

# Physical Properties of Crosslinked Cellulose Catalyzed with Nano Titanium Dioxide

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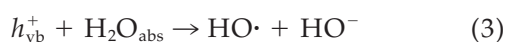
**ABSTRACT:** Four different carboxylic acids, 1,2,3,4-butane tetracarboxylic acid (BTCA), maleic acid (MA), succinic acid (SUA), and citric acid (CA), were used as crosslinking agents to treat cotton fabrics in the presence of nanometer titanium dioxide (TiO<sub>2</sub>) as a catalyst under UV irradiation. The dry crease recovery angle (DCRA) and wet crease recovery angle (WCRA) values of the treated fabrics were ranked BTCA > MA > CA > SUA and the tensile strength retention (TSR) values were ranked BTCA < MA < CA < SUA at a given resin concentration, catalyst concentration, and irradiation time period. The physical properties of the treated fabrics for nanometer silver/nanometer titanium dioxide (Ag/TiO<sub>2</sub>) catalyst showed the same tendency. At a given DCRA, the WCRA values were ranked in the order BTCA ≅ MA > CA ≅ SUA; and at a given value of the TSR, the WCRA and DCRA values were both ranked in the order BTCA > MA > CA > SUA. The softness values of the

carboxylic acid treated fabrics in the presence of nanometer TiO<sub>2</sub> catalyst were all better than that of the untreated fabric. Surface deposition of the treated fabrics for BTCA, which contains one vinyl double bond and four carboxylic acid groups, was higher than that for CA, which contains no vinyl double bond. IR spectra and electron spectroscopy for chemical analysis survey spectra showed the ester bond crosslink between the cellulose molecule and the various acids used in this study. The values of DCRA, WCRA, and add-on of the CA crosslinked fabrics for the mixed catalysts were in the order ZrO<sub>2</sub>/TiO<sub>2</sub> < SiO<sub>2</sub>/TiO<sub>2</sub> < Ag/TiO<sub>2</sub>. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 97: 2450–2456, 2005

**Key words:** crosslinking; resin; catalysts; FTIR; electron spectroscopy for chemical analysis

## INTRODUCTION

It is well known that the electron of the nanometer titanium dioxide (TiO<sub>2</sub>) particle at the valence band is excited to the conduction band under UV radiation with a wavelength of <400 nm for a few microseconds to raise the electron to the surface of the nanometer particle.<sup>1,2</sup> This phenomenon is called photoreduction, which can reduce the water molecule and hydroxyl ion that are absorbed on the surface of the other nanometer material in the reaction system and create a series reaction as follows<sup>1,3</sup>:



The carboxylic acid group and vinyl double bond of the acid crosslinking agents therefore could react with

the cellulose molecule (crosslink) and react with each other (condensation).

Acid crosslinking agents used for durable press finishing have been actively investigated in recent years.<sup>4–10</sup> Ester crosslinks have been researched for the benefit of not using formaldehyde and the improvement of the strength for tensile and abrasion. At the same time, using nanometer titanium dioxide or other nanometer materials as catalysts has been investigated for many years to enhance the catalytic effect<sup>11–15</sup> on the improvement of some properties. Using nanometer-grade TiO<sub>2</sub> as a catalyst<sup>16</sup> to improve the crease recovery property of silk fabrics has been studied and maleic anhydride has been found to be a useful crosslinking agent. However, detailed information is still lacking for the use of nanometer grade TiO<sub>2</sub> as a catalyst and carboxylic acids as crosslinking agents to treat cotton fabrics.

In this study, we use four carboxylic acid crosslinking agents, 1,2,3,4-butane tetracarboxylic acid (BTCA), maleic acid (MA), succinic acid (SUA), and citric acid (CA), whose reactive functional groups are vinyl double bonds and/or carboxylic acid groups, to treat cotton fabrics in the presence of nanometer TiO<sub>2</sub> as a catalyst under UV irradiation with a wavelength of

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**TABLE I**  
**Physical Properties of Treated Fabrics Catalyzed with Nano Titanium Dioxide**

Agents	Treating Conditions			Physical properties				
	Agents concn (%)	Cat. concn (%)	Radiation time (min)	Dry crease recovery angle ( $W + F$ )°	Wet crease recovery angle ( $W + F$ )°	Tensile strength retention (%)	$\Delta$ Stiffness (%)	
Control	—	—	—	99	169	100	0	
	2	0.8	30	211	173	96.6		
	4	0.8	30	218	178	95.2		
	6	0.8	30	220	181	94.5		
	8	0.8	30	223	184	93.7		
	10	1.0	30	221	180	92.1		
	BTCA	10	0.8	30	226	186	92.7	6.2
		10	0.6	30	223	181	94.4	
		10	0.4	30	219	174	95.7	
		10	0.2	30	202	161	96.3	
10		0.8	5	213	178	96.1		
10		0.8	15	219	181	94.8		
10		0.8	45	226	186	90.0		
2		0.8	30	209	172	96.6		
4		0.8	30	215	175	95.5		
6		0.8	30	218	178	94.8		
MA	8	0.8	30	221	181	93.9		
	10	1.0	30	220	179	92.7		
	10	0.8	30	223	183	93.6	6.0	
	10	0.6	30	221	180	94.8		
	10	0.4	30	214	172	96.1		
	10	0.2	30	200	160	97.6		
	10	0.8	5	209	175	96.5		
	10	0.8	15	215	178	95.4		
	10	0.8	45	225	183	90.9		
	2	0.8	30	206	167	97.0		
CA	4	0.8	30	213	172	95.9		
	6	0.8	30	215	175	95.3		
	8	0.8	30	218	177	94.4		
	10	1.0	30	216	176	94.0		
	10	0.8	30	218	177	93.8	5.5	
	10	0.6	30	217	174	95.0		
	10	0.4	30	212	169	96.4		
	10	0.2	30	199	159	97.8		
	10	0.8	5	205	172	96.8		
	10	0.8	15	212	173	95.9		
SUA	10	0.8	45	220	178	91.5		
	2	0.8	30	202	164	97.4		
	4	0.8	30	210	169	96.2		
	6	0.8	30	214	173	95.5		
	8	0.8	30	216	174	94.5		
	10	1.0	30	213	173	93.2		
	10	0.8	30	217	175	94.1	5.0	
	10	0.6	30	215	172	95.4		
	10	0.4	30	210	168	96.6		
	10	0.2	30	198	158	98.0		
10	0.8	5	200	171	97.2			
10	0.8	15	208	172	96.1			
10	0.8	45	217	175	91.9			

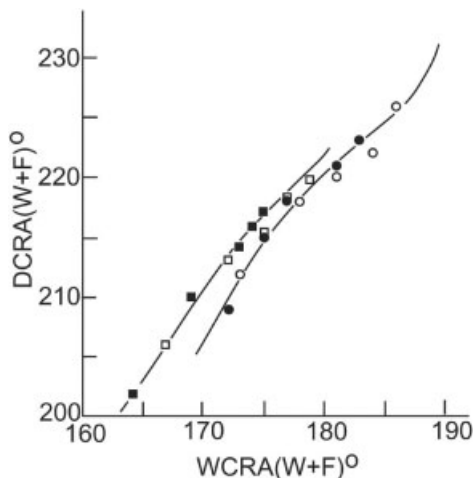
254 nm. The crease recovery angle and tensile strength of the treated fabrics are determined and/or measured by Fourier transform IR spectroscopy (FTIR), electron spectroscopy for chemical analysis (ESCA), and scanning electron microscopy (SEM) in order to research how the physical properties of the treated fabrics are affected in the presence of nanometer TiO<sub>2</sub> as a catalyst under UV irradiation, the agent distributions on

the treated fibers, and the possible reaction mechanism of crosslinking for different carboxylic acids.

## EXPERIMENTAL

### Materials

In this study, we used desized, scoured, and bleached cotton fabric with 32s × 32s ends (77) and picks (66).



**Figure 1** The relationship between the DCRA and WCRA of the various crosslinking agent treated fabrics in the presence of nano titanium dioxide catalyst: (○) BTCA, (●) MA, (□) CA, and (■) SUA.

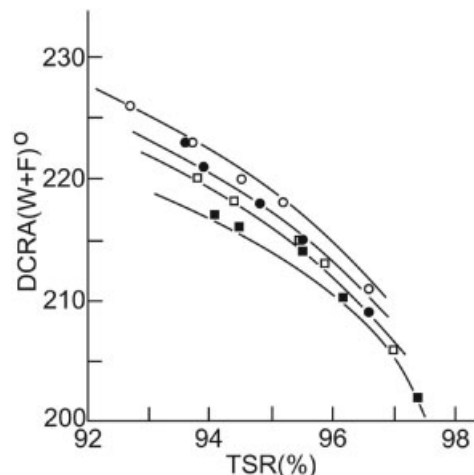
The crosslinking agents were BTCA, MA, SUA, and CA, which were supplied by First Chemical Co. (Taipei, Taiwan). Methylcellulose was obtained from Sigma Chemical Co. (St. Louis, MO). Nanometer titanium dioxide (particle size < 7 nm) was supplied by Sachtleben Chemie GmbH Co. (Duisburg, Germany). Silver powder, zirconium dioxide, and silicon dioxide (particle sizes ~ 300 nm) were obtained from Taiwan Nanotechnology Co. (Taipei, Taiwan). All other chemicals were reagent grade.

### Methods

Cotton fabric samples were padded twice to about 85% wet pickup with freshly prepared (2, 4, 6, 8, and 10%, w/w) aqueous solutions of mixtures of carboxylic acid crosslinking agents (BTCA, MA, SUA, and CA) and various concentrations of nanometer titanium dioxide catalyst. Padded fabrics were dried at 60°C for 3 min to remove the mobile water, irradiated with UV light (254 nm) for various time periods, soaped, washed, and dried.

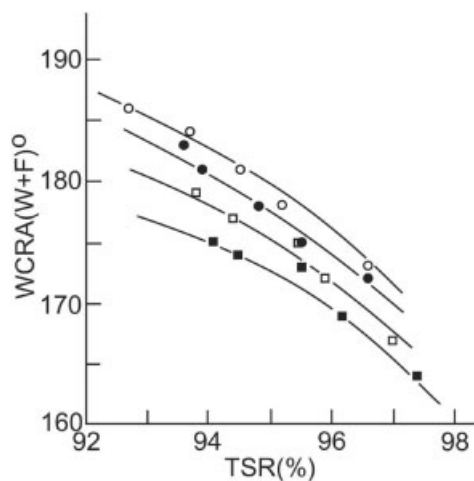
The tensile strengths of the treated fabrics were measured on an Instron tensile tester (KAO-TIEH, AT-7010 D1, Machinery Industrial Co. Ltd., Taichung, Taiwan) according to the ASTM D 1682-64 method. ASTM standard D 1295-67 was used to determine the dry crease recovery angle (DCRA) and wet crease recovery angle (WCRA). The softness values of the treated fabrics were measured with a 45° cantilever tester (Go-In International Co., Ltd., Taipei, Taiwan) according to the CNS 12915 L3233 method.

The IR spectra of the samples were obtained using a KBr disk technique.<sup>17</sup> Samples were prepared to give a dry weight of 1.8 mg after storage in 1-dram vials

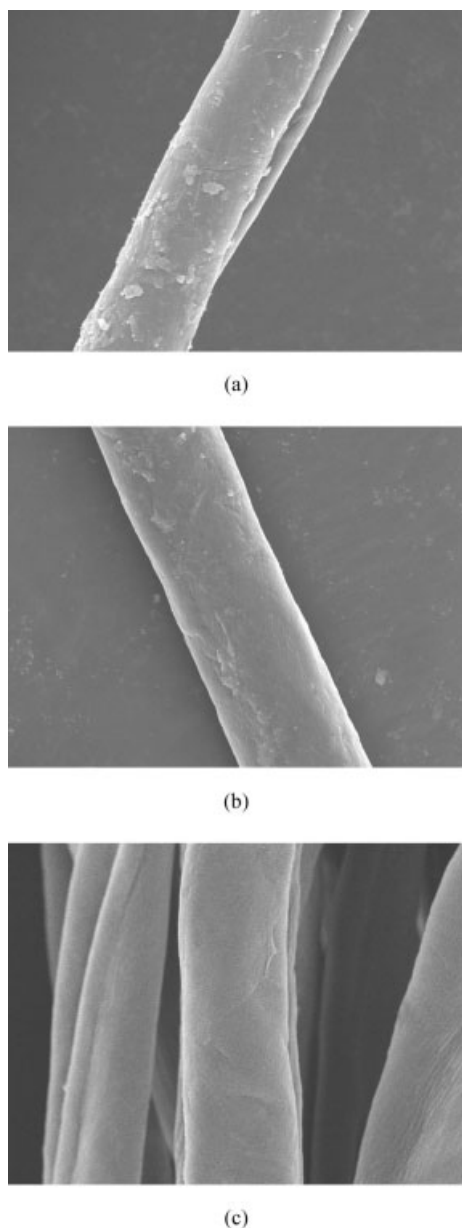


**Figure 2** The relationship between the DCRA and TSR of the various crosslinking agent treated fabrics in the presence of nano titanium dioxide catalyst: (○) BTCA, (●) MA, (□) CA, and (■) SUA.

over P<sub>2</sub>O<sub>5</sub> 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. Three mixing solutions were prepared. A mixing solution of methylcellulose (methylcellulose was selected as a model of cotton cellulose because cellulose powder is not soluble in water) and nanometer TiO<sub>2</sub> (1 wt % methylcellulose) was coated on glass, irradiated with UV for 30 min, washed with deionized water, and then dried. A mixing solution of methylcellulose and CA (10 wt % methylcellulose) was coated on glass, irradiated with



**Figure 3** The relationship between the WCRA and TSR of the various crosslinking agent treated fabrics in the presence of nano titanium dioxide catalyst: (○) BTCA, (●) MA, (□) CA, and (■) SUA.



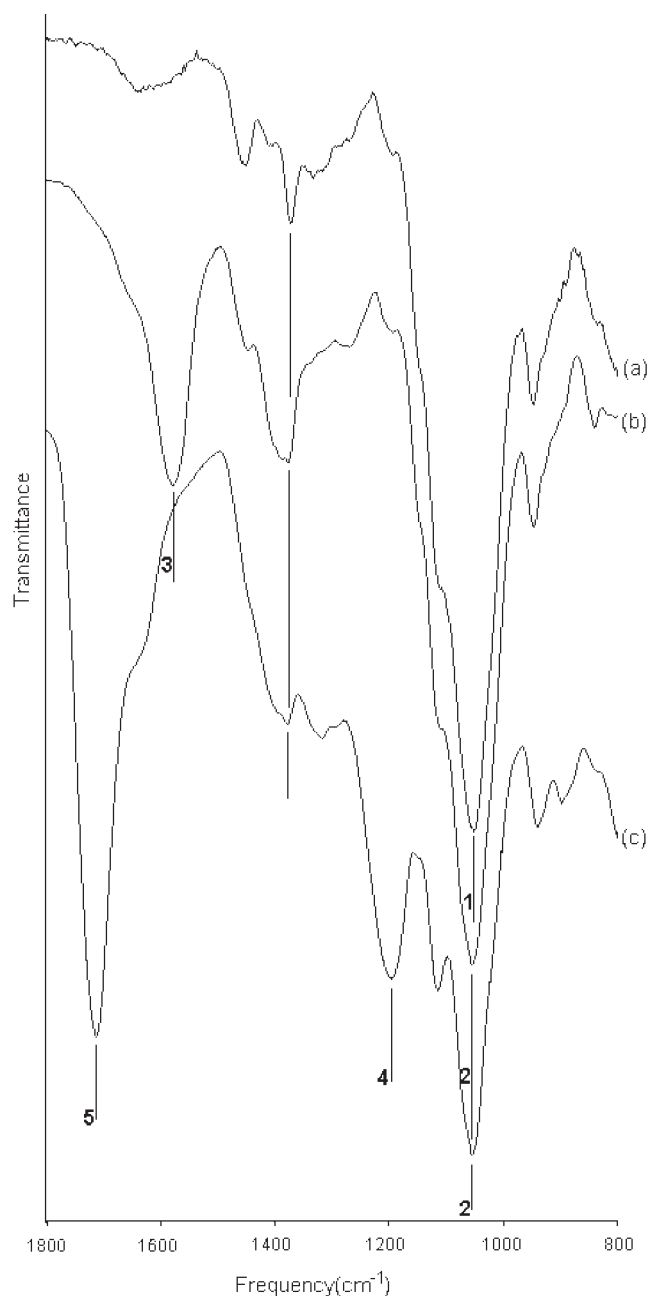
**Figure 4** SEM pictures of the (a) BTCA treated, (b) CA treated, and (c) nontreated fibers in the presence of nano titanium dioxide catalyst.

UV for 30 min, washed with 3% Na<sub>2</sub>CO<sub>3</sub> and deionized water, and dried. A mixing solution of methylcellulose, CA (10 wt % methylcellulose), and nanometer TiO<sub>2</sub> (1 wt % methylcellulose) was coated on glass, irradiated with UV for 30 min, washed with 3% Na<sub>2</sub>CO<sub>3</sub> and deionized water, and dried. All dried samples were mixed with KBr, ground, and dried in 1-dram vials over P<sub>2</sub>O<sub>5</sub> for 3 days, then pressed in an evacuated die under suitable pressure. An FTIR spectrophotometer (FTIR microscope, Perkin-Elmer) was used to obtain the spectra.

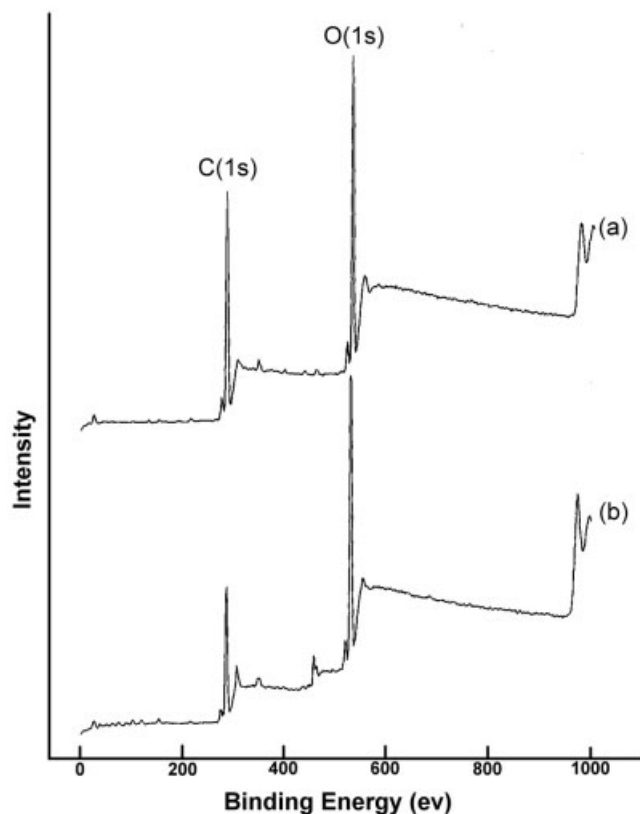
The ESCA (VG Scientific ESCALAB 250, Thermo Electron Co., Eastern Grinstead, UK) survey spectra of

the UV irradiated (10% resin concentration, 0.8% nanometer titanium dioxide catalyst, and 30-min irradiation) fabric and untreated fabrics were measured using Al K $\alpha$  irradiation operated at 15 kV and 200 W. The beam size was 650–120  $\mu$ m, and the sample size was 1  $\times$  1 cm.

The BTCA and CA treated (10% resin concentration, 0.8% nanometer titanium dioxide catalyst, and 30-min irradiation) and untreated fibers were examined with SEM (JSM-6335F, Jeol Ltd., Tachigawa, Japan) to compare the surface deposition of the crosslinking agents.



**Figure 5** The IR spectra of the UV radiated (a) mixture of methylcellulose and nano TiO<sub>2</sub>; (b) mixture of methylcellulose and citric acid; and (c) mixture of methylcellulose, citric acid, and nano titanium dioxide.



**Figure 6** The electron spectroscopy for chemical analysis (ESCA) survey spectra of the (a) CA-treated fabric in the presence of nano titanium dioxide catalyst and (b) untreated fabrics.

## RESULTS AND DISCUSSION

Table I lists the DCRA, WCRA, and tensile strength retention (TSR) of the fabrics treated with BTCA, MA, SUA, and CA under different conditions and catalyzed with nanometer titanium dioxide. The table shows that the DCRA and WCRA values for the various fabric samples gradually increase with increased crosslinking agent concentration and irradiation time period, but the TSR values decrease in all cases. It is interesting that the DCRA and WCRA values were gradually increased with the increase of the catalyst concentration and then decreased as the catalyst concentration increased, but the TSR values gradually decreased with the increase of catalyst concentration. The results obtained from the changes of the catalyst concentrations are somewhat different from the results obtained from normal crosslinking reactions in which higher DCRA and WCRA respond to lower TSR.<sup>18,19</sup> This difference may be caused by the damage of cellulose fiber and crosslinking agent under a higher nanometer titanium dioxide catalyst. Table I shows that, at a given resin concentration, catalyst concentration, and irradiation time period, the DCRA and WCRA values for the various crosslinking agents are ranked BTCA > MA > CA > SUA and the TSR

values are ranked BTCA < MA < CA < SUA. The crease recovery angles of the various treated fabrics are related to two factors: one is the number of carboxylic acid groups and the other is the content of vinyl double bonds. BTCA has four carboxylic acid groups and one vinyl double bond, MA has two carboxylic acid groups and one vinyl double bond, CA has three carboxylic acid groups and no vinyl double bond, and SUA has two carboxylic acid groups and no vinyl double bond. These results agree with the fact that a crosslinking agent with more carboxylic acid groups and vinyl double bonds will have a higher crease recovery angle in the treated fabrics under the nanometer titanium dioxide catalyst. We also found that the vinyl double bond seems to have more of an effect than the carboxylic acid group from a wet and dry crease recovery comparison between CA and MA treated fabrics. It is assumed that the carboxylic acid group and vinyl double bond act as the functional group of the crosslinking reaction in the presence of nanometer titanium dioxide catalyst. Walsh et al.<sup>20</sup> and Sumrell et al.<sup>21</sup> pointed out that the vinyl double bond of the crosslinking agents could crosslink with cellulose and react with other agents in the presence of base and irradiation conditions.

In addition, the softness values of the various treated fabrics are better than that of the untreated fabric and the improved values are ranked BTCA > MA > CA > SUA. It is believed that the crosslinking reaction for the various acids in the presence of nanometer titanium dioxide catalyst may mainly be a condensation reaction that causes the deposition of crosslinking agent in or on the treated fibers. In fact, we found that the treated fabrics have relatively lower crease recovery angles values and higher TSR values.

The relationships between the DCRA, WCRA, and TSR of the treated fabrics with the various resin concentrations are shown in Figures 1, 2, and 3, respectively. From the relationships between the DCRA and WCRA of the finished fabrics (Fig. 1), we can find that the rank of WCRA values is BTCA  $\approx$  MA > CA  $\approx$  SUA at a given DCRA. Figure 2 shows the plots of DCRA versus TSR of the finished fabrics, and the rank of DCRA values is BTCA > MA > CA > SUA for a

**TABLE II**  
Values of C<sub>1s</sub> and O<sub>1s</sub> of Untreated and Treated Fabrics Measured with ESCA

Samples	Atom (%)		O <sub>1s</sub> /C <sub>1s</sub>
	C <sub>1s</sub>	O <sub>1s</sub>	
Untreated fabric	67.75	32.25	47.60
CA-treated fabric	56.39	43.61	77.34

Treated conditions: 10% resin concentration, 0.8% nanometer titanium dioxide catalyst, and radiation time period of 30 min.

TABLE III  
Physical Properties of Treated Fabrics Catalyzed with Mixed Nanometer Grade TiO<sub>2</sub> (0.8%) and Various Compounds

Mixed catalysts	Treating conditions	Physical properties			
	Mixed ratios (w/w)	Dry crease recovery angle (W + F) <sup>o</sup>	Wet crease recovery angle (W + F) <sup>o</sup>	Tensile strength retention (%)	Add-on (%)
Control	—	199	169	100	—
—/TiO <sub>2</sub>	0/1	219	178	93.6	0.26
ZrO <sub>2</sub> /TiO <sub>2</sub>	2/1	228	191	91.7	0.92
	4/1	242	208	79.9	2.60
SiO <sub>2</sub> /TiO <sub>2</sub>	2/1	236	203	89.8	1.07
	4/1	258	219	79.5	2.69
Ag/TiO <sub>2</sub>	2/1	255	212	88.6	1.13
	4/1	266	228	79.1	2.81

Treated conditions: 10% citric acid under radiation for 30 min.

given value of the TSR. The plots of WCRA versus TSR values of the finished fabrics shown in Figure 3 reveal that the WCRA value is also ranked BTCA > MA > CA > SUA at a given TSR. The higher DCRA and WCRA of the treated fabrics may be caused by the different crosslinking conditions and deposition of different crosslinking agents in or on the treated fabrics. Our previous studies<sup>22,23</sup> showed that the surface distribution of the crosslinking agent could have a higher WCRA at the same crosslinking density and TSR.

Further, the BTCA and CA treated and the untreated fibers were examined with SEM and are shown in Figure 4(a–c). These pictures show that the surface of the BTCA treated fiber has a higher deposition of crosslinking agent, but CA treated and untreated fibers have little and no deposition of crosslinking agent, respectively. The better stiffness values of the BTCA treated fabrics are caused by the presence of the double bonds in BTCA, which can react with each other under the nanometer titanium dioxide catalyst, which can produce the hydroxyl ion and radical under UV irradiation [eqs. (2) and (3)].

Figure 5(a–c) provides the FTIR spectra of the UV irradiated mixtures of methylcellulose and nanometer titanium dioxide; methylcellulose and CA; and methylcellulose, CA, and nanometer titanium dioxide. The interesting and important absorption band for methylcellulose is the hydroxyl group at 1048 cm<sup>-1</sup> on Figure 5(a). We were interested to find that this absorption band of the hydroxyl group shifts to 1052 cm<sup>-1</sup> for both the UV irradiated samples of the mixed methylcellulose and CA and the mixed methylcellulose, nanometer titanium dioxide, and CA shown in Figure 5(b,c). At the same time, the carboxylic acid ionic group at 1581 cm<sup>-1</sup> shown in Figure 5(b) disappears and the spectrum of Figure 5(c) generates two new absorption bands at 1196 and 1714 cm<sup>-1</sup>. These are believed to be the ester and carbonyl group<sup>24</sup>

formed from the reaction between the carboxylic acid group of CA and methylcellulose. The slight shift of the hydroxyl group at 1048 cm<sup>-1</sup> is assumed to be the change of the environment around the group of methylcellulose. The crease recovery property of the various treated fabrics is caused by the crosslinking reaction between the cellulose molecule and the various acids used in this study to form the ester bond.

The ESCA survey spectra of the untreated and CA treated fabrics are shown in Figure 6(a,b). They reveal that the C<sub>1s</sub> values decreased and O<sub>1s</sub> values increased as the fabric sample was treated with CA in the presence of nanometer titanium dioxide catalyst (Table II). The results strongly support the crosslinking reaction between the carboxylic acid group of CA and the cellulose molecule in the presence of nanometer titanium dioxide catalyst.

In order to study the effect of the mixed catalysts only on the crosslinking reaction of the carboxylic acid group, CA was selected to treat the cotton fabric with 10% resin concentration, 0.8% nanometer titanium dioxide catalyst, and an irradiation time period of 30 min. Some of the physical properties obtained are listed in Table III. They show that the DCRA and WCRA and the values of the add-on increased when cocatalysts were used. The series of mixed catalysts are ranked ZrO<sub>2</sub>/TiO<sub>2</sub> < SiO<sub>2</sub>/TiO<sub>2</sub> < Ag/TiO<sub>2</sub>. It is interesting that this series agrees with the known series of redox potential values of cocatalysts ZrO<sub>2</sub>, SiO<sub>2</sub>, and Ag. Some studies<sup>1,25</sup> pointed out that the excited nanomaterials elongate the time period of the excited state of the nanomaterials by the transmission of the electron to the other nanomaterials and this mechanism may improve the catalytic effect of the nanometer titanium dioxide. Those results strongly support the idea that the degree of this transmission mechanism is related to the series of redox potential values of the cocatalysts.

TABLE IV  
Physical Properties of Treated Fabrics with Different Crosslinking Agents and Catalyzed with Nano Ag/TiO<sub>2</sub>

Agents	Treating conditions			Physical properties			
	Agents concn (%)	Cat. concn (%) Ag/TiO <sub>2</sub>	Radiation time (min)	Dry crease recovery angle (W + F) <sup>o</sup>	Wet crease recovery angle (W + F) <sup>o</sup>	Tensile strength retention (%)	Add-on (%)
Control	—	—	—	199	169	100	0
BTCA	10	2.4/0.8	30	270	235	81.4	2.8
MA	10	2.4/0.8	30	266	228	81.9	2.6
CA	10	2.4/0.8	30	256	212	83.7	2.3
SUA	10	2.4/0.8	30	251	209	84.9	2.1

Table IV shows some of the physical properties of fabrics treated with the various acid crosslinking agents in the presence of nanometer Ag/TiO<sub>2</sub> catalyst. It reveals that the values of DCRA, WCRA, and add-on of the treated fabrics are in the order BTCA > MA > CA > SUA at the same crosslinking agent concentration, but the rank for TSR values is inverse. This rank agrees with that obtained from a single nanometer TiO<sub>2</sub> catalyst shown in Table I. This result again confirms that the crosslinking reaction in the presence of a nanometer single catalyst (TiO<sub>2</sub>) and the mixed catalyst (Ag/TiO<sub>2</sub>) stemmed from the effects of the two factors (carboxylic acid group and vinyl double bond) on the crosslinking agents.

### CONCLUSIONS

In this study, we used four different carboxylic acid crosslinking agents to treat cotton fabrics in the presence of nanometer TiO<sub>2</sub> as a catalyst under UV light irradiation. The results showed that, at a given resin concentration, catalyst concentration, and irradiation time period, the DCRA and WCRA values for the various crosslinking agents were BTCA > MA > CA > SUA, and the TSR values were BTCA < MA < CA < SUA. The softness values of the various treated fabrics are better than that of the untreated fabric. The WCRA values were in the order BTCA ≈ MA > CA ≈ SUA at a given DCRA, and the WCRA and DCRA values were both BTCA > MA > CA > SUA for a given TSR value. From SEM pictures, we found that the surface of the BTCA treated fiber had a higher deposition of crosslinking agent, but CA treated and untreated fibers had little and no deposition of crosslinking agent, respectively. IR spectra and ESCA survey spectra showed that the crease recovery property of the various treated fabrics was caused by the crosslinking reaction between the cellulose molecule and the various acids used in this study to form an ester bond. The values of DCRA, WCRA, and add-on

of the CA crosslinked fabrics for the various mixed catalysts were ZrO<sub>2</sub>/TiO<sub>2</sub> < SiO<sub>2</sub>/TiO<sub>2</sub> < Ag/TiO<sub>2</sub>. Those values for the various crosslinking agents were in the order BTCA > MA > CA > SUA in the presence of nanometer Ag/TiO<sub>2</sub> catalyst.

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