CELLULOSE CHEMISTRY AND TECHNOLOGY

FUNCTIONALIZATION OF COTTON FABRICS BY ESTERIFICATION CROSS-LINKING WITH 1,2,3,4-BUTANETETRACARBOXYLIC ACID (BTCA)

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In this study, cotton cellulose was cross-linked with 1,2,3,4-butanetetracarboxylic acid (BTCA) via esterification to improve anti-pilling, wrinkle recovery and flame retardant properties of the fabric. Cross-linking was conducted in the presence of sodium hypophosphite (SHP) as an effective catalyst for anhydride formation. Fourier transform infrared (FT-IR) spectroscopy was used to investigate the structure of the cotton fabric cross-linked with BTCA. Water absorption, air permeability, bursting strength, and whiteness change in the fabrics treated with BTCA were also studied. The results indicated that the BTCA treatment could act as a multi-functional finishing agent to improve anti-pilling, crease resistance and flame retardant properties of the cotton fabric. However, it caused a decrease in the bursting strength, air permeability and whiteness of the fabric.

Keywords: BTCA, cross-linking, pilling, wrinkle recovery resistance, flame retardant, water absorbance

INTRODUCTION

The major undesirable properties of cotton fabric are shrinkage and wrinkling. To improve their dimensional stability and crease resistance, chemical finishes are applied on cellulosic textiles. Satisfactory dimensional stability and crease resistance for cotton fabrics can be achieved by cross-linking cellulose fibers, whose hydroxyl groups react with the cross-linkers, which results in the formation of a cross-linked structure.[1,2] Since it has been believed that chain slippage under moist conditions is responsible for wrinkling, cross-linking adjacent cellulose chains could be logically a way of improving crease recovery.[1]

Cellulose cross-linkers can be divided into two types: those that self-polymerize as well as cross-link cellulose, and those that only cross-link cellulose, known as cellulose reactants. Three-dimensional polymers are formed by condensation of small molecules into macromolecules inside the fibers when self-polymerizing cross-linkers are used. Reactant cross-linker agents can form covalent bonds by reacting with the cellulose hydroxyl groups.[1]

N-methylol cross-linker agents have long been used by the textile industry as durable press finishes producing wrinkle-resistant cotton fabrics. However, they release formaldehyde either from treated fabrics or during finishing processes,[1] which has been identified to have a negative impact on human health and the environment, and its industrial use requires significant investment to handle it safely.[3] Therefore, many efforts have been made to reduce the amount of formaldehyde released from finished fabrics.

Polycarboxylic acids have been confirmed as the most promising formaldehyde-free cross-linking agents for cotton cellulose among the various new reagents investigated. Among the various effective polycarboxylic acids, 1,2,3,4-butanetetracarboxylic acid (BTCA) is the most effective cross-linking agent for cotton fabric.[4-5]

Cellulose esterification with a polycarboxylic acid proceeds first to form a cyclic anhydride, and then an ester with the hydroxyl group (-OH group) of the cellulose macromolecule. The esterification of cotton cellulose with BTCA proceeds in two steps: (i) formation of a cyclic anhydride through the dehydration of two adjacent carboxylic acid groups and (ii) the formed acid anhydrides subsequently undergo an

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esterification reaction with the hydroxyl groups of the cellulose macromolecules to form an ester.[6] The cross-linking mechanism of cellulose hydroxyl groups with BTCA is shown in Figure 1.[1-8]

A literature survey on the cross-linking of cotton fabrics with BTCA indicates that BTCA has been generally cross-linked onto the cotton fabric for enhancing wrinkle resistance.[6,9-12] Besides durable press finishes, cross-linking agents have been used in antimicrobial,[12-15] flame retardant[16-19] and water repellent finishes.[20-21] In recent years, they have also been applied along with bio and nano treatments.[22-25] To the authors' knowledge, no investigation has been reported so far on the effect of BTCA treatment on the pilling properties and water absorbance of cotton fabrics. Pilling on fabrics causes unpleasant appearance and can seriously compromise a fabric's acceptability. Pilling arises during the usage of the fabric because of rubbing.

The migration of fibers to the external parts of yarns leads to the emergence of loose fibers, while a rubbing action on loose fibers forms pills on the fabric surface. Pilling is affected by various textile structural variables, including fiber, yarn, fabric, and finishing treatments.[26,27] Cross-linkers or resins that are used during many chemical finishing treatments decrease the pilling tendency of the fabrics.[28] Therefore, in this study we investigated the effect of cross-linking with BTCA on the pilling behavior of fabrics. Especially, we focused on improving anti-pilling, wrinkle-resistant and flame retardant properties of cotton fabrics cross-linked with BTCA. Water absorbance, whiteness, air permeability, bursting strength and chemical structure of treated fabrics have been also evaluated in the present study.

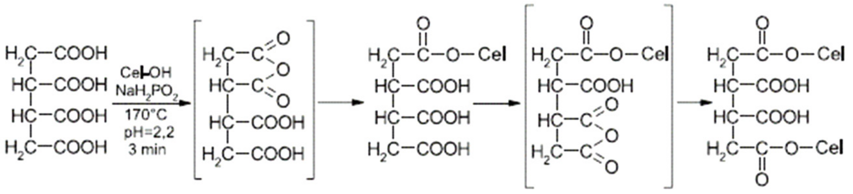

Figure 1: Mechanism for crosslinking hydroxyl groups of cellulose with BTCA[2]

Table 1
Conditions of padding and curing of BTCA on the fabrics

Sample	BTCA concentrations (%)	Padding conditions	Drying and curing conditions	Added on (%)
Sample 1	6 BTCA 4 SHP	2 bar pressure 2 m/min	Drying at 80 °C for 3 min & Curing at 170 °C for 3 min	% 8.93
Sample 2	15 BTCA 10 SHP			% 19.59

EXPERIMENTAL

Materials

Undyed, plain knitted 100% cotton fabric (16 courses per cm and 12 wales per cm) with the weight of 186 g/m^2 was desized, scoured, and bleached by the supplier. The cross-linking agent used was 1,2,3,4-butanetetracarboxylic acid (BTCA), and it was purchased from Sigma Aldrich company with the purity of 99%. The catalyst used was analytical grade sodium hypophosphite (SHP), and it was purchased from Sigma Aldrich company.

Cross-linking of fabrics

BTCA and SHP concentrations were equivalent to the weight of the agent in the 100 mL water solution. The fabric was treated in the solution comprising different concentrations of BTCA and SHP, as listed in Table 1. The fabric was then passed through squeeze rolls at a pressure of 2 bar and speed of 2 m/min by using foulard. The fabric was predried at 80 °C for 3 minutes and cured for 2 minutes at 170 °C. The added on value (%) was calculated dividing the weight of the cured fabrics to the weight of untreated dry fabric.

Chemical analysis of fabric by FT-IR spectroscopy

To investigate the chemical structure of cotton fabrics cross-linked with BTCA, the FT-IR spectra of untreated cotton fabric and treated ones were examined. The spectroscopic analyses of the BTCA, treated and untreated cotton were performed on KBr disks by using an FT-IR instrument (Perkin Elmer Spectrum BX model). The same amount of fabric was cut into small pieces, and mixed in KBr to prepare the KBr disks. The number of scans was 16 and the resolution was 4 cm^{-1} during the FT-IR analysis.

Testing of the fabrics

Tests of the treated and untreated fabrics were performed on conditioned fabric samples under standard atmospheric conditions (20 ±2 °C temperature and 65% ±2 relative humidity).

The pilling resistance test of treated and untreated fabrics was performed on a Nu-Martindale Abrasion Tester according to TS EN ISO 12945-2. According to the pilling test, after a certain rubbing cycle, the fabric was assessed and rated according to a standard fabric photograph. Samples were rated on a scale of 1 to 5 (1 for the worst, 5 for the best).

To determine the durability of the anti-pilling effect of the BTCA on the fabric, pilling tests were repeated after 1, 10 and 20 washing cycles. Therefore, the durability of BTCA cross-linking on the cellulose backbone was also investigated. The samples were washed according to TS EN ISO 105C 06/A15 at 40 °C for 30 minutes, using ECE standard detergent without optical brightener. The washed samples were rinsed by tap water and then they were dried by hanging.

The wrinkle recovery angle (WRA) was tested according to the TS 390 EN 22313/April 1996 in order to evaluate the wrinkle-resistant performance of the specimens. The obtained result for the conditioned WRA was the average of five measurements.

Flame retardant properties of the fabrics were determined according to ASTM D1230-94 ("Standard Test Method for Flammability of Apparel Textiles", reapproved in 2001) by using a 45° flammability tester BV AFC Auto test instrument. Untreated and treated fabric samples mounted in a specimen holder were brushed, dried at 105 °C for 30 min, cooled down in a desiccator containing a drying agent for 90 min, and then moved to the 45° flammability tester with the specimen positioning at an angle of 45°. The specimen was exposed to a standard butane flame for 3 s to cause ignition and combustion, then the burning time and burning characteristics were recorded. The arithmetic mean burning time of 5 specimens and the burning characteristics were used as the basis to determine the flammability classifications. The samples, untreated and treated with BTCA, were also tested according to AATCC Test Method 39-1980 to investigate the water absorbance time of the fabrics. Ten samples were tested for each fabric and the average time was calculated as water absorption time. When the water absorption time was lower than 5 s, according to the standard, the fabric was accepted as hydrophilic.

The static immersion test method was used to evaluate the water absorption amount according to BS 3449, and water absorption was calculated using Equation 1. Eight specimens were tested for each fiber type. The mean amount of water absorption was calculated.

$$\frac{\mathit{Absorbed\ Water\ Mass}\ (g)}{\mathit{Dry\ Fabric\ Mass}\ (g)} = \mathit{Water\ absorption} \qquad (1)$$

Textest FX 3300 Air Permeability Tester was used to measure air permeability of all fabrics according to TS EN ISO 9237 (1995) test method. Ten tests were done on each fabric at the same air pressure (100 Pa) and the average value was calculated.

The bursting strength test of all the fabrics was conducted by using the pneumatic method on a TruBurst Model 611 machine according to ISO 13938-2 1999 under standard atmospheric conditions (20 ±2 °C temperature and 65% ±2 relative humidity). Five pieces of 10 cm x 10 cm size were cut from different parts of each fabric and measurement was conducted using a test area of 7.3 cm^2.

The evaluation of the fabric test results was made by using SPSS 10.0 statistical software for Windows. ANOVA was applied to determine the statistical importance of the variations of the test results. To deduce whether the parameters were significant or not, p values were examined. If the value was greater than 0.05 ($p > 0.05$), the difference was considered as unimportant and thus was ignored.

Change in fabric whiteness

The change in whiteness of the cotton fabrics was evaluated in accordance with ASTM E313-05. For each specimen, four measurements were performed by using an X-Rite 938 spectrophotometer. Color studies were quantified by the CIELAB with a three-axis system, i.e. lightness (L*) from 0% (black) to 100% (white); a* from green (-a) to red (+a); and b* from blue (-b) to yellow (+b).[29] In this study, we focused on L value and b index to determine the yellowness of BTCA treated fabrics. The average color values were calculated.

RESULTS AND DISCUSSION

FT-IR analysis results

The transmittance spectra of the characteristic peaks of the cotton fabrics untreated and treated with 6% BTCA and 15% BTCA are shown in Figure 2. The band at the wavelength of 1728 cm^{-1} corresponds to the ester carbonyl group (which confirms the covalent bond between the cellulose and BTCA).[4,30] The intensity of this band increased in the spectrum of cotton treated with 15% BTCA, compared to the sample treated with 6% BTCA. This band was present in the spectra of both treated cotton samples, however, it could not be observed in the spectrum of the untreated one. The intensity of the H-bonded hydroxyl (O-H) stretching band of cellulose at the wavelength of 3352 cm^{-1} remarked in the untreated cotton spectrum decreased in the spectra of BTCA treated cotton samples, especially for the 15% BTCA treated cotton. This peak gets narrow because of the decreasing of H-bonded -OH groups of cross-linked cellulose. This is a proof of the esterification reaction between the carboxylic acid groups of polycarboxylic acid and hydroxyl groups of cellulose. Based on these results, it was concluded that the cellulose macromolecules were cross-linked by the esterification reaction of the BTCA.

Pilling properties of the fabrics

The pilling test results of the fabrics on the Martindale tester are presented in Table 2. We observed that untreated cotton fabrics pilled more, while no pilling occurred on cotton fabrics treated with BTCA. This tendency can be explained by the cross-linking of the fibers by BTCA esterification. The cross-linked fibers in the fabric were held together by the cross-linkages. Therefore, fiber migration decreased and the formation of entangled fibers could be reduced, which led to a decrease of pilling on the cross-linked fabrics. The stability and durability of the BTCA cross-linking on cotton fabrics were evaluated by pilling tests performed after various numbers of washing cycles. According to the pilling test results, even after 20 washing cycles, there was no pilling on both BTCA treated cotton fabrics. Therefore, it was concluded that BTCA was permanently cross-linked on the cotton fabric and 6% concentration of BTCA was enough to obtain durable pilling resistance.

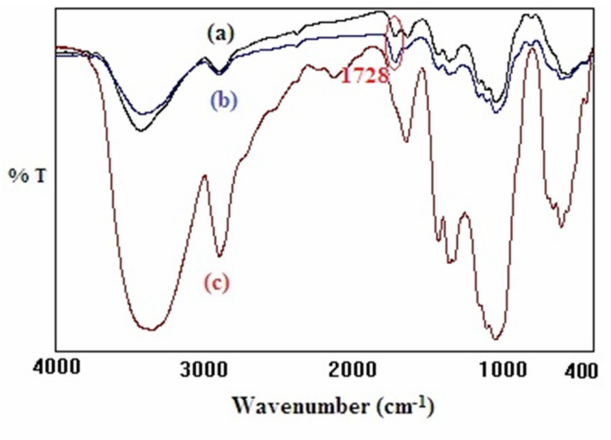

Figure 2: FT-IR spectra of cotton fabrics; a) 15% BTCA treated; b) 6% BTCA treated; c) untreated

Table 2
Pilling test results of the fabrics on Martindale tester

Evaluation								
Before washing			After 1 washing cycle		After 10 washing cycles		After 20 washing cycles	
Untreated	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2
4-5	5	5	5	5	5	5	5	5
4	5	5	5	5	5	5	5	5
3	5	5	5	5	5	5	5	5
2-3	5	5	5	5	5	5	5	5
2-3	5	5	5	5	5	5	5	5
2	5	5	5	5	5	5	5	5

Wrinkle recovery angle (WRA)

For the 6% and 15% BTCA treatments, the average of WRA of the fabrics is illustrated in Figure 3, which evidences that the WRA of the fabrics increased in the following sequence: no BTCA, 6% BTCA and 15% BTCA. The wrinkle recovery angle of the fabrics increased with increasing BTCA content. This finding is consistent with other results reported in the literature, according to which the wrinkle recovery angle of the cotton fabrics increases with cross-linking by BTCA in the presence of SHP catalyst.[6,12,21] In the presence of SHP, the BTCA molecules are able to cross-link the hydroxyl groups of the cellulosic macromolecules effectively by esterification, as proved by the FT-IR results presented in Figure 2.

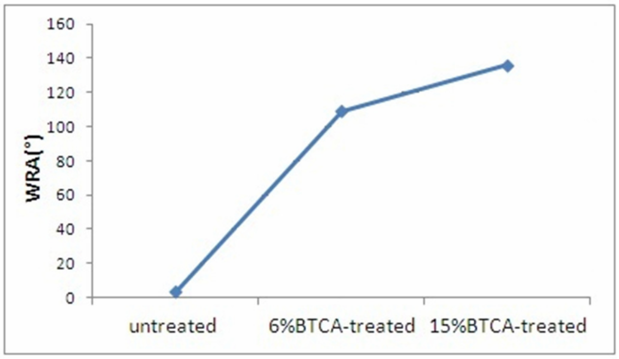

Figure 3: Wrinkle recovery angle of untreated fabric and the one treated with 6% and 15% BTCA

Table 3
Flame retardant test results of the fabrics

Sample	Average burning time (s)	Classification
Untreated	13.85 1.63*	Class 1
Sample 1	31.45 0.55*	Class 1
Sample 2	46.85 1.38*	Class 1

* The values are standard deviations

Flame retardant properties

Polycarboxylic acids are not traditional flame retardants. However, applications of polycarboxylic acids, such as BTCA, citric acid, malic acid and succinic acid, as flame retardants on cotton carpet and cotton/polyester blend fleece have been reported in the literature.[16-19] In this study, the flame retardant properties of the cotton fabrics cross-linked with BTCA were studied. To determine the flame retardant properties of the fabrics, a flame was applied to the fabrics for 3 seconds. As soon as the burning started in the fabric, the burning time was recorded and the fabric was classified according to the burning time. The burning times of the untreated samples and those treated with 6% and 15% BTCA are listed in Table 3. According to the results, all fabrics passed the test and were classified as "Class 1" flammability, which makes these textiles generally acceptable for apparel. However, the burning time and burning characteristics showed differences. Untreated, 6% BTCA treated and 15% BTCA treated cotton fabrics burned in 13.85 s, 31.45 s and 46.85 s, respectively. The burning time of the fabrics increased with increasing BTCA content in the fabric. In addition, the untreated samples completely burned, while the BTCA treated samples took fire, but the fire went out at a given time, as indicated in Table 3. Photos of the fabrics subjected to the flame retardant test are given in Figure 4 to illustrate visually the burning characteristics of the fabrics. According to the findings of the studies on cotton fleece, polycarboxylic acids having esterified the cellulose are effective in reducing the flammability of cotton.[17] It should be also pointed out that polycarboxylic acid becomes an effective flame retardant only when it esterifies the cellulose of cotton. The results obtained in this study are consistent with the findings of the literature.

Water absorption

The liquid sweat absorption ability of clothing is important in order to keep the wearer dry and hence comfortable. Therefore, we investigated the water absorption properties of the treated fabrics. The samples that were untreated and treated with BTCA were tested according to AATCC Test Method 39-1980 to investigate the water absorbance time of the fabrics. Figure 5 shows the drop test results of the fabrics. According to the results, the water absorption time of the 6% BTCA treated fabric was 1.55 s, which was very close to 1.44 s, the time for the untreated fabric. However, 15% BTCA treated fabric absorbed the water in an average of 5.54 s, which was slightly higher than 5 seconds. ANOVA results indicated that the water absorption time of the 15% BTCA treated fabric was higher statistically.

The water absorption amounts of the untreated and treated fabrics, which were calculated by using Equation 1, are presented in Figure 6. The absorbed water amount of the fabric decreased in the following sequence: no BTCA, 6% BTCA and 15% BTCA treatment. This finding is consistent with the results of the drop test. ANOVA results showed that the amount of the water absorbed by the 15% BTCA treated fabric was significantly lower than that of other fabrics, while there was no significant difference between the results of the untreated and 6% BTCA treated fabrics. In the cellulose macromolecules of the cotton, the crystalline regions did not contribute significantly to the process of water adsorption. Water molecules are absorbed by the effect of H-bonds and dipole forces between the water molecules and the –OH group of cellulose molecules, which are outer surfaces on crystals or in the amorphous region. The hydroxyl groups in the amorphous regions of cross-linked cellulose molecules, which can absorb water molecules, form cross-linkages by reacting with cross-linker molecules. This may inhibit the water absorption of the cellulose molecules.[31]

Air permeability

Air permeability is another important fabric property for clothing comfort. It is a measure of how well fabric allows air passage through the fabric and applied finishes should not change fabric air permeability significantly. As known, finishes including cross-linkers reduce the air permeability of the fabric at least partially by filling the interstices between the yarns of the fabrics. To investigate the effect of BTCA cross-linking on air permeability, the fabrics were tested and the test results are presented in Figure 7. Untreated fabrics have higher air permeability, compared to the fabrics treated with BTCA.

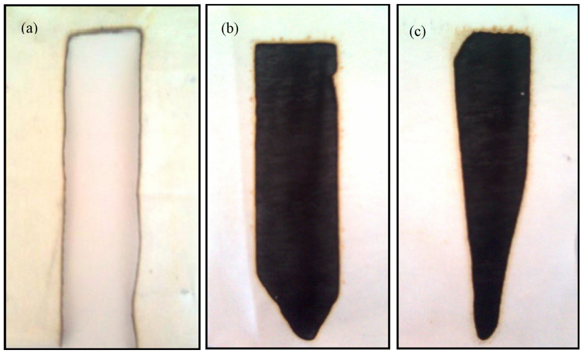

Figure 4: Photographs of the samples after the flammability test; a) untreated; b) 6% BTCA treated sample; c) 15% BTCA treated sample

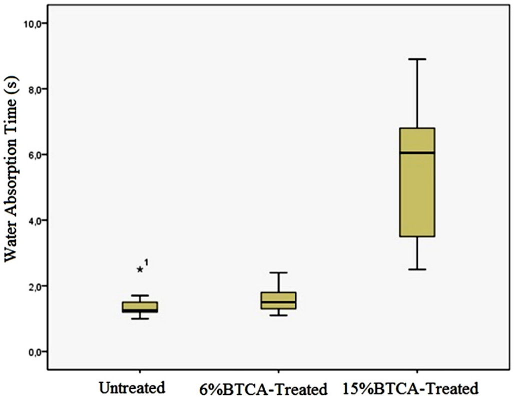

Figure 5: Drop test results

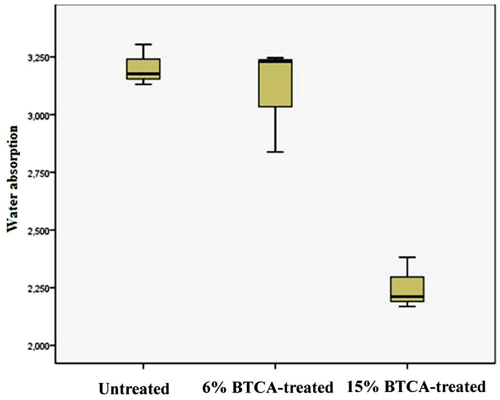

Figure 6: Static immersion test results

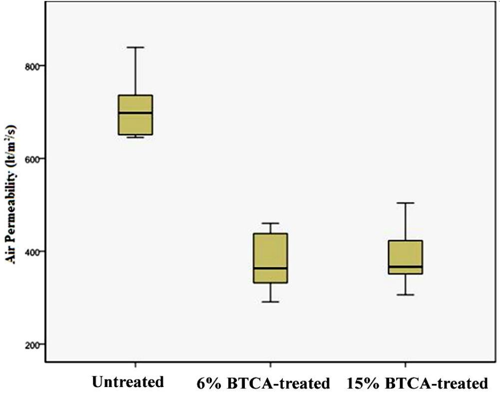

Figure 7: Air permeability test results

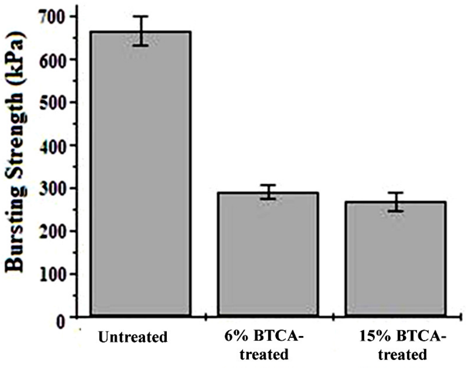

Figure 8: Bursting strength test results

Table 4
Surface color characteristics of the fabrics untreated and treated with BTCA

Sample	ΔL	+b	Whiteness index
Untreated	93.28	-0.58	86.16
6% BTCA treated	93.47	-0.51	81.58
15% BTCA treated	92.00	-0.12	81.16

We performed the ANOVA analysis of the air permeability values of the fabrics in order to demonstrate the importance of each variable. The results of the statistical analysis showed the air permeability of the BTCA treated fabrics to be considerably lower than that of untreated fabric at the 0.05 level. However, there was no significant difference between the air permeability values of the fabrics treated with 6% and 15% of BTCA.

Bursting strength

As noted in the literature, strength loss occurs in cotton fabrics cross-linked by polycarboxylic acid esterification because of acid-catalyzed cellulose depolymerization and cross-linking of cellulose. However, the strength loss of fabrics treated with more reactive polycarboxylic acids for esterifying cellulose, like BTCA, can be lower. Esterification reduces the concentration of proton on the fabric and causes less cellulose depolymerization.[16] Therefore, we investigated the bursting strength of cotton fabric cross-linked by BTCA esterification. Bursting strength test results of the fabrics are presented in Figure 8. It can be concluded that the bursting strength of the fabrics was reduced by cross-linking with BTCA. The bursting strength of the untreated fabric was 666.75 kPa. When the fabric was treated with 6%

and 15% BTCA, the bursting strength decreased significantly to 290.18 and 268.5 kPa, respectively. As the BTCA concentration increased, the burst strength decreased slightly, thus there was no significant difference statistically between the results of the fabrics treated with 6% and 15% BTCA. The results obtained in this study are consistent with the findings reported in the literature.

Color change of the fabric

CIELAB values are summarized in Table 4. Whiteness index, lightness and +b values were used to explain the change of whiteness in the fabric after BTCA treatment. The whiteness index and lightness of untreated fabric were 86.16 and 93.28, respectively. When the fabrics were treated with 6% and 15% BTCA, whiteness index decreased to 81.58 and 81.16, respectively. However, whiteness index decreased slightly as the BTCA concentration increased from 6% to 15% and there was no important change in the lightness values (ΔL) of the fabrics after BTCA treatment. Yellow-blue (b*) color coordinates changed significantly and yellowing of the fabric increased after BTCA application.

CONCLUSION

In this study, cotton fabric was functionalized by cross-linking of polycarboxylic acid (BTCA). The effect of BTCA cross-linking on the pilling, crease resistance and flame retardant properties of the fabrics were evaluated. The changes in chemical structures, water absorption, air permeability, bursting strength and whiteness and yellowness of the cotton fabric after BTCA treatment in the presence of SHP were also examined. It was found that the fabrics treated with BTCA had anti-pilling properties, even after 20 washing cycles there was no pilling on the fabrics. It was concluded that 6% concentration of BTCA was enough to obtain durable pilling resistance. In addition, BTCA treatment could enhance significantly the wrinkle-resistance and flame retardancy of cotton fabrics. Thus, BTCA could act as a multi-functional finishing agent to improve the afore-mentioned fabric properties. However, a reduction of fabric whiteness, bursting strength and air permeability was exhibited by the cotton fabrics onto which BTCA was applied. Water absorption of the fabrics decreased significantly at higher concentration of BTCA (15%), while there was no significant effect of the 6% BTCA treatment on water absorption. In short, the performance of BTCA-SHP formaldhyde-free finishing system could enhance the wrinkle-resistant, anti-pilling and flame retardant properties of the cotton fabrics in a single finishing process.

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