

Infrared Spectroscopic Studies of the Nonformaldehyde Durable Press Finishing of Cotton Fabrics by Use of Polycarboxylic Acids

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SYNOPSIS

A number of polycarboxylic acids have been used successfully as new nonformaldehyde cross-linking agents for cotton fabrics. In our previous research, Fourier transform infrared photoacoustic spectroscopy (FTIR/PAS) demonstrated the ability for characterizing ester cross-linkages in the finished cotton fabrics. In this research, the effects of different acid concentrations, different catalyst concentrations, different curing temperatures, and different curing times on the ester cross-linking of the cotton fabrics were determined by FTIR/PAS. The infrared spectroscopic data were also correlated to durable press (DP) ratings. FTIR appears to be useful for evaluating the effectiveness of polycarboxylic acids as cross-linking agents for cotton fabrics. FTIR definitely complements the existing methods for evaluating finished textile fabrics.

INTRODUCTION

Dimethylodihydroxyethyleneurea (DMDHEU) and its derivatives have long been used to produce highly effective durable press finishes for cotton and cotton/polyester blend fabrics in the textile industry.¹ However, the release of formaldehyde during processing and storage of the fabrics has caused increasing concern about the possible health risk.²⁻⁶ Intensive efforts have been made to develop new high-quality, low-cost nonformaldehyde cross-linking agents to replace the conventional finishing agents.⁷⁻¹² Among the new nonformaldehyde durable press finishing agents being developed, polycarboxylic acids demonstrated a number of advantages over the other proposed reagents, such as excellent durable press performance, high fabric strength retention, and good home laundry durability.¹³⁻¹⁵

In our previous research, Fourier transform infrared photoacoustic spectroscopy was used to characterize the ester cross-linkages in the polycarboxylic acid-treated cotton fabrics on a molecular level.¹⁶

The infrared spectroscopic data demonstrated that the ester carbonyl band in the infrared spectra of the finished cotton fabrics could be separated from the bands of carboxyl and carboxylate carbonyls. Therefore, the ester cross-linking of various acid-finished cotton fabrics can be examined and compared on a semiquantitative basis by measuring the ester carbonyl band intensities and the carbonyl band intensity ratios (ester/carboxylate) in the infrared spectra of those fabrics.¹⁶

In the textile science community, the effectiveness of durable press finishing agents is examined by measuring the final performance of finished fabrics, such as wrinkle recovery angle and durable press rating. The objective of this research was to evaluate the effectiveness of polycarboxylic acids as cross-linking agents for cotton fabrics using FTIR spectrometry, so that the effects of acid concentration, catalyst concentration, curing temperature, and curing time on the ester cross-linking of the fabrics could be determined. The infrared spectroscopic data were also correlated to the durable press rating results.

PAS is a nondestructive sampling technique. Fabric samples can be analyzed without being

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ground into powders to prepare potassium bromide pellets.¹⁷ Therefore, any alteration to finished fabrics can be avoided. FTIR photoacoustic spectroscopy is also a near-surface analytical technique.¹⁸ The photoacoustic infrared spectrum of a fabric represents a few microns of the fabric's near-surface.¹⁸ When a fabric is ground into a powder, the photoacoustic infrared spectrum of the powder represents the bulk of the fabric. Therefore, the distribution of finishes in a fabric can be determined by comparing the photoacoustic infrared spectrum of the fabric and the spectrum of the corresponding powder.^{19,20} Our previous research indicated that the ester groups in the acid-finished cotton fabrics were homogeneously distributed between the near-surface of a fabric and its interior.¹⁶ Therefore, FTIR/PAS can be used as a reliable method to examine the ester cross-linking throughout the fabrics.

EXPERIMENTAL

Infrared Spectroscopic Measurements

A Nicolet 20DXB FTIR spectrometer with an MTEch Model 100 photoacoustic cell at the Department of Chemistry, Marshall University, was used for all the FTIR measurements. Resolution for all the spectra presented was 8 cm^{-1} . The number of scans was 250. A 6 mm sample cup was used for data collection. Carbon black was used as a reference material, and helium was used to purge the photoacoustic cell prior to collecting data. The mirror velocity used was 0.139 cm/s . No smoothing function or base-line correction was used.

For the purpose of semiquantitative measurement of the finishes on the fabrics, the carbonyl intensities in the infrared spectra of the finished cotton fabrics were normalized against the 1317 cm^{-1} band, which is associated with the bending vibration mode of hydrocarbon structures in cellulose molecules. Even though the 1370 and 1430 cm^{-1} bands are more commonly used as references, the presence of a strong band at the $1440\text{--}1360\text{ cm}^{-1}$ region due to symmetric stretching of carboxylate groups prevents the use of these two bands for normalization.

Fabric Treatment

Fabric treatments were carried out at the Southern Regional Research Center, U.S. Department of Agriculture, New Orleans. The fabric was a desized, scoured, and bleached print cloth weighing 3.2 oz/yd^2 . The fabrics were impregnated with solutions containing butanetetracarboxylic acid (BTCA) as

a cross-linking agent and monosodium phosphate as a catalyst. The catalyst used was in a hydrated form ($\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$). The impregnated fabrics were dried at 85°C for 5 min, then cured at 180°C for 90 s unless specified otherwise. Cotton fabrics were also treated with tricarballic, succinic, and maleic acids using the same method. The cured fabrics were given a home laundering with a nonionic detergent and tumbled dry. The tap water used to wash the fabrics had pH of 9.5. The durable press (DP) appearance rating was measured according to the AATCC standard method (AATCC Test Method 124-1984).

RESULTS AND DISCUSSION

Characterization of the Intermolecular Ester Cross-Linking of Cotton Fabrics

When esterification occurs between a polycarboxylic acid and cotton cellulose, the carbonyls in the fabric

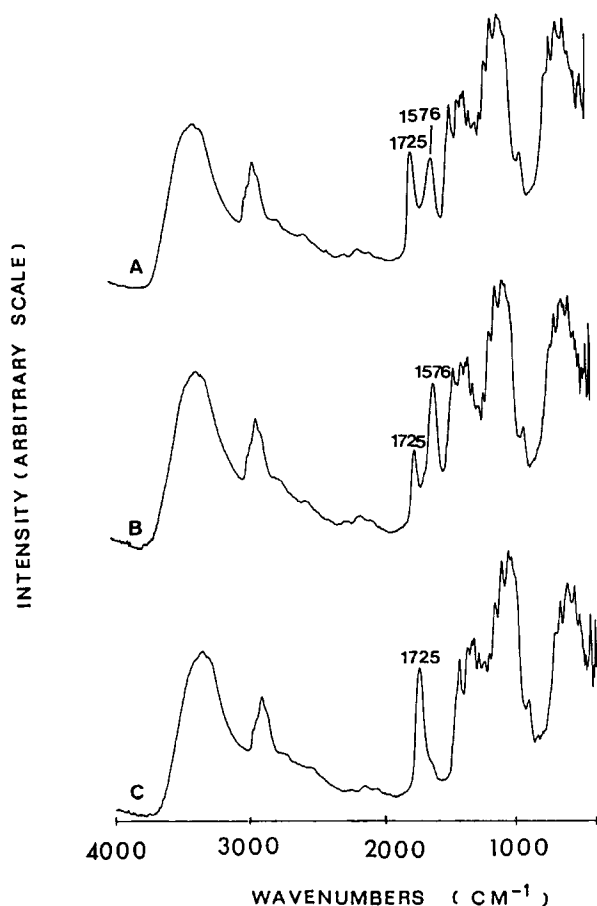
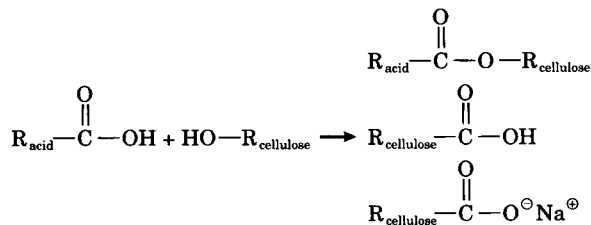


Figure 1 Photoacoustic infrared spectra of the cotton fabrics finished with BTCA and NaH_2PO_4 and cured at 180°C for 90 s: (A) no treatment; (B) treated in a 0.1 M NaOH solution; (C) treated in a 0.1 M HCl solution.

can exist in three forms: intermolecular ester linkage, carboxyl, and carboxylate. For polycarboxylic acids containing three or more carboxyl groups, all three forms of carbonyls could be present in one acid molecule. The generalized reaction scheme for the formation of the three species follows:



The photoacoustic infrared spectrum of the cotton fabric treated in a solution containing 6.6% BTCA and 3.7% NaH_2PO_4 is shown in Figure 1(A). Two carbonyl bands at 1725 and 1576 cm^{-1} , respectively, are observed in Figure 1(A). An increase in the 1576 cm^{-1} band intensity and a decrease in the 1725 cm^{-1} band intensity result after the finished cotton fabric was treated in a 0.1 M NaOH solution for 2 min at room temperature [Fig. 1(B)]. The 1576 cm^{-1} band disappears completely after the finished fabric was treated in a 0.1 M hydrochloric acid solution for 2 min at room temperature [Fig. 1(C)]. It can be concluded that the 1576 cm^{-1} band is due to the carbonyl of carboxylate and that the carboxyl and ester carbonyl bands overlap at 1725 cm^{-1} . Sodium hydroxide converts carboxyl to carboxylate so that a decrease in the 1725 cm^{-1} band intensity and an increase in the 1576 cm^{-1} band intensity are observed [Fig. 1(B)]. Hydrochloric acid converts all the carboxylate to carboxyl. As a result, the 1576 cm^{-1} band disappears completely, whereas the intensity of the 1725 cm^{-1} band is increased [Fig. 1(C)].

When a finished fabric is treated with a dilute NaOH solution, carboxyls in the fabric are converted to carboxylates. Consequently, the band of the ester carbonyl in an infrared spectrum of the fabric is separated from the carboxyl carbonyl band, which overlaps the ester carbonyl band at 1725 cm^{-1} . As a result, the total quantity of the ester groups in the finished fabrics can be determined by measuring the 1725 cm^{-1} band intensity. Because the ester groups in the finished fabric include both the ester cross-linkages and the ester groups between cotton cellulose and singly bonded acid molecules, the ester carbonyl band (1725 cm^{-1}) intensity alone does not represent the effectiveness of the acid as a cross-linking agent in the cotton fabric. The carbonyl band intensity ratio (ester/carboxylate), however, can be

used to evaluate the effectiveness of the acid for cross-linking cellulose. An acid molecule can form a cross-linkage between cellulose molecules only when more than one carboxyl groups in its molecule are esterified. The more carboxyl groups are esterified per each acid molecule, the more ester cross-linkages are formed for the same quantity of ester groups in the fabric. As a result, the acid becomes a more effective cross-linking agent. An increase in the carbonyl band intensity ratio (ester/carboxylate) indicates an increase in the average number of ester groups formed per each acid molecule. Even though the actual number of the carboxyl groups esterified per each acid molecule in the fabric is yet to be determined using another method, an increase in the carbonyl band intensity ratio (ester/carbox-

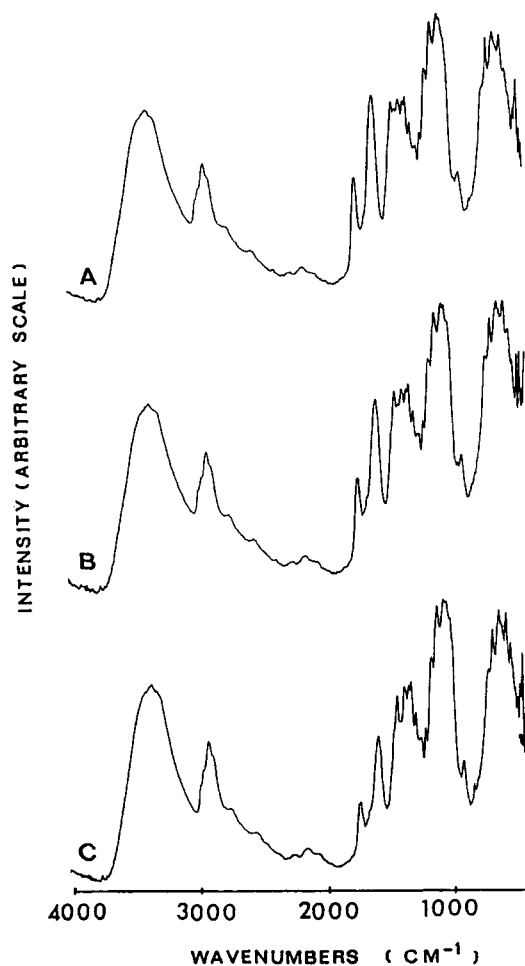


Figure 2 Photoacoustic infrared spectra of the cotton fabrics finished with solutions containing the following: (A) 10% BTCA, 5.8% NaH_2PO_4 ; (B) 6.0% BTCA, 3.5% NaH_2PO_4 ; (C) 2.0% BTCA, 1.1% NaH_2PO_4 . The fabrics were treated with a 0.1 M NaOH solution for 2 min at room temperature prior to analysis.

Table I The Ester Carbonyl Band Intensities, the Carbonyl Band Intensity Ratios, and the DP Ratings of the Cotton Fabrics Treated with BTCA and NaH₂PO₄ of Different Concentrations^a

	Acid Concentration (%)		
	10.0	6.0	2.0
	Catalyst Concentration (%)		
	5.8	3.5	1.1
Ester carbonyl band (1725 cm ⁻¹) intensity	0.65	0.57	0.37
Carbonyl band intensity ratio (1725 cm ⁻¹ /1576 cm ⁻¹)	0.58	0.55	0.49
DP rating	4.0	3.7	2.5

^a The cotton fabrics were cured at 180°C for 90 s. The fabrics were treated in a 0.1M NaOH solution for 2 min at room temperature prior to analysis.

ylate) indicates an increase in the effectiveness of a polycarboxylic acid as a cross-linking agent for cotton.

The Effects of Acid and Catalyst Concentrations

The effects of changes in acid and catalyst concentrations on the ester cross-linking of cotton were examined using FTIR/PAS. The cotton fabrics were treated in solutions containing 10.0%, 6.0%, and 2.0% BTCA, respectively, and monosodium phosphate as a catalyst. The three solutions contained a constant carboxyl/NaH₂PO₄ mol ratio of 3.5. The carboxyl groups were converted to carboxylate by a NaOH solution treatment before FTIR analysis. The

photoacoustic infrared spectra of the cotton fabrics thus treated are presented in Figure 2. The ester carbonyl band intensities, the carbonyl band intensity ratios (ester/carboxylate), and the DP ratings are summarized in Table I. It is observed that both the ester carbonyl band intensity and the carbonyl band intensity ratio increase as the acid/catalyst concentrations are increased. It indicates that an increase in acid/catalyst concentration results in both an increase in the total quantity of ester groups formed between BTCA and the cotton cellulose and an increase in the average number of carboxyl groups esterified per each BTCA molecule and, consequently, an increase in the effectiveness of BTCA as a cross-linking agent. Table I also shows that the fabric DP performance is improved as a result of the increase in acid/catalyst concentrations. Obviously, the improved fabric DP rating is not only due to the increase in the total amount of carboxylic acid substitution, but also to the increase in the average number of carboxyl groups esterified per BTCA molecule.

The effects of change in the catalyst concentration on the ester cross-linking was also investigated. The cotton fabrics were treated with solutions containing 6.3% BTCA and different catalyst concentrations. A 0.1M NaOH solution was used to convert the carboxyl groups to carboxylate groups in the fabrics before FTIR analysis. The ester carbonyl intensities, the carbonyl band intensity ratios (ester/carboxylate), and the DP ratings of the fabrics are shown in Table II. It appears that change in catalyst concentration alone has a more moderate effect on the effectiveness of BTCA than does change in both acid and catalyst concentrations. The cotton fabric treated using the highest catalyst concentration shows the highest ester carbonyl band intensity and

Table II The Ester Carbonyl Band Intensities, the Carbonyl Band Intensity Ratios, and the DP Ratings of the Cotton Fabrics Treated with BTCA and NaH₂PO₄^a

BTCA concentration (%)	6.3	6.3	6.3	6.3	6.3	6.3	6.3
NaH ₂ PO ₄ concentration (%)	5.5	5.0	4.3	3.7	3.0	2.4	1.2
Mol ratio (carboxyl/NaH ₂ PO ₄)	2.4	2.6	3.0	3.5	4.3	5.3	10.6
Ester carbonyl band (1725 cm ⁻¹) intensity	0.53	0.47	0.49	0.44	0.48	0.45	0.45
Carbonyl band intensity ratio (1725 cm ⁻¹ /1576 cm ⁻¹)	0.55	0.52	0.54	0.51	0.52	0.52	0.51
DP rating	4.2	4.0	4.1	3.7	3.6	3.7	3.4

^a All the cotton fabrics were cured at 180°C for 90 s. The fabrics were treated in a 0.1M NaOH solution for 2 min at room temperature prior to analysis.

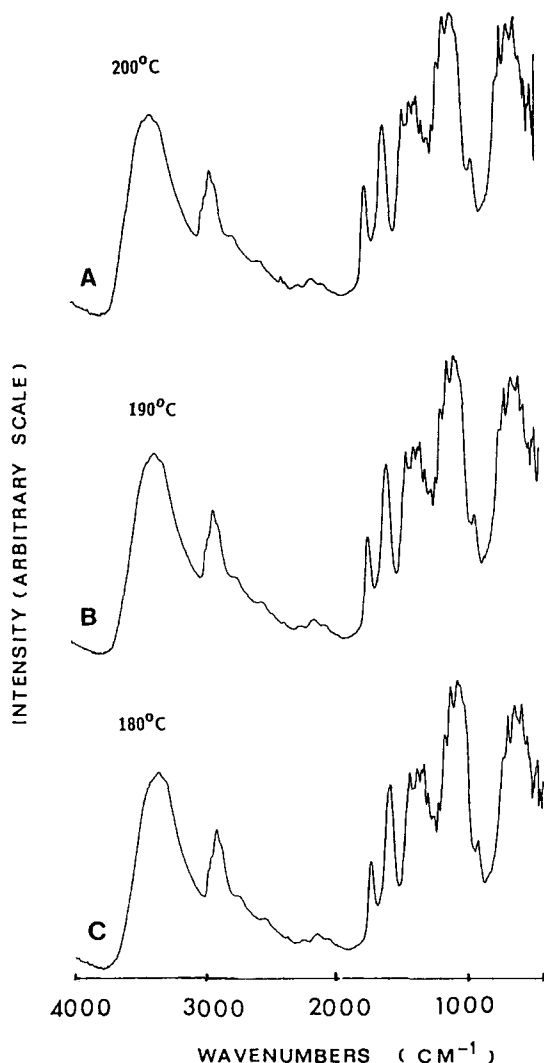


Figure 3 Photoacoustic infrared spectra of the cotton fabrics finished with 6.3% BTCA and 3.7% NaH_2PO_4 and cured for 60 s at different temperatures: (A) 200°C; (B) 190°C; (C) 180°C. The fabrics were treated with a 0.1M NaOH solution for 2 min at room temperature prior to analysis.

the highest carbonyl band intensity ratio and, consequently, the highest DP rating. The ester carbonyl band intensity decreases slightly when the catalyst concentration is reduced. It is also observed that a reduction in the catalyst concentration from 5.0% to 1.2% has little effect on the carbonyl band intensity ratio (ester/carboxylate). Those fabrics treated with reduced catalyst concentrations showed moderately reduced fabric DP ratings.

The Effect of Curing Temperature

The cotton fabrics finished with 6.3% BTCA/3.7% NaH_2PO_4 , cured at different temperatures for 60 s,

and, finally, treated with a 0.1M NaOH solution were analyzed by FTIR/PAS (Fig. 3). The ester carbonyl band intensities, the carbonyl band intensity ratios (ester/carboxylate), and the DP ratings of those fabrics are presented in Table III. When the curing temperature was increased from 180°C to 200°C, the average number of ester groups formed per each BTCA molecule increased substantially, as demonstrated by the increase in the carbonyl band intensity ratio (Table III). This was probably because an increase in curing temperatures caused an increase in the rate of esterification reaction. Consequently, the number of ester cross-linkages in the cotton fabric were increased during the same time period and so was the effectiveness of the acid as a cross-linking agent as measured by the DP rating (Table III).

Shown in Figure 4 are the photoacoustic infrared spectra of the same BTCA-treated cotton fabrics described above except that the fabrics were not treated in a 0.1M NaOH solution prior to analysis. The free carboxylic acid groups in the cured and washed cotton fabrics existed in two forms: acidic form (carboxyl) and basic form (carboxylate). The carboxyl and carboxylate carbonyls absorb at 1725 and 1576 cm^{-1} , respectively. Figure 4 demonstrates that the 1725 cm^{-1} band intensity increases and the 1576 cm^{-1} band intensity decreases substantially when curing temperature is increased. It indicates that an increase in curing temperature results in an increase in the carboxyl concentration, and therefore in the acidity in the near-surfaces of the cured and washed fabrics, since the photoacoustic infrared spectra represented a few microns of the fabrics' near-surfaces.¹⁸⁻²⁰ Similar phenomena were also ob-

Table III The Ester Carbonyl Band Intensities, the Carbonyl Band Intensity Ratios, and the DP Ratings of the Cotton Fabrics Treated with BTCA/ NaH_2PO_4 and Cured for 60 s^a

	Curing Temperature (°C)		
	200	190	180
Ester carbonyl band (1725 cm^{-1}) intensity	0.63	0.58	0.52
Carbonyl band intensity ratio (1725 cm^{-1} / 1576 cm^{-1})	0.65	0.60	0.54
DP rating	5.0	4.5	4.5

^a The fabrics were treated in a 0.1M NaOH solution for 2 min at room temperature prior to analysis. The same procedures were applied to the data presented in Tables IV-VI.

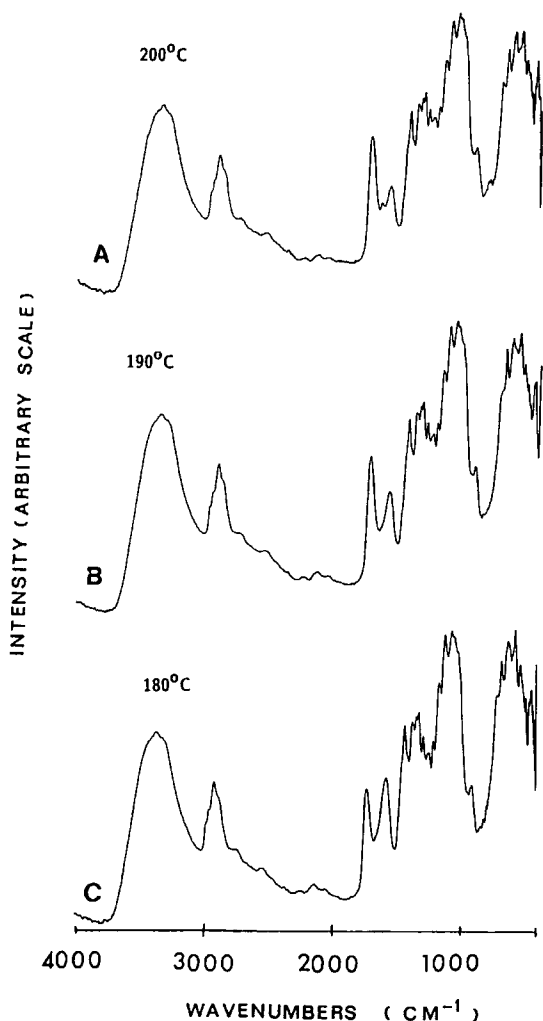


Figure 4 Photoacoustic infrared spectra of the cotton fabrics finished with 6.4% BTCA and 3.7% NaH_2PO_4 and cured for 60 s at different temperatures: (A) 200°C; (B) 190°C; (C) 180°C.

served for cotton fabrics treated with tricarballylic acid.

The cotton fabric treated with 6.4% maleic acid/3.7% NaH_2PO_4 and cured at different temperatures for 60 s were also studied using FTIR/PAS (Fig. 5 and Table IV). The data in Table IV indicate that the ester carbonyl band intensity and the carbonyl band intensity ratio (ester/carboxylate) increased slightly when the curing temperature was increased from 180°C to 200°C. An increase in curing temperature appears to have little influence on the effectiveness of maleic acid for cross-linking cotton fabrics. The DP rating shows a slight decrease as the temperature increases. Infrared spectroscopic data also show that succinic acid is even less effective for cross-linking cotton than is maleic acid. This phenomenon can be explained by the esterification

mechanism of polycarboxylic acids. Infrared spectroscopic data obtained in our laboratory support the hypothesis that cyclic anhydrides were formed as intermediates before polycarboxylic acid was esterified with cotton cellulose.²¹ It was also observed in our laboratory that in the absence of a catalyst a temperature much higher than the curing temperature used is necessary for esterification between a polycarboxylic acid and cotton cellulose to occur.²¹ Experimental evidence also suggests that a cyclic anhydride, once formed, can esterify cotton cellulose at a temperature much lower than these curing temperatures used.²² It is most likely that esterification between an acid and cotton cellulose can occur at a lower temperature through the formation of a cyclic anhydride. For a bifunctional polycarboxylic acid

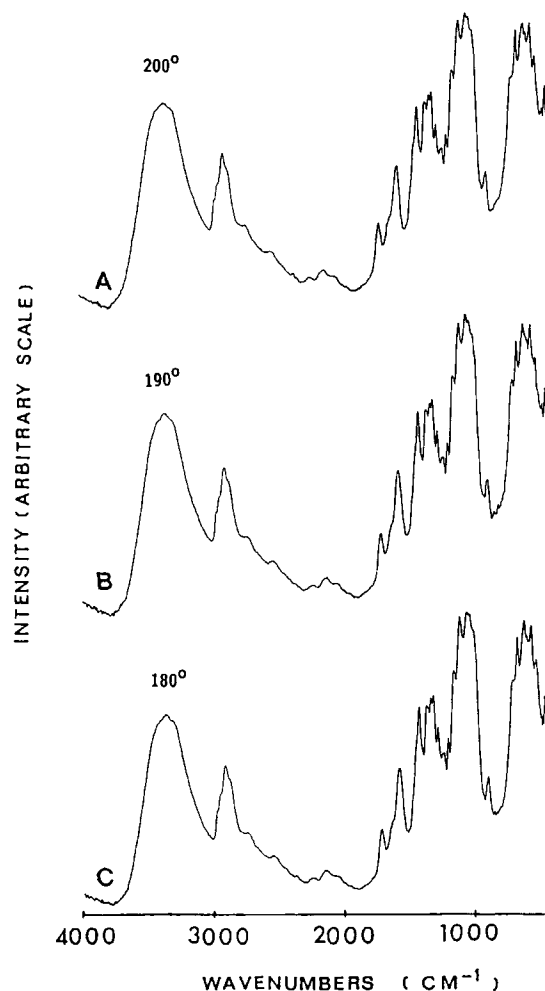


Figure 5 Photoacoustic infrared spectra of the cotton fabrics finished with 6.4% maleic acid and 3.7% NaH_2PO_4 and cured at different temperatures: (A) 200°C; (B) 190°C; (C) 180°C. The fabrics were treated with a 0.1M NaOH solution for 2 min at room temperature.

Table IV The Ester Carbonyl Band Intensities, the Carbonyl Band Intensity Ratios, and the DP Ratings of the Cotton Fabrics Treated with Maleic Acid/ NaH_2PO_4 and Cured for 60 s

	Curing Temperature (°C)		
	200	190	180
Ester carbonyl band (1725 cm^{-1}) intensity	0.36	0.34	0.32
Carbonyl band intensity ratio (1725 cm^{-1} / 1576 cm^{-1})	0.55	0.50	0.49
DP rating	2.5	2.5	3.0

such as maleic acid, once the first carboxyl group in an acid molecule is esterified, the second one is unable to form an anhydride. The esterification of the second carboxyl group has a much lower reaction rate than does the first one. This explains why an increase in curing temperature from 180°C to 200°C has little effect on the carbonyl band intensity ratio (ester/carboxylate).

The Effects of Curing Time

The cotton fabrics treated with 6.3% BTCA and 3.7% NaH_2PO_4 were cured at 190°C for different time periods. The cured fabrics were first treated in a 0.1M NaOH solution for 2 min at room temperature and then analyzed by FTIR/PAS. The ester carbonyl intensity and the carbonyl band intensity ratio (ester/carboxylate) are summarized in Table V. Both the ester carbonyl band intensity and the carbonyl band intensity ratio increase as curing time is increased. It is expected that an increase in curing time would improve the effectiveness of BTCA for cross-linking cotton cellulose, because a longer cur-

Table V The Ester Carbonyl Band Intensities, the Carbonyl Band Intensity Ratios, and DP Ratings of the Cotton Fabrics Treated with BTCA/ NaH_2PO_4 and Cured at 190°C

	Curing Time (s)		
	60	30	15
Ester carbonyl band (1725 cm^{-1}) intensity	0.58	0.54	0.45
Carbonyl band intensity ratio (1725 cm^{-1} / 1576 cm^{-1})	0.60	0.56	0.49
DP rating	3.3	3.5	3.0

ing time period permits more ester cross-linkages to be formed. However, the DP ratings in Table V show a fluctuation as the curing temperature was raised. Although DP rating is only a visual estimation of the final performance of a finished fabric, FTIR spectroscopy is able to examine the ester cross-linking on a molecular level. Thus, FTIR spectroscopy provides a more accurate measurement of the cross-linking in cotton fabrics. Infrared spectroscopy definitely complements the existing testing methods for evaluating finished fabrics, such as DP rating and wrinkle-recovery angle.

Presented in Table VI are the carbonyl band intensities and the carbonyl band intensity ratios (ester/carboxylate) for the cotton fabrics treated with 6.4% maleic acid/3.7% NaH_2PO_4 and cured at 190°C for different times. The data in Table VI show that an increase in curing time has little effect on the effectiveness of maleic acid as a cross-linking agent in the finished cotton fabrics. This can also be explained by the esterification mechanism of polycarboxylic acids discussed above. For a bifunctional carboxylic acid such as maleic acid, the esterification of the second carboxyl in its molecule has a much lower reaction rate than does the first carboxyl, which is able to form a cyclic anhydride with cotton cellulose. Therefore, an increase in curing time from 15 s to 60 s has little effect on the average number of ester groups formed for each maleic acid molecule.

The photoacoustic infrared spectra of the BTCA-treated cotton fabrics cured at 190°C for different time periods are presented in Figure 6. The fabrics were not treated in a 0.1M NaOH solution. It could be seen that an increase in curing time resulted in a dramatic increase in the 1725 cm^{-1} band intensity and a decrease in the 1576 cm^{-1} band intensity, indicating that the concentration of carboxyl, and

Table VI The Ester Carbonyl Band Intensities, the Carbonyl Band Intensity Ratios, and DP Ratings of the Cotton Fabrics Treated with Maleic Acid and Cured at 190°C

	Curing Time (s)		
	60	30	15
Ester carbonyl band (1725 cm^{-1}) intensity	0.34	0.34	0.32
Carbonyl band intensity ratio (1725 cm^{-1} / 1576 cm^{-1})	0.48	0.47	0.50
DP rating	2.5	2.5	2.0

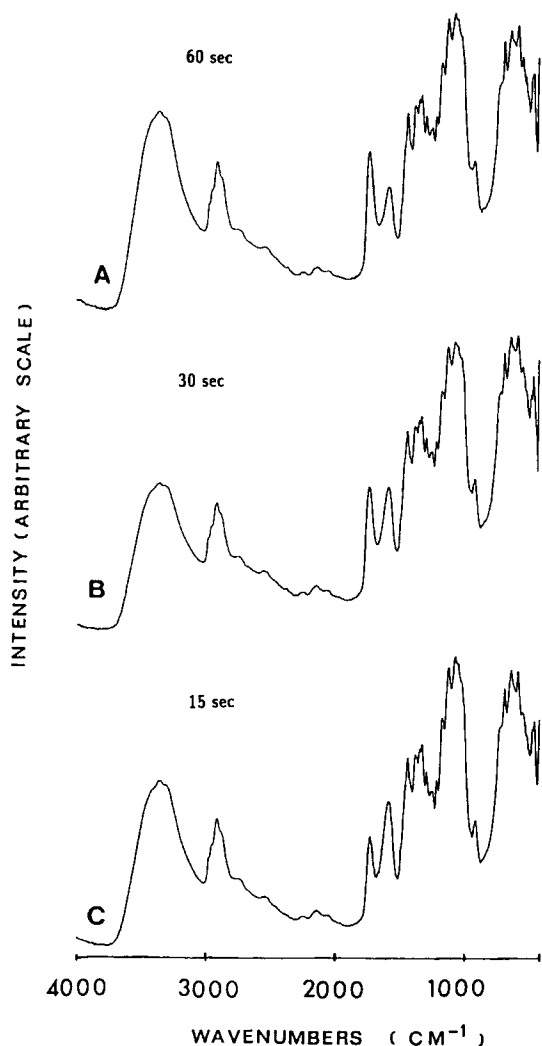


Figure 6 Photoacoustic infrared spectra of the cotton fabrics finished with 6.3% BTCA and 3.7% NaH_2PO_4 and cured at 190°C for different time periods: (A) 60 s; (B) 30 s; (C) 15 s.

therefore the acidity in the near-surfaces of the finished fabrics, was increased when the curing time was increased. Studies of the cotton fabric treated with tricarballic acid/ NaH_2PO_4 showed a similar result.

CONCLUSIONS

1. An increase in acid concentration and a corresponding increase in catalyst concentration improves the effectiveness of BTCA as a cross-linking agent in the finished cotton fabrics.
2. An increase in curing temperature or in curing time improves the effectiveness of BTCA and tricarballic acid for cross-linking cotton cellulose. However, it has little influence on

the effectiveness of dicarboxylic acids such as maleic acid in the treated cotton fabrics.

3. Infrared spectroscopy appears to be useful for evaluating the effectiveness of polycarboxylic acids in the finished fabrics and definitely complements the existing methods for evaluating finished textile fabrics.

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