# *Deposition of polyelectrolyte complexes as a mechanism for developing paper dry strength*

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**ABSTRACT:** Fibers from recycled copy paper were pretreated with two polymers, cationic poly-DADMAC followed by anionic CMC. Results were slightly different from the results of earlier work, which showed that the maximum strength was obtained when the amount of poly-DADMAC was just sufficient to saturate the fibers. In this study, higher tensile strength was obtained when poly-DADMAC addition exceeded the saturation level. It was hypothesized that the strength increase arises from the formation of polyelectrolyte complexes (PECs) in the bulk phase followed by deposition of PECs onto the fibers. Pre-formed complexes were retained efficiently by the fibers, especially when their surfaces had been pretreated with poly-DADMAC to saturation. Surprisingly, such pretreatment increased the retention efficiency of all of the PEC mixtures tested. PEC deposition yielded an additional increase of about 13% in dry strength beyond what could be achieved by treatments not involving complexation.

***Application:*** Dual treatment with dry-strength polymers may boost the strength of paper made from recycled copy paper.

The strength of bonds between cellulosic fibers plays a key role in meeting the dry-strength requirements of various paper products [1–2]. In cases where bond strength can be improved, there are opportunities for reducing basis weight [3], using less expensive fibers [4–5], and using more inexpensive mineral fillers [6–8]. One of the most promising ways to increase the bonding strength in a paper sheet is to use dry-strength chemical additives, including cationic starch and various synthetic polymeric additives [9–11].

A previous study focused on the optimization of a particular type of dry-strength additive program [12]. The system consisted of a highly charged cationic polymer followed by an anionic polymer. The fiber furnish was virgin unbleached kraft. Compression strength was maximized when the fibers were first treated with a sufficient amount of the cationic polymer to saturate the fiber surfaces. The furnish was then treated with a constant amount of the anionic polymer.

From that study it was proposed that the saturated condition yielded the greatest strength development because it maximized the net amount of adsorbed polymer. The strength benefit from the adsorbed polyelectrolytes was not limited to the first generation of paper being produced. Rather, the benefit carried over to provide increased strength when the same paper was redispersed and formed into another generation of recycled paper.

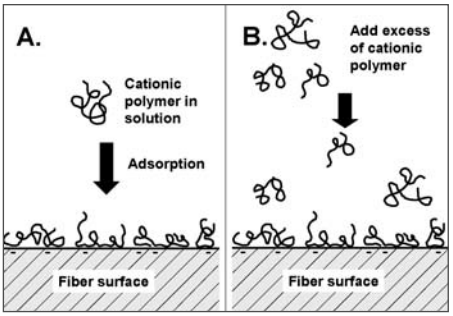
The current study was undertaken to find out if a similar chemical approach could be applied to fiber recycled from xerographic copy paper. This pulp is representative of the mixed office waste category of recycled fiber that is playing an increasingly important role as a component of modern paper products. Compared to virgin unbleached kraft fiber [12], fibers from mixed office waste have a lower density of negative charge [13–14], even aside from the charge effects of such papermaking additives as cationic starch [6, 7, 10]. Also, the fibers from mixed office waste have less pore volume as a result of pores collapsing during drying [15–17]. This factor would tend to decrease the amount of polyelectrolyte that could be adsorbed onto those surfaces.

## THEORY

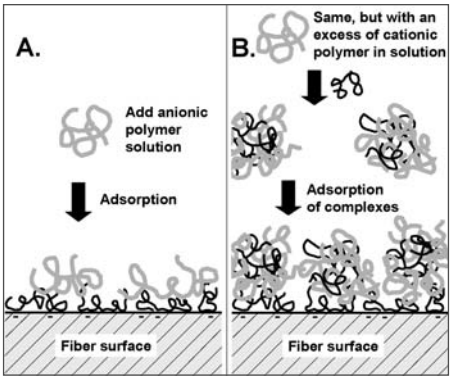
To lay the groundwork for analyzing the data, it is worth considering polyelectrolyte complexation and the deposition of polyelectrolyte complexes onto fibers. How do polyelectrolytes affect the amounts of strength-enhancing polymers that can adsorb onto fiber surfaces? Increased amounts of water-loving polyelectrolytes adsorbed on fiber surfaces lead to increased strength when the paper is dried, as previous studies have shown [2, 4–12].

A proposed mechanism is illustrated in **Fig. 1**, which contrasts two idealized cases. In Case A, the amount of highly charged, cationic polyelectrolyte added to an aqueous suspension of cellulosic fibers is just sufficient to cover the available surfaces completely. A previous study identified this condition of surface saturation [12] as being advantageous as a starting point for a two-polymer, dry-strength treatment in the case of unbleached kraft fibers. In Case A, little or no cationic polymer remains in the free solution after the fiber suspension is stirred for an adequate length of time. By contrast, when an excess of the same cationic polymer is added, as in Case B, the surface becomes saturated, and an excess of cationic polymer also remains in solution.

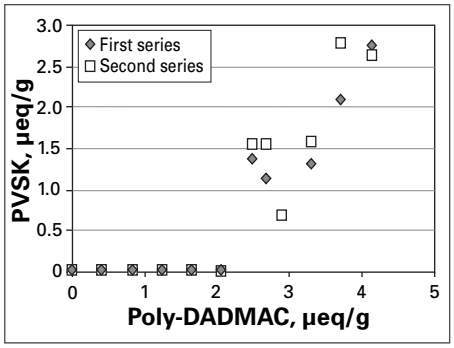
In **Fig. 2**, a solution of negatively charged dry-strength polymer is added in each idealized case. In Case A, the anionic polyelectrolyte molecules form a second layer, adsorbing on top of the layer of cationic polymer. Strong adsorption of polyelectrolytes, reversing the initial surface charge, has been demonstrated recently [18]. Charge reversal can be repeated with oppositely charged polyelectrolytes to build up multiple layers [19–22]. The model shown as Case A was used in a recent study to explain the development of a maximum in compression strength when poly-diallyldimethylammonium chloride (poly-DADMAC) was added at a saturation level to unbleached kraft fibers, prior to treatment with carboxylmethylcellulose (CMC) [12]. The same chemicals also were used in the current study.




***1. Idealized picture of the adsorption of cationic polymer onto negatively charged cellulose fibers under the conditions of (A) surface saturation and (B) an excess of cationic polymer, beyond the maximum that can be adsorbed.***




***2. Idealized dry-strength strategies based on (A) surface saturation and (B) an excess of cationic polymer added before the anionic dry-strength polymer.***




***3. Determination of the amount of poly-DADMAC required to saturate the surfaces of fibers from repulped xerographic copy paper.***

In Case B in Fig. 2, a large excess of the cationic polymer is first added to the fiber suspension. Anionic polymer added subsequently interacts first with the excess cationic polymer in solution. The outer surfaces of polyelectrolyte complexes formed between the cationic and anionic polymers [23–25] have a net charge, the sign of which will depend on the ratio of the charged groups [26–29]. The sign of charge, or the zeta potential, of the polyelectrolyte complex is assumed to be negative. If that is the case, polyelectrolyte complexation should allow a higher net level of polymer adsorption onto the fiber surfaces than in Case A [30]. It is hypothesized here that such a mechanism could also account for an increase in dry strength.

## EXPERIMENTAL

### *Chemicals*

The poly-DADMAC used for the adsorption and strength experiments was a medium-molecular-mass product from Ciba Specialty Chemicals. Solutions were prepared by dissolving the dry-bead product in deionized water. For calibration, charge titrations were carried out with poly-DADMAC from Aldrich Chemical Co. (molecular mass of 400,000–500,000 Daltons).

The anionic dry-strength additive was a 7*M* CMC from Hercules. The nominal degree of substitution of this product was 0.7 carboxymethyl groups per pyranose unit of sugar.

The anionic standard titrant solution used for determining charge was 0.0025*N* poly-vinylsulfate, potassium salt (PVSK) from Nalco.

### *Fiber preparation*

The fibers used in the experiments were from xerographic paper containing 35% recycled fiber. To prepare the fibers, we dispersed the copy paper with a TAPPI disintegrator [31] at 30,000 revolutions. Fine particles were removed from the slurry with a Bauer-McNett apparatus [32] fitted with a 200 mesh wire screen. The apparatus was run at a 200-mL/s flow rate for 10 min. We thickened the fibers for storage by collecting them on a TAPPI handsheet mold [31]. We diluted the fiber fraction with deionized water to 0.5% solids to prepare the fiber slurry used in adsorption and strength tests.

### *Procedures*

We determined the amounts of poly-DADMAC adsorbed onto fibers by adding an excess of the cationic polymer to the fiber slurry, filtering, then back-titrating the filtrate with a standard solution of PVSK. A streaming current detector from Mütek Analytic was used to detect the point of charge equivalence. The electrical conductivity was adjusted to 1000 µS/cm with sodium sulfate, and the pH was kept within the range of approximately 8.0–8.2 with $10^{-4}$ mol/L of sodium bicarbonate.

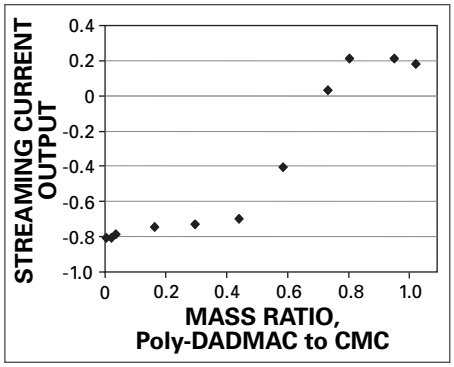
Paper handsheets with basis weights of approximately 60 g/m² were prepared from the long-fiber fraction of xerographic copy paper according to TAPPI T 205. Handsheets were pressed and then equilibrated at 50% RH. The tensile properties of paper were determined according to TAPPI T 494 with four replicate tests.

Some of the results did not fit well with the surface saturation model demonstrated earlier [12]. Consequently, additional tests were carried out to evaluate the hypothesis for the situation depicted in Part B of Fig. 2, in which polyelectrolyte complexes (PECs) are considered to deposit on fiber surfaces.

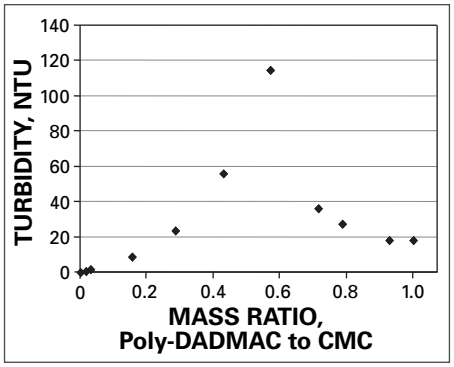
One step in validating this hypothesis is to show that such complexes exist within a certain range of concentrations of ingredients. Increasing amounts of 0.1% CMC solution were added to poly-DADMAC (final concentration 1.63 g/L) with 50 mL of buffer solution, followed by manual swirling for 30 s. The contents were transferred to a cuvette, and the turbidity was evaluated with at DRT-15CE Turbidimeter from HF Scientific. Results shown are the averages of five measurements, with the cuvette being swirled between each reading.

A second step in evaluating the PEC deposition hypothesis involved tests to determine the efficiency of retention under the conditions of testing. PECs were prepared at different ratios of the two polyelectrolytes, poly-DADMAC and CMC, and their contributions to turbidity were evaluated. Next, similarly prepared PECs were added to 500 mL portions of a 0.5% solids pulp slurry in a Britt Jar apparatus [33–34] with a constant impeller speed of 800 rpm.

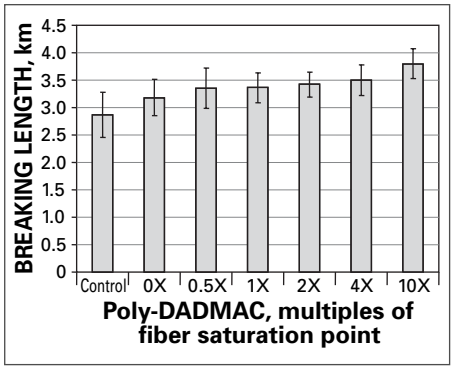
The turbidity of filtrate passing through a standard screen with 0.72 µm openings was determined after 30 s, after we discarded the first 15 mL to pass through the screen. In each case, we obtained a second set of turbidity measurements by discontinuing the stirring and allowing a fiber mat to form on the Britt Jar screen. Again, we obtained samples of filtrate after discarding the first 15 mL. We carried out blank experiments with no chemicals added to evaluate the contribution to turbidity of the small amount of fine materials remaining in the pulp after fractionation.




*4. Streaming current titration of a CMC solution (0.01% solids) by poly-DADMAC.*




*5. Turbidity of solutions of CMC to which different amounts of highly cationic poly-DADMAC have been added.*




*6. Effect on tensile strength of different concentrations of cationic polymer added before 2% CMC.*

## RESULTS AND DISCUSSION

### *Saturation level*

As shown in **Fig. 3**, no residual poly-DADMAC was detected in the filtrate solution until the amount added to fibers from recycled copy paper exceeded 2.1 μeq of charge per gram of filterable solids in the treated suspension. Since the pulp fibers were well rinsed, all of this charge demand was attributed to adsorption of the polymer onto solids. The calculated saturation level was 1.8 μeq/g based on a linear regression of the data points above 2 μeq/g.

To put the results shown in Fig. 3 into perspective, a previous study in which similar methods were used yielded a saturation level of 54 μeq/g for unbleached kraft fibers. The current results indicated only about 4–5% as much poly-DADMAC depositing onto recycled bleached kraft fibers. The difference is tentatively attributed to the lower charge density of bleached kraft fibers [13–14] and the usage of cationic starch and other cationic additives in the production of copy paper.

### *Complexation stoichiometry between poly-DADMAC and CMC*

The repeating unit of poly-DADMAC has a single equivalent of charge and a mass of 161.5 g/mole. By contrast, the CMC used had an average of 0.7 charges per anhydro-glucose unit. Based on an assumption that the CMC is in the form of the sodium salt, these numbers indicate one molar equivalent of charge per 209.6 g. A charge-balanced complex formed between these polyelectrolytes will have a mass ratio of approximately 0.77:1 of poly-DADMAC to CMC solids.

We also determined the stoichiometry of interaction between the two polyelectrolytes experimentally, by streaming current titrations and by measuring turbidity. As mentioned, the electrical conductivity of the starting solution of CMC was adjusted to 1000 μS/cm with $Na_2SO_4$. As shown in **Fig. 4**, the mass ratio at the streaming current end point was approximately 0.7 poly-DADMAC to CMC.

**Figure 5** shows corresponding results for the suspension turbidity. The starting solutions of both poly-DADMAC and CMC contributed very little to turbidity. A turbidity maximum was observed at a mass ratio of approximately 0.6. This result agrees approximately with the point of charge neutralization as determined by streaming current analysis. Exact agreement is not expected, since turbidity is a complex function of the size and density of the polyelectrolyte complexes suspended in solution. This finding of maximum turbidity near the point of charge neutralization is consistent with the findings of others [27, 29, 35].

### *Poly-DADMAC pretreatment level vs. strength properties*

Handsheets were prepared from the long-fiber fraction from repulped xerographic copy paper. The first set of experiments involved a variable level of cationic polymer treatment (poly-DADMAC), followed each time by a constant 2% addition of anionic polymer (CMC) to the furnish, on a dry fiber basis.

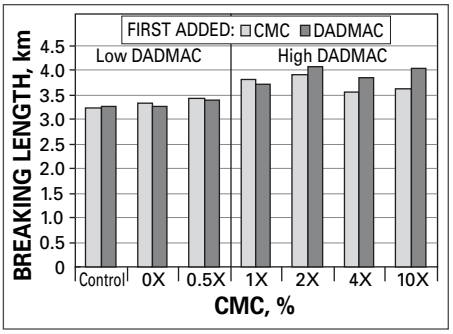
As shown in **Fig. 6**, the tensile strength of paper treated with poly-DADMAC then CMC generally increased with an increasing level of poly-DADMAC. The labels below the histogram bars indicate the amounts of poly-DADMAC addition relative to the saturation level. Even at zero poly-DADMAC, there was an apparent strength improvement from CMC, although the difference was not significant at the 95% confidence level. What had not been anticipated based on previous work [12] was that the strength continued to increase at levels of poly-DADMAC addition significantly greater than the saturation level.

The results shown in Fig. 6 cannot be explained in terms of the surface saturation model that was demonstrated with recycled unbleached kraft fibers [12]. Instead, the contribution to strength continued to grow when the initially added poly-DADMAC was ten times greater than the amount required to saturate the fiber surface. This outcome indicates a process similar to what is shown in Case B of Fig. 2.

### *Order of addition*

Follow-up tests to help elucidate the mechanism involved two levels of poly-DADMAC addition, with each followed by selected levels of CMC. The low level of poly-DADMAC addition was 0.029%, which is the approximate amount required to saturate the fiber surfaces. The high level of poly-DADMAC addition was ten times higher, 0.29% on a dry-fiber basis. Also, two different orders of polymer addition were compared. Each value shown in **Fig. 7** is the average from two independent experimental runs.

As Fig. 7 shows, the highest tensile strength values were obtained at the high level of poly-DADMAC addition. Within that class of tests, the highest tensile strength involved the same addition order, cationic polymer followed by anionic polymer. The order of addition made the largest difference, in terms of strength, at the high levels of CMC addition.



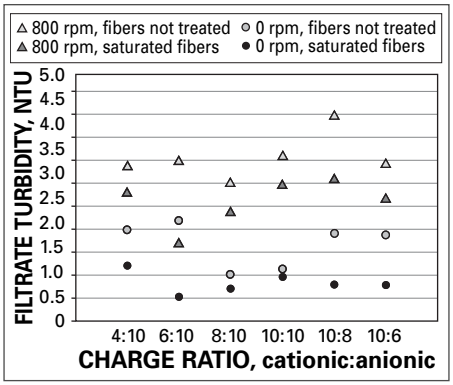

***7. Effect of the amounts and order of addition on tensile strength development after treatment with poly-DADMAC and CMC.***

Apparently, or at least as one way to explain these results, the initial treatment of the slurry with an excess of cationic polymer caused the fibers to have a strong positive charge. The addition of CMC then resulted in complexation of the excess cationic polymer that remained in solution. The right-most histogram bar corresponds to a situation in which there was a large excess of CMC. It is reasonable to expect such complexes to be retained efficiently on the positively charged fiber surfaces, contributing to paper strength, since the net charge of those complexes should be negative.

### *Demonstration of deposition of complexes*

**Figure 8** shows the results of tests in which pre-formed mixtures of poly-DADMAC and CMC were added to slurries of fibers obtained by fractionating recycled copy paper. The turbidity of filtrate from the Britt Jar tests did not depend only on the ratio of the two polymers. It also depended on whether or not the fibers had been pretreated with poly-DADMAC at a saturation level and whether or not the slurry was being stirred as the filtrate was obtained.

To evaluate the efficiency of retention of PECs, based on Fig. 8, it is necessary to account for the contributions of PECs to turbidity at different polymer ratios. Experiments were carried out using exactly the same procedures as used for the experiments of Fig. 8, except that no fibers were present. The results are shown in **Fig. 9**. As the data points show, the turbidity contribution of the PECs depended on the ratio of the two polymers, but it was also strongly influenced by whether or not the mixture was being stirred when each filtrate sample was collected.




***8. Turbidity of the filtrate from Britt Jar experiments with mixtures of poly-DADMAC and CMC added at different ratios to slurries of fiber from recycled copy paper. (The numbers on the horizontal axis are multiples of the amount of poly-DADMAC needed to saturate the fiber surfaces.)***

The influence of stirring is attributed to an effect noted by Davison [36], who proposed that the retention of fine substances during paper formation increases if those substances agglomerate into particles large enough to be sieved. Evidence supporting this hypothesis involved Britt Jar tests of mineral filler retention in the presence or absence of fibers. Surprisingly, the addition of retention chemicals decreased the amount of filler passing through the screen even when there were no fibers for the particles to adhere to. Li and Scott have reported further work showing the retention of agglomerated fine particles on a Britt Jar screen [37].

The results shown in Fig. 9 are consistent with these ideas, since agitation tends to break PECs into smaller units. The flow may also drive PECs through openings in the screen.

A further contribution to the turbidity values in Fig. 8 involves the contribution of fine particulate matter that remains in the fiber slurry after fractionation. Results depended on the stirring rate and on whether or not the fibers had been pretreated with a saturation level of poly-DADMAC. For untreated fibers, the filtrate turbidity was 2.90 NTU at 800 rpm and 1.05 NTU in the absence of stirring. Pretreated fibers yielded corresponding values of 1.48 NTU with stirring and 0.48 NTU without stirring.

Results in **Fig. 10** are based on the following equation. In this equation, it is assumed for the sake of simplification that the PECs do not have a measurable effect on the retention of fine solids originally present in the fiber suspension.

$$R = \frac{t_{polymer} - t_{test} - t_{blank}}{t_{polymer}}$$

where $100 \times R$ is the percent retention, $t_{polymer}$ is the turbidity of the polymer mixture, as in Fig. 9, $t_{test}$ comes from Fig. 8, and $t_{blank}$ values were given in the preceding paragraph. The standard deviations of the turbidity quantities shown in Eq. 1 had average values in the range of 0.12–0.17 NTU in cases involving pretreated fibers and in the range of 0.24–0.33 NTU in cases in which the fibers had not been treated with poly-DADMAC before PEC was added. These standard deviations suggest standard error values equal to about 3–10 percentage points on the vertical axis of Fig. 10.

As Fig. 10 shows, the retention of the PECs was most efficient when the charge ratio between the two polymers was close to one. For instance, for tests in which the solution was not stirred when the samples were collected (circles and filled circles), the efficiency of PEC retention reached about 80% when the charge ratio was either 10:10 or 10:8 as cationic:anionic groups on the two polymers. The fact that retention was reduced in the presence of stirring indicates that a filtration mechanism can be important for the retention of PECs.

A surprising finding is indicated in Fig. 10. Pretreating fibers with a saturating amount of poly-DADMAC was observed to increase the retention of the complexes in almost every case, irrespective of whether the PECs had an excess of either positive or negative groups. The greatest relative benefits of pretreatment were found with PECs having a charge ratio of 6:10, corresponding to a net negative charge. In this respect, the results are consistent with the model of Case B in Fig. 2.

Is it fair, however, just to consider the net charges of the complexes? It might be more useful to envision each PEC as being covered by alternating patches of positive and negative polymeric groups [24]. We will seek answers to such questions in future work.

## CONCLUSIONS

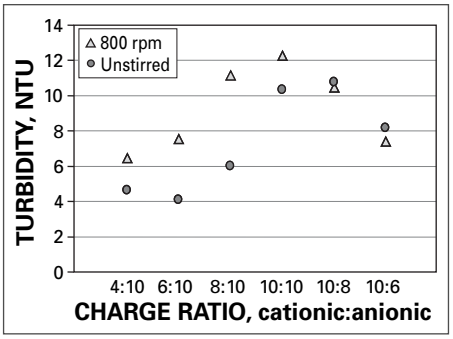
The tensile strength of handsheets formed from recycled copy paper fibers

was maximized when the amount of cationic polymer used to pretreat the fibers was increased in excess of that needed to saturate the fiber surfaces. It was hypothesized that a mechanism contributing to dry strength development involves the deposition of polyelectrolyte complexes (PECs). This hypothesis was supported by the finding that turbidity increased for mixtures of the two charged polymers.

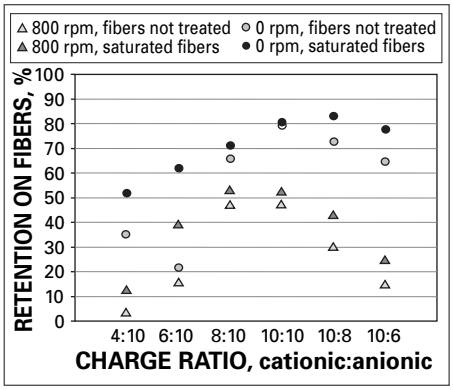
Consistent with this hypothesis, higher strength was achieved when the cationic polymer was added first, followed by a relatively high amount of the anionic polymer. The cationic polymer may provide anchoring sites for the adsorption of PECs with net negative charges. The opposite order of addition yielded significantly less strength gain. The retention of PECs was found to be highest when the ratio between the two linear polyelectrolytes was within about 20% of a one-to-one match between charged groups.

The pretreatment of the fibers with cationic polymer at a saturation level increased the retention of PECs over the whole range of compositions considered, not just in cases in which PECs had a net negative charge.

Finally, in addition to providing insight into the molecular mechanisms of dry-strength chemical use, the results suggest that pre-formed polyelectrolyte complexes can be used in the wet end to increase paper strength beyond that which can be achieved by separate additions of the same polymers. **TJ**




***9. Turbidity of mixtures of poly-DADMAC and CMC under the same conditions of dilution and mixing as in Fig. 8, except that no fibers were present.***




***10. Calculated retention of polyelectrolyte complexes onto fibers from recycled copy paper, based on Britt jar tests carried out at an impeller speed of either 800 rpm or 0 rpm, with the fibers optionally pretreated with sufficient poly-DADMAC to saturate their surfaces.***

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*Received: September 7, 2004*
*Accepted: July 27, 2005*

This paper is also published on TAPPI's web site <www.tappi.org> and summarized in the September *Solutions! for People, Processes and Paper* magazine (Vol. 88 No. 9).

## INSIGHTS FROM THE AUTHORS

Previously we had imagined that charged polymers mainly interact with fiber surfaces in the wet end of a paper machine. Now we realize the importance of polymer interactions with each other before they become attached to fibers. This seems to be a trendy area, since other research groups around the world have shown interest in pursuing related work. We chose this area of study due to the importance of paper dry-strength, especially when recovered fiber is being used. It also was important to choose a subject area suitable for a pair of summer-long undergraduate research projects.

The amount of effort and attention to detail required to obtain good data is often overlooked. For us, that was the most difficult aspect of this research. A lot of care is needed when one sets out to observe effects of strength additives in handsheets. Turbidity tests also require very consistent lab technique in order to get meaningful data.

In our work, the most amazing finding was that strength keeps going up when you continue increasing the dosage of the polyelectrolyte mixtures that we studied. This is quite different from what one observes in the case of traditional dry-strength additives, such as cationic starch.

The approach described in this article is most likely to make sense if there is a serious gap between the achievable dry strength and that which is required in a certain paper or paperboard product. The cost of the chemical dosages required then will need to be justified versus potential savings in fiber costs or other production strategies.

We have already taken the next step of this research. Follow-up work with fibers from recycled paper has confirmed the basic mechanism proposed in this work. Tests with non-bonding glass fibers have documented the dramatic nature of strength improvements that can be achieved. The follow-up research also helped to clarify the effects of different polymer charge ratios, as well as effects due to pre-treatment of the fibers to reverse their sign of electrical charge.

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