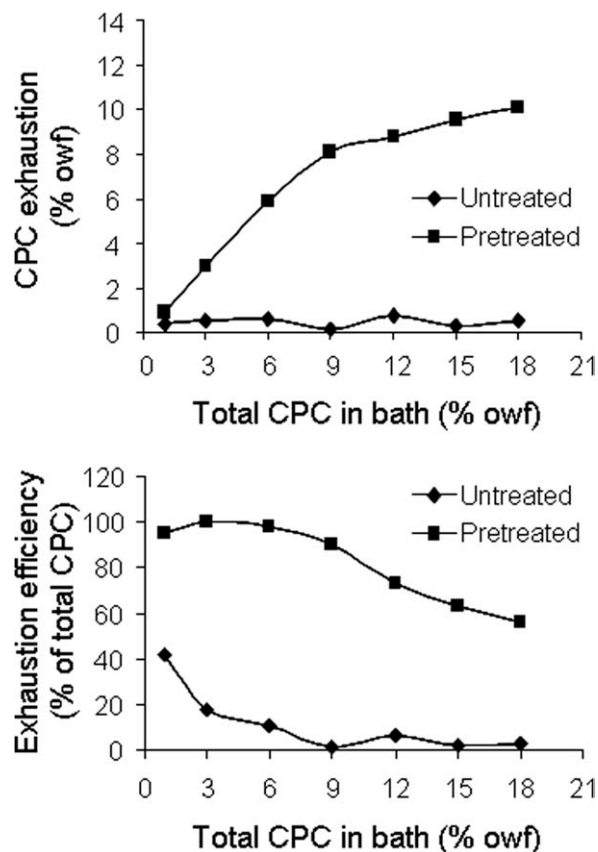


Figure 6 Exhaustion of CPC onto the PMS/sulfite pretreated wool at various amounts of CPC at 40°C for 1 h.

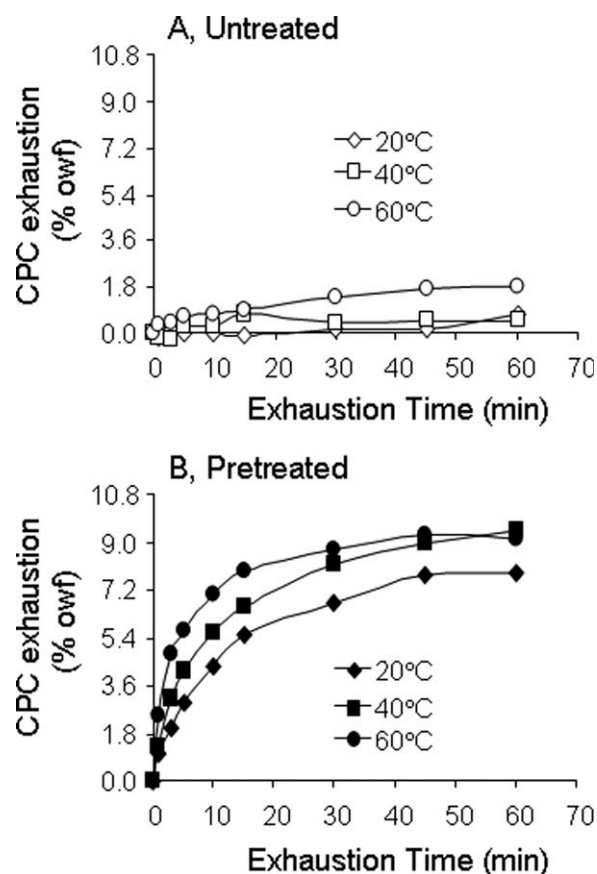


Figure 7 Effect of the temperature on CPC exhaustion on the untreated (open symbols) and PMS/sulfite pretreated (closed symbols) wool fabrics: (∇) 20, (\square) 40, and (\circ) 60°C. The initial amount of CPC in the exhaustion bath was 18% owf.

[Fig. 7(B)]. After reaching a steady state (i.e., after 60 min), the exhaustion increased marginally from 20 to 40°C but stayed steady when the temperature was further increased to 60°C. Such a temperature profile was consistent with those reported for wool with other QACs.^{13,14} It was possible that such a modest temperature effect was partly due to the temperature-facilitated diffusion of CPC into the wool fiber.

The effect of pH on the CPC exhaustion was studied by careful adjustment of the pH of the exhaustion solutions with appropriate buffers. pH values in the range 4.2–10.0 were found to have little effect on the CPC exhaustion (data not shown).

Effect of dyeing on the CPC exhaustion

Investigations were made to examine whether CPC could be applied to wool fabrics that had been pretreated with PMS/sulfite and dyed. Four different types of dyes, that is, reactive, acid, premetalized, and chrome dyes, were used. In the untreated wool fabrics, the exhaustion was small (0.4% owf) and was not affected by any of the dyes [Fig. 8(A)]. Pre-

treated fabrics, on the other hand, took up 9.2% owf CPC without dyeing [Fig. 8(B)]. The reactive dye had very little effect on CPC exhaustion. On the other hand, the acid, premetalized, and chrome dyes decreased the CPC exhaustion to 6.9, 6.0, and 4.6% owf, respectively [Fig. 8(B)]. These values represented reductions of 25, 35, and 50%, respectively, when compared with that of the undyed fabrics.

Antimicrobial activity

After exhaustion with different amounts of CPC (2–18% owf in the bath), the wool fabrics were rinsed extensively before being used to determine their antimicrobial activity against the Gram-negative bacteria *E. coli*. The pretreated fabrics (i.e., with 0% CPC finishing) did not have any antimicrobial activity (Table I). The fabrics finished with 2 and 4% owf CPC in the bath exhausted 1.96 and 3.81% owf CPC, respectively, but did not cause any bacterial reduction. When CPC in the bath was increased to 6%

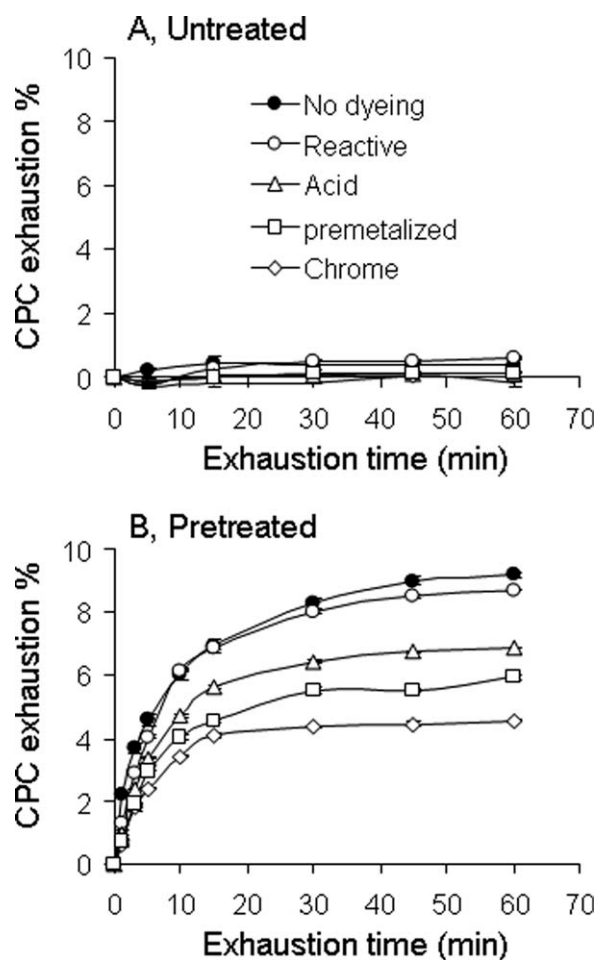


Figure 8 Effect of dyeing on CPC exhaustion: (A) untreated or (B) PMS/sulfite pretreated wool fabrics dyed with reactive dye, acid dye, metal dye, and chrome dye before use for CPC exhaustion at 40°C. The total amount of CPC in the baths was 18% owf.

TABLE I
Quantitative Antimicrobial Assays of Wool Fabrics that
Were Pretreated with PMS/Sulfite and Finished with
Various Amounts of CPC in the Bath at 40°C for 1 h

CPC for exhaustion (% owf)	Exhaustion (% owf)	Antimicrobial test (%)
0	0	NR
2	1.96	NR
4	3.81	NR
6	5.89	96.3
9	8.09	98.8
12	8.80	99.9
18	9.25	99.9

The test bacteria are *E. coli*.

owf, the fabrics were able to deactivate 96.3% of the bacteria. A further increase in the amount of CPC in the bath enabled the fabrics to kill 99.9% of the bacteria inoculated onto the fabrics. These results indicate that the CPC-treated fabrics had strong antimicrobial activity, but there was a minimal amount of CPC on the fabrics for effective bacterial control.

DISCUSSION

In this report, we have demonstrated that the PMS/sulfite pretreatment enabled wool to exhaust large amounts of the quaternary ammonium salt CPC. The pretreated wool fabrics rapidly and efficiently exhausted up to 10% owf CPC under mild exhaustion conditions. The exhausted CPC formed an even coating on the fibers and conferred the fabrics with strong antimicrobial abilities.

The pretreatment consisted of a PMS oxidation step and a sulfite reduction step. For the pretreatment to be most effective, both steps were required. PMS oxidation alone and, to a lesser extent, sulfite reduction alone did not generate a wool surface that was receptive to CPC exhaustion (Fig. 5). Similar differences in the interaction between wool and other cationic substances have been discussed in the literature. For instance, colloidal cationic polymer particles and the cationic polymeric biocide PHMB could only be adsorbed onto PMS/sulfite-treated wool but not onto PMS-treated wool,^{15,21} and the cationic polymer Hercosett did not exhaust well onto PMS/sulfite-treated wool. Such differences may have been due to the chemistry on the wool surface after these treatments. Oxidation with PMS partially breaks disulfide bonds, whereas reduction with sulfite causes nucleophilic fission of disulfide or partially oxidized residues.^{18,19} It has been observed that Bunte salts (wool-SSO₃) were produced by the PMS oxidation/sulfite reduction but not by PMS oxidation alone.²⁰

Bunte salts may have been involved in the exhaustion of CPC on the pretreated wool. They were nega-

tively charged, and their ionic interactions with CPC led to the deposition of some CPC on the wool surface. However, this ionic interaction may not entirely explain the deposition of such a large quantity of CPC on the wool surface. As described previously, the wool was able to exhaust up to 10% owf CPC (or 280 μmol/g of wool), which formed a coating approximately 0.4 μm in thickness. However, the amount of Bunte salt on the PMS/sulfite-treated wool was rather small, 6 μmol/g of wool.²⁰ Once the Bunte salt sites on the wool surface were saturated by CPC, other interaction forces had to come into play. It is likely that the mechanism of CPC exhaustion on the pretreated wool fibers was similar to that of cationic PHMB exhaustion on cotton.²² Blackburn et al.²² found that at low concentrations of PHMB, ionic bonding was the dominant force in the adsorption. As the concentration of PHMB increased, hydrogen bonding became increasingly important, and at high concentrations of PHMB, adsorption was due to monolayer aggregation and multilayer stacking of the PHMB molecules.

It has been reported that reactive and acid dyes enhance the uptake of QACs through the introduction of additional anionic sites on synthetic fibers.^{6-8,11} Therefore, we examined whether CPC exhaustion could be further enhanced by dyeing the fabrics first. This work also served to establish whether the CPC finishing process was compatible with dyeing. Of the four different types of dyes used, none increased CPC exhaustion (Fig. 8). In fact, the acid dye, premetalized dye, and chrome dye decreased CPC exhaustion by 25–50%, whereas the reactive dye had no effect on the uptake of CPC onto the pretreated fabrics. The difference between our work and that in the literature may have been due to the inherent differences between proteinaceous and synthetic fibers. In addition, we note that the exhaustion of QACs in these studies was quite small, that is, 1–2% owf,^{6-8,11} whereas it was as much as 10% owf in our study (Fig. 5–8). This appears to suggest that the role of dye molecules in the exhaustion of CPC onto the pretreated wool was very minor and may have been overshadowed by other interaction mechanisms. The four types of dyes had different effects on CPC exhaustion. The reason for this is not known but may be of interest for further investigations. In any case, this work showed that CPC finishing was compatible with reactive dyes, and for best results, CPC should be used in conjunction with reactive dyes.

The finishing of PMS/sulfite-pretreated wool fabrics with CPC conferred them with strong antimicrobial activity. This was expected, as CPC is a strong biocide with a minimal inhibition concentration as low as 10 μM (3.4 ppm).²³ There was a minimal amount of CPC, that is, about 5% owf, on the wool

that was required for effective antimicrobial activity (Table I). This appeared to be a relatively large amount when we considered the potency of CPC. It is possible that CPC immobilized on a solid surface becomes less readily available for interaction with bacterial cells. This is similar to PHMB, which has a minimal inhibition concentration of 0.05–10 ppm against bacterial species but still needs 2–4% owf on cotton or wool for effective antimicrobial activity.^{15,24}

Finally, untreated wool was reported by Sun and colleagues^{13,14} to exhaust relatively large quantities (i.e., ca. 5% owf) of QACs, such as CPC, benzyldimethylhexadecylammonium chloride, and aminopyridinium salts. The exhaustion appeared to be effective over a broad range of temperature (e.g., 20–90°C) and pH values (e.g., pH 5–9) over 20–80 min. However, we were unable to observe such exhaustions. In our studies, the untreated wool could only exhaust 0.5–1.8% CPC at 20–60°C over 1 h. We repeated the work with wool from three different sources and also used alkylbenzyldimethylammonium chloride (i.e., benzalkonium chloride). In all cases, the untreated wool had a very limited ability to exhaust these compounds (data not shown). The reasons for the discrepancy between our studies and those of Sun and colleagues are unknown.

CONCLUSIONS

We demonstrated that a wool pretreatment consisting of PMS oxidation and sulfite reduction produced a wool surface receptive for the exhaustion of QACs. The exhaustion proceeded rapidly and efficiently under mild conditions (e.g., room temperature and a broad pH range). The process was compatible with reactive dyes, although other types of dyes reduced the QAC uptake by up to half. Wool fabrics finished with QAC exhibited strong antimicrobial activity. The work may form the basis for the production of antimicrobial wool textiles.

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