Crosslinking Structures and Dyeing Kinetics of Cotton Cellulose Treated with a Steeped Process

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ABSTRACT: A steeped procedure was introduced to the durable press finishing process in this study to study some physical properties, the crosslinking structure, and the pore structure. The results showed that the values of the dry and wet crease recovery angles of fabrics treated with the steep–pad–dry–cure process were higher than those of fabrics treated with the pad–dry–cure process, and those phenomena increased with higher steeped temperatures and longer times. The lengths of the crosslinks for the steep–pad–dry–cure process were higher than those for the pad–dry–cure process at a given number of crosslinks per anhydroglucose unit. The fabrics treated with the steep–pad–dry–cure process had higher values of the dye absorption, rate constant, and structural diffusion resistance constant than those treated with the pad–dry–cure process at the same dyeing temperature; however, they had lower values of the equilibrium absorption and activation energy than those treated with the pad–dry–cure process. The results of thin-layer chromatography, H-NMR, ^{13}C-NMR, and IR analyses suggested that self-condensation between the crosslinking agents occurred during the steeped procedure. Additionally, the steep–pad–dry–cure-treated fabric had more inner agent distribution than the pad–dry–cure-treated fabric. © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 98: 2555–2562, 2005

Key words: activation energy; adsorption; crosslinking; resins

INTRODUCTION

Some previous studies[1–3] have shown that the finishing process, the swelling state of the crosslinked fibers, and the crosslinking agents can affect the crease recovery properties of crosslinked fabrics. Our previous report[4] showed that some physical properties were improved by the polymerization of crosslinking agents in the treated fabrics. Franklin et al.[5] revealed that the use of dimethyloldihydroxyethyleneurea and acrylic acid (AA) under a redox catalyst system could improve some physical properties of the finished cotton fabrics. They suggested that the polymerization occurred between the *N*-methylol compound and AA during the curing process.

It is interesting to us that the dry and wet crease recovery properties of treated fabrics increase with the use of the steeped procedure before the traditional pad–dry–cure process for a longer time and higher temperature. Detailed information about the crosslinking structures and pore structures of fabrics treated with the steeped procedure is highly interesting to us.

Self-condensation during the steeped procedure was confirmed with IR, ^{1}H-NMR, and ^{13}C-NMR spectra and thin-layer chromatography (TLC), and we also examined the crosslinking of cotton fabrics with the steep–pad–dry–cure and pad–dry–cure processes with respect to the nitrogen and formaldehyde content, number of crosslinks per anhydroglucose unit (CL/AGU), and length of the crosslinks (CL length) of the treated fabrics.

Also, Color Index (C. I.) Direct Red 81 with a molecular weight of 676 g/mol was used to study the pore structures of the steeped-procedure-treated fabrics. We studied the pore structure by using the following diffusion equation:[6,7]

$$a = a_e[1 - \exp(-kC_0 t)]^n = a_e[1 - \exp(-k't)]^n \quad (1)$$

where a is the absorption of the dye on cotton in time t, a_e is the equilibrium absorption at $t \rightarrow \infty$, k is the specific rate constant, k' is the rate constant, C_0 is the concentration of cotton in a finite bath, and n is the structural diffusion resistance constant. The higher n is, the larger the pore size is of the treated fabric.

EXPERIMENTAL

In this study, we used desized, scoured, and bleached cotton fabric 20 s × 20 s ends (60) and picks (60).

The crosslinking agents were dimethyldimethyloldihydroxyethyleneurea (DMDMDHEU) and AA. DMDMDHEU was obtained from Cytec Industries, Inc. (Taipei, Taiwan) and had a solid content of 40%: AA, hydrogen peroxide, and other chemicals were all reagent-grade.
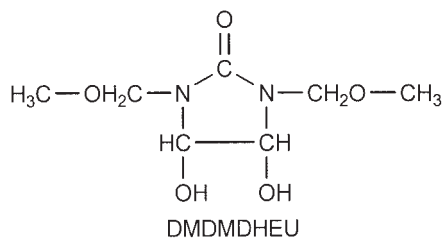
DMDMDHEU

The fabric samples were steeped with freshly prepared mixtures of DMDMDHEU (2, 4, 6, and 8% w/w) and AA at a molar ratio of 1 to 1 in the presence of hydrogen peroxide [the H_2O_2 initiator was 0.067% H_2O_2 (35% v/v)] under an environment of N_2 at different temperatures and for different times; then, the ammonium sulfate catalyst (0.1 × the amounts of DMDMDHEU) was added to the solutions, and they were stirred for about 3 min. This process proceeded in a glass container, which was placed in a water bath, with an N_2 inlet, condensing tube, and thermometer on top of this instrument. For a steeped time of 0 min, the fabric samples were dipped in the solutions contained the ammonium sulfate catalyst at the specific temperature for 3 min. The fabric samples were padded twice to about 80% wet pickup. The padded fabrics were dried at 80°C for 5 min, cured at 160°C for 3 min, soaped, washed, and dried.

Formaldehyde and nitrogen determinations were made with chromatropic acid[8] and kjeldahl methods, respectively. The tensile strength of the warp yarns was measured on an Instron tensile tester (Taichung, Taiwan). ASTM Standard D 1295-67 was used to determine the dry crease recovery angle (DCRA) and wet crease recovery angle (WCRA).

The self-condensation between the crosslinking agents was detected with the following methods. IR spectra of the samples were obtained with a KBr disk technique.[9] The solid residue of a mixture of DMDMDHEU and AA (1 : 1 mol/mol), which was stirred under an environment of N_2 at 70°C for 60 min, was obtained by the method of deep-freezing and vacuuming the solution to remove the water. Samples (solid residue, DMDMDHEU, and AA) were prepared to give a dry weight of 1.8 mg after storage in 1-dram vials over P_2O_5 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. The

samples were ground and mixed with KBr and pressed in an evacuated die under suitable pressure. AA and KBr were mixed, ground, and dried in 1-dram vials over P_2O_5 for 3 days, and then the samples were pressed in an evacuated die under suitable pressure. A Fourier transform infrared spectrophotometer (model FT/IR-3, Jasco, Tokyo, Japan) was used to obtain the spectra.

An NMR spectrometer (Avance 400, Bruker, Billerica, MA) provided H-NMR and ^{13}C-NMR spectra of DMDMDHEU alone and the residue of the mixture of DMDMDHEU and AA after the steeped procedure. The probe temperature was 25°C, and the solvent was dimethyl sulfoxide-d_6.

The TLC plate was a TLC aluminum sheet precoated with cellulose (0.1-mm-layer thickness), and the solvent was BEW (40 mL of n-butanol, 10 mL of ethanol, and 10 mL of water). The visualizing method was described by Moore and Babb.[10] The samples (a fresh mixture of DMDMDHEU and AA with a molar ratio of 1 to 1 and the residue of the mixture of DMDMDHEU and AA stirred under an N_2 environment at 70°C for 60 min) were applied to the TLC sheet as 2 μL of an aqueous solution, and the TLC sheet was then installed in a glass vessel to be developed for 30 min.

Two crosslinked cotton fabrics were cut into pieces of approximately 0.5 cm^2 and dyed with C. I. Direct Red 81 (dye concentration = 0.05 g/L, NaCl concentration = 10 g/L) at a 60 : 1 liquor ratio. The dyeing temperatures were 40, 50, and 60°C and were kept constant in a water bath. The dye solution and cotton fabric were stirred slowly with a magnetic stirrer. After selected time intervals, the dye concentration in the dye solution was measured directly on a Hitachi (Osaka, Japan) U-3010 spectrophotometer.

For the observation of the agent distribution on the treated fabrics, the various treated fabric samples were dyed with C. I. Direct Red 81 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 with a CS-5 Chroma-Sensor (Applied Color System, D&D Textilvetricles, GmbH, Cadolzburg, Germany), and the $[D]_f$ values (g of dye/kg of cotton) were determined spectrophotometrically with a U-3010 spectrophotometer (Hitachi) after the extraction of the dyed fabrics with pyridine/water (1 : 3) at 60°C.

RESULTS AND DISCUSSION

Table I shows DCRA, WCRA, and the tensile strength retention (TSR) of the fabrics treated under different steeped conditions and shows that the values of DCRA and WCRA of the fabrics treated with the steep–pad–dry–cure process are higher than the values of those treated with the pad–dry–cure process.

TABLE I
Physical Properties of Cotton Fabrics-Treated with a Mixture of DMDMDHEU and AA (8% DMDMDHEU and Equal Moles of AA) with the Steep–Pad–Dry–Cure Process Under Various Conditions

Steeped temperature (°C)	Steeped time (min)	Properties of finished fabrics			
		Nitrogen (%)	DCRA [(W + F)°]	WCRA [(W + F)°]	TSR (%)
—	—	—	182	162	100
50	0	0.73	291	279	52.5
	10	0.74	292	284	50.1
	20	0.74	294	285	48.7
	40	0.75	298	288	45.9
	60	0.77	302	291	42.3
70	0	0.74	284	274	54.1
	10	0.75	285	276	53.5
	20	0.76	288	278	51.7
	40	0.79	292	280	49.0
	60	0.81	296	283	45.9

The initiator used was 0.067% H_2O_2 (35% v/v).

The higher the steeped temperature and time are, the higher DCRA and WCRA are and the lower TSR is for the treated fabrics. Meanwhile, the nitrogen contents of the finished fabrics under different steeped conditions are also listed in Table I, which shows that the value with the steep–pad–dry–cure process at any specific steeped condition is higher than that with the pad–dry–cure process. The higher values of DCRA, WCRA, and the nitrogen content for the fabrics treated with the steeped procedure may be caused by changes in the crosslinking structure and distribution of the crosslinking agent on those treated fabrics.

The nitrogen and formaldehyde contents and the number and length of the crosslinks for crosslinked cotton fabrics with the steeped procedure with various resin concentrations are presented in Table II. As expected, the nitrogen and formaldehyde contents show a gradual increase with increasing resin content in the bath in all cases. At the same time, the values of the nitrogen content for the steep–pad–dry–cure process are higher than those for the pad–dry–cure process, but the values of the formaldehyde con-

tent are inversed at the same resin concentration (Fig. 1). These results imply that the crosslinking structure of the fabric treated with the steep–pad–dry–cure process differs from that of the fabric treated with the pad–dry–cure process.

The CL/AGU, CL length, and moles per anhydroglucose unit (AGU) of nitrogen and formaldehyde of the finished fabrics, listed in Table II and obtained with the methods of Frick et al.,[11,12] indicate that all increase as the concentration of the resin in the bath increases. Figure 2(a,b) shows the increase in the CL length and CL/AGU with the increase in the resin concentration in the bath separately. The curvilinear relationship between the CL length and CL/AGU for samples treated with the pad–dry–cure and steep–pad–dry–cure, shown in Figure 3, is similar to that reported in our previous study.[13] For a given number of CL/AGU, the values of the CL length for the steep–pad–dry–cure process are higher than those for the pad–dry–cure process. This may be attributed to the self-condensation of the crosslinking agents and other factors caused by the steep–pad–dry–cure process.

TABLE II
Structural Characteristics of Cotton Fabrics Treated with Mixtures of 2, 4, 6, and 8% DMDMDHEU and AA (DMDMDHEU/AA 1 : 1 mol/mol) with the Steeped Procedure at 70°C for 60 min

Process	DMDMDHEU (%)	Formaldehyde (%)	Nitrogen (%)	Moles/AGU		CL/AGU	CL length	[D]	K/S
				Nitrogen	Formaldehyde				
Pad-dry-cure	2	0.35	0.19	0.0221	0.0190	0.0080	1.39	2.23	0.36
	4	0.71	0.40	0.0470	0.0389	0.0154	1.52	1.25	0.23
	6	1.00	0.59	0.0698	0.0552	0.0203	1.72	0.79	0.16
	8	1.22	0.76	0.0905	0.0678	0.0256	2.01	0.64	0.13
Steep-pad-dry-cure	2	0.35	0.21	0.0245	0.0191	0.0068	1.80	2.89	0.54
	4	0.68	0.43	0.0506	0.0373	0.0120	2.10	1.40	0.27
	6	0.97	0.62	0.0734	0.0535	0.0168	2.19	0.83	0.18
	8	1.18	0.79	0.0942	0.0656	0.0186	2.54	0.65	0.14

The initiator used was 0.067% H_2O_2 (35% v/v).
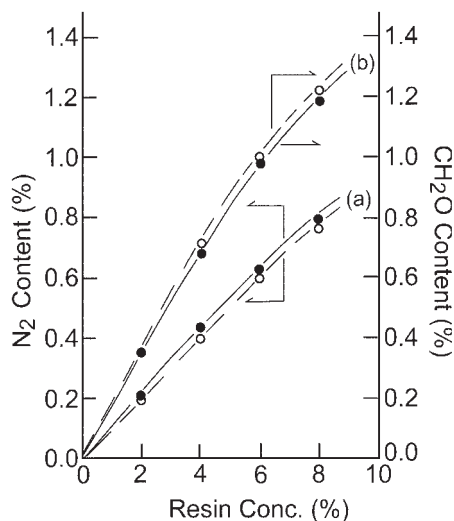
Figure 1 (a) Nitrogen contents and (b) formaldehyde contents of (○) pad–dry–cure- and (●) steep–pad–dry–cure-treated fabrics for different resin concentrations.

To confirm that AA can graft with cotton cellulose in the presence of H_2O_2 during the steeping process to affect the crosslinking reaction, we used the same conditions for the steeping process but without the crosslinking agent DMDMDHEU to treat the cotton fabric. The results show that the add-on values are
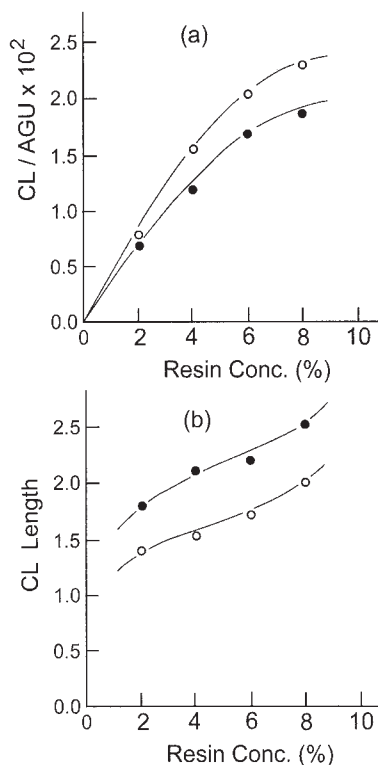
Figure 2 (a) CL/AGU and (b) CL length of (○) pad–dry–cure- and (●) steep–pad–dry–cure-treated fabrics for different resin concentrations.
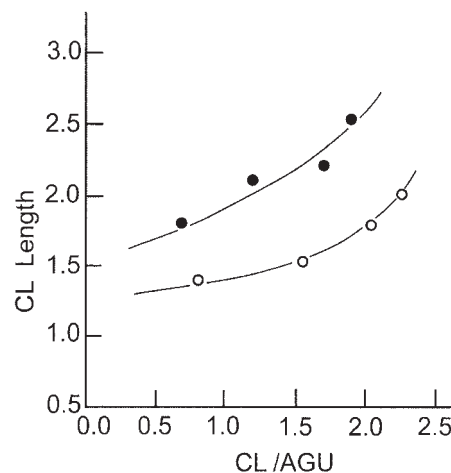

Figure 3 Relationship between the CL length and CL/AGU of (○) pad–dry–cure- and (●) steep–pad–dry–cure-treated fabrics for different resin concentrations.

almost zero for all the steeping-treated fabrics without DMDMDHEU after 5 min of washing with tap water; this indicates the impossibility or almost nonexistence of the graft of AA onto the cotton cellulose under such conditions. Additionally, the concentration of H_2O_2 used in this study was only about $1/20–1/30$ times that of the traditional grafting process, and so the oxidation of cellulose molecules could be ignored. We believe that the tensile strength of the treated fabrics under such a lower concentration of H_2O_2 will mainly be affected by the crosslinks of the crosslinking agent.
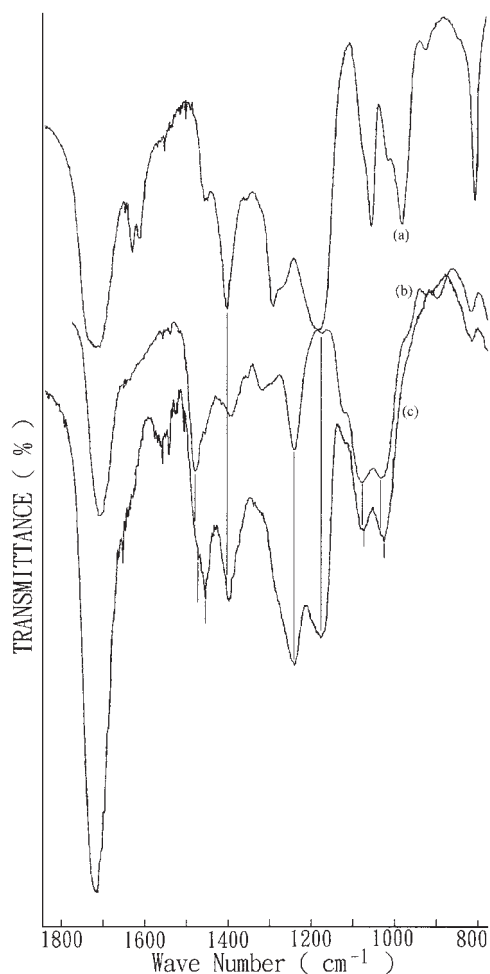
To confirm the self-reaction of the crosslinking agents, DMDMDHEU and AA were mixed and stirred in aqueous solutions in the presence of hydrogen peroxide under a nitrogen environment at 70°C for 60 min. The residue was studied with the following methods. First, a fresh mixture of DMDMDHEU and AA (1 : 1 mol/mol) and a mixture of DMDMDHEU and AA (1 : 1 mol/mol) at 70°C for 60 min were used as the samples for TLC analysis. These results [Fig. 4(a,b)] show that the migration for the fresh mixture of DMDMDHEU and AA is different from that of the mixture of DMDMDHEU and AA (1 : 1 mol/mol) at 70°C for 60 min. This result may be caused by the different structure of the residue of the mixture of DMDMDHEU and AA at 70°C for 60 min; that is, the condensation reaction between the crosslinking agents occurred with the steeped procedure.

If the condensation between the crosslinking agents indeed occurred, the absorption bands of the functional groups on the IR spectrum obtained from the residue of the mixture of DMDMDHEU and AA at 70°C for 60 min should differ from those obtained from DMDMDHEU alone and AA alone. Figure 5(a–c) shows the IR spectra of AA, DMDMDHEU, and the residue of the mixture of DMDMDHEU and AA at 70°C for 60 min. In the spectrum obtained from acrylic
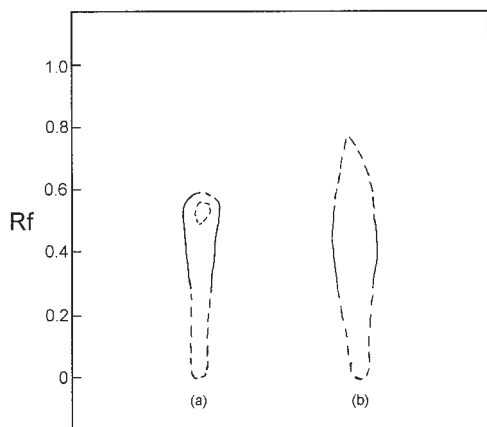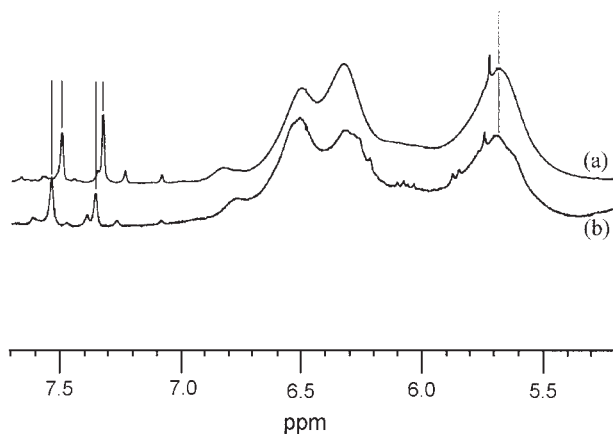
Figure 4 Migration (R_f) of TLC of (a) a fresh mixture of DMDMDHEU and AA (1 : 1 mol/mol) and (b) a mixture of DMDMDHEU and AA (1 : 1 mol/mol) at 70°C for 60 min.

Figure 6 H-NMR spectra of (a) DMDMDHEU alone and (b) the residue of a mixture of DMDMDHEU and AA (1 : 1 mol/mol) at 70°C for 60 min.

alone, the important absorbing band is that for H_2C=CH— (vinyl group, 1293 cm^{-1}). The important absorbing band for DMDMDHEU alone is that for


Figure 5 IR spectra of (a) AA, (b) DMDMDHEU, and (c) the residue of a mixture of DMDMDHEU and AA (1 : 1 mol/mol) at 70°C for 60 min.

—CH—OH (1478 cm^{-1}). As expected, some important absorbing bands for the residue of the mixture of DMDMDHEU and AA at 70°C for 60 min are changed. H_2C=CH—(1293 cm^{-1}) of AA disappears from the residue of the mixture of DMDMDHEU and AA at 70°C for 60 min. Meanwhile, the absorbing band of —CH—OH of DMDMDHEU weakens and shifts from 1478 to 1467 cm^{-1}, and a new absorbing band at 1451 cm^{-1} is generated in the spectrum of the residue of the mixture of DMDMDHEU and AA at 70°C for 60 min. These results affirm the self-condensation between the crosslinking agents.
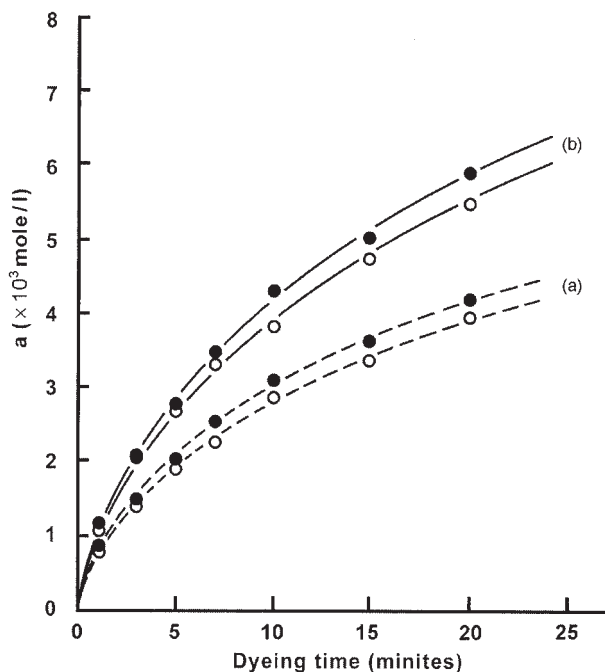
Figure 6(a,b) shows the H-NMR spectra of DMDMDHEU alone and the residue of the mixture of DMDMDHEU and AA at 70°C for 60 min separately. In Figure 6(a), the interesting and important chemical shifts for DMDMDHEU are those for —CH—OH (5.73 ppm) and —CH—OH (7.33 and 7.49 ppm). For the residue of the mixture of DMDMDHEU and AA at 70°C for 60 min [Fig. 6(b)], the chemical shifts for —CH—OH (5.74 ppm) show no obvious change, but the chemical shifts for —CH—OH shift to 7.36 and 7.54 ppm separately. The ^{13}C-NMR spectra of DMDMDHEU alone and the residue of the mixture of DMDMDHEU and AA at 70°C for 60 min are separately shown in Figure 7(a,b). Figure 7(a) shows that the interesting and important chemical shifts for DMDMDHEU alone are those for —CH_2—O—CH_3 (60.39 ppm), —CH_2—O—CH_3 (72.37 ppm), and —CH—OH (82.78 and 83.13 ppm). From Figure 7(b), we find that the chemical shift for —CH_2—O—CH_3 shifts to 60.57 ppm, —CH_2—O—CH_3 shifts to 72.47 ppm, and —CH—OH shifts to 82.98 and 83.32 ppm separately. Meanwhile, two new chemical shifts at 88.76 and 90.00 ppm can be found in the spectrum of the residue of the mixture of DMDMDHEU and AA at 70°C for 60 min. The values of the two new chemical shifts would not be the chemical shift of —CH_2—or —CH— of AA
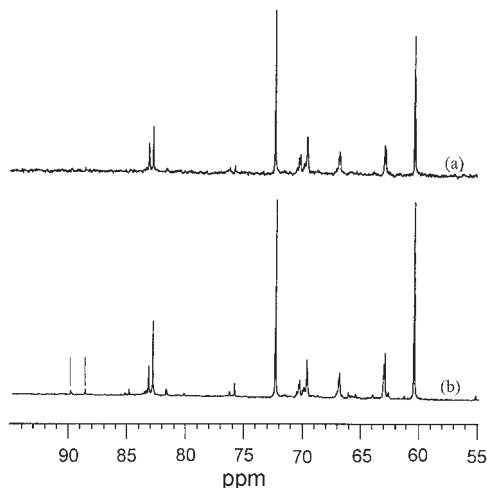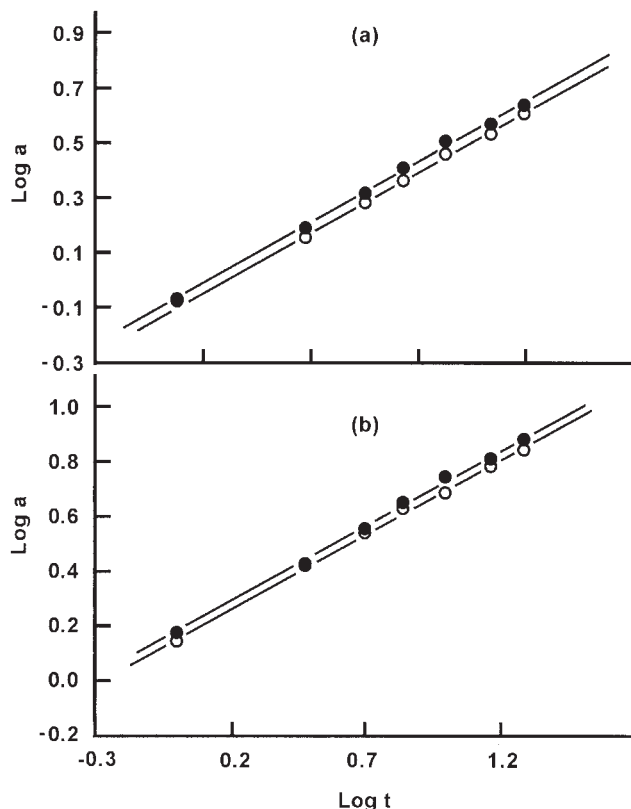
Figure 7 ^{13}C-NMR spectra of (a) DMDMDHEU alone and (b) the residue of a mixture of DMDMDHEU and AA (1 : 1 mol/mol) at 70°C for 60 min.

alone because the values of the chemical shift of these carbons would have lower values. The aforementioned results again strongly support self-condensation between the crosslinking agents.

Figure 8(a,b) shows the rates of dye absorption with C. I. Direct Red 81 under 40 and 60°C for the crosslinked cotton with the steep–pad–dry–cure process and pad–dry–cure process separately. The type of dye absorption is the same for both processes. How-

Figure 9 Plots of log a versus log t at (a) 40 and (b) 60 for fabrics treated with (○) the pad–dry–cure process and (●) the steep–pad–dry–cure process.

ever, from the overall dyeing time, we find that the values of the dye absorption for the two processes are in the order of steep–pad–dry–cure > pad–dry–cure at the same dyeing temperature. The higher dye absorption values for the steep–pad–dry–cure process may be caused by the different pore structures of the crosslinked fabrics with the steep–pad–dry–cure process and pad–dry–cure process. Our previous study suggested that the pore structures of fabrics treated with different processes are different from each other.[14]

Figure 9(a,b), separately for the rates of dye absorption of the crosslinked cotton with the steep–pad–dry–cure process and pad–dry–cure process, shows the linear relationships between the logarithmic values of the dye (C. I. Direct Red 81) absorption (log a) at different temperatures and the logarithmic values of the time intervals of dyeing (log t) over the initial dyeing time. From the results of the linear relationships, we believe that the use of the diffusion kinetic equation reported by Chrastil and coworkers[6,7] to study the equilibrium absorption, rate constant, and structural diffusion resistance constant (the index of the pore structure) of the various treated cotton fabrics is suitable. Several data obtained from the dyeing kinetics of eq. (1) are listed in Table III.

Figure 8 Rates of dye (Direct Red 81) adsorption at (a) 40 and (b) 60°C for fabrics treated with (○) the pad–dry–cure process and (●) the steep–pad–dry–cure process.

TABLE III
Values of the Equilibrium Adsorption (a_e), Rate Constant (k'; the Data Were Obtained from a Dyeing Time Interval of 20 min), Structural Diffusion Resistance Constant (n), and Activation Energy (ΔE) of Cotton Fabrics Treated with a Mixture of DMDMDHEU and AA (4% DMDMDHEU and Equal Moles of AA) with the Steep–Pad–Dry–Cure Process

Direct dye C.I. no.	Molecular weight (g/mol)	Dyeing temperature (°C)	Pad–dry–cure process				Steep–pad–dry–cure process			
			$a_e \times 10^{-3}$ (M)	n	$k \times 10^{-3}$ (min^{-1})	ΔE^* (kcal/mol)	$a_e \times 10^{-3}$ (M)	n	$k \times 10^{-3}$ (min^{-1})	ΔE^* (kcal/mol)
		40	12.51	0.52	5.54		11.92	0.54	7.60	
81	676	50	12.35	0.53	7.96	8.61	11.80	0.55	11.38	7.94
		60	12.14	0.54	12.73		11.65	0.56	16.36	

The initiator used was 0.067% H_2O_2 (35% v/v).

Table III shows that the equilibrium absorption values of the direct red dye for pad–dry–cure process are higher than those for the steep–pad–dry–cure process at a given temperature. This phenomenon may be caused by the self-condensation reaction between the crosslinking agents for the steep–pad–dry–cure process to form a loose crosslink. This is supported by the results of Figure 3: the crosslinking length for the steep–pad–dry–cure process is longer than that for the pad–dry–cure process at a given value of CL/AGU.

For all cases, the rate constants increase as the dyeing temperature increases. The rate constants for the two crosslinked fabrics that were dyed with the same direct dye and at the same dyeing temperature show the rank of steep–pad–dry–cure process > pad–dry–cure process. These results may be caused by the pore structure dependence of the dye absorption.[15,16]

Structural diffusion resistance constants are only slightly changed with increasing dyeing temperature (Table III). Table III also shows that the structural diffusion resistance constants for the two processes reveal the following rank of steep–pad–dry–cure process > pad–dry–cure process. Several reports[17–19] have revealed that the crosslinks reduce the structural diffusion resistance constant. Those results agree with the results in another report[17] and indicate that the pore structure of the crosslinked cotton changes with the different processes.

Arrhenius plots (two crosslinked fabrics) for C. I. Direct Red 81 are shown in Figure 10. The activation energies from the figure are also listed in Table III. The activation energies for the two crosslinked fabrics are ranked as steep–pad–dry–cure process < pad–dry–cure process. This ranking also agrees with the pore structure of the crosslinked fabrics, that is, the structural diffusion resistance constant.

The results for the pore sizes determined by the dyeing method and the lengths of the crosslinks both confirm that the crosslinking structures of the crosslinked cotton fabrics with the steep–pad–dry–cure process are different from those of the pad–dry–cure process, and this could improve the dry and wet crease recovery properties of the treated fabrics. Our

previous results[20] revealed that the deposition of crosslinking agents in cotton fiber produces longer crosslink lengths and higher DCRAs and WCRAs.

Figure 11 reveals the relationships between the values of K/S and $[D]_f$ for the steep–pad–dry–cure and pad–dry–cure processes and shows that K/S of the fabrics treated with the steep–pad–dry–cure process are higher than those of the fabrics treated with the pad–dry–cure process at the same value of $[D]_f$. These results clearly suggest that the amount of dye adsorbed onto the surface of the steep–pad–dry–cure-treated fabric is higher than that adsorbed onto the surface of the pad–dry–cure-treated fabric; that is, the crosslinks of the steep–pad–dry–cure-treated fabric have more inner distribution than those of the pad–dry–cure process-treated fabric. More inner distribution of the crosslinking agent may also create the higher DCRA and WCRA values of the steep–pad–dry–cure-treated fabric.
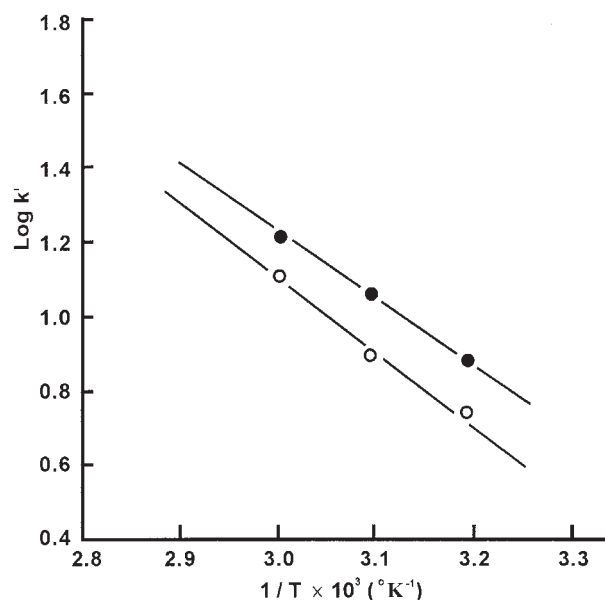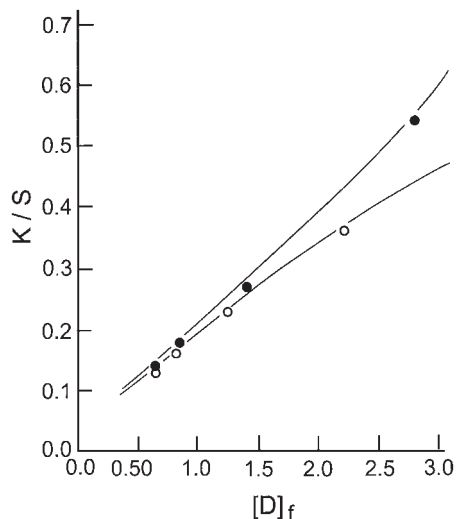

Figure 10 Arrhenius plots of fabrics treated with (○) the pad–dry–cure process and (●) the steep–pad–dry–cure process dyed with C. I. Direct Red 81.

Figure 11   Relationship between the values of K/S and $[D]_f$ of fabrics treated with (○) the pad–dry–cure process and (●) the steep–pad–dry–cure process dyed with C. I. Direct Red 81.

CONCLUSIONS

In this study, the steeped procedure was introduced to the durable press finishing process, and the DCRA and WCRA values of the fabrics treated with the steep–pad–dry–cure process were higher than those of the fabrics treated with the pad–dry–cure process. The higher the steeped temperature and time were, the higher the nitrogen content, DCRA, and WCRA were and the lower TSR was of the treated fabrics at the same resin concentration. For a given number of CL/AGU, the values of the CL length for the steep–pad–dry–cure process were higher than those for the pad–dry–cure process. The values of the dye absorption, rate constant, and structural diffusion resistance constant for the two finished processes were in the rank of steep–pad–dry–cure > pad–dry–cure at the same dyeing temperature. The equilibrium absorption values of the direct dye and the activation energies for the two crosslinked fabrics were ranked as steep–pad–dry–cure process < pad–dry–cure process. From the results of TLC, H-NMR, ^{13}C-NMR, and IR analyses, we conclude that the self-condensation between the crosslinking agents occurred with the steeped procedure. Additionally, the crosslinks of the steep–pad–dry–cure-treated fabric had more inner distribution than those of the pad–dry–cure-treated fabric.

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