

Degree of Crosslinking and Physical Properties of Dimethyloldihydroxyethyleneurea/Acrylic Acid Crosslinked Cotton Fabrics after Treatment with Various Metallic Salts

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ABSTRACT: Three metallic salts were used to posttreat dimethyloldihydroxyethyleneurea (DMDHEU)/acrylic acid (AA) crosslinked cotton fabrics, and the results showed that at a given value of the tensile strength retention (TSR), the dry crease recovery angle (DCRA) and wet crease recovery angle (WCRA) of the crosslinked and posttreated fabrics were higher than those of the DMDHEU-AA-treated fabrics, and those of the crosslinked and posttreated fabrics were in the order of $\text{Ag}^+ > \text{Cu}^{+2} > \text{Al}^{+3}$. The DCRA and TSR values for the crosslinked and posttreated fabrics were higher than those for the DMDHEU-AA-crosslinked fabrics, and those for the crosslinked and posttreated fabrics were in the order of $\text{Ag}^+ > \text{Cu}^{+2} > \text{Al}^{+3}$; however, WCRA values for the

crosslinked and posttreated fabrics were lower than those for the DMDHEU-AA-crosslinked fabrics, and those for the crosslinked and posttreated fabrics were ranked as $\text{Ag}^+ > \text{Cu}^{+2} > \text{Al}^{+3}$ at a given number of crosslinks per anhydroglucose unit. IR spectra clearly revealed the different interactions and bonding states between the hydroxyl group of the cellulose and the various metallic ions and the strength of the interaction. All crosslinked and posttreated fabric samples showed good odor absorption and antibacterial and washing-fastness properties. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 97: 584–594, 2005

Key words: adsorption; crosslinking; FT-IR; resins

INTRODUCTION

It is well known that metallic salts can significantly improve the antibacterial properties of materials.^{1–3} Park et al.¹ showed that the addition of metals to the grafted polypropylene fabric improved the antibacterial properties significantly. Additionally, previous works^{4–6} have revealed that the physical properties of finished fabrics are affected by the crosslinking structure. Steven and Smith⁷ pointed out that the hydrogen bonds in the finished fibers could affect the physical properties of the treated fabrics. Our previous study^{8,9} showed that the physical properties, reaction rate constants, and reaction energy consumption of the treated fabrics were affected by the addition of a carboxyl group to the crosslinking agent dimethyloldihydroxyethyleneurea.

In this study, we used prereacted dimethyloldihydroxyethyleneurea (DMDHEU)/acrylic acid (AA) to

treat cotton fabrics, and the treated fabric samples were then posttreated with various metallic salts under different conditions. We expected the crosslinking and physical properties of the crosslinked and posttreated fabric samples to be affected by the metallic ions. However, detailed information is lacking on the physical properties and degree of crosslinking formed in DMDHEU-AA-crosslinked cotton fabrics posttreated with metallic salts. We examined the antibacterial and physical properties and the crosslinking structures of DMDHEU-AA (DMDHEU/AA molar ratio = 1/1) crosslinked cotton fabrics posttreated with various metallic salts.

EXPERIMENTAL

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

The crosslinking agent was prereacted DMDHEU-AA.^{8,9} Poly(vinyl alcohol) (PVA; Showa Chemical Co., Ltd., Tokyo, Japan), silver nitrate (Nihon Shiyaku Industries, Ltd., Osaka, Japan), cupric sulfate (Nihon Shiyaku Industries), and aluminum sulfate (Osaka, Ltd., Osaka, Japan) were reagent-grade, as were the other chemicals:

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TABLE I
Physical Properties of DMDHEU-AA (DMDHEU/AA = 1/1) Crosslinked Cotton Fabrics after Treatment with AgNO_2 , CuSO_4 , and $\text{Al}_2(\text{SO}_4)_3$ at Different Concentrations at 30°C for 2 h

Metallic salts	DMDHEU concentration (%)	Ion concentration of metallic salts (M)	Physical properties			
			DCRA (W + F) ^o	WCRA (W + F) ^o	TSR (%)	MR (%)
Control	—	—	200	188	100	7.8
	2	—	226	221	80	6.9
	4	—	246	239	70	6.3
	6	—	261	252	63	5.9
	8	—	270	269	57	5.6
Ag^+	2	0.012	238	215	89	7.4
	4	0.024	258	234	78	7.0
	6	0.036	273	251	72	6.7
	8	0.048	282	265	64	6.5
Cu^{+2}	2	0.012	235	214	87	7.3
	4	0.024	254	231	77	6.7
	6	0.036	269	249	70	6.4
	8	0.048	279	262	63	6.1
Al^{+3}	2	0.012	230	211	86	7.2
	4	0.024	248	227	76	6.5
	6	0.036	264	242	69	6.2
	8	0.048	274	258	62	5.8

W, warp; F, filling.

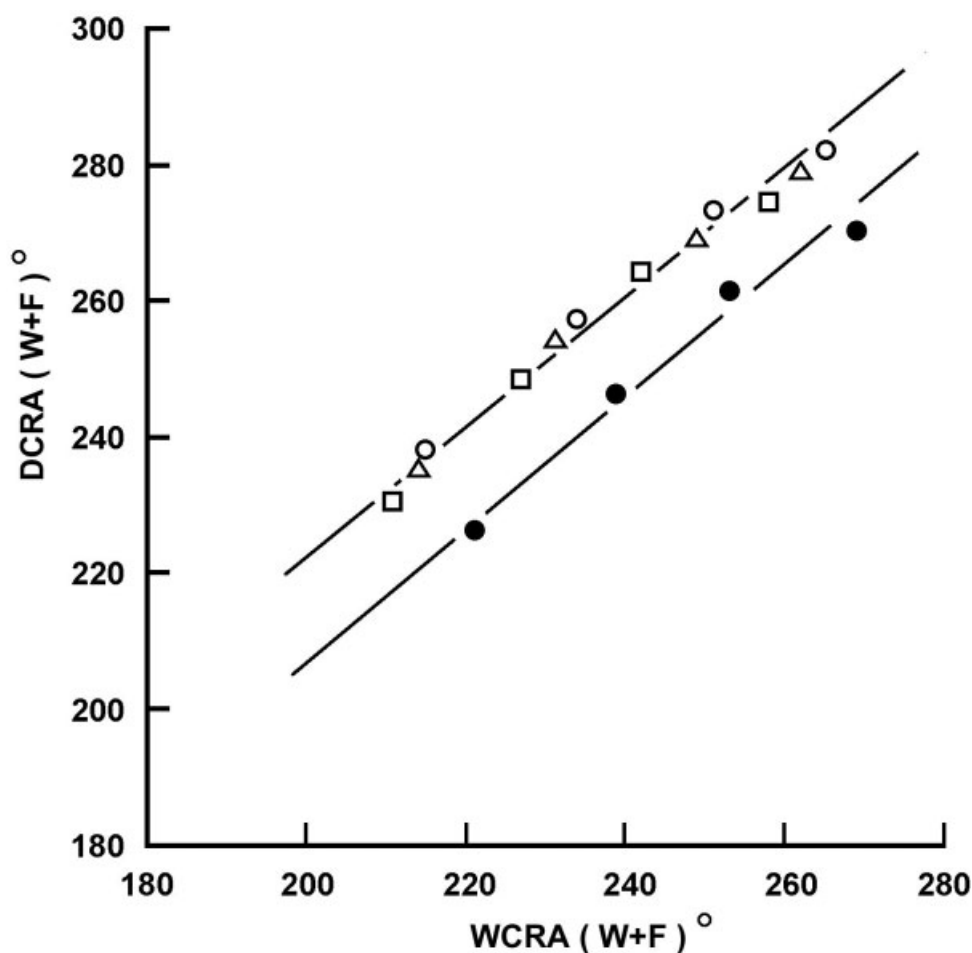


Figure 1 Relationship between DCRA and WCRA of DMDHEU-AA-crosslinked and posttreated fabric samples with various metallic salts: (●) none after treatment, (○) AgNO_2 after treatment, (△) CuSO_4 after treatment, and (□) $\text{Al}_2(\text{SO}_4)_3$ after treatment.

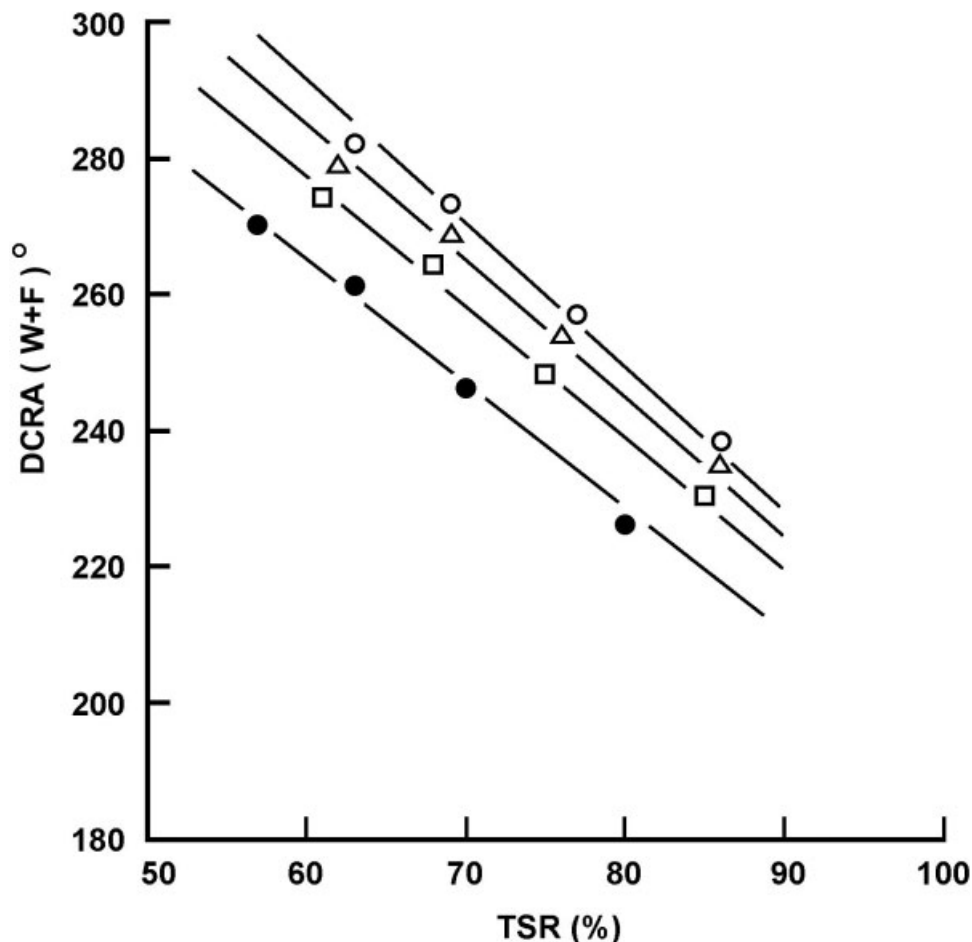
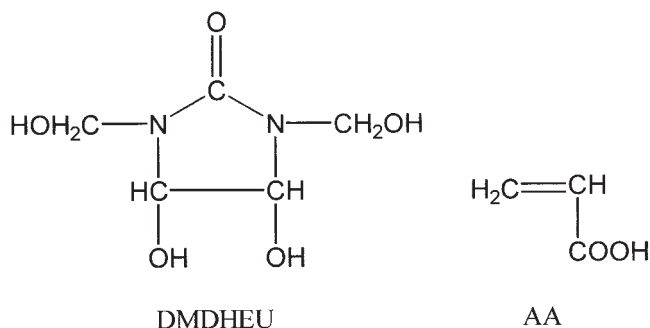


Figure 2 Relationship between DCRA and TSR of DMDHEU-AA-crosslinked and posttreated fabric samples with various metallic salts: (●) none after treatment, (○) AgNO_2 after treatment, (△) CuSO_4 after treatment, and (□) $\text{Al}_2(\text{SO}_4)_3$ after treatment.



The cotton fabric samples were padded twice to about 90% wet pickup with freshly prepared aqueous solutions of prereacted DMDHEU-AA (DMDHEU/AA = 1/1) in the presence of an ammonium sulfate catalyst. The catalyst concentration was 0.10 times the amount of DMDHEU. The padded fabrics were dried at 80°C for 5 min, cured at 160°C for 3 min, soaped with 1.0 g/L soap at 40°C for 5 min, washed with tap water at room temperature for 5 min, and dried at 60°C. After that, various metal salts [AgNO_2 , CuSO_4 , and $\text{Al}_2(\text{SO}_4)_3$] were selected to posttreat the DMD-

HEU-AA-finished fabrics under different conditions. The posttreated fabric samples were then washed and dried.

The tensile strengths of the warp yarns were measured on an Instron tensile tester. ASTM Standard D 1295-67 was used to determine the dry crease recovery angle (DCRA) and wet crease recovery angle (WCRA). Formaldehyde and nitrogen determinations were made with chromatropic acid¹⁰ and Kjeldahl methods, respectively. The antibacterial property (bacterial inhibition values) of the treated cotton fabrics was tested with AATCC Test Method 100-1998, and *Staphylococcus aureus* and *Escherichia coli* were used.

Odor absorption values were measured with the following method described by Kazuto.¹¹ The treated fabric sample was hung in a bottle of poly(tetrafluoroethylene), in which 50 mL of ammonia water contained a total ammonia concentration of 200 ppm, that was kept at a constant temperature of 50°C under a water bath for 1 min and then was cooled at room temperature for 60 min. Finally, the residue of ammo-

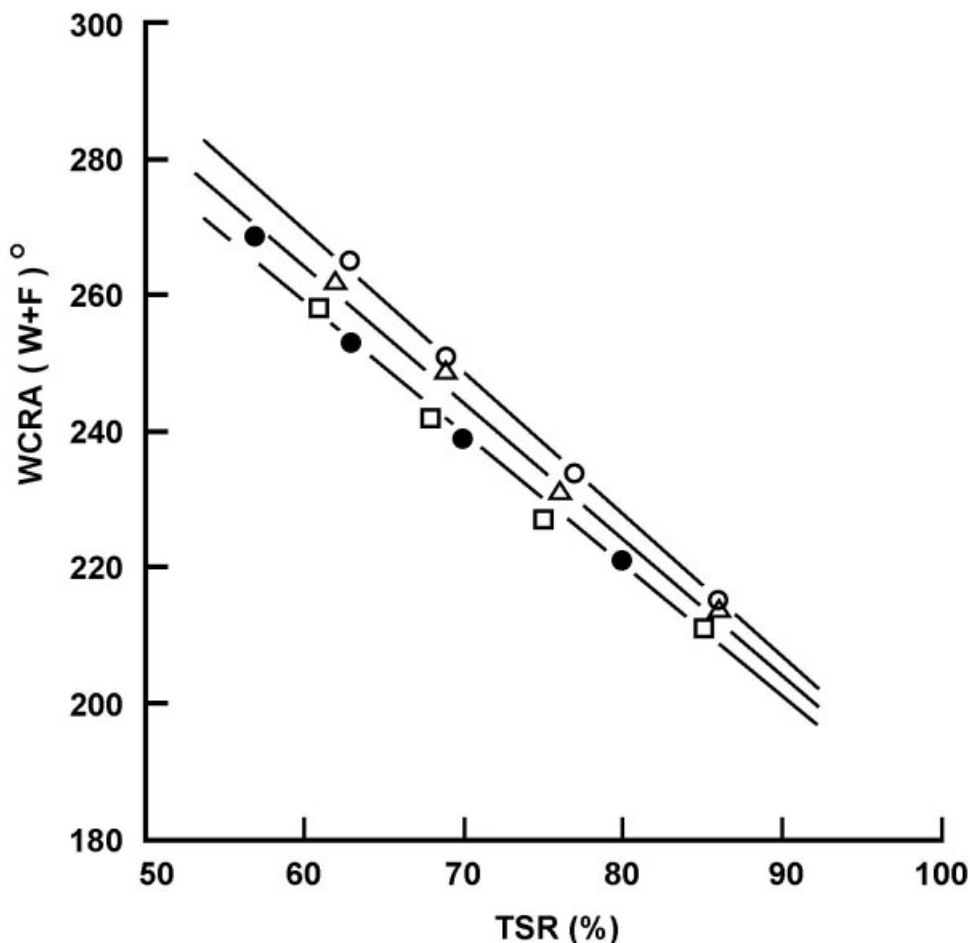


Figure 3 Relationship between WCRA and TSR of DMDHEU-AA-crosslinked and posttreated fabric samples with various metallic salts: (●) none after treatment, (○) AgNO_2 after treatment, (△) CuSO_4 after treatment, and (□) $\text{Al}_2(\text{SO}_4)_3$ after treatment.

nia gas in the bottle was measured with a Gastec pump and a Gastec detector tube (Gastec Corp., Kanagawa, Japan).

IR spectra of the samples were obtained with a KBr disk technique.¹² The samples 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 PVA (because cellulose powder is not soluble in water, PVA was selected as a model of cotton cellulose) and a mixture of dissolved PVA and metal salts (the molar ratio of the repeat unit of PVA to the metallic ion was 2/1) were coated onto a piece of glass and dried at 30°C . These materials were mixed with KBr, ground, and dried in 1-dram vials over P_2O_5 for 3 days, and the samples were then pressed in an evacuated die under a suitable pressure. A Fourier transform infrared spectrophotometer (model FT/IR-3; Jasco, International Co., Ltd., Tokyo, Japan) was used to obtain spectra.

RESULTS AND DISCUSSION

Relationships between the physical properties

Table I shows the DCRA, WCRA, tensile strength retention (TSR), and moisture regain (MR) of DMDHEU-AA-crosslinked samples and samples posttreated with various metallic salts. DCRA and WCRA increased with an increased concentration of DMDHEU-AA in the bath, but TSR and MR decreased in all cases. The DCRA, TSR, and MR values of the DMDHEU-AA-crosslinked and posttreated fabrics were higher than those of the DMDHEU-AA-crosslinked fabrics; however, the WCRA values were lower than those of DMDHEU-AA. The DCRA, WCRA, TSR, and MR values of the fabrics crosslinked and posttreated with metallic salts were in the order of $\text{Ag}^+ > \text{Cu}^{+2} > \text{Al}^{+3}$. The higher DCRA, TSR, and MR values of the fabrics posttreated with metallic salts may have been caused by the formation of new bonding between the metallic ions and the functional groups of the cellulose. Steven and Smith⁷ pointed out that hydrogen

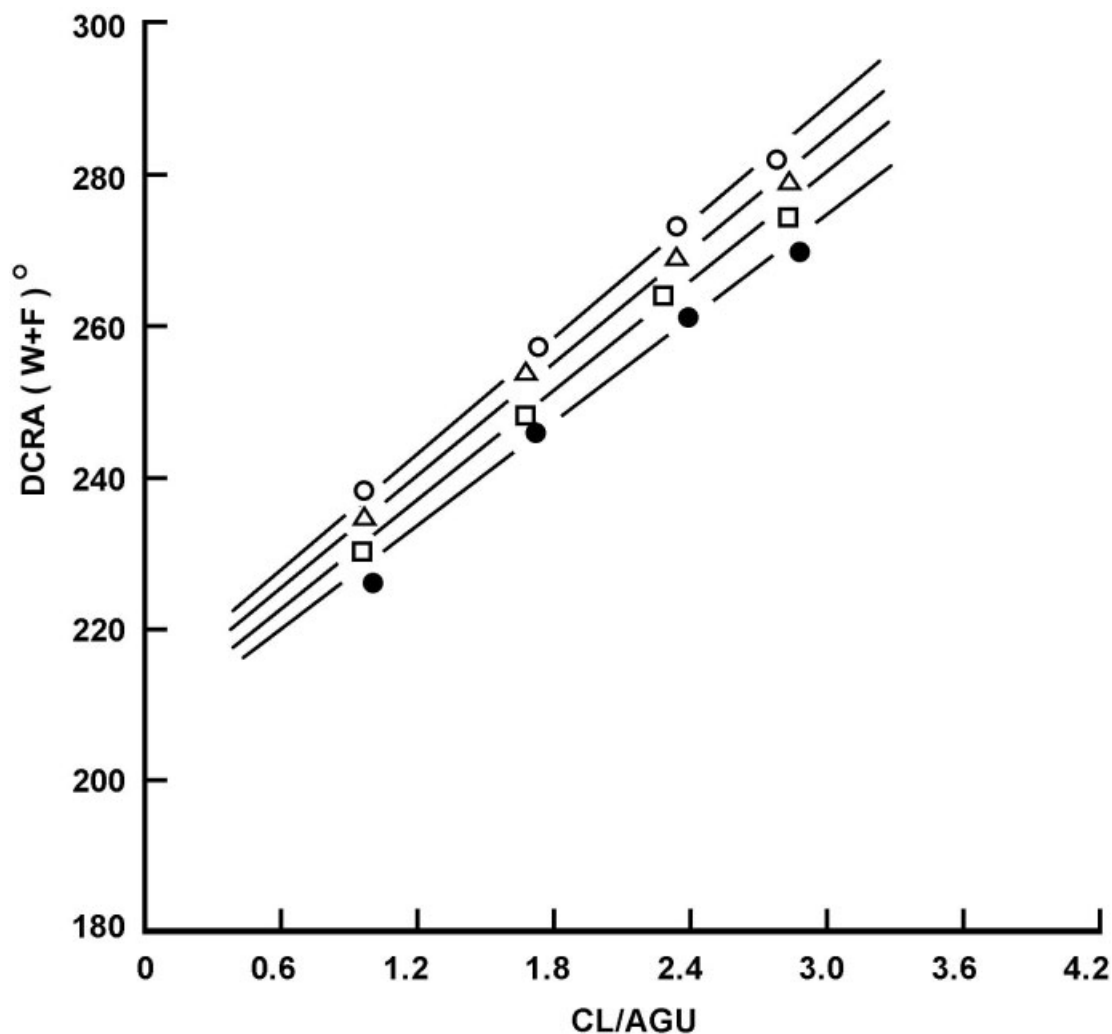


Figure 4 Plots of DCRA versus CL/AGU of DMDHEU-AA-crosslinked and posttreated fabric samples with various metallic salts: (●) none after treatment, (○) AgNO_2 after treatment, (Δ) CuSO_4 after treatment, and (□) $\text{Al}_2(\text{SO}_4)_3$ after treatment.

bonds that formed in the finished fibers could affect the physical properties of the treated fabrics. Our recent study⁸ has shown that physical properties, such as DCRA, WCRA, and MR, are affected by the carboxylic acid of the crosslinking agent, which can react and bond with the functional groups of cellulose.

The relationships between DCRA, WCRA, and TSR of the various finished fabrics are shown in Figures 1–3. According to the relationships between DCRA and WCRA of the finished fabrics (Fig. 1), DCRA of the DMDHEU-AA-crosslinked fabrics posttreated with metallic salts was higher than DCRA of the DMDHEU-AA-crosslinked fabrics, and the DCRA values of fabrics posttreated with metallic salts were similar at a given value of WCRA. Figure 2 shows the plots of DCRA versus TSR of the various finished fabrics. For a given value of TSR, the DCRA values of the DMDHEU-AA-crosslinked and metallic-salt-posttreated fabrics were higher than those of the DMDHEU-AA-

crosslinked fabrics, and those for metallic-salt-posttreated fabrics were in the order of $\text{Ag}^+ > \text{Cu}^{+2} > \text{Al}^{+3}$. The plots of WCRA versus TSR of the finished fabrics shown in Figure 3 reveal that the WCRA values of the DMDHEU-AA-crosslinked and metallic-salt-posttreated fabrics were almost all higher than those of the DMDHEU-AA-crosslinked fabrics, and those for the metallic-salt-posttreated fabrics were ranked as $\text{Ag}^+ > \text{Cu}^{+2} > \text{Al}^{+3}$ at the same value of TSR. The other factors that could affect the physical properties of the various metallic-salt-posttreated fabrics were the degree of crosslinking and the difference in the bonding effect between the metallic ions and the functional groups of the cellulose.

Degree of the crosslinking and physical properties

The nitrogen and formaldehyde contents, number of crosslinks, and length of crosslinks (CL length) for the

TABLE II
Structural Characteristics of DMDHEU-AA (DMDHEU/AA = 1/1) Crosslinked Cotton Fabrics after Treatment with AgNO_2 , CuSO_4 , and $\text{Al}_2(\text{SO}_4)_3$ at Different Concentrations at 30°C for 2 h

Metallic salts	DMDHEU concentration (%)	Ion concentration of metallic salts (M)	Nitrogen (%)	Formaldehyde (%)	Moles ($\text{AGU} \times 10^2$)		CL/AGU	CL length
					Nitrogen	Formaldehyde		
—	2	—	0.18	0.38	2.10	2.07	1.018	1.030
	4	—	0.34	0.68	3.99	3.72	1.728	1.154
	6	—	0.50	0.97	5.90	5.34	2.392	1.233
	8	—	0.68	1.25	8.08	6.93	2.892	1.397
Ag^+	2	0.012	0.17	0.36	1.98	1.96	0.967	1.024
	4	0.024	0.33	0.67	3.87	3.67	1.732	1.117
	6	0.036	0.49	0.95	5.78	5.23	2.340	1.235
	8	0.048	0.67	1.22	7.96	6.76	2.748	1.429
Cu^{+2}	2	0.012	0.17	0.36	1.98	1.96	0.967	1.024
	4	0.024	0.34	0.67	3.99	3.67	1.673	1.191
	6	0.036	0.49	0.94	5.78	5.23	2.285	1.265
	8	0.048	0.69	1.23	7.96	6.82	2.834	1.401
Al^{+3}	2	0.012	0.18	0.37	2.10	2.01	0.964	1.088
	4	0.024	0.33	0.66	3.87	3.61	1.677	1.154
	6	0.036	0.50	0.95	5.90	5.23	2.282	1.293
	8	0.048	0.67	1.23	7.96	6.82	2.839	1.401

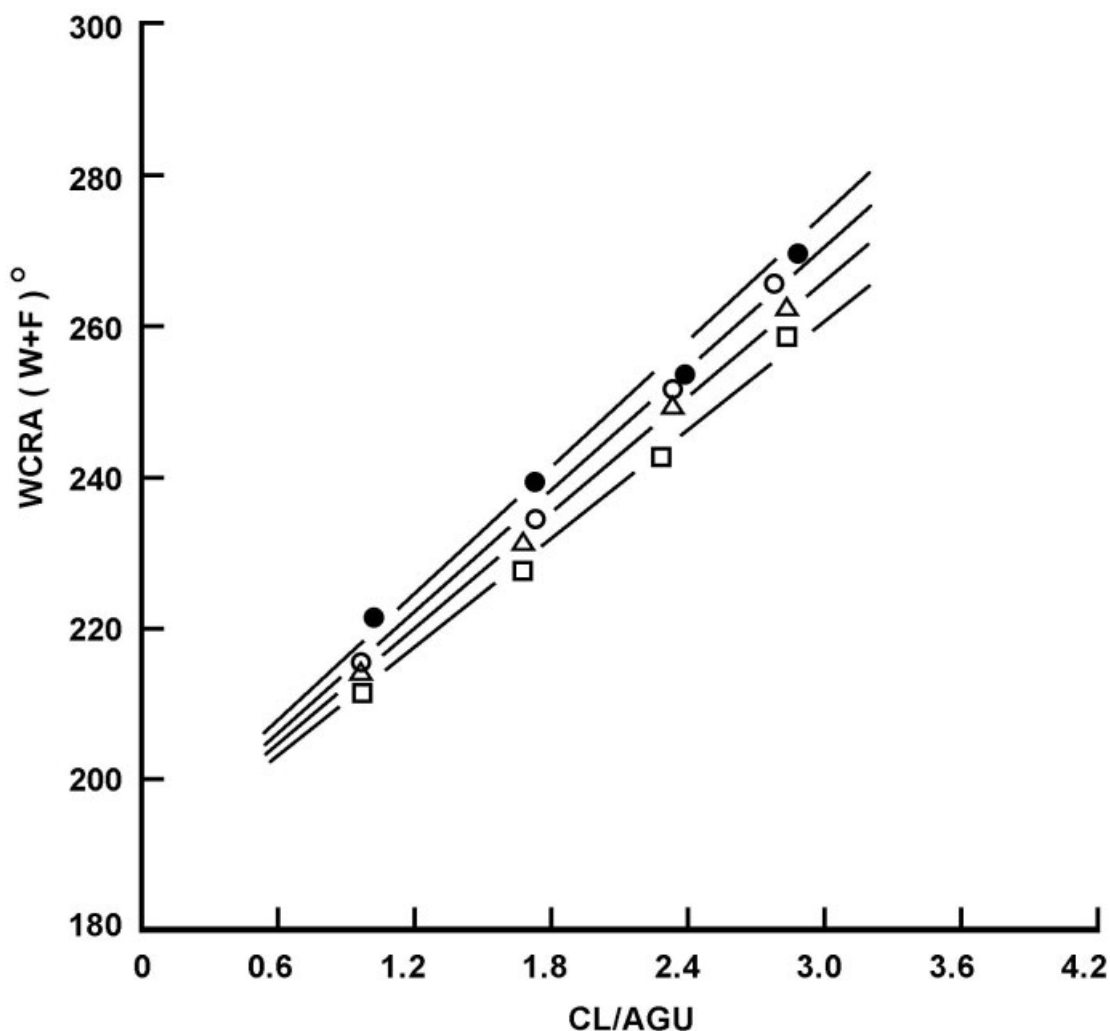


Figure 5 Plots of WCRA versus CL/AGU of DMDHEU-AA-crosslinked and posttreated fabric samples with various metallic salts: (●) none after treatment, (○) AgNO_2 after treatment, (△) CuSO_4 after treatment, and (□) $\text{Al}_2(\text{SO}_4)_3$ after treatment.

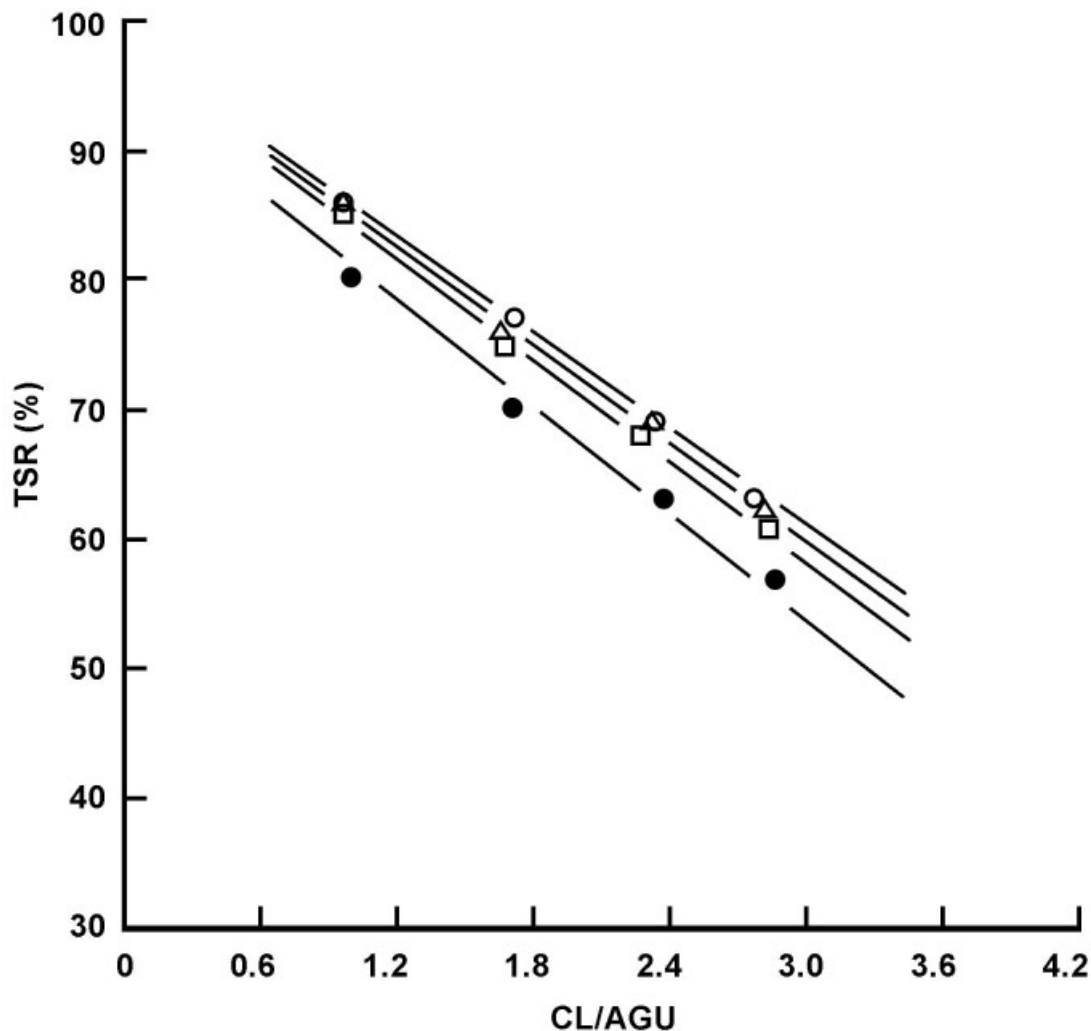


Figure 6 Plots of TSR versus CL/AGU of DMDHEU-AA-crosslinked and posttreated fabric samples with various metallic salts: (●) none after treatment, (○) AgNO₂ after treatment, (△) CuSO₄ after treatment, and (□) Al₂(SO₄)₃ after treatment.

cotton fabrics crosslinked with various DMDHEU-AA concentrations and posttreated with various metallic salts are presented in Table II. As expected, the nitrogen and formaldehyde contents gradually increased with increasing DMDHEU-AA concentration in the bath in all cases. The nitrogen and formaldehyde contents of the finished samples were only slightly varied with the change in the metallic salts used in the post-treatment process.

The number of crosslinks per anhydroglucose unit (CL/AGU) and the CL length of the crosslinked and posttreated fabrics (Table II), obtained with the methods of Frick et al.,^{13,14} indicated that both increased as the concentration of DMDHEU-AA in the bath increased. These results revealed that the crosslinks were only slightly affected by the change in the metallic salts used in the posttreatment and could only slightly affect the physical properties.

The physical properties are plotted versus CL/AGU for the finished fabrics in Figures 4–6. For a given

value of CL/AGU, DCRA and TSR values for the DMDHEU-AA-crosslinked and metallic-salt-posttreated fabrics were higher than those for the DMDHEU-AA-crosslinked fabrics, and those for the crosslinked and metallic-salt-posttreated fabrics were in the order of Ag⁺ > Cu⁺² > Al⁺³ (Figs. 5 and 7). However, the WCRA values of the various treated fabrics (Fig. 6) showed an unexpected and interesting tendency: the values for the DMDHEU-AA-crosslinked and metallic-salt-posttreated fabrics were lower than those for the DMDHEU-AA-crosslinked fabrics, and those for crosslinked and metallic-salt-posttreated fabrics were in the order of Ag⁺ > Cu⁺² > Al⁺³. Generally, if the degree of crosslinking of the finished fabric was not changed, an increase in DCRA lowered WCRA of the finished fabric,¹⁵ which was crosslinked in the dry state. Therefore, the unexpected higher values of WCRA for Ag⁺-posttreated fabrics may have been affected by the bonding effect between Ag⁺ and cellulose molecules.

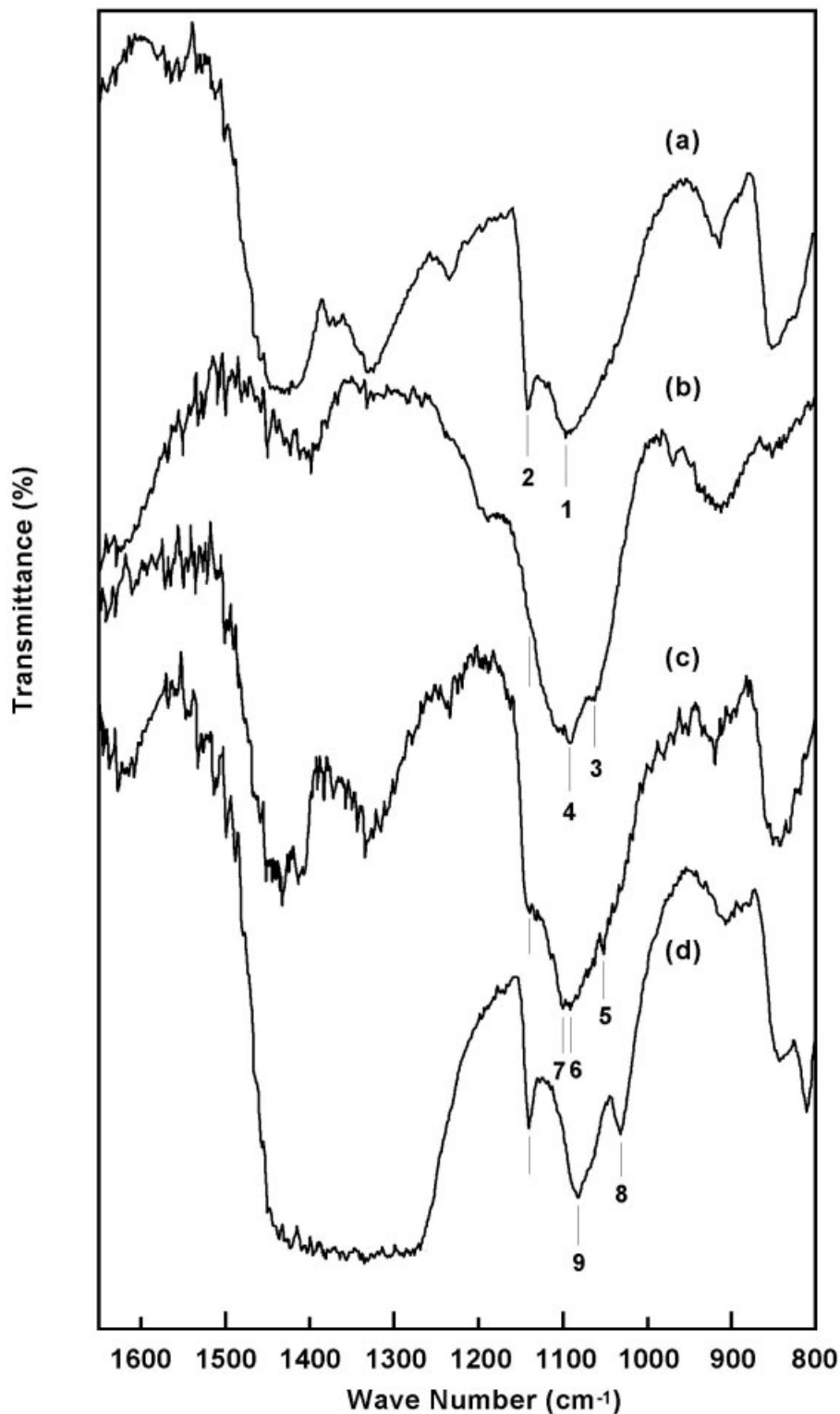


Figure 7 IR spectra of (a) PVA, (b) PVA/ $\text{Al}_2(\text{SO}_4)_3$, (c) PVA/ CuSO_4 , and (d) PVA/ AgNO_2 : (1) 1020, (2) 1072, (3) 1045, (4) 1015, (5) 1085, (6) 997, (7) 1010, (8) 1041, and (9) 1068 cm^{-1} .

Interactions between the metallic ions and cellulose molecules

To confirm the bonding effect between the various metallic ions and cellulose molecules (PVA was se-

lected as a model molecule of cotton cellulose for the undissolved cellulose powder in water), a model molecule of PVA was dissolved in water and mixed with various metallic salts, and then the various mixtures were dried at 30°C. The IR spectra of PVA, a dried

TABLE III
Physical Properties of DMDHEU-AA (DMDHEU/AA = 1/1 or 1/3) Crosslinked Cotton Fabrics after Treatment with 0.3% (w/w) Ionic Concentrations of AgNO₂, CuSO₄, and Al₂(SO₄)₃ at 30°C for 2 h

Metallic salts	DMDHEU concentration (%)	DMDHEU/AA (molar ratio)	NH ₃ gas absorption (ppm/g of cotton)	Antibacterial activity ^a	
				Bacterial inhibition for S.A. ^b (%)	Bacterial inhibition for E.C. ^c (%)
Control	—	—	13	0	0
Ag ⁺	2	1/1	46	100	99.7
	4	1/1	65	100	100
	6	1/1	81	100	100
	8	1/1	90	100	100
Cu ⁺²	2	1/1	40	58.2	33.9
	4	1/1	56	79.8	59.3
	6	1/1	72	97.4	71.3
	8	1/1	80	100	81.9
Cu ⁺²	2	1/3	70	69.7	53.0
	4	1/3	100	99.9	78.6
	6	1/3	100	100	93.4
	8	1/3	100	100	100
Al ⁺³	2	1/1	34	47.2	24.0
	4	1/1	49	72.0	45.7
	6	1/1	62	87.4	57.7
	8	1/1	71	94.4	65.5

^a Time of exposure to the environment is 1 h.

^b S.A. = *Staphylococcus aureus*.

^c E.C. = *Escherichia coli*.

sample of a mixture of dissolved PVA and Al₂(SO₄)₃, a dried sample of a mixture of dissolved PVA and CuSO₄, and a dried sample of a mixture of dissolved PVA and AgNO₂ are shown in Figure 7(a–d), respectively. The interesting and important absorption bands are those of —CHOH (1140 and 1097 cm⁻¹)¹⁶ for PVA [Fig. 7(a)]. As the dissolved PVA and various metallic salts were mixed together, the IR spectra [Fig. 7(b–d)] changed significantly. The spectrum in Figure 7(b) for PVA/Al₂(SO₄)₃ shows that the absorption band of 1140 cm⁻¹ disappeared, and a relatively small

but important absorption band was generated at 1063 cm⁻¹; the main absorption band at 1097 cm⁻¹ shifted to 1092 cm⁻¹. In Figure 7(c), the spectrum for PVA/CuSO₄ reveals that the absorption band of 1140 cm⁻¹ almost disappeared, and a relatively sharp and important absorption band was generated at 1062 cm⁻¹; the main absorption band at 1097 cm⁻¹ was disrupted and shifted to 1091 and 1100 cm⁻¹. Meanwhile, the main and important absorption band of 1097 cm⁻¹ in Figure 7(d) for PVA/AgNO₂ shifted to 1082 cm⁻¹, and a new absorption band at 1032 cm⁻¹ was sharp and signifi-

TABLE IV
Washing Fastness of DMDHEU-AA (DMDHEU/AA = 1/1 or 1/3) Crosslinked Cotton Fabrics after Treatment with a 0.3% (w/w) Ionic Concentration of CuSO₄ at 30°C for 2 h

DMDHEU concentration (%)	DMDHEU/AA (molar ratio)	NH ₃ gas absorption (ppm/g of cotton)		Antibacterial activity ^a			
		Washing 1 cycle	Washing 30 cycles	Bacterial inhibition for S.A. ^b (%)		Bacterial inhibition for E.C. ^c (%)	
				Washing 1 cycle	Washing 30 cycles	Washing 1 cycle	Washing 30 cycles
2	1/1	40	28	58.2	39.1	33.9	25.3
4	1/1	56	41	79.8	69.5	59.3	47.3
6	1/1	72	52	97.4	86.7	71.3	63.7
8	1/1	80	60	100	89.9	81.9	72.0
2	1/3	49	40	69.7	51.6	53.0	40.7
4	1/3	72	58	99.9	83.0	78.6	63.7
6	1/3	87	72	100	97.4	93.4	86.9
8	1/3	95	82	100	98.9	100	95.1

^a Time of exposure to the environment is 1 h.

^b S.A. = *Staphylococcus aureus*.

^c E.C. = *Escherichia coli*.

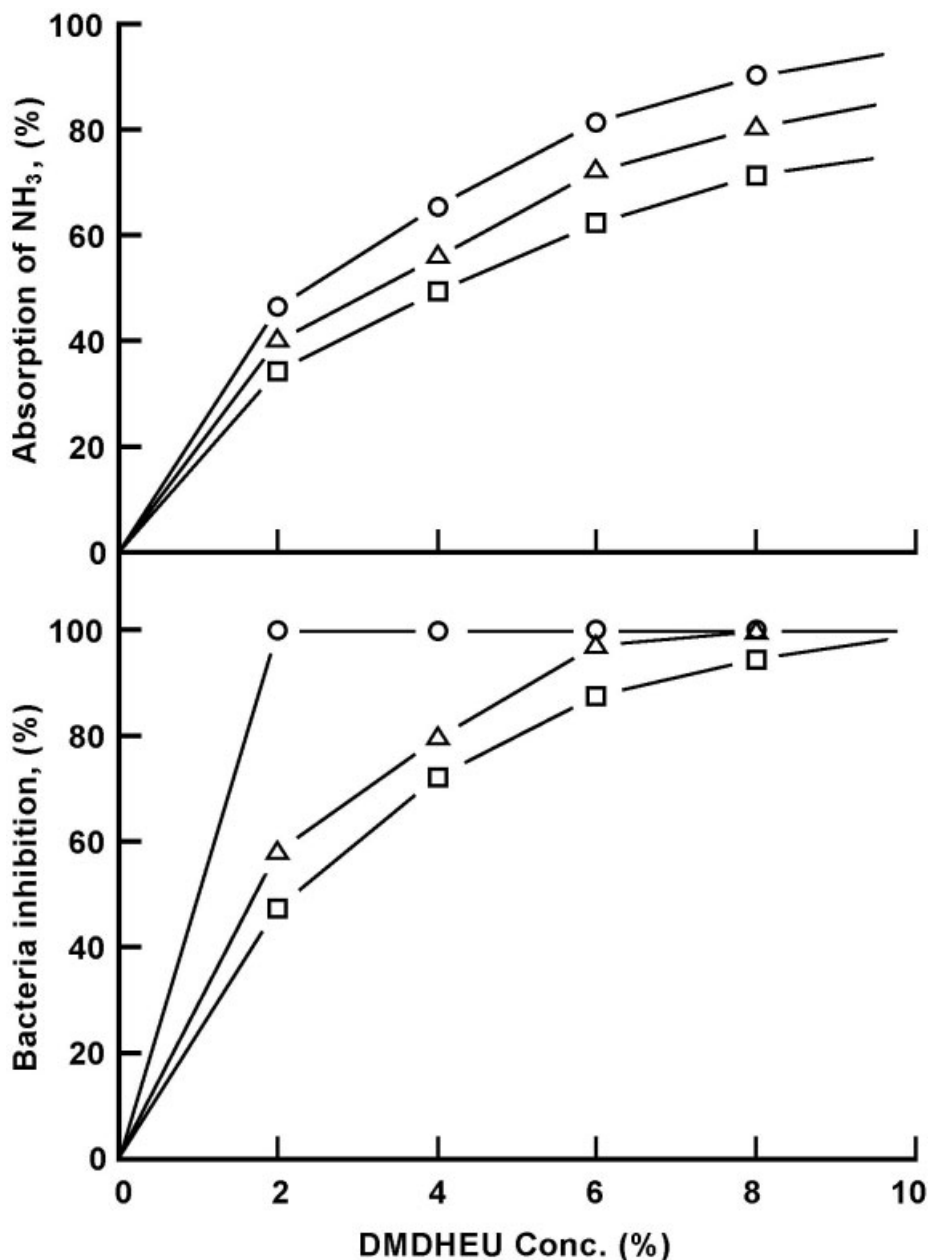


Figure 8 Odor absorption and bacterial (*Staphylococcus aureus*) inhibition for DMDHEU-AA (DMDHEU/AA = 1/1) crosslinked cotton fabrics posttreated with a 0.3% (w/w) ionic concentration of (○) AgNO₃, (△) CuSO₄, or (□) Al₂(SO₄)₃.

cant. These results clearly showed that the interaction, which was believed to be coordinated bonding between the —OH group of the cellulose and the metallic ions, which were different from one another in the bonding state, could improve the crease recovery angle and TSR, as shown in Figures 5–7, the strength of the bonding for the various metallic ions being in the order of Ag⁺ > Cu⁺² > Al⁺³.

Antibacterial property and odor absorption

The bacterial inhibition values are defined as $(M_b - M_a)/M_b \times 100\%$, where M_b and M_a are the numbers

of bacteria of the finished fabrics for the nourishment at 0 h and a specific number of hours, respectively. A higher value of bacterial inhibition indicates a greater effect of the antibacterial property. Tables III and IV show that the values of bacterial inhibition for both bacteria (*S. aureus* and *E. coli*) used in this study and odor absorption (NH₃ absorption) increased with an increase in the resin concentration and AA content in the padding bath; they are in the order of Ag⁺ > Cu⁺² > Al⁺³ for the various posttreated fabrics at a given DMDHEU-AA concentration and molar ratio of DMDHEU to AA, as shown in Figure 8(a,b). Additionally, Table IV shows that the washing fastness of the

crosslinked and metallic-salt-posttreated fabrics were excellent and increased with an increase in the AA content and DMDHEU-AA concentration in the padding bath. The higher odor absorption and antibacterial property for Ag^+ could be attributed to the larger ion diameter. Park et al.¹ showed that the Ag^+ -posttreated fabric grafted with AA had a higher antibacterial property than the fabrics treated with other metallic ions. Additionally, from the previous discussions of IR spectra, we can reasonably suggest that NH_3 gas can also be adsorbed by metallic ions with coordinated bonding between the $-\text{NH}_2$ group of ammonia gas and the metallic ions, and the strength of the bonding and odor absorption of the crosslinked and posttreated fabrics with various metallic ions are also in the order of $\text{Ag}^+ > \text{Cu}^{+2} > \text{Al}^{+3}$.

CONCLUSIONS

In this study, we used three metallic salts to posttreat DMDHEU-AA-crosslinked cotton fabrics, and we found that the DCRA, WCRA, TSR, and MR values of the crosslinked and posttreated fabrics were in the order of $\text{Ag}^+ > \text{Cu}^{+2} > \text{Al}^{+3}$ at the same DMDHEU-AA concentration in a padding bath. The DCRA and WCRA values of the DMDHEU-AA-crosslinked and posttreated fabrics were higher than those of the DMDHEU-AA-crosslinked fabrics, and those for crosslinked and posttreated fabrics were in the order of $\text{Ag}^+ > \text{Cu}^{+2} > \text{Al}^{+3}$ at a given values of TSR. For a given value of CL/AGU, the DCRA and TSR values for the DMDHEU-AA-crosslinked and posttreated fabrics were higher than those for the DMDHEU-AA-crosslinked fabrics, and those for crosslinked and posttreated fabrics were in the order of $\text{Ag}^+ > \text{Cu}^{+2} > \text{Al}^{+3}$; however, the WCRA values of the various

treated fabrics showed that the values for the DMDHEU-AA-crosslinked and posttreated fabrics were lower than those for the DMDHEU-AA-crosslinked fabrics, and those for crosslinked and posttreated fabrics were in the order of $\text{Ag}^+ > \text{Cu}^{+2} > \text{Al}^{+3}$. The IR spectra clearly showed the interaction between the $-\text{OH}$ group of the cellulose and the metallic ions, which were different from one another in the bonding state, and the strength of the bonding for the various metallic ions was in the order of $\text{Ag}^+ > \text{Cu}^{+2} > \text{Al}^{+3}$. All crosslinked and metallic-salt-posttreated fabric samples had good values of odor absorption, antibacterial activity, and washing fastness.

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