

Crosslinking Cotton with Poly(itaconic acid) and *In Situ* Polymerization of Itaconic Acid: Fabric Mechanical Strength Retention

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Received 4 December 2001; accepted 12 January 2002

ABSTRACT: Polycarboxylic acids have been used as non-formaldehyde durable press finishing agents for cotton fabrics. Previously, we found that itaconic acid (IA) polymerized *in situ* on cotton fabric and also in an aqueous solution in the presence of a $K_2S_2O_8/NaH_2PO_2$ initiation system. Both poly(itaconic acid) (PIA) and the polymer formed by *in situ* polymerization of IA are able to crosslink cotton cellulose, thus imparting wrinkle resistance to cotton. In this research, we compared the performance of the cotton fabric crosslinked by PIA and that crosslinked by *in situ* polymerization of IA. The fabric treated with PIA and that treated with IA had similar wrinkle recovery angles. The cotton fabric treated with IA, however, lost more tensile strength than that treated with PIA due to cellulose degradation. We

determined the magnitude of the fabric tensile strength loss attributed to crosslinking by separating the tensile strength loss due to cellulose degradation from the total tensile strength loss, and found that the tensile strength loss caused by crosslinking for the fabric treated with PIA was significantly higher than that for the fabric treated with IA. This can probably be attributed to more concentrated crosslinkages formed on the near surface of the PIA-treated cotton fabric. PIA had poorer penetration into the amorphous cellulose region in fiber interior due to its much larger molecular size, thus increasing its concentration on the fabric's near surface. The data also suggest that more concentrated crosslinkages on the fabric surface reduced fabric abrasion resistance. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 87: 2023–2030, 2003

INTRODUCTION

Polycarboxylic acids were used as crosslinking agents for cotton cellulose.^{1–3} However, the performance of the crosslinked cotton fabrics was not satisfactory because the catalysts used were not efficient enough to ensure effective crosslinking of cellulose. Since the late 1980s, extensive efforts have been made to use polycarboxylic acids as durable press finishing agents for cotton to replace the formaldehyde-based dimethyloldihydroxyethyleneurea (DMDHEU) due to the increasing concern with the toxicity of formaldehyde.^{4,5} Polycarboxylic acids, such as 1,2,3,4-butanetetracarboxylic acid (BTCA), have shown high levels of effectiveness for crosslinking cotton when sodium hypophosphite (NaH_2PO_2) was used as the catalyst.^{6–9} Multifunctional carboxylic acids can also be used as crosslinking agents for wood pulp cellulose to improve paper wet strength.^{10–14}

Itaconic acid (IA) is difficult to polymerize in aqueous solutions under conditions normally used for vinyl monomers. The first homopolymerization of IA

reported in 1959 was carried in a 0.5M hydrochloric acid solution with $K_2S_2O_8$ as an initiator, and it took several days to achieve 35% yield.¹⁵ IA was also polymerized in methanol at room temperature with 2,2'-azo-bis-isobutyronitrile (AIBN) as a free-radical initiator, and it took 30 days to reach 70% yield.¹⁶ In our previous research, we found that in the presence of a redox initiation system consisting of NaH_2PO_2 and $K_2S_2O_8$, IA can polymerize in an aqueous solution.¹⁷

Choi reported that treating cotton fabric with a mixture of maleic acid (MA) and IA, in the presence of $K_2S_2O_8$ as an initiator, significantly increases the wrinkle resistance of the treated cotton fabric.¹⁸ Previously, we found that MA and IA polymerize *in situ* on the cotton fabric at elevated temperatures when both $K_2S_2O_8$ and NaH_2PO_2 are present.¹⁹ We also found that IA is able to homopolymerize *in situ* on cotton fabrics at elevated temperatures.^{20,21} Both PIA and the polymer formed by *in situ* polymerization of IA are able to crosslink cotton cellulose, thus imparting wrinkle resistance to cotton.^{20,22}

In this research, we evaluated the performance of the cotton fabric crosslinked by PIA and that crosslinked by *in situ* polymerization of IA. We compared the mechanical strength and abrasion resistance of the crosslinked cotton fabric so that we can elucidate the relationship between the mechanical strength of crosslinked cotton fabric and the size of the crosslinking agent.

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Contract grant sponsor: National Textile Center; contract grant number: C00-C01.

EXPERIMENTAL

Materials

The cotton fabric was a 100% cotton scoured, desized, and bleached printcloth (Testfabrics Style 400). IA, BTCA, sodium hypophosphite (NaH_2PO_2), and potassium persulfate ($\text{K}_2\text{S}_2\text{O}_8$) were reagent grade chemicals supplied by Aldrich.

Polymerization

One hundred twenty-five grams (125 g) IA and 50 g NaH_2PO_2 were dissolved in 325 mL water at 65°C. Sixteen grams (16 g) $\text{K}_2\text{S}_2\text{O}_8$ were then added to the solution in batches for 30 min. The polymerization continued for another 120 min. Extra NaH_2PO_2 was added to the solution after the polymerization process was complete so that the IA-to- NaH_2PO_2 ratio reached 6:5.3 in the PIA solution. The PIA concentration was then diluted to 6% based on the amount of IA added to the solution, and the solution thus prepared was used directly to treat the cotton fabric.

Cotton fabric treatment

A cotton fabric sample was first impregnated in an aqueous solution containing PIA/ NaH_2PO_2 , or that containing IA, $\text{K}_2\text{S}_2\text{O}_8$, and NaH_2PO_2 with a weight ratio of 6:2:4.9. Another cotton fabric sample was treated with 5.4% BTCA and 2.7% NaH_2PO_2 . The IA and BTCA solutions contained approximately the same carboxylic mole concentration (0.92 mol/L). The fabric was then passed through two dips and two nips in a laboratory padder with wet pick-up ranging from 95 to 100%. The impregnated fabric was dried at 85°C for 3 min, and then cured at a specified temperature for 3 min in a Mathis curing oven. All the concentrations were presented as percent weight of bath (%wob).

Evaluation of fabric performance

Tensile strength and flex abrasion resistance were tested according to ASTM Method D 5035-95 and D3885-92, respectively, and only the tensile strength at filling direction was measured. Wrinkle recovery angle (WRA) was measured according to AATCC Standard Method 66-1996. The home laundering washing/drying procedure was conducted according to AATCC Standard Method 124-1996. The initial properties of the cured fabric samples were measured after one washing cycle without the use of a detergent.

Hydrolysis of the crosslinked cotton fabric and the calculation of fabric tensile strength loss

We used alkaline hydrolysis with a 0.1M NaOH solution at 50°C for 50 h to remove the ester crosslinking in

the cotton fabric treated with PIA or IA. The WRA of the treated fabric decreased to the level of an untreated cotton fabric sample after 50 h of hydrolysis, indicating complete removal of the ester crosslinking on the fabric. The total tensile strength loss (%) of a crosslinked cotton fabric sample was calculated by the following formula: $[\text{tensile strength of a control sample} - \text{tensile strength of the crosslinked sample}] \div [\text{tensile strength of a control sample}] \times 100$. The tensile strength loss (%) due to crosslinking was calculated by the following formula: $[(\text{tensile strength of a crosslinked sample after hydrolysis} - \text{tensile strength of the crosslinked sample before hydrolysis}) \div \text{tensile strength of a control sample}] \times 100$, assuming that the contribution to the total tensile strength loss by crosslinking and acid degradation are additive. The tensile strength loss due to cellulose degradation (%) was calculated by the following formula: $[(\text{tensile strength of a control sample} - \text{tensile strength of a crosslinked sample after hydrolysis}) \times \text{tensile strength of a control sample}] \times 100$.

Fourier transform (FT)-Raman spectroscopic measurement

A Nicolet 950 FT-Raman spectrometer with a liquid sample accessory and an InGaAs detector was used to collect the Raman spectra of the solutions. For the Raman spectra, the resolution was 8 cm^{-1} , and there were 128 scans for each spectrum.

RESULTS AND DISCUSSION

IA and NaH_2PO_2 were first dissolved in water and the temperature was raised to 65°C. The FT-Raman spectrum of the solution is shown in Figure 1(A). The intense band at 1645 cm^{-1} is the C=C stretching mode of IA, and the peak of 1705 cm^{-1} is attributed to the stretching mode of the carboxylic carbonyl of IA. The asymmetric and symmetric stretching modes of unsaturated $=\text{CH}_2$ of IA were shown at 3118 and 3008 cm^{-1} , respectively, whereas the asymmetric stretching mode of $-\text{CH}_2-$ appeared at 2938 cm^{-1} in Figure 1(A). The sharp peak at 1044 cm^{-1} is due to the P—O stretching mode of H_2PO_2^- .^{23,24}

$\text{K}_2\text{S}_2\text{O}_8$ was added to the solution in batches during a period of 30 min, and the polymerization proceeded for another 30 min. The FT-Raman spectrum of the reaction mixture [Fig. 1(B)] shows that the intensity of the bands at 3118, 3008, and 1645 cm^{-1} due to unsaturated C—H and C=C, all are characteristic bands of IA, were reduced, whereas a band at 2942 cm^{-1} due to saturated CH_2 emerged, indicating the formation of PIA. One also observes that the carboxylic acid carbonyl band shifted from 1705 cm^{-1} in Figure 1(A) to 1710 cm^{-1} in Figure 1(B). The two bands at 1078 and

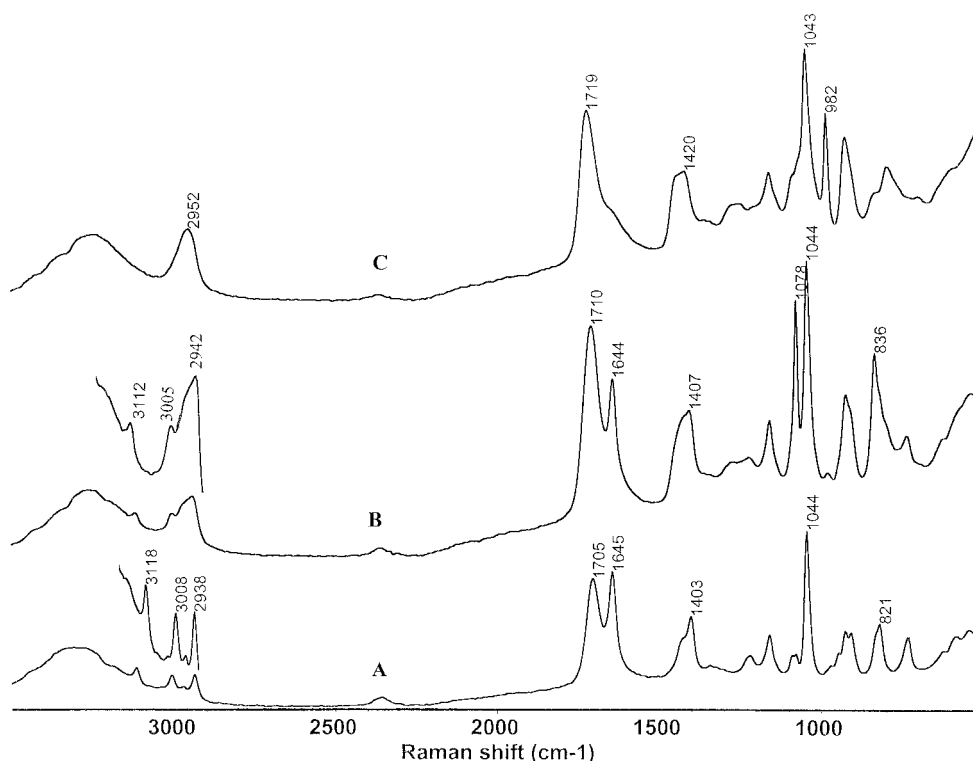


Figure 1 FT-Raman spectra of the aqueous solution of IA, NaH_2PO_2 , and $\text{K}_2\text{S}_2\text{O}_8$: (A) before polymerization; (B) polymerized for 60 min; (C) polymerized for another 90 min.

836 cm^{-1} in Figure 1(B) are attributed to the stretching modes of $\text{S}=\text{O}$ and $\text{S}-\text{O}$, respectively, of $\text{S}_2\text{O}_8^{2-}$.²⁵

After the polymerization was continued for another 90 min, the characteristic bands of IA at 3118, 3008, and 1645 cm^{-1} disappeared completely in Figure 1(C), indicating that the polymerization was complete. The carboxylic acid carbonyl band increased its frequency further to 1719 cm^{-1} in Figure 1(C), because the conjugation between $\text{C}=\text{C}$ and $\text{C}=\text{O}$ shifted the carboxylic acid carbonyl stretching mode to a lower frequency,²⁶ and it no longer existed when the $\text{C}=\text{C}$ bond became saturated after polymerization. The band at 821 cm^{-1} in Figure 1(A) due to out-of-plane deformation of $=\text{CH}_2$ of IA also disappeared completely in Figure 1(C). The band at 1403 cm^{-1} in Figure 1(A), attributed to the combination of $\text{C}-\text{O}$ stretching and $\text{O}-\text{H}$ deformation of IA,²³ shifted to 1420 cm^{-1} in Figure 1(C) the polymerization was complete. We can observe that the two bands at 1078 and 836 cm^{-1} due to $\text{S}_2\text{O}_8^{2-}$ disappeared whereas a band at 982 cm^{-1} due to SO_4^{2-} emerged, showing that all the $\text{K}_2\text{S}_2\text{O}_8$ added to the reaction mixture decomposed. The polymerization mechanism of IA was investigated using mass spectroscopy and ^{31}P nuclear magnetic resonance (NMR) spectroscopy, and the molecular weight of PIA was found to be approximately 700. The results will be discussed in a subsequent paper.

The cotton fabric was treated with PIA, IA, and BTCA with carboxylic acid concentration ranging

from 2 to 10%, dried, and finally cured at 180°C for 3 min. The WRA of the cotton fabric thus treated is shown in Figure 2. The data indicated that IA imparted slightly higher wrinkle resistance to cotton than PIA, whereas BTCA was a much more effective crosslinking agent than IA and PIA. The BTCA-treated cotton fabric had significantly higher WRA than that treated with IA or PIA. The slightly lower WRA for the PIA-treated fabric than the IA-treated one can possibly be attributed to the difference in the ability of PIA and IA to penetrate into the interior of cotton fibers. The molecular size of IA is much smaller than PIA. The smaller IA molecules had better penetration into the amorphous regions of cotton fibers to form a more homogeneously distributed crosslinking than PIA. More homogeneous distribution of crosslinks in cotton fibers is beneficial to the wrinkle resistance of the crosslinked cotton fabric.²⁷ One also observes in Figure 2 that the slope (WRA vs acid concentration) decreased as the acid concentration was raised above the 6% concentration level, probably due to the lack of cellulosic hydroxyl groups to esterify the acid at the high acid levels.

The cotton fabric treated with 6% PIA, 6% IA, and 5.4% BTCA, and then cured at different temperatures for 3 min. The WRA of the cotton fabric thus treated is presented in Figure 3. BTCA was apparently more effective in crosslinking cellulose and imparting significantly higher wrinkle resistance to cotton than IA

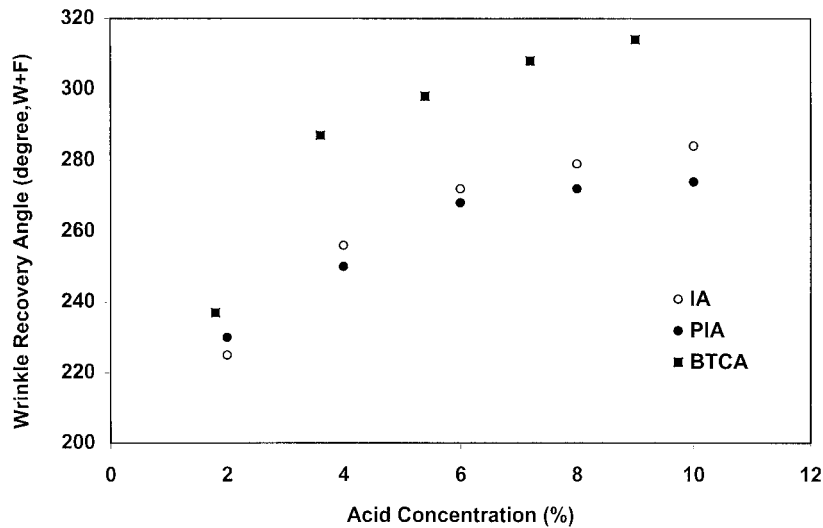


Figure 2 Wrinkle recovery angle of the cotton fabric treated with IA, PIA, and BTCA of different concentrations and cured at 180°C for 3 min.

and PIA. The IA-treated fabric showed slightly higher WRA than that treated with PIA (Fig. 3). We also can observe that WRA for the cotton fabric treated with PIA, IA and BTCA had a more steep increase as the curing temperature was increased from 140 to 160°C, and the increase in WRA became much more moderate as the curing temperature was increased further to 180 and 200°C (Fig. 3).

The percent tensile strength loss of the cotton fabric treated with IA and PIA of different concentration and cured at 180°C for 3 min, and that treated with 6% IA and 6% PIA and cured at different temperatures for 3 min are presented as a function of WRA of the treated fabric in Figures 4 and 5, respectively. The data presented show that the fabric tensile strength loss increased as the WRA increased as a result of higher

concentration of the crosslinking agent and higher curing temperature, as shown in Figures 3 and 4, respectively. We can observe that the tensile strength loss for the fabric treated with PIA was significantly lower than that treated with IA at the same WRA in both Figures 4 and 5.

In our previous research, we found that the strength loss for cotton fabric crosslinked by a polycarboxylic acid is attributed two factors: irreversible cellulose degradation and reversible crosslinking of cellulose by the polycarboxylic acid.²⁸ In order to study the strength loss of the cotton fabric treated with PIA and that treated with IA, we first evaluated the effect of the $K_2S_2O_8/NaH_2PO_2$ initiation system on cellulose degradation. NaH_2PO_2 functions as the reducing agent in the initiation system and also as the catalyst for cellu-

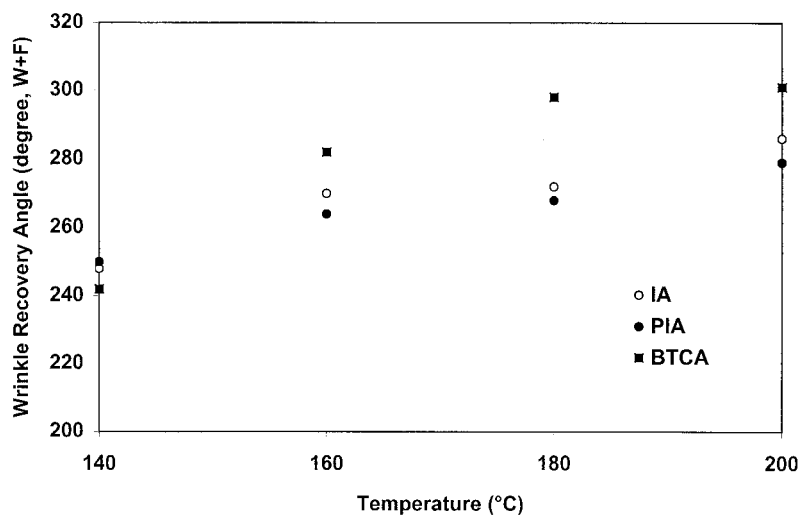


Figure 3 Wrinkle recovery angle of the cotton fabric treated with 6% IA, 6% PIA, and 5.4% BTCA and cured at different temperatures for 3 min.

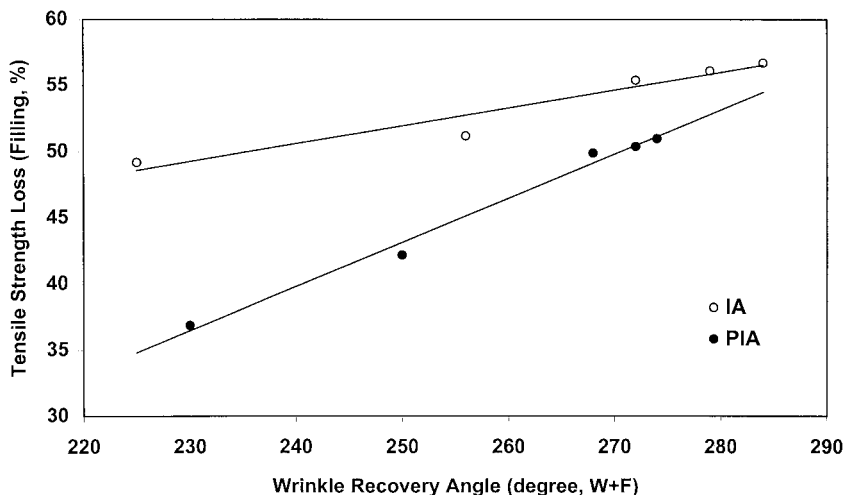


Figure 4 Tensile strength (filling) loss for the cotton fabric treated with IA and PIA of different concentrations and cured at 180°C for 3 min vs wrinkle recovery angle.

lose esterification. We found that the effect of NaH_2PO_2 on the fabric strength is negligible. The cotton fabric was treated with $\text{K}_2\text{S}_2\text{O}_8$ and the combination of $\text{K}_2\text{S}_2\text{O}_8$ and NaH_2PO_2 , with a $\text{K}_2\text{S}_2\text{O}_8$ -to- NaH_2PO_2 ratio of 2:4.9 (w/w), and the treated fabric was heated at 180°C for 3 min. Presented in Figure 6 is percent strength loss of the fabric treated with $\text{K}_2\text{S}_2\text{O}_8$ and the combination of $\text{K}_2\text{S}_2\text{O}_8$ and NaH_2PO_2 as a function of the concentration of $\text{K}_2\text{S}_2\text{O}_8$. One observes a significant loss in fabric strength caused by $\text{K}_2\text{S}_2\text{O}_8$ alone. The cotton fabric lost 40.2% of its original strength when it was treated with 0.25% $\text{K}_2\text{S}_2\text{O}_8$, and the strength loss increased to 76.6% as the $\text{K}_2\text{S}_2\text{O}_8$ concentration was increased to 1% (Fig. 6). The diminished fabric strength is evidently due to the oxidative effects of $\text{K}_2\text{S}_2\text{O}_8$. The strength loss of the cotton fabric treated with $\text{K}_2\text{S}_2\text{O}_8/\text{NaH}_2\text{PO}_2$ was much more mod-

erate than that treated with $\text{K}_2\text{S}_2\text{O}_8$ alone, because NaH_2PO_2 reduced $\text{K}_2\text{S}_2\text{O}_8$ on the fabric, thus minimizing the oxidative effect of $\text{K}_2\text{S}_2\text{O}_8$. The fabric treated with 1% $\text{K}_2\text{S}_2\text{O}_8$ and 2.45% NaH_2PO_2 lost only 41.3% of its strength. All the $\text{K}_2\text{S}_2\text{O}_8$ decomposed during polymerization process of IA as indicated by the complete disappearance of the two bands at 1078 and 836 cm^{-1} due to the stretching mode of $\text{S}=\text{O}$ and $\text{S}-\text{O}$, respectively, of $\text{S}_2\text{O}_8^{2-}$ in Figure 1(C). Therefore, the cotton fabric treated with PIA was not degraded by $\text{K}_2\text{S}_2\text{O}_8/\text{NaH}_2\text{PO}_2$ initiation system under the curing conditions.

The cotton fabric was treated 6% PIA and 6% IA, and then cured at different temperatures for 3 min. The cured cotton fabric was then hydrolyzed in 0.1M NaOH at 50°C for 50 h to remove the crosslinking in the fabric. Our previous research showed that all the

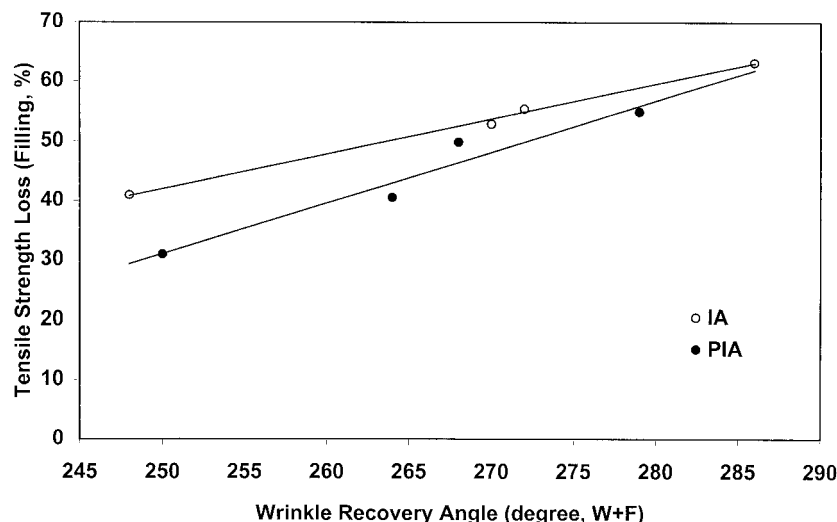


Figure 5 Tensile strength (filling) loss for the cotton fabric treated with 6% IA and 6% PIA and cured at different temperatures for 3 min.

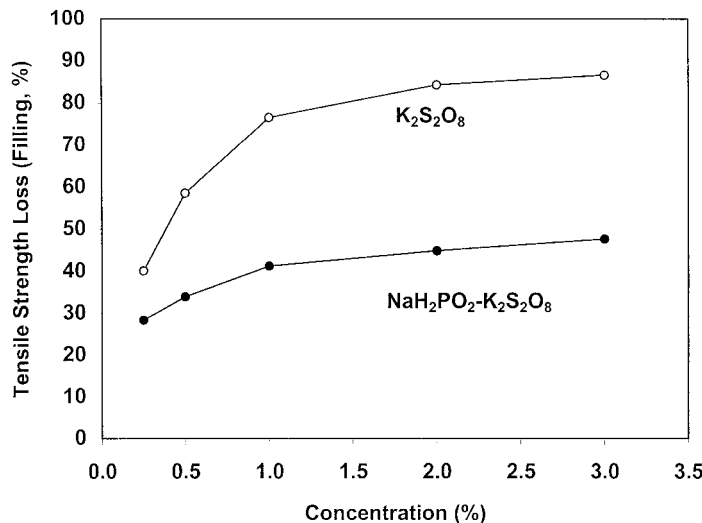


Figure 6 Tensile strength (filling) loss for the cotton fabric treated with $K_2S_2O_8$ and $NaH_2PO_2/K_2S_2O_8$, and heated at $180^\circ C$ for 3 min.

crosslinking in the cotton fabric treated with 6% BTCA and cured at $180^\circ C$ for 2 min was totally removed after hydrolysis in 0.1M NaOH at $50^\circ C$ for 48 h.²⁸ Thus, the percent tensile strength loss of the crosslinked cotton fabric due to degradation can be determined based on the difference between the tensile strength of control fabric and that of the crosslinked fabric after hydrolysis. The percent tensile strength loss due to degradation of the cotton fabric treated with 6% IA and that treated with 6% PIA are shown as a function of curing temperature in Figure 7. One observes that the IA-treated cotton fabric lost from 37 to 49% of its original strength due to cellulose degradation whereas the PIA-treated fabric lost 15 to 34% of its strength due to degradation when the curing temperature was increased from 150 to $190^\circ C$ (Fig. 7). The data indicate that the strength loss of the fabric treated with IA was

12–19% higher than that treated with PIA in the temperature range (150 – $190^\circ C$). Evidently, the oxidative effect of $K_2S_2O_8$ on the IA-treated fabric caused more strength loss than the PIA-treated fabric.

We determined the percent strength loss of the crosslinked cotton fabric due to crosslinking based on the difference between the strength of the fabric before hydrolysis and that after hydrolysis. The percent strength loss due to crosslinking of the fabric treated with 6% IA and that treated with 6% PIA presented in Figure 8 show that the tensile strength loss due to crosslinking for the IA-treated fabric was 8–11% lower than that for the PIA-treated fabric at different temperatures. The data presented here suggest that the mechanical strength of crosslinked cotton fabric is affected by the size of the crosslinking agent, and the consequent distribution of the crosslinks across the

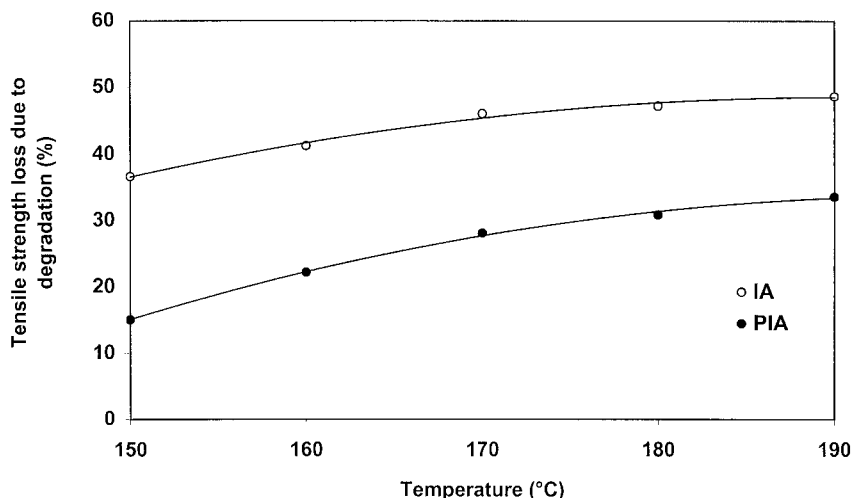


Figure 7 Tensile strength (filling) loss due to degradation for the cotton fabric treated with 6% IA and 6% PIA and cured at different temperatures for 3 min.

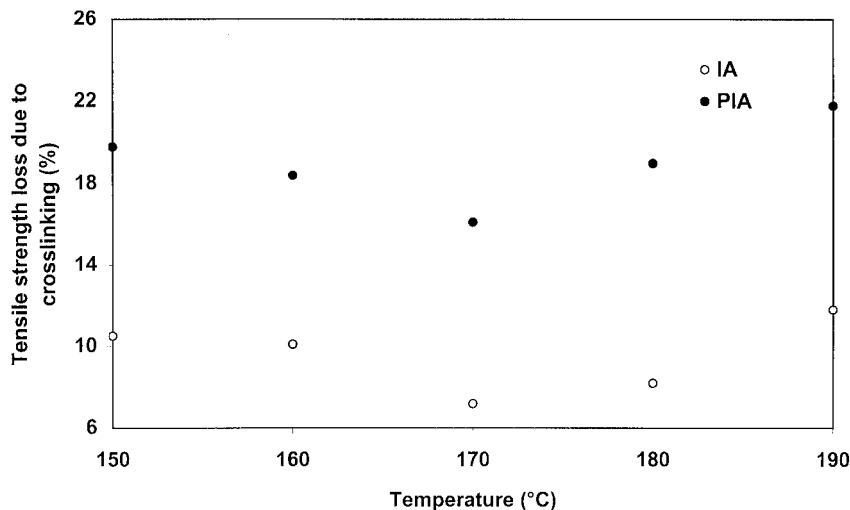


Figure 8 Tensile strength (filling) loss due to crosslinking for the cotton fabric treated with 6% IA and 6% PIA and cured at different temperatures for 3 min.

fabric. IA, with its molecular size much smaller than that of PIA, had better penetration into the fiber interior, therefore had more homogeneous distribution in the fabric. Consequently, the IA-treated fabric had significantly lower tensile strength loss due to crosslinking than the PIA-treated fabric because of a more homogeneous distribution of the crosslinks across the fabric cross section. Our observation here is consistent with what reported in the literature.^{29,30} De Bore and Borsten observed that mercerized and DP finished cotton fabric had more homogeneous distribution of crosslinks and improved fabric tensile and tearing strength.²⁹ Shin and co-workers used “wet fixation” and “steam fixation” to facilitate the penetration of monomeric crosslinking agents into fiber interiors and observes higher fabric strength.³⁰

Shown in Figure 9 is the flex abrasion resistance vs

WRA of the cotton fabric treated with PIA and BTCA of different concentrations and cured at 180°C for 3 min. The BTCA-treated cotton fabric appeared to have significantly higher abrasion resistance than the PIA-treated fabric at the same levels of wrinkle resistance. The difference in fabric abrasion resistance is most likely due to the different distribution of the crosslinks in the treated fabric. The molecular size of PIA is much larger than that of BTCA, and consequently its ability to penetrate into the fiber interior is reduced compared with that of BTCA. Therefore, PIA-treated cotton fabric with more concentrated crosslinks on the fabric surface had lower abrasion resistance than BTCA-treated cotton fabric.

We also evaluated the home laundering durability of the cotton fabric crosslinked by PIA. The cotton fabric was treated with 6% PIA and cured at 180°C.

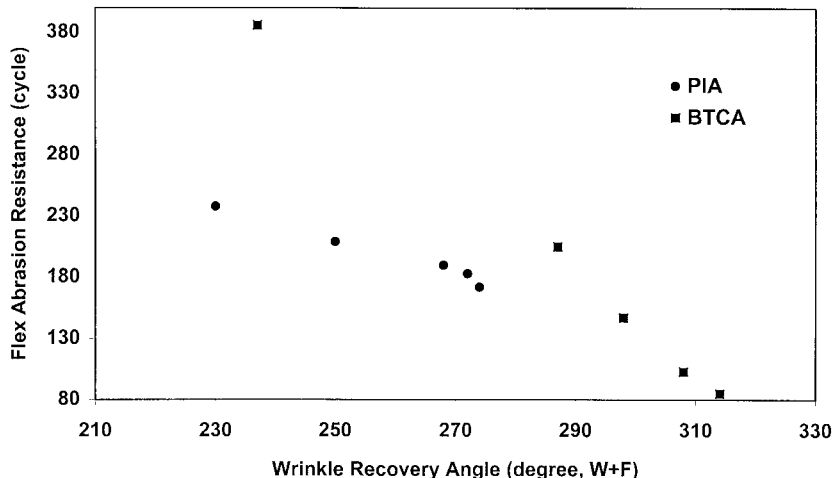


Figure 9 Flex abrasion resistance (filling) of the cotton fabric treated with PIA and BTCA of different concentrations and cured at 180°C for 3 min s wrinkle recovery angle.

TABLE I
The Performance of Cotton Fabric Treated with 6% PIA and 5.4% BTCA and Cured at 180°C for 3 min

	DP rating					WRA (degree, W+F)					Tensile strength (filling, kgf)	Flex abrasion resistance (filling, cycle)
	Washing cycle					Washing cycle						
	0	5	10	20	30	0	5	10	20	30		
PIA	3.6	3.5	3.2	3.1	3.0	269	263	259	257	245	10.1	190
BTCA	3.8	3.6	3.5	3.4	3.2	305	282	276	270	262	7.7	147
Control			–			200					20.5	595

The treated fabric was subjected to 30 home laundering washing/drying cycles. The cotton fabric was also treated with 6% BTCA for comparison. The performance of the cotton fabric thus treated is shown in Table I. The WRA of the BTCA-treated cotton fabrics decreased from 305° to 262° after 30 washing cycles, whereas the WRA of the PIA-treated fabric decreased from 269° to 245° (Table I). BTCA is apparently more effective than PIA for crosslinking cotton. Nevertheless, the cotton fabric treated with PIA still maintained at 3.0 DP rating after the 30 washing cycles (Table I).

CONCLUSIONS

Tensile strength and abrasion resistance of crosslinked cotton fabric are affected by the size of the crosslinking agent. A crosslinking agent with smaller molecular size has better penetration into the fiber interiors and more homogeneous distribution in the fabric, which result in higher tensile strength and abrasion resistance.

Although IA is a more effective crosslinking agent than PIA, oxidative effect of the initiator diminishes the mechanical strength of the crosslinked cotton fabric. The cotton fabric treated with PIA shows satisfactory durable press performance and good home laundry durability.

We are grateful to National Textile Center for providing funding for this research project (C00-C01).

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