Improving Easy Care Nonformaldehyde Finishing Performance Using Polycarboxylic Acids via Precationization of Cotton Fabric

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ABSTRACT: Cotton fabric were first subjected to quaternization (cationization) reaction using 3-chloro-2-hydroxypropyl trimethyl ammonium chloride commercially known as Quat®-188. Cationization was carried out under different conditions for optimization of the preparation of cationized cotton with different degrees of cationization, using the pad-batch method. Also, established was the optimal condition for cationization that involves Quat-188/NaOH molar ratio 1/2 at 70°C for 4 h. Besides, a thorough investigation of factors affecting reaction of these cationized cotton with citric acid (CA) or 1,2,3,4-butanetetracarboxylic acid (BTCA) was carried out with a view of improving the ease of care characteristics of nonformaldehyde finishing.

The dependence of fabric performance as measured by strength properties, dry wrinkle recovery angles, whiteness index, and dyeability with reactive dyes was also evaluated. It was postulated that reaction of cationized cotton with either CA or BTCA involves estercrosslinking as well as ionic crosslinking. This, indeed, was largely positively reflected on the fabric performance especially when the properties of both uncationized cotton were compared with those of the cationized cotton. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 100: 2697–2704, 2006

Key words: crosslinking; cotton; cationization; esterification; finishing

INTRODUCTION

Since the identification of formaldehyde as a probable human carcinogen, extensive efforts have been made to find formaldehyde-free crosslinking agents for cotton to replace the traditional *N*-methylol reagents.¹ In 1988, Welch reported that 1,2,3,4-butanetetracarboxylic acid (BTCA) was able to provide effective crosslinking for cotton cellulose.²

In recent years, multifunctional carboxylic acids have been used as nonformaldehyde durable press finishing agents for cotton.^{3–5} Among the various polycarboxylic acids investigated, BTCA is the most effective crosslinking agent for cotton.⁶

Crosslinking of cotton cellulose imparts wrinkle resistance, but reduces mechanical strength of the treated cotton fabric. Severe tensile strength loss and yellowing have been the major obstacles for applications of polycarboxylic acids in durable press finishing processes to a wider range of cellulose fabrics. On the other hand, crosslinking of cotton cellulose with either *N*-methylol-based agents or polycarboxylic acids alters both chemical and physical properties of the cot-

ton. Such chemical treatments eliminate or greatly restrict the ability of the cellulosic fibers to absorb dyes of various classes. Dyeing properties are adversely affected because of the inability of the crosslinked fiber to swell sufficiently in an aqueous environment. Under such circumstances, dyeability of finished cotton is markedly reduced.⁷

Several studies have been made to decrease the mechanical strength loss and yellowing of durable press finished cotton fabric. ⁸⁻¹³ With polycarboxylic acids, all such trials have varying degree of success. In another studies, dye sorption by crosslinked cotton is greatly improved by inclusion of reactive hydroxyal-kyl nitrogenous agents in finishing bath. ¹⁴⁻¹⁹

The present work was designed to tackle problems associated with finishing of cotton fabric with polycarboxylic acids, such as fabric yellowing high tensile strength losses and difficult dyeability. To achieve the goal, cationic groups were introduced in the molecular structure of cotton cellulose prior to polycarboxylic finishing treatments. Hence, cationization of cotton fabric using 3-chloro-2-hydroxypropyl trimethyl ammonium chloride (Quat®-188) in alkaline medium was carried out under a variety of conditions. This was done to discover the optimum cationization conditions of cotton fabric. Following this, crosslinking of the so-obtained cationized cotton fabric (CCF) was effected at elevated temperatures using polycarboxy-

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lic acid along with sodium hypophosphite (SHP) as catalyst. 1,2,3,4-Butanetetracarboxylic acid (BTCA) and citric acid (CA) were independently used as crosslinkers for CCF. Fabric performance was assessed through monitoring wrinkle recovery angle, strength properties, and dyeability before and after the crosslinking treatments.

EXPERIMENTAL

Materials

Cotton fabric

Mill desized, scoured, and bleached 100% cotton fabric (poplin) was supplied by Misr Company for Spinning and Weaving, Mehala El-Kura, Egypt. The fabric has the following specification: Plan weaved, warp 36 yarn/cm, weft 30 yarn/cm, fabric weight, 150 g/m². The fabric was further purified in the laboratory by washing at 100°C for 60 min using a solution containing 2 g/L Na₂CO₃ and 1 g/L Egyptol® (nonionic wetting agent based on ethylene oxide condensate). The fabric was then washed several times with boiling water, then with cold water, and finally dried at ambient conditions.

Chemicals

Sodium hydroxide, sodium carbonate, acetic acid, citric acid (CA), 1,2,3,4-butanetetracarboxylic acid (BTCA), acetone, and sodium hypophosphite (SHP) were of analytical grade. 3-Chloro-2-hydroxypropyl trimethyl ammonium chloride (65 wt % aqueous solution under a commercial name Quat®-188) was kindly supplied by DOW Chemical Company, USA.

Cationization of cotton fabric

Cationization of cotton fabric was carried out as per pad-batch method using Quat-188 as follows. The fabric was padded in solution containing Quat-188 (5–20 g/L) and NaOH (0–10 g/L) then squeezed between two nips and dips to a wet pick up 100%. The fabric was then batched in plastic bag at different temperature (30–90°C) for different time intervals. And then, the cationized fabric was washed several times with water, acidified with 1% acetic acid, and finally washed with water and dried at ambient conditions. Acetone and acetone/water mixture at different ratios were also used to establish the most appropriate cationization media.

Ester crosslinking of cationized cotton by polycarboxylic acid

BTCA and CA were separately used as an ester crosslinker for CCF. The CCF was first impregnated in

a solution containing BTCA or CA along with 65 g/L SHP as catalyst. All concentrations of BTCA and CA presented here were based on weight of bath. No softener or wetting agents were used in the formulation. The fabric was squeezed to a wet pick up of ca. 100%. The fabric was then dried at 85°C for 5 min, and then cured at a specified temperature in a curing oven. Then, the fabric was washed several times with cold water and dried at ambient conditions.

No salt dyeing of cationized ester crosslinked cotton fabric

Dyeing of cationized ester crosslinked cotton fabric without addition of salt was carried out using a reactive dye namely, Suncion® Red HE3B (C.I: Reactive Red 52). Thus, 0.25 g of the dye was dissolved in 1000-mL water at room temperature. The fabric sample was then introduced into this dye solution. A material to liquor ratio 1:100 was employed (in this case, the dye shade will be 2.5% based on weight of fabric). The aqueous solution of the dye containing the sample was put in shaking water bath and the temperature kept at 30°C for 10 min, then 20 g/L Na₂CO₃ was added. The temperature was raised to 80°C and kept at this temperature for 60 min. Slow heating rate was exercised, and shaking was adjusted at 120 rpm. At the end of dyeing, the sample was washed several times with boiling water and 1 g/L Egyptol® (nonionic wetting agent) and finally washed with cold water and dried at ambient conditions. Dyeing with Sumofix® Supra Brilliant Red F3B (C.I: Reactive Red 194) was performed under conditions similar to those described earlier, except that the temperature was raised to 50°C.

Testing and analysis

- Nitrogen content of the cationized samples was determined by the Microkjeldahl method.²⁰
- Carboxyl content (expressed as meq/100 g fabric) was determined according to reported method.²¹
- Tensile strength and elongation at break were determined according to ASTM standard test method.²²
- Wrinkle recovery angles of the treated and untreated sample were determined according to AATCC standard test method.²³
- Whiteness index (WI), expressed as CIE unit, was measured as per AATCC standard test method.²⁴
- *K/S* of the dyed fabric was measured using spectrophotometer type Milton Roy Color Mate.

RESULTS AND DISCUSSION

Reaction of Quat-188 with cotton fabric

The reaction of cotton cellulose with 3-chloro-2-hydroxypropyl trimethyl ammonium chloride (Quat-

	O				
0 (0)	4 (0.28)	6 (0.35)	8 (0.43)	10 (0.40)	12 (0.34)
50 (0.39)	60 (0.41)	70 (0.44)	80 (0.43)	90 (0.31)	
1 (0.25)	2 (0.32)	3 (0.41)	4 (0.44)	5 (0.44)	
5 (0.07)	10 (0.19)	15 (0.32)	20 (0.45)		
0:100 (0.44)	25:75 (0.46)	33.5:66.5 (0.48)	50.50 (0.52)	66.5:33.5 (0.57)	75:25 (0.73)
	50 (0.39) 1 (0.25) 5 (0.07)	50 (0.39) 60 (0.41) 1 (0.25) 2 (0.32) 5 (0.07) 10 (0.19)	50 (0.39) 60 (0.41) 70 (0.44) 1 (0.25) 2 (0.32) 3 (0.41) 5 (0.07) 10 (0.19) 15 (0.32)	50 (0.39) 60 (0.41) 70 (0.44) 80 (0.43) 1 (0.25) 2 (0.32) 3 (0.41) 4 (0.44) 5 (0.07) 10 (0.19) 15 (0.32) 20 (0.45)	50 (0.39) 60 (0.41) 70 (0.44) 80 (0.43) 90 (0.31) 1 (0.25) 2 (0.32) 3 (0.41) 4 (0.44) 5 (0.44) 5 (0.07) 10 (0.19) 15 (0.32) 20 (0.45)

TABLE I
Factors Affecting the Reaction Between Quat-188 and Cotton Fabric

Conditions used: The effect of NaOH conc., temp, and time was carried out in aqueous solution and each factor was studied while keeping other factors constant.

Values in parentheses are N (%) values.

188) requires the addition of alkali in a multistep process, which is time, temperature, and pH-dependent reaction process. During the reaction of Quat-188

with cotton cellulose, several reaction steps occur simultaneously as suggested by eqs. (1)–(3).

$$\begin{array}{c} \text{OH} & \text{CH}_{3} & \text{CH}_{3} \\ \text{Cl-} \text{CH}_{2} \text{-CH-} \text{CH}_{2} \text{-N-} \text{CH}_{3} \text{Cl-} + \text{NaOH} \longrightarrow \text{CH}_{2} \text{-CH-} \text{CH}_{2} \text{-N-} \text{CH}_{3} \text{Cl-} + \text{NaCl} \\ \text{CH}_{3} & \text{CH}_{3} & \text{CH}_{3} & \text{CH}_{3} \\ \text{CH}_{2} \text{-CH-} \text{CH}_{2} \text{-N-} \text{CH}_{3} \text{Cl-} + \text{Ho-Cell} \longrightarrow \text{Cell-} \text{O-CH}_{2} \text{-CH-} \text{CH}_{2} \text{-N-} \text{CH}_{3} \text{Cl} \\ \text{CH}_{3} & \text{Cl-} \text{CH}_{3} & \text{CH}_{3} & \text{Cl} \\ \text{CH}_{3} & \text{Cl-} \text{CH}_{2} \text{-CH}_{2} \text{-CH}_{2} \text{-CH}_{2} \text{-CH}_{3} \text{Cl} \\ \text{CH}_{3} & \text{Cl-} \text{CH}_{3} & \text{Cl-} \text{CH}_{3} & \text{Cl} \\ \text{Cl-} \text{CH}_{2} \text{-CH-} \text{CH}_{2} \text{-N-} \text{CH}_{3} \text{Cl-} + \text{NaOH} \longrightarrow \text{CH}_{2} \text{-CH}_{2} \text{-CH}_{2} \text{-CH}_{2} \text{-CH}_{3} \text{Cl-} + \text{NaCl} \\ \text{CH}_{3} & \text{OH} & \text{CH}_{3} & \text{Cl-} \\ \text{CH}_{3} & \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-} \\ \text{CH}_{3} & \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-} \\ \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-} \\ \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-} \\ \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-} & \text{Cl-}$$

Considering the above reaction scheme, the magnitude of quaternization (cationization) of cotton fabric would rely on NaOH concentration, reaction temperature, reaction time, solvent used, and method of application. The extent of the quaternization reaction was expressed as nitrogen content of the CCF and summarized in Table I. Results of Table I highlight the following points:

- i. Increasing NaOH concentration from 4 to 8% is accompanied by an increase in N% of the CCF from 0.28 to 0.43%. Further increase in NaOH concentration decreases the N% of CCF. Logically, higher NaOH concentrations enhance the hydrolysis of Quat-188 [eq. (3)].
- ii. Raising the batching temperature from 50 to 80°C increases the N% from 0.03 to 0.19%. Further increase in temperature decreases the N% of cationized cotton. Here too, higher temperature seems to act in favor of alkaline hydrolysis [eq. (3)].
- iii. The *N*% increases by prolonging duration of quaternization up to 3 h then levels of where further prolongation of duration has practically no effect on the extent of reaction between Quat-188 and cotton fabric. This could be associated with depletion in Quat-188 concentration and shortage of accessible cellulose hydroxyls as the reaction proceeds. However, changes in the physical or chemical structure of cotton or both during cationization and the onset of this on the ability of cotton to react further with Quat-88 cannot be ruled out.
- iv. Increasing Quat-188 concentration is accompanied by increasing the *N*% of CCF; a point that can be interpreted in terms of greater availability of Quat-188 molecules that are mobile in the proximity of the immobilized cellulose hydroxyls at higher Quat-88 concentrations.
- v. When the reaction between Quat-188 and cotton fabric was conducted in aqueous medium, the obtained CCF exhibits nitrogen content of

0.44%. The nitrogen content of the cationized cotton increases upon using a mixture of acetone and water. This increase in N% depends on acetone/water ratio. For instance increasing the acetone/water ratio from 25/75 to 75/25 increases the N% from 0.46 to 0.73%. It was also observed that no reaction between Quat-188 and cotton cellulose takes place in 100% acetone. This means that presence of water in the reaction medium is essential to dissolve NaOH and Quat-188 and help to establish swelling of the cotton fabric. Furthermore, current results are in accordance with previous studies reported by us, as well as by others. $^{25-32}$

Crosslinking of CCF with CA and BTCA

Bleached cotton fabrics were subjected to cationization reaction using different concentrations of Quat-188 to obtain CCF having different degree of cationization, which was monitored and calculated as N% as detailed in the preceding section. Cotton fabric treated at zero Quat-188 concentration represents the noncationized cotton fabric. Cationized and noncationized cotton fabrics were subjected to finishing treatment using different concentrations of either CA or BTCA. These two acids serve as nonformaldehyde crosslinkers. The effect of concentration of the said polycarboxylic acids on the N% of CCF was examined, and the onset of this on fabric performance was assessed. Assessment of fabric performance was made through monitoring dry wrinkle recovery angle (WRA), WI, tensile strength (TS), elongation at break (E), and dyeability.

Effect of easy care finishing conditions with CA or BTCA on nitrogen content of CCF

Table II shows the nitrogen content of CCFs before and after crosslinking with CA or BTCA. It is clear that: (a) at the same carboxyl content, the *N*% increases as the Quat-188 concentration increases. This is observed when the CCFs were treated independently with CA and BTCA and (b) at the same Quat-188 concentration, increasing the concentration of CA or BTCA increases the carboxyl content of the treated cotton fabric while keeping the nitrogen content practically intact.

On the basis of the aforementioned observations, it may be concluded that finishing treatment of CCF with CA or BTCA in the presence of SHP at 160° C using the pad-dry-cure method has no noticeable effect on the N% of CCF. That is, the CCFs undergo no change in their N% when they are subjected to finishing treatments using CA or BTCA as crosslinkers in the presence of SHP catalyst.

Dry wrinkle recovery angle

Table III shows the combined effect of both polycar-boxylic acid (CA or BTCA) and Quat-188 concentrations on wrinkle recovery angle of crosslinked-CCF. Zero Quat-188 concentration represents uncationized cotton fabric.

Results of Table III reveal that the DWRA of the cotton fabric (not treated with PCA) is increased when it was treated with increasing amounts of Quat-188. This could be attributed to two reasons: the first is the ionic interaction between the cationic group in Quat-

TABLE II

Nitrogen Content of Cationized Cotton Fabric with Different Levels Before and After Crosslinking with Citric Acid

(CA) or 1,2,3,4 Butanetetracarboxylic Acid (BTCA)

Polycarboxylic	Carboxylic content (meq/100 g)						
acid conc. (%)		0	5	10	15	20	
Blank (untreated)	4.25	0	0.41	0.54	0.61	0.73	
CA							
3	19.704	0	0.41	0.53	0.61	0.71	
5	30.903	0	0.42	0.54	0.60	0.72	
8	55.106	0	0.41	0.55	0.62	0.73	
10	78.307	0	0.42	0.54	0.62	0.72	
BTCA							
4	30.2	0	0.41	0.53	0.62	0.75	
6	65.40	0	0.43	0.52	0.63	0.74	
8	95.13	0	0.42	0.51	0.62	0.73	
10	108.98	0	0.41	0.53	0.61	0.72	

^a Polycarboxylic acid concentration was calculated based on weight of bath. Conditions used: Cationization procedures were carried out in aqueous/acetone (1:3) solution at 70°C for 4 h. Finishing with CA or BTCA are detailed in the Experimental Section.

TABLE III
Dry Wrinkle Recovery Angle of Cotton and Cationized Cotton with Different Levels Before and After Crosslinking
with CA or BTCA

		Dry wrinkle recovery angle (W + F) Quat-188 conc. (%) ^a					
Polycarboxyl acid	Carboxyl content						
content conc. (%)	(meq/100 g)	0	5	10	15	20	
Blank (untreated)	425	140	154	160	194	200	
CA							
3	19.704	240	244	250	255	260	
5	30.903	255	260	264	265	268	
8	55.106	255	266	268	270	272	
10	78.307	260	268	266	273	273	
BTCA	30.2	0	0	0	0	0	
4	30.2	250	255	260	265	261	
6	65.40	265	265	268	270	275	
8	95.13	270	275	276	278	280	
10	108.98	274	278	278	280	288	

^a Polycarboxylic acid and Quat-188 concentrations were calculated based on weight of bath. Conditions used: Cationization procedures were carried out in aqueous/acetone (1:3) solution at 70°C for 4 h. Finishing with CA or BTCA are detailed in the Experimental Section.

188 and the residual carboxyl groups of raw cotton and the second is the morphological changes of cotton fiber under the influence of sodium hydroxide used in cationization reaction. It is also seen from Table III that DWRA of uncationized cotton fabric increases by increasing the concentrations of each of the polycarboxylic acids under investigation. Similarly, WRA of a given CCF increases by increasing the concentration of each of these polycarboxylic acid. Also, WRA in-

creases by increasing the degree of cationization of cotton fabric when the latter was treated with the same concentrations of CA or BTCA. The results (Table III) reveal further that at 20% Quat-188 concentration and 100 g/L CA or BTCA, the wrinkle recovery angle values are higher with BTCA than with CA. On the other hand, the WRA of the finished fabric increases as the Quat-188 concentration increases when a constant CA or BTCA concentration was used. This

TABLE IV

Tensile Strength and Elongation at Break of Cotton and Quaternized Cotton Fabric with Different Levels Before and After Crosslinking with CA or BTCA

			Tensile strength (kgf) (elongation at break %)						
Polycarboxyl acid	Carboxyl content		Quat-188 conc. (%) ^a						
	(meq/100 g)	0	5	10	15	20			
Blank (untreated)	4.25	100 (25.5)	100 (25)	100 (25)	98 (24)	95 (24)			
CA									
3	19.704	68 (22.3)	66 (21.5)	65.2 (21)	63.1 (21)	62.5 (20)			
5	30.903	65 (23.9)	63.9 (22)	62.3 (21.5)	62 (22)	61.5 (21)			
8	55.106	62.4 (25)	60 (23)	62.0 (22.5)	63 (23)	62.4 (22)			
10	78.307	60 (21)	59.8 (22.5)	60.2 (22.1)	62.1 (23)	60.5 (20)			
BTCA									
4	30.2	65 (20)	68 (20.3)	61 (21.1)	65 (20.4)	65 (20.1)			
6	65.40	56 (18.5)	65 (19.9)	65 (18.5)	64 (20.3)	64 (19.2)			
8	95.13	55 (18.8)	60 (18.5)	63 (18.7)	64 (19.5)	63 (18.5)			
10	108.98	52 (18.7)	60 (18.5)	60 (18.3)	61 (18.8)	61 (18.8)			

Values in parentheses represent the elongation at break of the samples.

^a Polycarboxylic acid and Quat-188 concentrations were calculated based on weight of bath. Conditions used: Cationization procedures were carried out in aqueous/acetone (1:3) solution at 70°C for 4 h. Finishing with CA or BTCA are detailed in the Experimental Section.

observation could be associated with ionic crosslinking occurring between residual unesterified carboxyl groups of CA or BTCA and the cationic groups of Quat-188 during treatment of the CCF with either CA or BTCA. 32,33

To shed more insight about the reaction involved during the treatments of CCF with polycarboxylic acid, for example CA, the reaction scheme suggested by the eqs. (4)–(6) are shown below:

A. Formation of ionic bonds during the finishing treatments

Cell-O-CH₂ - CH -CH₂ -
$$\stackrel{+}{N}$$
-(CH₃)₃ OOC-CH₂

$$+ OOC - \stackrel{-}{C} - OH + HCI$$

$$+ OOC - CH2$$

B. Esterification of cationized cotton with CA and formation of ionic crosslinking

(Ionic crosslinked cellulose structure)

(5)

I
$$\xrightarrow{SHP}$$
 Cell-O-CH₂ - CH -CH₂ -N-(CH₃)₃ + OC-CH₂
OC-C-OH

Cell-OOC-CH₂

OH
Cell-OOC-CH₂
Cell-O-CH₂ -N-(CH₃)₃ OOC-C-OH
Cell-OOC-CH₂

(II)
(Ester and ionic crosslinked cellulose structure)
(6)

Tensile strength and elongation at break

Table IV shows the tensile strength and elongation at break of CCF before and after ester crosslinking with CA or BTCA. Obviously, for a given CA or BTCA concentration, the tensile strength of crosslinked-CCF exhibits marginal decrement by increasing the concentration of Quat-188, while the elongation at break remains practically unchanged. It is also obvious that, for a given Quat-188 concentration, the tensile strength decreases as the CA or BTCA concentration increases, while the results of elongation at break show no clear-cut trend. On the other hand, for a given Quat-188 concentration and at the same CA or BTCA, the values of tensile strength and elongation at break of the crosslinked-CCF are comparable.

Whiteness index (WI) of CCFs before and after ester and ionic crosslinking with CA or BTCA

Table V shows the dependence of WI of CCF on Quat-188 concentration as well as on CA or BTCA concentration. It is seen that for a given Quat-188 concentration, the WI decreases by increasing CA or BTCA concentration. Similarly for a given CA or BTCA concentration, the WI of cationized-crosslinked cotton fabric decreases as the concentration of Quat-188 increases. On the other hand, at roughly equal concentrations of CA or BTCA, the values of carboxyl content of the crosslinked-CCFs are much higher in case of BTCA as compared with those of CA; meanwhile, the values of WI are comparable.

The observed differences among CA and BTCA could be associated with differences in: (i) ability of the polycarboxylic acid to form the anhydride intermediate, (ii) energy required to expedite reactions suggested by eqs. (4)–(6), (iii) thermal stability of the polycarboxylic acid and its mode of reaction with the cellulose hydroxyls, whether single-ended or crosslinking, and (iv) number and eventual location and length of the crosslinker. The ultimate effect of these parameters would certainly affect the WI.

TABLE V
Whiteness Index (WI) of Cotton and Quarternized Cotton Fabric with Different Levels Before and After Crosslinking
with CA or BTCA

Polycarboxyl acid content conc. (%)		Whiteness index Quat-188 conc. (%) ^a					
	Carboxyl content (meq/100 g)						
		0	5	10	15	20	
Blank (untreated)	4.25	73.7	68.03	68.0	64.0	63.0	
CA							
3	19.704	70.5	70.0	69.2	68.3	68.1	
5	30.903	70.01	68.3	69.0	67.5	67.8	
8	55.106	68.09	65.5	65.3	65.1	65.0	
10	78.307	62.79	60.9	60.3	60.2	60.0	
BTCA							
4	30.2	73.0	69.5	68.5	64.5	64.0	
6	65.40	75.01	60.2	68.2	63.9	62.5	
8	95.13	70.2	68.5	67.1	64.0	62.0	
10	108.98	72.0	69.0	67.0	65.0	62.0	

^a Polycarboxylic acid and Quat-188 concentrations were calculated based on weight of bath. Conditions used: Cationization procedures were carried out in aqueous/acetone (1:3) solution at 70°C for 4 h. Finishing with CA or BTCA are detailed in the Experimental Section.

Dyeability of CCF before and after ester and ionic crosslinking with CA or BTCA

Table VI shows K/S values (measure for color strength) of CCF before and after crosslinking with CA or BTCA. The reactive dye used was Suncion Reactive Red H-E3B (Reactive Red 52). It is seen that, for a given CA or BTCA concentration, increasing Quat-188 concentration enhances K/S values of the dyed samples. It is also seen that, for a given Quat-188

concentration, increasing CA or BTCA concentration decreases K/S values of the dyed samples. On the other hand, treatment of cotton fabric and CCF with CA or BTCA concentration causes significant decrements in K/S values.

Nevertheless, the K/S values for crosslinked-CCFs are 10-fold higher than those of crosslinked uncationized cotton fabric. This is observed with both polycarboxylic acids used, but with the cer-

TABLE VI

K/S Values of Quaternized Cotton Fabric with Different Levels Before and After Crosslinking with CA or BTCA and Dyed Using Suncion Reactive Red H-E3B

D.1. 1. 1.				K/S				
Polycarboxyl acid content	Carboxyl content (meq/100 g)	Quat-188 conc. (%)						
conc. (%)		0	5	10	15	20		
Blank (untreated)	4.25	6.51	17.1	18.3	19.5	21.1		
CA								
3	19.704	3.13	8.1	9.3	8.3	10.5		
5	30.903	1.5	5.8	6.2	7.2	10.0		
8	55.106	1.2	6.4	6.3	6.5	9.5		
10	78.307	0.8	6.71	6.5	6	9.2		
BTCA								
4	30.2	4.21	9.1	9.9	10.1	12.5		
6	65.40	3.52	10.5	10.5	11.3	11.9		
8	95.13	2.34	11.1	11.2	12.1	12.1		
10	108.98	1.41	10.5	10.9	11.3	11.7		

^a Polycarboxylic acid and Quat-188 concentrations were calculated based on weight of bath. Conditions used: Cationization procedures were carried out in aqueous/acetone (1:3) solution at 70°C for 4 h. Dyeing conditions are detailed in the experimental section.

TABLE VII
K/S Values of Quarternized Cotton Fabric with Different Levels Before and After Crosslinking with CA or BTCA and
Dyed Using Sumifix Supra Brill Red S2B

Polycarboxyl acid content conc. (%)						
	Carboxyl content (meq/100 g)		o) ^a			
		0	5	10	15	20
Blank (untreated)	4.25	5.24	18.9	19.3	20.4	25.2
CA						
3	19.704	2.28	10.2	14.6	12.7	13.6
5	30.903	1.93	9.7	13.1	13.4	14.5
8	55.106	1.40	10.1	12.2	13.1	14.3
10	78.307	1.22	11.1	11.3	12.2	13.5
BTCA						
4	30.2	3.98	11.3	12.2	11.7	12.0
6	65.40	2.83	10.2	11.5	10.5	11.1
8	95.13	1.5	9.9	10.1	9.2	10.5
10	108.98	1.3	9.5	10.0	9.5	10.1

^a Polycarboxylic acid and Quat-188 concentrations were calculated based on weight of bath. Conditions used: Cationization procedures were carried out in aqueous/acetone (1:3) solution at 70°C for 4 h. Dyeing conditions are detailed in the experimental section.

tainty that fabrics crosslinked using BTCA display higher K/S values than those crosslinked by CA. This means that there are differences between CA and BTCA as detailed earlier, and such differences are responsible for the higher K/S observed with BTCA than with CA.

Similar results are obtained when cationzied cotton fabrics before and after crosslinking were dyed with Sumofix Supra Brilliant Red F3B (Reactive Red 194) as shown in Table VII. The K/S values depend largely on the magnitudes of both cationization and crosslinking irrespective of the polycarboxylic acid used. Here too, the K/S values for cationized cotton crosslinked using BTCA are higher than those for CA.

Based on the results of Tables VI and VII, it may be concluded that cationiziation of cotton fabrics using Quat-188 prior to crosslinking with CA or BTCA enhances the dyeability of the cotton fabric towards reactive dyes.

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