Mechanism of Paper Wet Strength Development by Polycarboxylic Acids with Different Molecular Weight and Glutaraldehyde/Poly(vinyl alcohol)

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ABSTRACT: In our previous research, we found that crosslinking paper using poly(carboxylic acid)s with different molecular weight or using the combination of glutaraldehyde and poly(vinyl alcohol) (PVA) significantly improved the wet strength of the paper. In this research, we studied the mechanism of paper wet strength development using crosslinking systems with different molecular weight by measuring scanning electron microscopic (SEM) images, wet strength, folding endurance, wet thickness, water retention, and Z-direction tensile strength of the treated paper. The paper crosslinked by a high-molecular weight (MW) poly(carboxylic acid) shows more swelling by water than that crosslinked by a low-MW polycarboxylic acid in the SEM micrographs even though both treated paper samples have similar wet strength. Thus, the data suggest that high-MW poly(carboxylic acid)s promote the formation of interfiber crosslinking. Crosslinking paper by glutaraldehyde, a

INTRODUCTION

Paper loses most of its strength when it is exposed to a high-humidity environment or soaked in water. The lost of paper strength is attributed to the penetration of water into paper web and destruction of hydrogen bonds, which hold the cellulosic fibers together.¹ Crosslinking agents have been used to improve the wet strength of paper.² However, the wet-strength resins used by the industry are currently under scrutiny because of their emission of toxic formaldehyde or absorbable organic halides during production and use of paper products.³

Polymeric carboxylic acids have been investigated as environment-friendly wet strength agents of paper.^{4–9} We found that polymeric carboxylic acids of small molecular sizes, such as 1,2,3,4-butanetetracarboxylic acid (BTCA), significantly reduces paper flexibility and diminishes its folding endurance, whereas acids with high-molecular weight (MW), such as poly-

Journal of Applied Polymer Science, Vol. 101, 277–284 (2006) © 2006 Wiley Periodicals, Inc. crosslinking agent of small molecular size, improves wet strength and reduces flexibility and swellability of paper because of the formation of intrafiber crosslinking. Combining glutaraldehyde with PVA as a coreactant increases wet strength and also retains flexibility and swellability of the treated paper because of the formation of interfiber crosslinking. The hypothesis that PVA reacts with glutaraldehyde to form a polymeric pentanedialated-PVA crosslinking system and promotes the formation of interfiber crosslinking on the paper is supported by the data of wet strength, folding endurance, wet thickness, water retention, and Z-direction tensile strength of the treated paper. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 101: 277–284, 2006

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(methyl vinyl ether-*co*-maleic acid) (PMMA), are able to retain the flexibility and folding endurance of the treated paper.^{7,8} In our previous research, we also found that the use of fully hydrolyzed PVA as a coreactant improves the wet strength without reducing the folding endurance of the paper crossliniked by a poly-(maleic acid) with a small molecular size (PMA) and that crosslinked by glutaraldehyde.^{9–11}

The objective of this research was to study the macrostructure, wet strength retention, folding endurance, water sorption, wet thickness, and Z-direction tensile strength of the paper crosslinked by polymeric carboxylic acids with different molecular sizes and that by the combination of glutaraldehyde and PVA, so that we can understand the mechanism of paper wet strength development by crosslinking systems with different molecular sizes.

EXPERIMENTAL

Materials

The paper used was an unbleached Kraft paper with grammage of 65 g/m². PMA with a number-averaged molecular weight of \sim 800 was a 50% aqueous solution

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with the trade name of "Belclene 283" supplied by FMC (Princeton, NJ). PMMA, a solid polymer with a number-averaged MW (Mn) of 1,130,000, and sodium hypophosphite (NaH₂PO₂) were supplied by Aldrich, WI. Glutaraldehyde was a 50% aqueous solution supplied by Union Carbide Specialty Polymers and Products (Bound Brook, NJ). The fully hydrolyzed PVA with a viscosity of 62–72 cps (abbreviated as "PVA") and another PVA with a viscosity of 28–32 cps (abbreviated as "PVA-LMW") were supplied by Air Products (Allentown, PA).

Scanning electron microscopy

The paper was treated by 2.0 PMA or 2.0% PMMA with 1% NaH₂PO₂ as the catalyst, and cured at 170°C for 1.5 min as described previously.⁸ Paper swelling was measured by immersing the paper stripes of 1.5 cm width in a 50% aqueous methanol solution. The solution was brought to boiling, and then cooled down. The paper strips were soaked in the solution overnight. A critical point drying (CPD) method, which was used to preserve the swollen state of the fiber network, was performed using the paper specimens, which underwent exchanging by a series of aqueous ethanol solutions with ascending concentrations (50, 70, 85, 95, and 100%), followed by drying in a Samdri 780 Critical Point Dryer. For scanning electron microscopy (SEM) measurement, the paper stripes were cut into small pieces with a sharp razor. The specimens were mounted perpendicularly on aluminum stubs with a carbon tape for observation of the transverse section. The specimens were then coated with gold on a SPI sputter coater. The specimens were examined with a JEOL JSM 5800 scanning electron microscope.

Measurement of paper strength and folding endurance

The wet strength and Z-direction tensile strength of the paper were evaluated according to TAPPI test methods T456 om-87 and T-541 om-99, respectively. For wet tensile strength measurement, the specimens were soaked in distilled water for 24 h before testing. Ten measurements were performed, and the data were averaged for each tensile strength value. The wet strength retention (%) was expressed as a ratio of wet strength of treated sample to the dry strength of control sample (*W/D* ratio). Folding endurance of the treated paper sheets was evaluated according to TAPPI standard test methods T511 om-96.

Measurement of paper thickness

The thickness of paper sheets was measured according to TAPPI test method T411 om-97. The data from 20

measurements were averaged for each thickness value.

Measurement of paper water retention

Water retention was measured using a centrifuge method.¹² Small paper stripes (~0.5 g) were soaked in water for 24 h, and then placed in centrifuge tubes with perforated support. The sample was centrifuged at 3500 rpm for 6 min to remove mechanically held water from the capillaries in the fiber networks of the paper, and then transferred to a weighing bottle to measure wet weight (W_w). The sample was then dried at 90°C for 10 min before being measured for its dry weight (W_d). The water retention (%) was determined according to the following formula: [($W_w - W_d$)/ W_d] × 100%.

RESULTS AND DISCUSSION

Paper crosslinked by polycarboxylic acids with different molecular weight

The cross sections of the dry and CPD wet paper samples are shown in Figure 1. The dry paper shows a dense cross section and all the lumens appear to be collapsed [Fig. 1(A)]. The fibers are held together with a few cavities among them. The paper was first swollen by water, and then dried by the CPD method. The purpose of using the CPD method was to preserve the swollen state of the fibers on the paper. The fibers in the wet paper [Fig. 1(B)] are obviously separated, which is a sharp contrast to the fibers in the dry paper [Fig. 1(A)]. The wet paper shown in Figure 1(B) has a thickness approximately twice as that of the dry sample [Fig. 1(A)] due to the swelling of the paper web by water and the consequent separation of the fibers.

The cross sections of the CPD wet paper, which was treated by PMA with molecular weight (MW) of approximately 800, are shown in Figure 2(A). It can be seen in Figure 2(A) that few fibers are swollen and the spaces between fibers are abundant. The reduced fiber swelling is probably caused by the intrafiber crosslinks formed by the low MW PMA, whereas the separation between fibers is an indication of lack of interfiber crosslinks. The data presented here suggest that the PMA molecules are small enough to penetrate through pores into the fiber wall and produces predominantly intrafiber crosslinks, but few interfiber crosslinks.

For the paper treated by PMMA with MW of 1,130,000, the topographical feature of the cross section of its CPD wet sample [Fig. 2(B)] is different. The SEM micrograph [Fig. 2(B)] shows closer contact among the fibers than that of the paper treated with PMA [Fig. 2(A)]. More fibers are shown to be swollen in Figure 2(B) than in Figure 2(A), as indicated by





Figure 1 SEM micrographs of the cross sections of untreated paper: (A) dry; (B) CPD wet.

more visible and larger lumens seen in Figure 2(B). Fiber interior swelling indicates a low level of intrafiber crosslinks, whereas close contact between fibers suggests strong interfiber bonding.

Swelling is one of the physical properties closely associated with water content in a cellulosic fiber.^{13,14} Cellulosic fibers consist of a large number of discontinuous crystalline regions separated by amorphous regions of cellulose macromolecules with fairly strong interchain hydrogen bonds. Liquid water flows into the interfiber capillaries and also penetrates though the fiber wall and exerts an osmotic pressure, which causes the fibers to swell.¹⁵ Water molecules break the hydrogen bonds between the cellulose molecules and form their own hydrogen bonds with cellulose, thus loosening fiber structure and diminishing paper strength. For a crosslinked cellulosic fiber, the swelling of the cellulosic networks in the fiber reflects the distribution of crosslinks in the treated paper, which is dependent upon the accessibility of cellulosic fibrous substrate by a crosslinking agent. The drastic difference in molecular size between PMA and PMMA results in different depth of penetration and consequently different locations in the fiber networks where crosslinking takes place.

PMA with MW of 800 is able to diffuse freely into a fiber's interior and produces mostly interfibrillar and interlamellar crosslinks inside the fibers. Those intrafiber crosslinks restrict the accessibility of the fiber microstructure by water and reduces swellability of the fiber, thus protecting the fiber-to-fiber hydrogen bonding and improving the fiber's wet strength. The intrafiber also reduced the mobility of the microcomponents in the fibers. Reduced fiber flexibility is the reason why the paper treated with the low MW crosslinking agents, such as BTCA and PMA described earlier, has low-folding endurance as discovered in our previous research.^{5,6}

PMMA, on the other hand, has much larger molecular size with MW of 1,130,000. Because of its size,





Figure 2 SEM micrographs of the cross sections of the CPD wet paper, which was treated with (A) 2.0% PMA; (B) 2.0% PMMA and cured at 170°C for 1.5 min.

	W/D (%) ^a Curing temperature (°C)			Folding endurance (times) ^a Curing temperature (°C)		
Crosslinker						
	140	150	160	140	150	160
2% PMA 2% PMMA	31 36	42 44	54 51	292 596	202 600	126 581

^a The W/D% for the untreated control sample was 11%. The folding endurance for the untreated control sample was 496.

PMMA stays on fiber surface or moves to the crossover areas of fibers, thus producing fiber-polymerfiber bonds. PMMA may also partly diffuse into fiber wall through large pores, leading to stronger resinfiber anchoring. The increased interfiber crosslinks protect the fiber–fiber hydrogen bonds from disruption by water, thus increasing the wet strength. Interfiber crosslinks do not have drastic impact on the swellability and flexibility of fibers. Water molecules are still able to penetrate freely into fiber interior and cause significant fiber expansion.

The paper was treated with the low MW PMA (2.0%) and the high MW PMMA (2.0%), both in combination with 1% NaH₂PO₂ as the catalyst, and cured at temperatures ranging from 140 to 160°C for 1.5 min. The wet strength and folding endurance of the paper thus treated are presented in Table I. The wet strength (*W/D* ratio) of the control sample (untreated paper) is 11%. The wet strength for the PMA-treated paper increases from 31 to 54% whereas that for the PMMAtreated paper increases from 36 to 51% as the temperature increases from 140 to 160°C (Table I). The improvement in the wet strength for the PMA-treated paper and that for the PMMA-treated paper is comparable. The folding endurance for the paper treated by the two different crosslinkers, however, is completely different. The control has the folding endurance of 496. The folding endurance drastically reduced to 292 when the paper treated with PMA was cured at 140°C (Table I). The folding endurance decreases further to 126 as the curing temperature increases to 160°C (Table I). For the PMMA-treated paper, its folding endurance increases to 581-600 after the treatment (Table I). The data presented here show that the predominantly interfiber crosslinks on the treated paper formed by the high MW PMMA increases both the wet strength and the flexibility of the treated paper, whereas the intrafiber crossliks formed by the low MW PMM increases paper wet strength at the expense of its folding endurance.



Figure 3 Wet strength (W/D ratio) of the paper treated by 2.0% glutaraldehyde with and without PVA as a function of the curing temperatures.

Paper treated with glutaraldehdye and poly(vinyl alcohol)

The paper was treated with 2.0% glutaraldehyde, 1.0% PVA, and 0.76% Zn(NO₃)₂ as a catalyst and then cured at 120, 130, 140, and 150°C for 1.5 min. The paper was also treated with glutaraldehyde, but without PVA, for the purpose of comparison. The W/D ratio of the paper thus treated is presented in Figure 3. The W/D ratio of the treated paper increases as the curing temperature is increased with or without the presence of PVA, but the paper treated with PVA shows considerably higher wet strength retention than that treated without PVA at all four temperatures (Fig. 3). It is obvious that the use of PVA as a coreactant brings about significant increase in the wet strength retention of the paper crosslinked by glutaraldehyde.

The W/D ratio of the paper treated with 2.0% glutaraldehyde and cured at 140°C for 1.5 min is presented as a function of the concentration of PVA added in Figure 4. The W/D ratio increases from 51% without PVA to 71% with 3.0% PVA added (Fig. 4). The amount of PVA added shows such a profound impact on the wet strength retention of the treated



Figure 4 Wet strength (W/D ratio) of the paper treated by 2.0% glutaraldehyde and cured at 140°C for 1.5 min as a function of PVA concentration.



Figure 5 Wet strength (W/D ratio) of the paper treated by 2.0% glutaraldehyde without PVA, with a high-MW PVA ("PVA"), and with a low-MW PVA ("PVA-LMW"), and cured for 1.5 min as a function of curing temperature.

paper, indicating that PVA plays an important role in the crosslinking of cellulose fibers by glutaraldehyde.

The importance of PVA in increasing the W/D ratio of the paper crosslinked by glutaraldehyde is also shown by the effect of the MW of PVA on the wet strength retention of the treated paper. The paper was treated with 2.0% glutaraldehyde and 0.76% $Zn(NO_3)_2$ in combination with two different PVA samples (1.0%). The PVA used previously has higher MW and a viscosity of 62–72 cps, whereas the new one (abbreviated as PVA-LMW) has lower MW and a viscosity of 28–32 cps. The wet strength retention of the treated paper is presented as a function of the curing temperature in Figure 5. In the entire curing temperature range, the paper treated with the higher MW PVA as the coreactant shows significantly higher W/D ratio than that treated with lower MW PVA, which, in turn, has higher W/D ratio than that treated without PVA (Fig. 5). When the curing temperature is 130°C, the W/D ratio increases from 44% without PVA to 47% when 1.0% PVA-LMV is added, and it increases further to 53% when 1.0% PVA is used. Evidently, the molecular size of PVA as a coreactant also has a profound impact on the wet strength retention of the paper crosslinked by glutaraldehyde.

The folding endurance of the paper treated with 2.0% glutaraldehyde and cured at 130°C for 1.5 min is shown as a function of the concentration of PVA added in Figure 6. The folding endurance of the untreated paper (control) is 496. It decreases to 348 when the paper is treated with 2.0% glutaraldehyde (Fig. 6). Crosslinking the paper with a small MW reagent, such as the low MW PMA discussed previously, reduces paper flexibility and diminishes the folding endurance of the crosslinked paper. When 0.5% PVA is used as a coreactant for the treatment, the folding endurance increases to 522, which is higher than that of control (Fig. 6). It increases further to 633 and 748 when 1.0 and 3.0% PVA, respectively, is used (Fig. 6). Thus, the

data demonstrate that using PVA as a coreactant significantly improves the flexibility of the paper crosslinked by glutaraldehyde.

Glutaraldehyde has a small molecular size with MW of 110; therefore, it is able to penetrate into a cellulose fiber and to form hemiacetal and acetal crosslinks between the adjacent cellulose chains inside the fiber. The crosslinking by glutaraldehyde restricts the accessibility and swelling of the cellulosic fiber by water, protects the interfibril and interlamellae hydrogen bonds from disruption by water, thus preserving the paper wet strength. Consequently, raising the curing temperature increases the amount of crosslinking inside a fiber, thus improving the W/D ratio of the treated paper.

For crosslinking agents of small molecular sizes, the improvement in paper wet strength is always achieved in the expense of paper folding endurance, indicating formation of intrafiber crosslinking reduces paper flexibility.⁴⁻⁷ When a high-molecular weight polymeric crosslinking agent is used, we found that both wet strength and folding endurance of the treated paper is increased in our previous research.^{6,7} The phenomenon of adding PVA in a crosslinking system increases both the wet strength retention and the folding endurance of the crosslinked paper (Figs. 3–6) is most likely due to the reactions of PVA with glutaraldehyde. PVA reacts with glutaraldehyde and chains up the small glutaraldehyde to form a bulky pentanedialated-PVA, as shown in Scheme 1. The pentanedialated-PVA carries highly reactive aldehyde and hemiacetal groups, which are able to react with cellulose to form a crosslinking network. Because of its bulky size, the pentanedialated-PVA cannot penetrate into fiber interior but stays on fiber surface, thus producing interfiber crosslinks. It may also partly diffuse into large pores and voids on the fiber surface. Consequently, the amount of intrafiber crosslinking is reduced when compared with the paper treated with glutaraldehyde alone, thus leading to less restrain on the fiber swelling. As a result, pentanedialated-PVA



Figure 6 Folding endurance of the paper treated by 2.0% glutaraldehyde and cured at 130°C for 1.5 min as a function of PVA concentration.

Scheme 1 Pentanedialated-PVA.

not only shows higher effectiveness in increasing paper wet strength, but it is also capable of preserving and improving the folding endurance than glutaraldehyde does. The combination of glutaraldehyde and the higher MW PVA (PVA) probably forms more interfiber bonding, thus becomes even more effective in improving the wet strength of the crosslinked paper than the combination of glutaraldehyde and the lower MW PVA (PVA-LMW).

Presented in Figure 7 is the wet thickness of the paper treated with 2.0% glutaraldehyde as a function of the W/D ratio. The different W/D ratios of the treated paper are a result of different curing temperature as discussed earlier. The data presented here show that the wet thickness of the paper treated with both glutaraldehyde and glutaraldehyde/PVA decreases as the wet strength retention increases and that the paper treated with glutaraldehyde/PVA has notably higher wet thickness than that treated with glutaraldehyde alone at all four different curing temperatures (Fig. 7). Shown in Figure 8 is the wet thickness of the paper treated with 2.0% glutaraldehyde as a function of the concentration of PVA added. One observes that the wet thickness of the treated paper increases as the amount of added PVA increases (Fig. 8).



Figure 7 Wet thickness for the paper treated by 2.0% glutaraldehyde with and without 1.0% PVA and cured at 120, 130, 140, and 150°C for 1.5 min as a function of wet strength retention (wet thickness for control: 4.30×10^{-4} in.).



Figure 8 Wet thickness for the paper treated with 2.0% glutaraldehyde and cured at 140°C for 1.5 min as a function of PVA concentration.

As we discussed earlier, the reactions of PVA with glutaraldehyde and the formation of a polymeric pentanedialated-PVA crosslinking system (Scheme 1) promotes the formation of interfiber crosslinking, thus leading to less restrain on the fiber swelling. As a result, the wet thickness of the paper treated with glutaraldehyde/PVA increases as shown in Figures 7 and 8. The wet thickness data presented here is consistent with the data of wet strength retention and folding endurance of the crosslinked paper presented previously.

Another phenomenon accompanying the swelling of cellulosic substrate is the water retention. The sorption of water by cellulose substrate is generally considered as surface adsorption, and the sorption capacity of the fiber is related to its internal surface.¹⁶ There are two types of sorbed water: (1) the water in which energy of binding to the cellulose is higher than the energy of interaction among the molecules of water; and (2) the water that condenses inside the cellulosic structure without a thermal effect. The sorption of water reflects the submicroscopic structure of fibers. Since the retained water is attached only to fiber surface as well as imbedded inside the capillaries, it is necessary to remove the mechanically held water from the capillary and interfiber areas, so that the true water content in the fiber wall can be measured.¹⁵ The centrifuge method is designed for this purpose.

Figure 9 shows the water retention of the paper treated with 2.0% glutaraldehyde with and without 1.0% PVA, and cured at 120, 130, 140, and 150°C for 1.5 min. The water retention is expressed as a function of W/D in Figure 10. The control sample has water retention of 83.0% and a W/D ratio of 10%. The water retention of the treated paper decreases with increasing wet strength for both the paper treated with 2.0% glutaraldehyde without PVA and that treated with 2.0% glutaraldehyde with 1.0% PVA (Fig. 9). The data presented in Figure 9 also show that the water retention of the paper treated with glutaraldehyde/PVA is 5–8% higher than that treated with glutaraldehyde



Figure 9 Water retention (%) of the paper treated by 2.0% glutaraldehyde with and without 1.0% PVA and cured at 120, 130, 140, and 150°C for 1.5 min as a function of the wet strength retention (water retention for control: 83%; *W/D* ratio for control: 10%).

alone at the same wet strength levels (Fig. 9). Addition of 1.0% PVA as a coreactant causes significantly less reduction in water sorption of the crosslinked cellulose fiber.

Presented in Figure 10 is the water retention (%) of the paper treated with 2.0% glutaraldehyde as a function of the amount of added PVA. The water retention increases from 51.6 to 56.7% as the amount of added PVA increases from 0 to 3.0% (Fig. 10). The increase in water retention is an indication of the loosening of the fiber wall structure.¹⁷ Water sorption of paper generally decreases with increasing wet strength. The paper wet strength data presented here, however, show the contrary. The wet strength retention increases as the amount of added PVA increases (Fig. 4). A most probable explanation is that PVA reacts with glutaraldehyde on the fiber surfaces and produces predominately interfiber bonding, thus enhancing the paper wet strength. The reaction between PVA and glutaraldehyde also reduces the intrafiber crosslinking by glutaraldehyde, thus increasing water retention. The data presented earlier support the hypothesis that the



Figure 10 Water retention (%) of the paper treated by 2.0% glutaraldehyde and cured at 140°C for 1.5 min as a function of PVA concentration.



Figure 11 *Z*-direction tensile strength of the paper treated by 2.0% glutaraldehyde and cured at 140°C for 1.5 min as a function of PVA concentration.

glutaraldehyde/PVA crosslinking system produces interfiber crosslinking and improves fiber–fiber bonds.

Z-direction tensile strength is frequently used to characterize the fiber-fiber bonds of paper.¹⁸ The Zdirection tensile strength of the paper treated with 2.0% glutaraldehyde in combination with PVA of different concentrations and cured at 140°C is shown as a function of PVA concentration in Figure 11. The Zdirection tensile strength of the paper treated with glutaraldehyde increases from 107 psi without PVA to 122 psi with the addition of 3.0% PVA (Fig. 11). As discussed earlier, the increase in Z-direction tensile strength is evidently attributed to the enhancement of fiber-fiber bonds. Therefore, the Z-direction tensile strength data provides additional evidence that adding PVA to the glutaraldehyde crosslinking system as a coreactant promotes the formation of interfiber binding.

CONCLUSIONS

This study elucidates the mechanisms of wet strength development of the paper crosslinked by polycarboxylic acids with different MW and that crosslinked by the combination of glutaraldehyde and PVA. The high-MW copolymer of maleic acid (PMMA) produces predominantly interfiber crosslinks, whereas the low-MW polymer of maleic acid (PMA) produces predominantly intrafiber crosslinks. PVA, used as coreactant for glutaraldehyde, reacts with glutaraldehyde under curing conditions, thus directly participating in the crosslinking of cellulose by glutaraldehyde and promoting the formation of interfiber crosslinks. The interfiber crosslinking enhances the effectiveness of a crosslinking system for improving paper wet strength without sacrificing its flexibility. The hypothesis that the polymeric crosslinking system based on reaction of glutaraldehyde and PVA promotes the formation of interfiber crosslinking is supported by the data of wet strength, folding endurance, wet thickness, water retention, and Z-direction tensile strength of the treated paper.

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