

Recovered Kraft Fibers and Wet-End Dry-Strength Polymers

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ABSTRACT

The drying and recycling of paper profoundly affects the bulk and surface properties of kraft fibers. Recovered kraft fibers tend to be less porous on a sub-microscopic scale, less flexible, and less able to swell with water, compared to never-dried, refined fibers. Recovered fibers also tend to be less able to form inter-fiber bonds. This review considers how changes associated with the drying and recycling of kraft fibers affect their interactions with dry-strength polymers such as cationic starch, copolymers of acrylamide, and high-charge cationic polymers. A key finding of recent research is that the listed changes in fiber properties occur independently of whether or not dry-strength polymers are present. There are two main ways that strength-enhancing additives can compensate for losses in bonding ability. First, dry-strength polymers present in the original paper can contribute to bonding when the same material is recycled. Second, additional wet-end polymers applied during production of the recycled paper can help compensate for deficiencies of bonding ability. Recently a dual-polymer dry-strength program was adjusted to match the ability of recovered kraft fibers to retain cationic polymers. This approach makes it possible to tailor a treatment system to the type of furnish that is being used.

INTRODUCTION

Recovered kraft, soda, and sulfite pulps, obtained by recycling of corrugated boxes, mixed office waste, *etc.*, differ in various respects, compared to never-dried fibers of the same type. The nature and extent of the differences between primary and recovered fibers have been described in previous articles [1-10]. The present review focuses on how various differences between primary and recycled fibers affect the strategy for using dry-strength polymers.

For purposes of discussion, it is useful to distinguish between changes in fiber structure *vs.* effects due to the presence of various chemicals introduced during previous stages of papermaking, converting, or recycling. The sections that follow deal with three key structural aspects, as follows: (1) fine-scale porosity of cell walls; (2) swelling of fibers in water; and (3) flexibility of individual fibers in the wet state. Advances in our understanding of each of these structural aspects have implications for the use of dry-strength agents, not only when paper is made from recovered fibers, but also in the case of paper made from never-dried fibers, which may later become recycled. Later sections of this review will deal with effects of chemical changes and contaminants in recovered kraft furnish on the performance of strength-enhancing polymer additives.

CHANGES IN CELL WALL POROSITY

Never-dried kraft and sulfite pulp fibers have an abundance of fine-scale void spaces within them, having widths in the range of about 2 to 50 nm [11-13]. Though these spaces are incredibly narrow, the cumulative volume may be in the range of 0.1 to 0.4 cm³ per gram of fiber solids.

Berthold and Salmén [12] used a solute exclusion method to contrast the pore-size distribution of kraft fibers with those of a mechanical pulp. Tests were carried out with well-defined dextran polysaccharides, which are known to have little tendency to adsorb onto the surfaces of fibers in aqueous solution. Such analyses are based on an assumption that the ability of a certain kind of polymer to penetrate into a given pore will depend on its molecular mass and its mean size when partially stretched out in solution. Though questions have been raised regarding the relationship between pore size and the average dimensions of

polymers in solution [13], there has been no dispute about the qualitative importance of the results, such as those shown in Fig. 1. As shown, the pores in mechanical pulp fibers tend to be much narrower compared to those in the cell walls of kraft fibers. It is reasonable to assume that the pores observed in never-dried kraft pulps correspond to spaces that were formerly occupied by lignin [14].

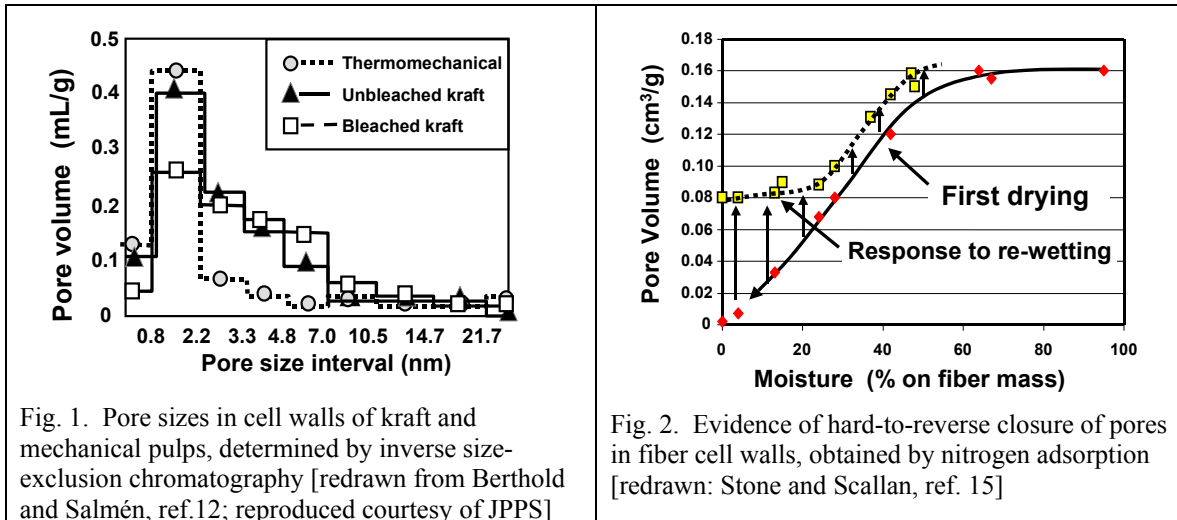


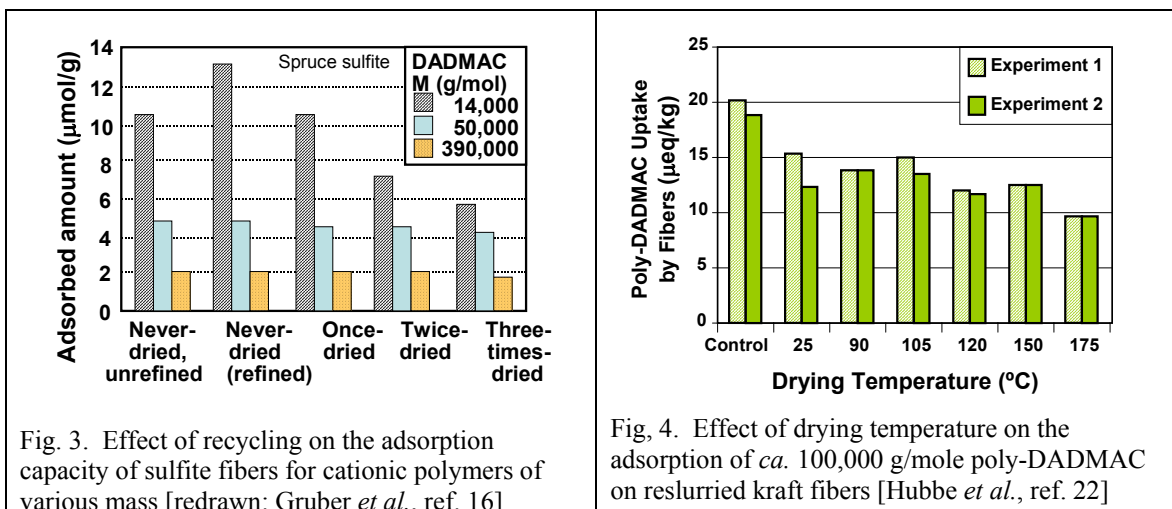
Fig. 1. Pore sizes in cell walls of kraft and mechanical pulps, determined by inverse size-exclusion chromatography [redrawn from Berthold and Salmén, ref.12; reproduced courtesy of JPPS]

Fig. 2. Evidence of hard-to-reverse closure of pores in fiber cell walls, obtained by nitrogen adsorption [redrawn: Stone and Scallan, ref. 15]

Some of the first evidence that drying can cause hard-to-reverse closure of fine-scale porosity in the cell walls of chemically pulped fibers was obtained by Stone and Scallan [15]. As shown in Fig. 2, the pore volume, as determined by nitrogen adsorption isotherms, depended on how much water was removed from the fibers during the initial drying (solid curve). Complete drying of the fibers appeared to collapse most of the pores; the remaining pore volume was close to zero, within the precision of measurement. As shown by the dotted line, much of this pore volume opened up again when the same fibers were redispersed in water. However, it appeared that about half of the total pore volume had been lost irreversibly in cases where the fibers had been completely dried.

Since the focus of the review article is on interactions between polymeric additives and fibers – especially recycled fibers – it is important to consider whether typical wet-end polymer additives are able to penetrate into the fine-scale pores of a kraft fiber. Some results of research work related to this question are shown in Fig. 3 [16]. It is evident that the amount of cationic polymer taken up by the negatively charge surfaces of the fibers depended strongly on molecular mass. Such a dependency is consistent with the hypothesis mentioned earlier, in which polymers above a certain molecular mass may be excluded from pores of a certain size. Also, the amount of the lowest-mass polymer able to adsorb onto fiber increased strongly with increased refining. Another striking feature of Fig. 3 is the strong decrease in the amount of the lowest-mass polymer adsorbed on the fibers as the number of times that the pulp had been previously dried increases. Since charged polymers having a molecular mass of 14,000 g/mole are expected to have a mean size higher than about 5 nm under the conditions of electrical conductivity prevailing in a typical paper machine [11, 17-18], it follows that pores of that size or somewhat larger are opening in response to refining and closing in response to drying of the fibers. In this respect the results shown in Fig. 3 are in general agreement with those shown in Fig. 2.

Cationic starch molecules of the kind often used at the wet end of a paper machine usually consist of a mixture of linear and branched molecules that differ in average molecular mass. The linear molecules, called amylose, are generally the smaller of the two types of starch molecule. Typical values for the degrees of polymerization (number or repeating units) in an amylose molecule are within the range of 600-3000 [19]. By contrast, the branched amylopectin molecules have about 6000 to 60,000 repeat units, corresponding to molecular masses of approximately 1 to 10 million grams per mole. Based on these numbers it can be expected that most of the cationic starch will stay out at the surface of fibers. At most, the starch might penetrate within the microfibrils, hemicellulose, and other macromolecules extending from the fiber surface [20-21].



Results of more recent work, shown in Fig. 4, reveal the effect of drying temperature on the adsorption of a high-charge cationic polymer, poly-DADMAC [22]. The molecular mass of the poly-DADMAC was approximately 100,000 g/mole, which corresponds to a diameter in solution of at least 15 nm [11]. As shown, drying reduced the uptake of the cationic polymer.

Further work has shown that the effects of drying on the polymer adsorption onto fibers can be described in terms of two main steps. The first step-change loss of adsorption capacity occurred even when fibers were dried at room temperature. This loss has been attributed to the effects of capillary forces [23-24]. Further reduction in adsorption capacity occurred only when the fibers were heated to 120 °C or higher. These high-temperature effects may be related to the shrinkage effects reported by Weise and Paulapuro [25].

CHANGES IN WATER RETENTION

Another way to quantify the changes in void volume within fiber cell walls is to measure the amount of water that remains in the fibers when they are subjected to centrifugation at a specified speed. Though this method does not provide information about pore sizes, the tests are relatively quick. As shown in Fig. 5 [24], drying of bleached kraft fibers at increasing temperatures strongly decreased the water-retention values (WRV), expressed as the mass of water after centrifugation per dry mass of fiber [26-27]. In agreement with results shown in Fig. 4, most of the change was already apparent at the lowest temperature of drying. There was no further loss of WRV until the fibers were heated above 120 °C.

More recent work by Maloney *et al.* [1997] showed that the irreversible changes in pore volume can begin to take place rather early in the process of removing water from wet kraft fibers. For instance, an irreversible loss in WRV was observed even if water was merely pressed from mats of wet fibers, approximating the effect of wet-press nips on a paper machine. As shown in Fig. 6, the loss in swelling ability was progressive; the loss in WRV depended in a roughly linear fashion on how much water was removed during pressing and subsequent evaporative drying. The lower line in the figure shows that after the relatively severe drying conditions of 105 °C in an oven, a second cycle of pressing and drying did not induce further significant changes in the ability of fibers to swell in water when they were reslurried.

WRV tests have been used to answer some further questions about the interactions of chemicals with fibers during the papermaking process. In particular, it is of interest to know whether it is possible to block the closure of pores in the fiber cell walls by pretreatment of the furnish with various chemicals before the fibers are dried for the first time. Figure 7 shows the results of experiments in which the fibers were dried in the presence of 2% sugar solutions [29]. For comparison, the first pair of 95% confidence intervals, corresponding to untreated fibers, shows a significant loss of WRV when the fibers were dried at room temperature in the absence of sugar. When comparing data corresponding to once-dried fibers, the 95%

confidence intervals overlap. However, it was possible to demonstrate a significant difference when using a 90% confidence criterion, comparing the control versus drying in the presence of sucrose. The drops in WRV values of fibers dried in the presence of sugar were about 50% to 80% of what happened in when fibers were dried in the absence of sugar. This seems like a disappointing result in light of the practical difficulties inherent in use of sugar solutions. Results were in general agreement with those of Laivins and Scallan [30], who used solute exclusion as a means of determining the amount of water held within the cell walls of fibers after drying them in the presence or absence of sugar.

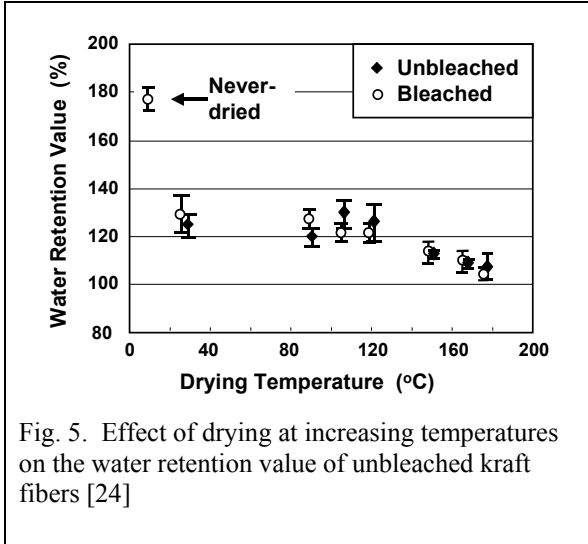


Fig. 5. Effect of drying at increasing temperatures on the water retention value of unbleached kraft fibers [24]

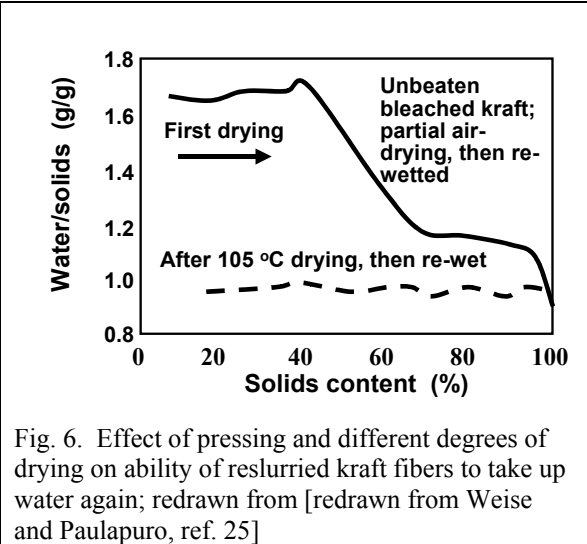


Fig. 6. Effect of pressing and different degrees of drying on ability of reslurried kraft fibers to take up water again; redrawn from [redrawn from Weise and Paulapuro, ref. 25]

Another question that can be addressed by using WRV experiments is whether pretreatment of never-dried fibers with water-loving polymers of various types can affect the water-holding ability of the fibers after they have been dried. Figure 8 shows results from a series of this type of experiment in which the fibers were treated at the 2% level (solids basis) with various starch products [31]. As indicated by the 95% confidence intervals, certain of the starch products had a significant effect on the WRVs obtained after the fibers were dried and reslurried. There are two ways to interpret these results. On the one hand, the results are consistent with a hypothesis that some of the starch products inhibited pore closure when the fibers were dried. On the other hand, the results also are consistent with a hypothesis that the polymers themselves were responsible for holding onto a significant amount of water. Further research, possibly involving starch films in the absence of swellable fibers, will be needed in order to discriminate between these two possible explanations of the results.

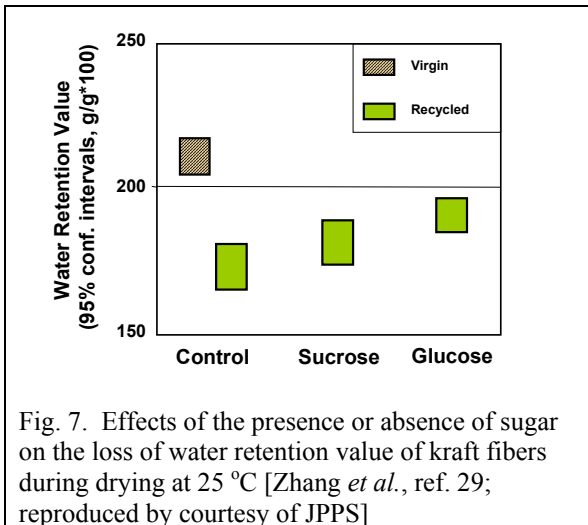


Fig. 7. Effects of the presence or absence of sugar on the loss of water retention value of kraft fibers during drying at 25 °C [Zhang *et al.*, ref. 29; reproduced by courtesy of JPPS]

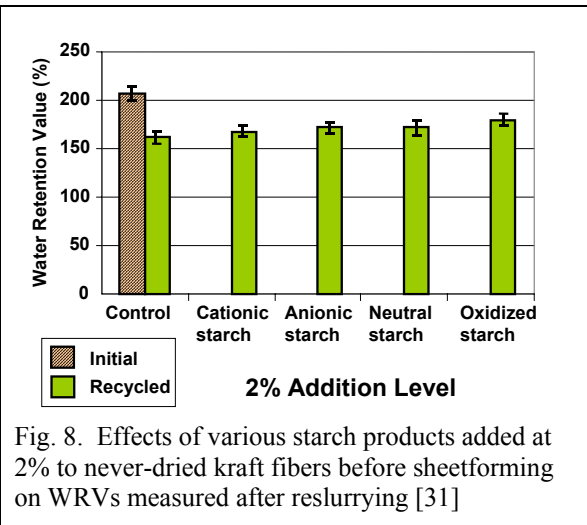


Fig. 8. Effects of various starch products added at 2% to never-dried kraft fibers before sheetforming on WRVs measured after reslurrying [31]

Further tests of a similar nature were carried out in which unbleached kraft fibers were treated sequentially with a highly charged cationic polymer (poly-DADMAC), followed by anionic carboxymethylcellulose (CMC) [22]. The latter chemical was always added at the 2% level (dry fiber basis), and the amounts of cationic polymer are shown in the horizontal axis labels of Fig. 9. As shown, treatment of the furnish with CMC resulted in higher mean values of WRV when the amount of poly-DADMAC used to pretreat the fibers was below 54 $\mu\text{eq/g}$. Higher amounts of poly-DADMAC negated the initial positive effect of CMC on WRV. To put these results into context, earlier work showed that treatment of similar fibers with poly-DADMAC alone yielded a significant decrease in WRV [31]. In summary it can be said that some conditions of polymer treatment have the potential to yield higher post-drying WRVs, but it is still uncertain whether such effects are due to the fiber pore structure or to the abilities of the polymers themselves to hold water.

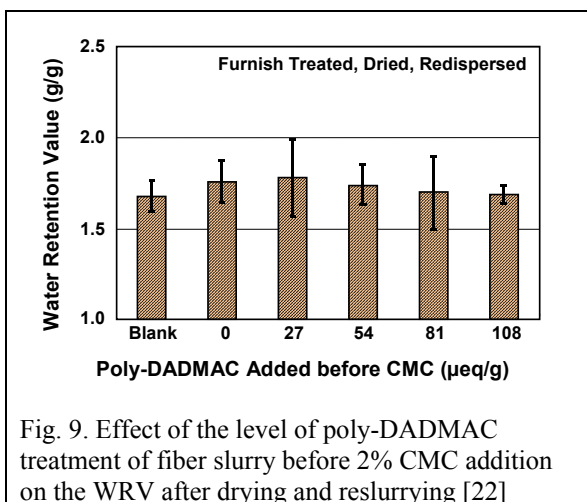


Fig. 9. Effect of the level of poly-DADMAC treatment of fiber slurry before 2% CMC addition on the WRV after drying and reslurrying [22]

CHANGES IN FLEXIBILITY AND STRENGTH

As shown by Parsons [32] and Page [33], there is often a direct relationship between the relative bonded area (RBA) of fibers in a sheet of paper and the paper's tensile strength. It seems that one of the main purposes of refining is to render the fibers sufficiently conformable so that they can flatten out into ribbons and drape over each other, forming a relatively large area of inter-fiber contact.

Unpublished work by Paavilainen and Luner at the State University of New York in Syracuse in 1995 showed that papermaking fibers of various types suffer large losses in flexibility when they are dried, during the formation of paper, and then later reslurried in water. Related results, using the same basic procedure to evaluate the wet flexibility of fibers [34], are shown in Fig. 10 [29]. As shown, the mean values follow a trend towards decreasing flexibility of the reslurried fibers following each of the dewatering methods. The effects were significant at the 95% level of confidence in the case of oven drying.

The reported results seem reasonable in the sense that fibers would be expected to become increasingly flexible as a result of refining. However, there was a concern that refining might also render the fibers even more susceptible to loss of flexibility when the fibers are dried. Results shown in Fig. 11 tend to lay such concerns to rest, at least under the conditions of testing of unbleached kraft fibers [35]. As shown, the flexibility of the refined fibers remained higher than the original flexibility of the unrefined fibers, even after oven drying.

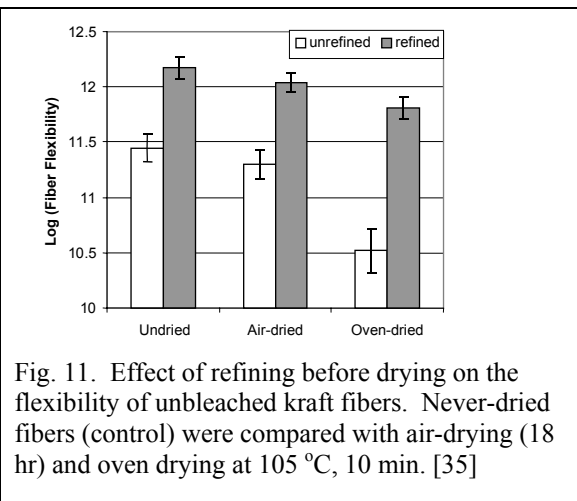
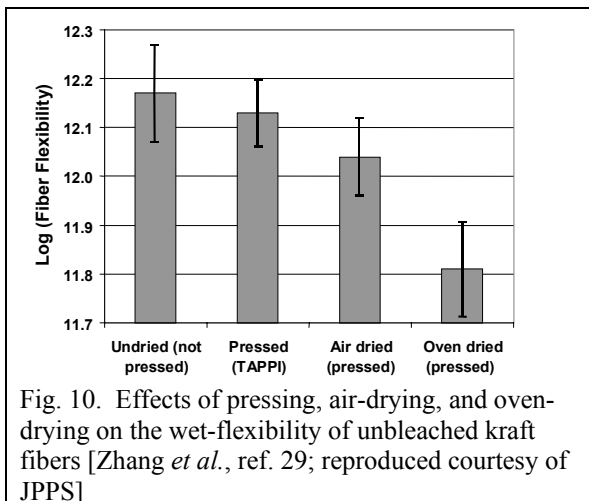
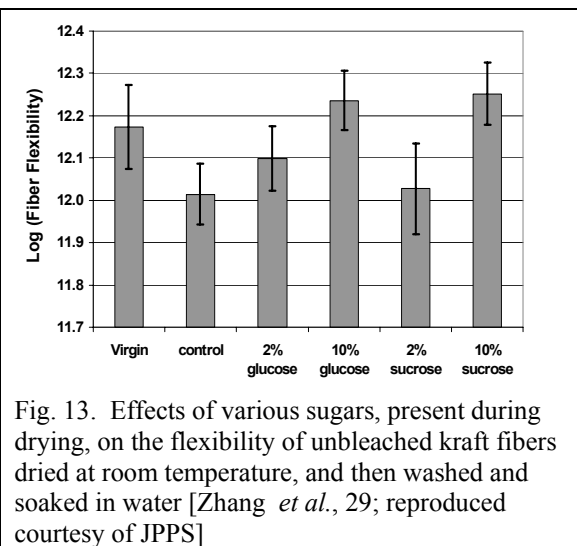
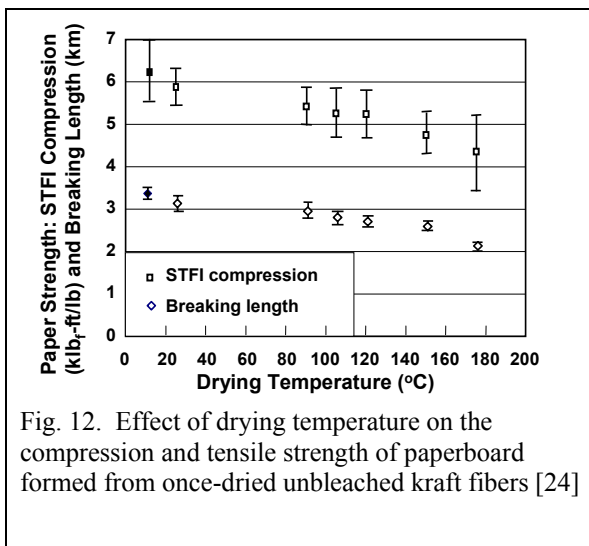


Figure 12 shows the consequences of drying-induced changes on the tensile and compression strength of paperboard handsheets prepared from unbleached kraft fibers. Paper strength fell under all conditions of drying, and the strength fell further when the drying temperature was higher than about 120 °C. These results follow the same general trends as was seen earlier in Figs 4 and 5 corresponding to the polymer adsorption capacity and the WRV of the same type of fibers.



In summary, it is reasonable to expect a functional relationship between fiber porosity, swelling ability, and flexibility. Since drying in the presence of sugar tended to reduce the loss in water retention (Fig. 7), it is reasonable to expect there to be some effect on fiber flexibility. Results shown in Fig. 13 [29] support this idea. The highest level of sugar treatment (10% solutions of glucose or sucrose) yielded results that were statistically indistinguishable from the flexibility of the corresponding never-dried fibers. Though the fibers were extensively washed, one cannot completely rule out the possibility that a small residual of sugar in the fibers might be functioning as a plasticizing agent, making the fibers more flexible than they would have been otherwise, regardless of their drying history.

RESIDUAL POLYMERS IN RECOVERED FURNISH

The fact that chemicals added during an initial papermaking operation can affect the properties of paper made from the same fibers after they are recycled, was shown most clearly by Sjöström and Ödberg [36].

These authors showed, for instance, that less AKD sizing agent was needed to make the recycled paper resist water penetration if the original paper had been treated with AKD. The same study quantified the effect of cationic acrylamide polymers, adsorbed onto fibers during the first cycle of papermaking, on the ability of the same fibers to take up more of the same polymer in a subsequent cycle of papermaking. Surprisingly, the process of drying and redispersing appeared to restore the surfaces of the fibers in some way, so that they were able to adsorb almost as much of the cationic polymers, compared to reference fibers that had not been treated during the original cycle of papermaking. Lee and Joo [37] carried out related work, showing that the adsorption of cationic acrylamide copolymer onto bleached hardwood kraft pulp did not suffer as a result of the fibers having been dried. On the other hand, refining of the stock resulted in large increases in the amount of cationic polymer that could be adsorbed onto the fibers. This extra increment of adsorption capacity was lost whenever the fibers were dried, causing the effective surface area to decrease again.

Grau *et al.* [38] showed that there continued to be a strength advantage in recycled handsheets, if the original paper had been produced with either an acrylamide-type strength additive or starch added to the furnish. The relative benefits decreased with each cycle of papermaking, when no further chemicals were added. Strength benefits attributable to the recycling of dry-strength chemicals, along with the fibers, were also reported by Higgins and McKenzie [39] and by Eichinger [40]. Figure 14 shows a further example of this type of strength benefit in the case of guar gum, which was added only during the first cycle of papermaking with unbleached kraft fibers [41].

Figure 15 shows the corresponding results for tensile strength and for the water retention value of the pulp sampled before formation of the recycled paper handsheets. Although the guar gum yielded relative increases in strength both in the original paper and in the recycled sheets, there was no significant effect of the guar gum on WRV. Thus, the strength benefits are more likely attributable to increased bonding per unit area, rather than on a mechanism involving development of more contact area between the fibers. It is worth noting that a similar conclusion has been reached in studies of the mechanism of cationic starch as a strength-enhancing agent [42].

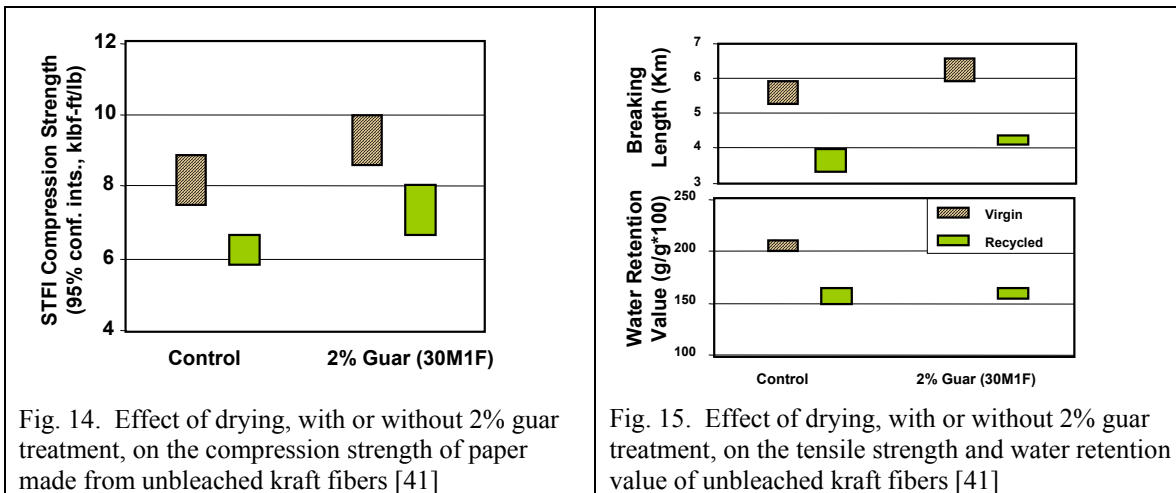


Fig. 14. Effect of drying, with or without 2% guar treatment, on the compression strength of paper made from unbleached kraft fibers [41]

Fig. 15. Effect of drying, with or without 2% guar treatment, on the tensile strength and water retention value of unbleached kraft fibers [41]

Figures 16 and 17 show the consequences of pretreating unbleached kraft furnish with different kinds of starch products during the first cycle of papermaking [31]. As shown in Fig. 16, treatment with cationic starch more than compensated for the expected loss of inter-fiber bonding ability, when the fibers were recycled without further chemical treatment. Anionic, neutral pearl starch, and oxidized starch products also yielded strength benefits, but not to as great a degree as cationic starch. It is likely that these differences are related to the superior ability of cationic starch to be retained on fiber surfaces [43]. Figure 17 shows corresponding results in the case of breaking length.

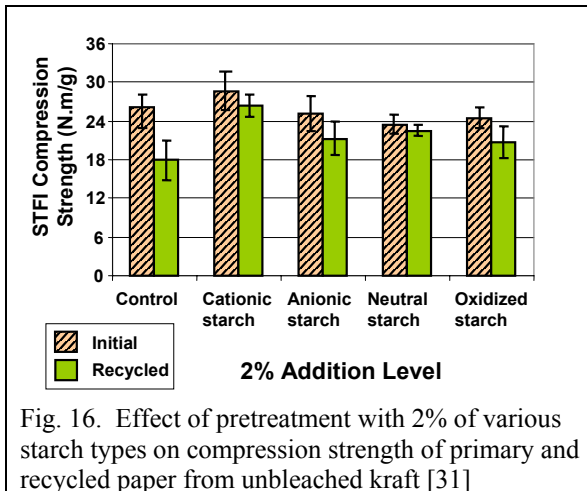


Fig. 16. Effect of pretreatment with 2% of various starch types on compression strength of primary and recycled paper from unbleached kraft [31]

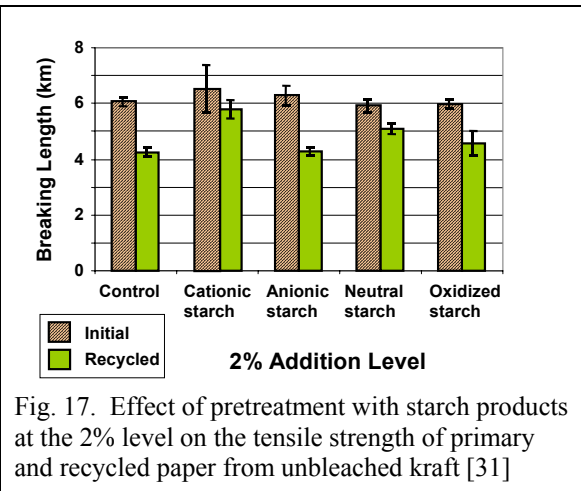


Fig. 17. Effect of pretreatment with starch products at the 2% level on the tensile strength of primary and recycled paper from unbleached kraft [31]

DRY-STRENGTH TREATMENTS FOR RECOVERED FIBERS

In principle, the addition of dry-strength chemicals to recovered fibers ought to have similar effects to when the same treatments are applied to never-dried fibers or to stock made from dry-lap pulps. However, the changes in fine-scale porosity and conformability caused by drying, usage, and recycling of the fibers can be expected to change both the relative importance and the detailed recipes needed in order to achieve the desired strength goals.

Figure 18 shows results from a study in which never-dried linerboard pulp was treated sequentially with a variable amount of highly cationic poly-DADMAC, followed by a constant amount of the anionic dry-strength additive carboxymethylcellulose (CMC) [22]. This is the same system that was considered in Fig. 9. As shown, the highest compression strength was obtained when the amount of the first additive was 54 $\mu\text{eq/g}$. Separate tests showed that this amount of poly-DADMAC corresponded to saturation of the fiber surface. The saturation level was defined as the maximum amount of cationic polymer that would adsorb onto the fibers, leaving no significant excess in the bulk solution. Further addition of poly-DADMAC, above the saturation level, before the CMC addition, resulted in lower compression strength. A strategy based on “surface saturation” was proposed to take advantage of this type of situation [22]. Corresponding results for tensile strength are shown in Fig. 19. Although the strength seemed to reach a plateau when the amount of poly-DADMAC was raised to the saturation level, it is interesting that further addition of poly-DADMAC did not hurt tensile strength.

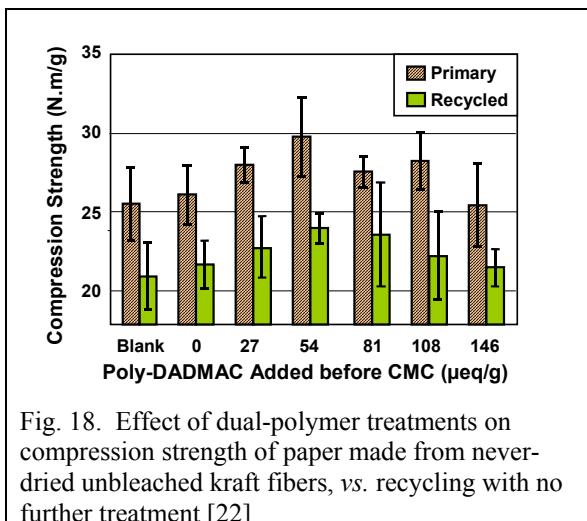


Fig. 18. Effect of dual-polymer treatments on compression strength of paper made from never-dried unbleached kraft fibers, vs. recycling with no further treatment [22]

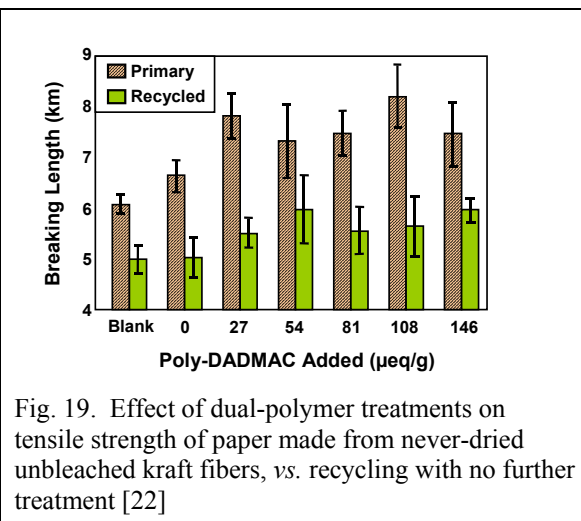


Fig. 19. Effect of dual-polymer treatments on tensile strength of paper made from never-dried unbleached kraft fibers, vs. recycling with no further treatment [22]

Recently we have been working to evaluate the saturation strategy in the case of redispersed xerographic copy paper. Preliminary results indicated that the fibers from the copy paper had a much lower ability to take up poly-DADMAC, compared to the unbleached kraft fibers considered in Figs. 18 and 19. Some results of the recent work are shown in Fig. 20. The horizontal axis labels are given in units of saturation amounts of poly-DADMAC. As shown, the strength continued to rise when the amount of the first additive exceeded the saturation level by as much as a factor of ten. Since the excess of cationic polymer clearly was not being retained directly on the fiber surfaces, it has been proposed that the further increase in strength towards the right-hand-side of the figure is due to formation of polyelectrolyte complexes (PECs) in the bulk solution phase, followed by their retention onto the fiber surfaces, where they function as bonding agents. Further work is under way to evaluate this mechanism.

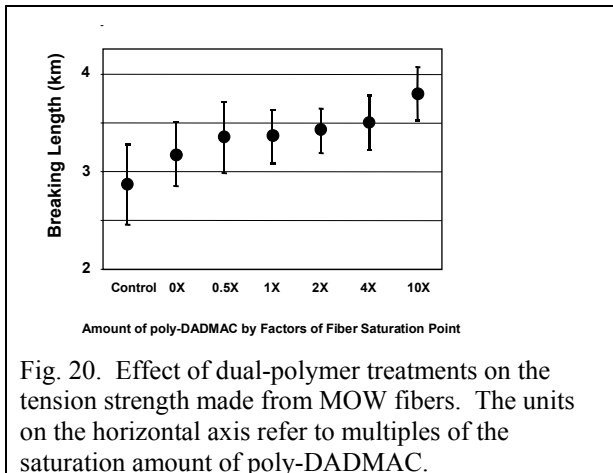


Fig. 20. Effect of dual-polymer treatments on the tension strength made from MOW fibers. The units on the horizontal axis refer to multiples of the saturation amount of poly-DADMAC.

EFFECTS OF CONTAMINANTS

The word “contaminants” will be used here to describe substances present in recovered kraft fibers at levels that are significantly higher compared to fibers obtained directly from a chemical pulping process. Even a component that is generally regarded as being beneficial, *e.g.* calcium carbonate filler, can create problems if its content in the recovered stock is highly variable or when it is attached to something objectionable, such as xerographic toner.

Some of the most important contaminants, in terms of their expected impact on dry-strength polymer performance, are the fines fraction (including fillers), additives affecting the charge demand of the furnish, surface-active agents – especially those used for de-inking processes, and ink particles themselves. Table 1 provides a brief summary of how each of these kinds of contaminant is likely to affect the performance of cationic starch and other dry-strength additives.

Each of the effects listed in Table 1 will depend on how much of each class of contaminant present in the furnish. Problems will mainly arise when there are large shifts in composition, possibly associated with different batches of recovered fibers. Though many such issues probably are best dealt with during repulping and de-inking processes [see 49], it is to be expected that changes in recovered furnish will tend to shift the balance of charges away from its optimum point for a given paper grade and dry-strength strategy. Kuys and Zhu [50] evaluated the charge characteristics of various substances likely to be present in recovered fiber stock. They found, for instance, that xerographic toner materials can be a major source of anionic charge of the recovered stock. In that type of situation online control systems for charge-control additives [51] and for retention aid flows [52] ought to make it easier to optimize the performance of dry-strength polymer additives.

Table 1. Summary of effects of various contaminants on the performance of dry-strength polymers in recovered kraft fiber stock	
Contaminant type	Expected effect on Dry-Strength Additive Performance
Mineral filler particles	These will tend to increase the need for strength enhancement [44-45]. Also, the effects of strength additives will be diluted by spreading them over a larger total surface area.
Fiber fines (recovered)	Again, the effects of strength additives will be diluted by spreading them over a larger total surface area. Strength-enhancing polymers adsorbed onto fibers are expected to give much greater contribution to strength, compared to those on fiber fines [46].
Anionic substances	Reduced performance of cationic polymer additives is expected, due to charge-neutralization and resulting inefficient retention on fiber surfaces [47].
Cationic substances	An excess of cationic substances in the recovered furnish may require changes in the dry-strength treatment strategy, such as some of those outlined in the previous main section.
Surface-active agents	While surfactants, such as those used in deinking processes generally tend to hurt paper strength [23-24], they don't necessarily interfere with the action of dry strength additives.
Ink and dirt particles	If the furnish contains a lot of ink or dirt particles, then there is a possibility that some uses of dry-strength agents, retention aids, and other polymers may increase the retention of dirt in the product.

The question of whether or not the fine-particle and fiber-fines fractions of recycled pulps play a significant role with respect to dry-strength development will depend strongly on the manner in which the fibers are recycled. Fine materials can reach very high levels in recycling operations where there is no attempt to remove ink, especially in cases where the pulp is subjected to significant levels of additional refining. On the other hand, the floatation, agglomeration, screening, and washing operations in various deinking systems will tend to remove a significant proportion of fiber fines and mineral particles along with the ink. Because of such contrasting situations, it is advisable to test various proposed dry-strength additive programs with the actual furnish that will be used in producing a certain grade of paper or board.

CONCLUSIONS

1. A tendency for recovered kraft and related pulp fibers to be less porous on a sub-microscopic scale after drying and reslurrying implies a reduced tendency for dry-strength chemicals to be absorbed into the fiber walls, thus losing their ability to affect bonding. This difference between primary and recovered fibers implies the possibility of a more efficient use of strength agents in the case of recovered fibers, especially if the additives have a relatively low molecular mass.
2. Though the presence of dry-strength polymers during the first cycle of papermaking does not prevent loss of ability of kraft fibers to swell again after they have been dried and then reslurried, the polymers do tend to increase the net amount of water that is held on or within recovered fibers.
3. The wet-flexibility of kraft fibers is reduced as a result of the drying of paper. This effect is consistent with a reduced ability of the fiber surfaces to conform to each other, allowing formation of hydrogen bonds. The loss of strength potential may need to be made up by a combination of refining (with further loss of freeness) and by judicious use of chemicals such as cationic starch and other dry-strength agents.
4. It is possible to optimize certain dual-polymer dry-strength treatments such that they are suited to recycled pulps having different surface charge characteristics. In some cases it may be advantageous to add an amount of cationic polymer just sufficient to saturate the fiber surfaces, and then add an anionic dry-strength polymer.
5. Variations in the contaminant levels in recovered fiber may require adjustments in the flows of different polymeric additives used to achieve dry-strength goals. Online control technologies may play a role in achieving the best performance from the dry-strength polymers that are used.

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