

Physical Properties and Reaction Kinetics of Cotton Cellulose Crosslinked with Dimethyloldihydroxyethyleneurea–Maleic Acid

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ABSTRACT: Two dicarboxylic acids were used to join with dimethyloldihydroxyethyleneurea (DMDHEU) as crosslinking agents to treated cotton fabrics. These results reveal that the dry crease recovery angle values of the treated fabrics for DMDHEU–maleic acid are higher than those for DMDHEU–tartaric acid at a given wet crease recovery angle and tensile strength retention. The IR spectra show that the reaction between the –OH of DMDHEU and cellulose and the vinyl groups of maleic acids occurred. The cross section of the DMDHEU–maleic acid and DMDHEU–tartaric acid treated fibers and the energies of activation and

other data of reaction kinetics for DMDHEU–maleic acid and DMDHEU–tartaric acid strongly suggest that the reaction of vinyl groups of maleic acid with cellulose molecules can take place during the pad/dry-cure process. Additionally, the surface distribution of crosslinking agent on the finished fabrics for DMDHEU–maleic acid is slightly lower than that for DMDHEU–tartaric acid. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 92: 3886–3893, 2004

Key words: crosslinking; kinetics (polym.); activation energy; catalysts; infrared spectroscopy

INTRODUCTION

Some previous studies have shown that carboxylic acid groups of polycarboxylic acid^{1–3} or acrylic acid⁴ can react with cellulose to improve the crease recovery property of the finished fabrics. Our recent study⁵ showed that the carboxylic acid of the α -amino acids, which were coreactants of crosslinking reagent, can react with the hydroxyl group of the *N*-methylol crosslinking agent [dimethyloldihydroxyethyleneurea (DMDHEU)] to affect the physical properties of the treated fabrics.

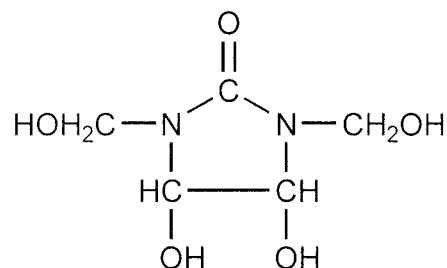
In this study, we were interested in the effects of the vinyl group of the polycarboxylic acid, joined with the *N*-methylol compound together to treat the cotton fabric samples, on the physical properties of the treated fabrics and the reaction kinetics. The DMDHEU and two dicarboxylic acid compounds (one is the vinyl group contained in maleic acid and the other is the nonvinyl group contained in tartaric acid) were used as the crosslinking agent and coreactants, respec-

tively. Here, we expected that the reaction between the vinyl group of polycarboxylic acid and the hydroxyl group of cellulose and/or DMDHEU, during a pad/dry-cure process, would change the physical properties and the reaction kinetics of those finished cotton fabrics.

EXPERIMENTAL

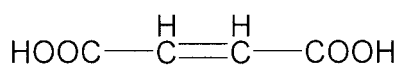
In this study, we used desized, scoured, and bleached cotton fabric 20s × 20s end (80) and picks (80).

The crosslinking agent used was DMDHEU, represented structurally as

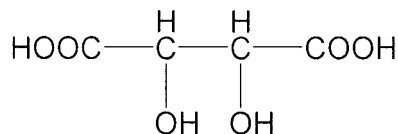


Dicarboxylic acids were maleic acid and tartaric acid, with the following structures:

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Maleic Acid



Tartaric Acid

Aluminum sulfate was reagent grade, as were the other chemicals.

The cotton fabric samples were padded twice to about 90% wet pickup, with freshly prepared (1, 2, 4, and 6 w/w) aqueous solutions of the mixtures of DMDHEU/dicarboxylic acids (weight ratio of 2/1) and the aluminum sulfate catalyst. The catalyst concentration was 0.12 times the crosslinking agent solid concentration. Padded fabrics were dried at 80°C for 5 min, cured at 130°C for 3 min, soaped, washed, and dried. It is believed that the finished fabric samples prepared under these conditions were relatively sufficient.^{6,7}

Tensile strength of the warp yarns was measured on an Instron tensile tester (Instron, Canton, MA). ASTM standard method D 1295-67 was used to determine both dry and wet crease recovery angles. Nitrogen determinations were made using the Kjeldahl method.

Infrared spectra of the samples were obtained using a KBr disk technique.⁸ Samples were prepared to give a dry weight of 1.8 mg after storage in 1-dram vials over P₂O₅ for 3 days. Spectral-grade KBr (250–300 mg) was ground, transferred to individual sample vials, dried in an oven at approximately 200°C for several hours, and stored in an oven at 110°C. Samples were ground and mixed with the KBr and pressed in an evacuated die under suitable pressure. One set consisted of (1) DMDHEU, (2) maleic acid, and (3) cured DMDHEU–maleic acid [1 : 1 mole ratio of DMDHEU to maleic acid was mixed with 12% (w/w) of ammonium sulfate, then coated on a piece of glass, dried at 80°C for 5 min, and cured at 160°C for 3 min]. The second set consisted of (1) polyvinyl alcohol (because cellulose powder is not soluble in water, polyvinyl alcohol was selected as a model of cotton cellulose), (2) maleic acid, and (3) cured methyl cellulose–maleic acid [1 : 1 mole ratio of polyvinyl alcohol repeat unit to maleic acid was mixed with 12% (w/w) of ammonium sulfate, then coated on a piece of glass, dried at 80°C for 5 min, and cured at 160°C for 3 min]. Each of the samples and KBr was mixed, ground, and dried in 1-dram vials over P₂O₅ for 3 days. The samples were then pressed in an evacuated die under suitable pressure. A Fourier transform infrared spectrophotometer (Jasco model FT/IR-3; Jasco, Tokyo, Japan) was used to obtain spectra.

For the investigation of agent distribution on the treated fabrics, the various treated fabric samples were

dyed with C.I. Direct Red 81 (Polysciences Inc., Warrington, PA) at 80°C for 50 min (dye concentration: 0.1 g/L, NaCl: 10 g/L, liquor ratio = 1 : 100). The *K/S* values (depth of color of the various treated fabrics) were measured by a ChromaSensor CS-5 applied color system (D&D Textilvertriebs GmbH, Cadolzburg, Germany), and the [*D*]_f values (g dye/kg cotton) were determined spectrophotometrically with a U-3010 spectrophotometer (Hitachi, Osaka, Japan) after extraction of the dyed fabrics with pyridine/water (1/3) at 60°C.

Treated fibers were brought to the boil in a 50% (by volume) aqueous solution of methanol containing 0.5% wetting agent (Triton X-100) for 1 h and allowed to cool and soak overnight in this wetting solution. Then the wet fibers were laid on a glass slide and covered immediately with nitrocellulose, which had been dissolved in acetone. The cross sections of the treated fibers were observed by a scanning electron micrograph.

For reaction kinetic studies, the cotton fabric samples were padded twice to about 90% wet pickup with freshly prepared DMDHEU–dicarboxylic acids [0.24M; the mole ratio of DMDHEU to dicarboxylic acids was 2 : 1 in the presence of the aluminum sulfate catalyst (0.024M)]. To obtain the bound nitrogen contents (of the heated fabrics), which changed with the change of heating temperatures (in these heated cases, they are not the fully cured conditions), padded fabric samples were heated at 80, 90, and 100°C for different time intervals, soaped, washed, and dried.

RESULTS AND DISCUSSION

Physical properties

The nitrogen content (N content), dry crease recovery angle (DCRA), wet crease recovery angle (WCRA), and tensile strength retention (TSR) of the finished fabrics are given in Table I, which shows that the N content, DCRA, and WCRA values for the various fabric samples gradually increase with increased crosslinking agent, but TSR values decrease in all cases. At a given resin concentration, the DCRA and WCRA values for the DMDHEU–dicarboxylic acid crosslinking agents are in the rank of DMDHEU–maleic acid > DMDHEU–tartaric acid; however, the TSR and nitrogen content values are in the inverse rank.

TABLE I
Physical Properties of Cotton Fabrics Treated with DMDHEU–Maleic Acid and DMDHEU–Tartaric Acid

Dicarboxylic acid	Dicarboxylic acids concentration (%)	DMDHEU concentration (%)	Nitrogen (%)	Properties of finished fabrics		
				Dry crease recovery angle, W + F (°C)	Wet crease recovery angle W + F (°C)	Tensile strength retention (%)
—	—	—	—	192	172	100
Maleic acid	1/3	2/3	0.127	245	200	92
	2/3	4/3	0.267	278	213	64
	4/3	8/3	0.401	290	238	51
	6/3	12/3	0.535	306	258	38
Tartaric acid	1/3	2/3	0.155	236	192	93
	2/3	4/3	0.281	253	207	72
	4/3	8/3	0.414	280	230	56
	6/3	12/3	0.561	293	252	42

The higher DCRA and WCRA, and the lower TS and N content for DMDHEU–maleic acid, may be caused by the presence of maleic acid's vinyl group.

The relationships between DCRA, WCRA, and TSR of the finished fabrics with the two crosslinking agent systems are shown in Figures 1, 2, and 3, respectively. From the relationships between DCRA and WCRA of the finished fabrics (Fig. 1), we find that the DCRA values of the treated fabrics for DMDHEU–maleic acid are higher than those for DMDHEU–tartaric acid at a

given WCRA. Figure 2 shows the plots of DCRA versus TSR of the finished fabrics. For a given value of TSR, the DCRA values of the treated fabrics for DMDHEU–maleic acid are higher than those for DMDHEU–tartaric acid. The plots of WCRA against TSR of the finished fabrics shown in Figure 3 reveal that the WCRA values of the treated fabrics are similar to those for DMDHEU–maleic acid and DMDHEU–tartaric acid at the same TSR. The higher DCRA of the DMDHEU–maleic acid treated fabrics may be caused by the

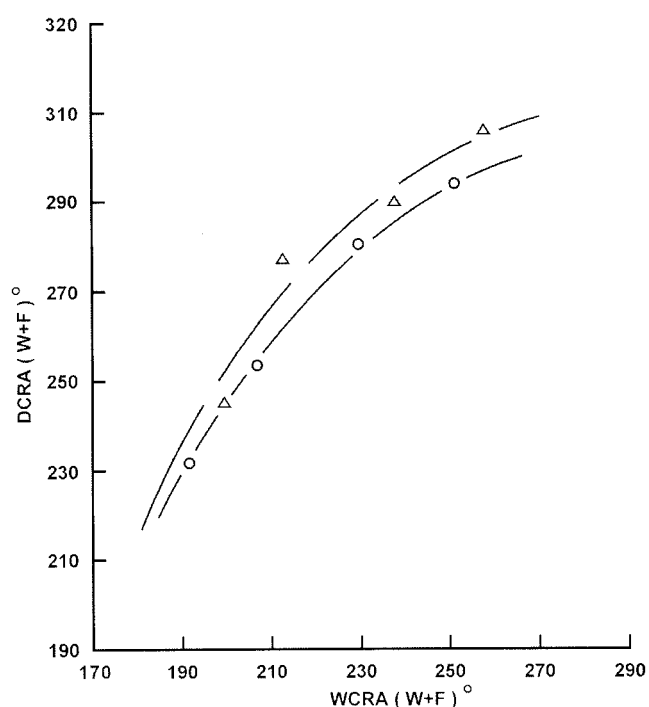


Figure 1 Relationship between DCRA and WCRA of the fabric samples treated with DMDHEU–maleic acid (Δ) and DMDHEU–tartaric acid (\circ).

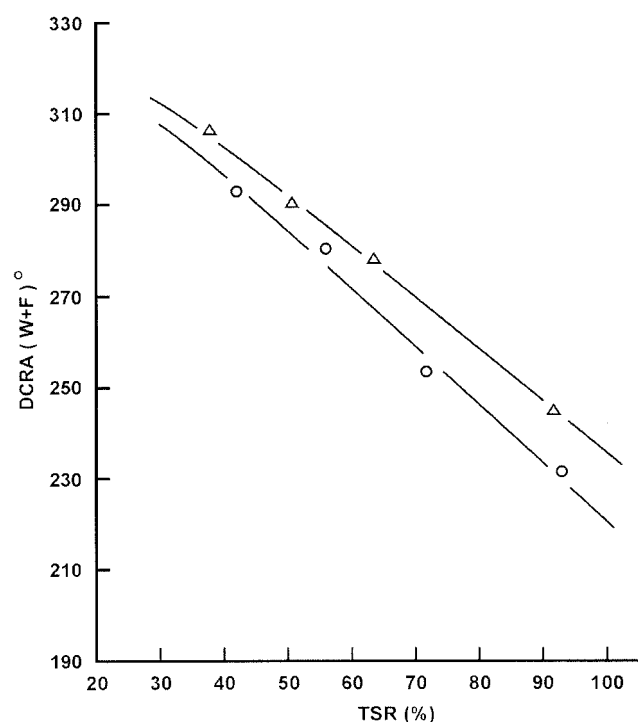


Figure 2 Relationship between DCRA and TSR of the fabric samples treated with DMDHEU–maleic acid (Δ) and DMDHEU–tartaric acid (\circ).

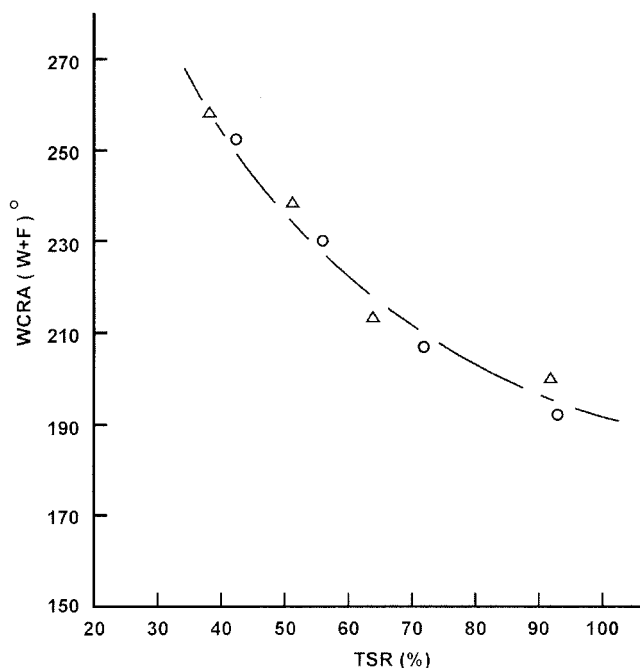


Figure 3 Relationship between WCRA and TSR of the fabric samples treated with DMDHEU-maleic acid (Δ) and DMDHEU-tartaric acid (\circ).

reaction between the hydroxyl group of DMDHEU and cellulose and the vinyl group of maleic acid. Our recent study⁹ revealed that the vinyl group of crosslinking agent systems could react with the hydroxyl group of cellulose and DMDHEU.

Interaction between the components

To confirm the crosslinking reaction between DMDHEU and the vinyl group of maleic acid molecule, DMDHEU was used to react with maleic acid under a pad/dry-cure process in the presence of ammonium sulfate as catalyst. The IR spectra of DMDHEU, maleic acid, and cured DMDHEU-maleic acid are shown in Figure 4(a), (b), and (c), respectively. The interesting and important absorbing bands are those of $-CH_2OH$ ($1027, 1077\text{ cm}^{-1}$)¹⁰ for DMDHEU [Fig. 4(a)], and the vinyl group (947 cm^{-1}) of maleic acid [Fig. 4(b)]. We find that the IR spectrum for cured DMDHEU-maleic acid [Fig. 4(c)] almost disappears at 947 cm^{-1} (vinyl group of maleic acid), but generates two new absorbing bands of 1156 and 1363 cm^{-1} . Additionally, 1027 and 1077 cm^{-1} are shifted to 1017 and 1065 cm^{-1} , respectively. From the description of Silverstein et al.,¹⁰ we determined that the new absorbing bands of

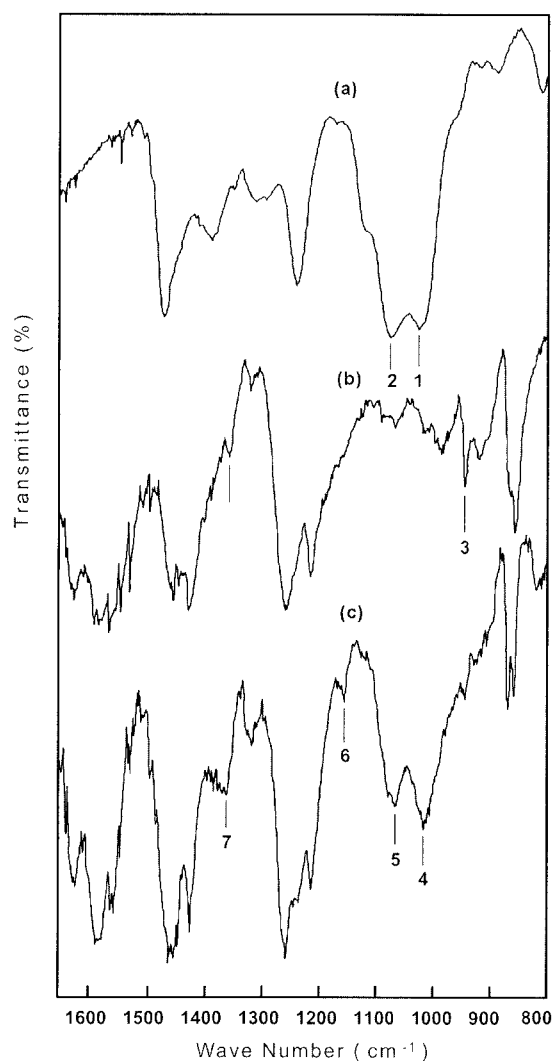
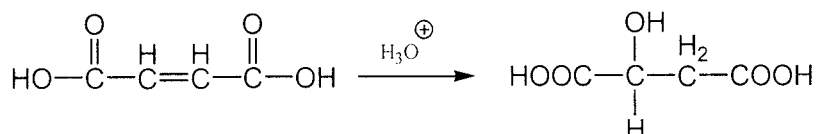
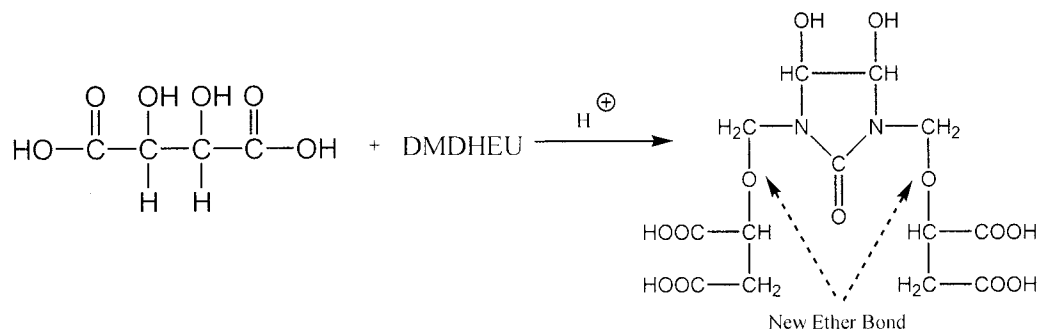


Figure 4 IR spectra of (a) DMDHEU, (b) maleic acid, and (c) cured DMDHEU-maleic acid. 1, 1027 cm^{-1} ; 2, 1077 cm^{-1} ; 3, 947 cm^{-1} ; 4, 1017 cm^{-1} ; 5, 1065 cm^{-1} ; 6, 1156 cm^{-1} ; 7, 1363 cm^{-1} .

1363 cm^{-1} are attributed to the ester group, and that of 1156 cm^{-1} is attributed to aliphatic ether. The formation of the new ether group at 1156 cm^{-1} strongly suggests that the reaction between DMDHEU and maleic acids can occur during the pad/dry-cure process. This reaction is likely attributable to the fact that the double bond of maleic acid is hydrolyzed to form hydroxysuccinic acid [eq. (1)] under drying condition and then to react with DMDHEU [eq. (2)]. (The ester group at 1363 cm^{-1} is suggested to be formed from the reaction between carboxylic acid of maleic and $-OH$ of DMDHEU.)





IR spectra of polyvinyl alcohol (PVA), maleic acid, and cured PVA-maleic acid are shown in Figure 5(a), (b), and (c), respectively. The interesting and important absorbing bands are those of -C-O- of alcohol (1097 cm^{-1})¹⁰ for PVA [Fig. 5(a)] and the vinyl group (947 cm^{-1}) of maleic acid [Fig. 5(b)]. We find that the IR spectrum for cured PVA-maleic acid [Fig. 5(c)] obviously decreases at 947 cm^{-1} (vinyl group of ma-

leic acid), but generates two new absorbing bands of 1093 and 1099 cm^{-1} , believed to be attributed to the aliphatic ether group. These results support the reaction between the -OH of polyvinyl alcohol and the vinyl groups of maleic acids.

Distribution of crosslinking agents

The K/S and $[D]_f$ values of the fabrics treated with DMEU-DMDMe3EU, DMEU/DMDMe3EU, and DMDMe3EU/DMEU processes (combined ratio of DMEU to DMDMe3EU is 5:5) are listed in Table II, and are plotted in Figure 5 according to the method described by Rowland et al.,^{11,12} and the linear relationships are similar to their results.¹² K is the coefficient of absorption, S is the coefficient of scattering, K/S is the color intensity calculated from the equation $K/S = (K/S)_{\text{dye}} - (K/S)_{\text{white}}$, and $[D]_f$ (g dye/kg cotton) is the dye content of the finished fabric, determined spectrophotometrically after extraction with pyridine/water (1/3) at 60°C . Color intensity is an inverse measure of crosslinking on fabric surfaces, and dye content is an inverse measure of total crosslinking content through fabric thickness. K/S results are averaged for the two sides of the fabric samples.

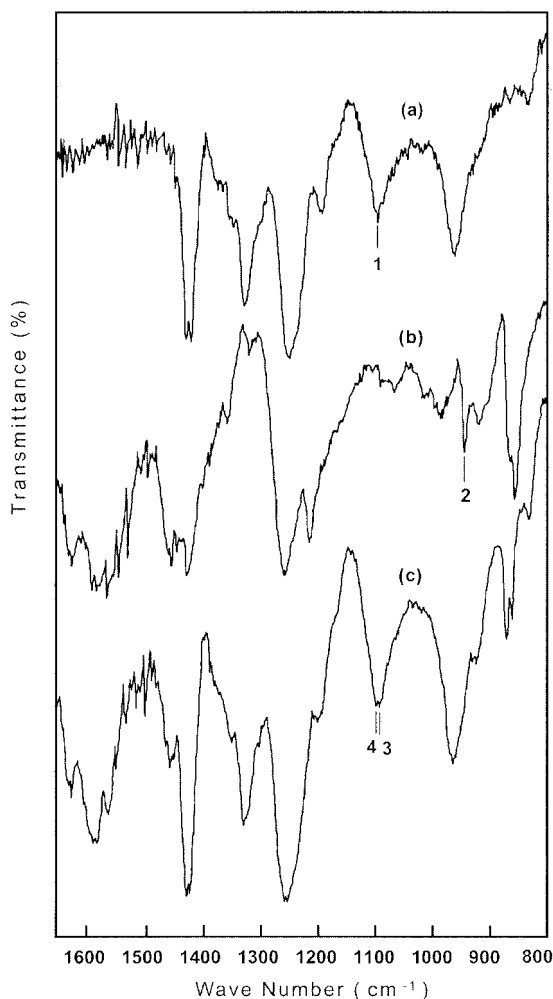


Figure 5 IR spectra of (a) PVA, (b) maleic acid, and (c) cured PVA-maleic acid. 1, 1097 cm^{-1} ; 2, 947 cm^{-1} ; 3, 1093 cm^{-1} ; 4, 1099 cm^{-1} .

TABLE II
 $[D]_f$ and K/S Values of Cotton Fabric Samples Treated with DMDHEU-Maleic Acid and DMDHEU-Tartaric Acid at a Mole Ratio of 2 to 1 Under Different Conditions

Dicarboxylic acid	Dicarboxylic acids concentration (%)	DMDHEU concentration (%)	$[D]_f$, g dye/kg cotton	K/S
—	—	—	18.42	4.23
Maleic acid	1/3	2/3	7.91	1.88
	2/3	4/3	2.85	0.75
	4/3	8/3	1.48	0.33
	6/3	12/3	1.19	0.28
Tartaric acid	1/3	2/3	9.05	2.03
	2/3	4/3	4.03	0.87
	4/3	8/3	2.02	0.46
	6/3	12/3	1.52	0.32

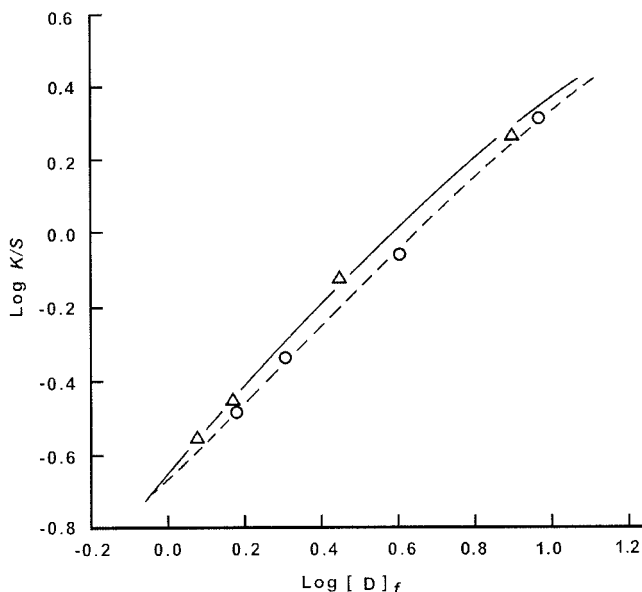


Figure 6 Plots of $\log K/S$ versus $\log [D]_f$ for fabric samples treated with DMDHEU-maleic acid (Δ) and DMDHEU-tartaric acid (\circ).

For a negative staining condition, agent distribution follows the rule that the lower the value of K/S at a specific level of dye content and the lower the dye fixation on the surface of the fabric, the higher the concentration of crosslinks on the surface of the fabric and the greater the agent distribution on the fabric. Figure 6 reveals lower values of $\log K/S$ for DMEU/DMDMe3EU-treated fabrics at the same value of $\log [D]_f$; that is, the surface distribution of crosslinking agent on the finished fabrics for DMDHEU-tartaric acid is slightly higher than that for DMDHEU-maleic acid. The lower surface distribution for DMEU-maleic acid may be caused by the reaction between cellulose molecule and maleic acid to decrease the migration of agents on the treated fiber.

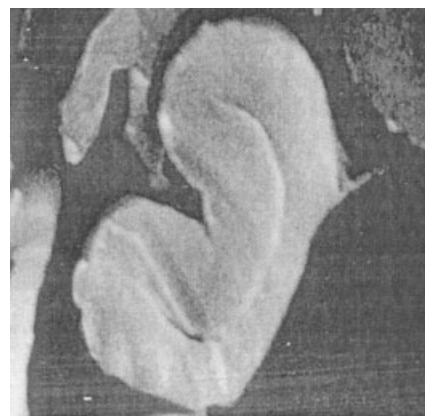
SEM micrographs of the expansion patterns of cross sections of fibers treated with 5% DMDHEU-maleic acid and 5% DMDHEU-tartaric acid are shown in Figure 7(a) and (b), respectively. These figures show that the expansion degree of the DMDHEU-maleic acid treated fiber is lower than that of the DMDHEU-tartaric acid treated fiber. This result agrees with the phenomenon that the crosslinking reactions between DMDHEU-maleic acid and DMDHEU-tartaric acid and cellulose molecules are different from each other, which is suggested to be caused by the presence of maleic acid's vinyl group.

Reaction kinetics

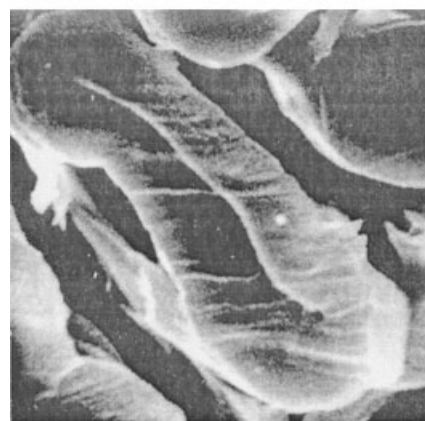
The data in Figure 8(a) and (b) show the changes in nitrogen content of cotton fabrics treated with DMDHEU-maleic acid and DMDHEU-tartaric acid, respec-

tively. All samples were catalyzed by aluminum sulfate with heating time in minutes. The figures reveal that the nitrogen contents of the treated fabrics increase with increase of heating time for all cases. Here, we can see the initial, slightly concave upward shape of the relationships between nitrogen content and the initial curing time in all cases, which we attribute to the time needed to raise the temperature of the fabric in the oven.¹³

Semilogarithmic plots of $(\%N_0 - \%N)/\%N_0$ against heating time in minutes are shown in Figure 9(a) and (b) for DMDHEU-maleic acid and DMDHEU-tartaric acid, respectively, where $\%N_0$ is the nitrogen content of the treated fabric after a pad/dry-cure process (i.e., 90% wet pickup, dried at 80°C for 5 min, cured at 135°C for 3 min; the samples are thought to be fully cured under these conditions^{6,13}). $\%N$ is the bound nitrogen after a given time interval at the tem-



(a)



(b)

Figure 7 SEM micrographs showing expansion patterns of cross sections of (a) 5% DMDHEU-maleic acid treated fiber ($\times 5000$) and (b) 5% DMDHEU-tartaric acid treated fiber ($\times 5100$), respectively.

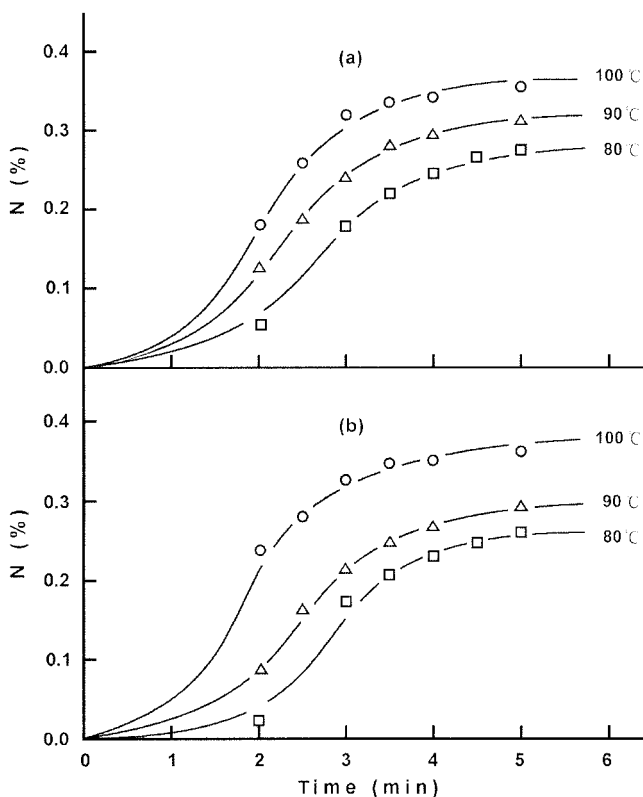


Figure 8 Changes in nitrogen content of fabrics treated with 0.24M DMDHEU-maleic acid (a) and DMDHEU-tartaric acid (b) in the presence of 0.24M ammonium sulfate as catalyst versus curing time in minutes (○, 100°C; △, 90°C; □, 80°C).

perature indicated (80, 90, and 100°C). We obtained straight lines for all cases, the linearity indicating that the reaction is pseudo-first-order for the time shown.

Table III shows data calculated from the semilogarithmic plots using least-squares analysis for each data set at 80, 90, and 100°C. Based on pseudo-first-order reaction kinetics, the specific reaction rate constants (k) were calculated from the slopes of lines. The k values shown in Table III reveal that the rate constants for DMDHEU-maleic acid are higher than those for DMDHEU-tartaric acid at all heating temperatures. The higher specific reaction rate constant may be contributed by the additional reaction between the vinyl group of maleic acid and the hydroxyl group of cellulose and DMDHEU. Our recent study⁵ revealed the crosslinking reaction between vinyl groups of alkyl diallyl ammonium salts and cellulose.

Figure 10 shows the semilogarithmic Arrhenius plots of the specific reaction rate constants. Lines were determined using the least-squares method and activation parameters for the various crosslinking agent systems are recorded in Table III, using the method of Ziifile et al.¹⁴ Free energies of activation (ΔG^*) based on nitrogen for each fabric were positive. The energies needed for the crosslinking for DMDHEU-tartaric

acid are slightly higher than those for DMDHEU-maleic acid. The positive enthalpies (ΔH^*) of activation for each fabric indicate that the transition state complex is endothermic. The enthalpies of activation for DMDHEU-maleic acid are significantly higher than those for DMDHEU-tartaric acid. These results suggest that the transition state complex is obviously different for DMDHEU-maleic acid and DMDHEU-tartaric acid. Entropies (ΔS^*) of activation for both samples are negative, and are ranked as: DMDHEU-maleic acid > DMDHEU-tartaric acid. The entropies of activation are slightly affected by the existence of vinyl group in the DMDHEU-maleic acid. These data again support that the crosslinking between DMDHEU-maleic acid and cellulose molecules is different from that between DMDHEU-tartaric acid and cellulose molecules, which indicates the vinyl group of maleic acid can participate in the crosslinking reaction. The data of reaction kinetics again suggest that the reaction between the vinyl group of maleic acid and cellulose molecules occurs in the crosslinking reaction during the pad/dry-cure process.

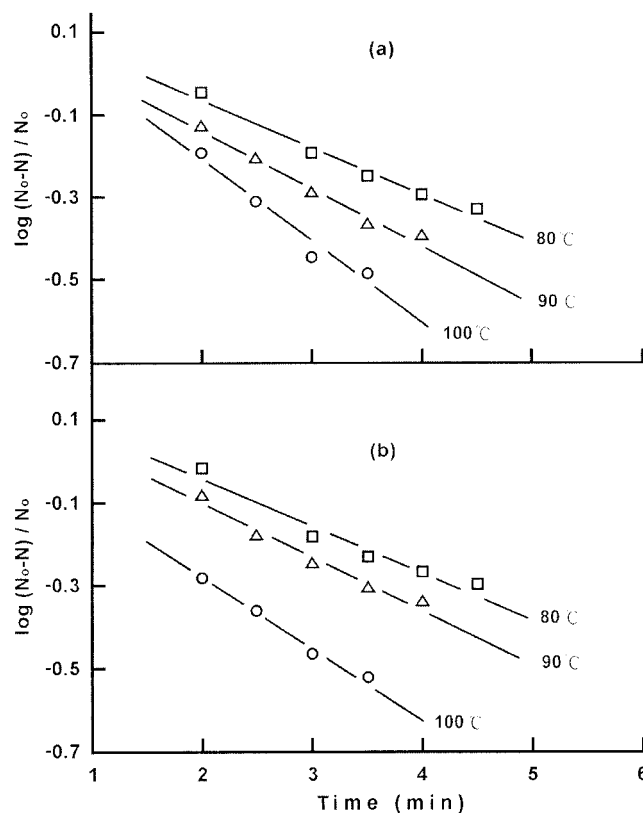


Figure 9 Semilogarithmic graphs of $(\%N_0 - \%N)/\%N_0$ versus curing time in minutes for fabrics treated with 0.24M DMDHEU-maleic acid (a) and DMDHEU-tartaric acid (b) in the presence of 0.24M ammonium sulfate as catalyst versus curing time in minutes (○, 100°C; △, 90°C; □, 80°C).

TABLE III
Reaction Rate Constants and Activation Parameters of Cotton Fabric Samples Treated with DMDHEU–Maleic Acid and DMDHEU–Tartaric Acid at a Mole Ratio of 2 to 1 Under Different Conditions

Crosslinking agent (M)	Heating temperature (°C)	$k' \times 10$ (min ⁻¹)	ΔH^* (kcal/mol)	ΔS^* (cal/°K ⁻¹ /mol)	ΔG^* (kcal/mol)
DMDHEU–maleic acid	80	0.262	7.111	-33.246	18.847
	90	0.319	7.091	-33.522	19.260
	100	0.467	7.071	-33.397	19.528
DMDHEU–tartaric acid	80	0.256	6.026	-36.369	18.865
	90	0.293	6.006	-36.682	19.322
	100	0.378	5.987	-36.726	19.685

CONCLUSIONS

In this study, we used two dicarboxylic acids (maleic acid and tartaric acid) to join with DMDHEU as the crosslinking agents to treat cotton fabric samples. The DCRA and WCRA values for DMDHEU–maleic acid were higher than those for DMDHEU–tartaric acid; however, the TSR and nitrogen content values were in the inverse at a given resin concentration. The IR spectra strongly support the reaction between the –OH of DMDHEU and cellulose and the vinyl group of maleic acid. The surface distribution of crosslinking agent on the finished fabrics for DMDHEU–tartaric acid was slightly higher than that for DMDHEU–ma-

leic acid. The cross section of the DMDHEU–maleic acid treated fiber was smaller than that of DMDHEU–tartaric acid, which is also suggested to be caused by the reaction of vinyl group of maleic acid with cellulose molecules. The values of reaction rate constant, enthalpy of activation, and entropy for DMDHEU–maleic acid were significantly higher than those for DMDHEU–tartaric acid. These results suggest that the transition state complex is obviously different for DMDHEU–maleic acid and DMDHEU–tartaric acid. Additionally, the free energies of activation (ΔG^*) based on nitrogen for each treated fabric were positive, and the energies needed for the crosslinking for DMDHEU–tartaric acid were slightly higher than those for DMDHEU–maleic acid. The reaction between the vinyl group of maleic acid and cellulose molecules that can occur in the crosslinking reaction during the pad/dry-cure process was thus proven.

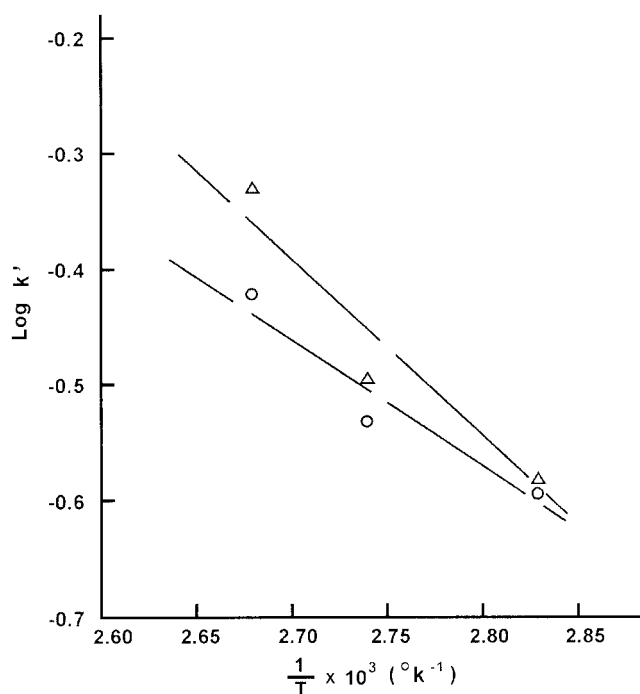


Figure 10 Plots of $\log k'$ based on nitrogen versus reciprocal of absolute temperature: Δ , DMDHEU–maleic acid; \circ , DMDHEU–tartaric acid.

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