Novel techniques for enhancing the strength of secondary fiber

Ganapati R. Bhat, John A. Heitmann, and Thomas W. Joyce

Graduate student, professor, and professor, respectively, Department of Wood and Paper Science, North Carolina State University, Raleigh, N.C. 27695

ABSTRACT *One impediment to the wider use of recycled fibers is the loss of strength that occurs when these materials are repulped. This report examines the effects of several techniques for enhancing the strength of secondary fiber. Repulping under alkaline conditions and refining are the most commonly used methods to improve the strength of secondary fibers. High-shear-field (HSF) treatment produces an effect similar to refining while producing less fines. The best results were obtained using a combination method of alkali treatment followed by HSF treatment. Strength properties were higher than refining and were comparable with virgin pulp in some cases. The report also shows that enzymes can be used to increase the freeness of secondary fiber without affecting its mechanical properties.*

KEYWORDS
Cellulose fibers
Freeness
Kraft pulps
Mechanical strength
Pulp properties
Reclaimed fibers
Recycling
Refining
Swelling

Annual consumption of recycled fiber has been predicted to grow at a rate of 3-4% per year, from 20.5 million tons in 1989 to 26-30 million tons per year by the year 2000 (*1-3*). Two of the main areas of predicted growth include the use of secondary fiber as a pulp substitute in container board and as a deinked chemical-fiber substitute in tissue grades. Environmental pressure and, in many cases, legislation are also stimulating very strong efforts to recycle old newsprint to supply 20-40% of the 16-million-ton/year newsprint market, thus diverting a significant volume of newsprint from the solid-waste stream. Until recently, recycled newsprint accounted for only about 10% of the newsprint market. Finally, public awareness of environmental issues is creating pressure to recycle office papers and create a new category of recycled fine papers. This 12-million-ton/year market has traditionally used only about 11% secondary fiber because of concerns about product quality.

Secondary fibers are defined as fibers that have been through at least one papermaking cycle. Recycled fibers have lower strength and higher drainage resistance than virgin fibers. The mechanical properties of fibers as well as their ability to swell are diminished after they are exposed to the pulping and drying conditions imposed during the papermaking cycle. The reduction in swelling and the loss of fiber flexibility after drying reduce the strength potential of recovered fibers. Contamination and age degradation also contribute to the reduced strength of secondary fiber.

When a fiber is dried, physical discontinuities in the cell wall are collapsed by high surface-tension forces that pull the surfaces together. These surfaces become hydrogen bonded, which reduces swelling in the next cycle. In subsequent beating stages, the recycled fiber will not be able to delaminate and swell as well as virgin fiber. This mechanism is confirmed by experimental work where hydroxyl groups on the cellulose fiber were blocked by derivatization, thus reducing the amount of irreversible shrinkage during drying. With irreversible shrinkage reduced, there were fewer differences between the properties of sheets made from once-dried and never-dried pulp (*4*).

Freeness reduction during beating is much faster for secondary fibers. For equivalent beating times, a sheet containing recycled fiber is less dense and usually more absorptive than virgin fiber stock. The fines created when secondary fibers are beaten consist largely of microfibrils that were strongly coupled to each other when they were originally dried on the paper machine. When liberated during refining, they increase the specific surface area of suspension more than the swelling potential. They start to behave as fillers, with a small effect on strength but a large effect on the drainage properties (*4*).

In general, the greater the degree of refining of the virgin fibers, the lower is the recovery potential of sheet properties that are a direct function of fiber bonding, e.g., burst strength and tensile strength (*5*). Folding endurance of recycled paper is also considerably lower than for sheets made from virgin stock. Sheet density decreases each time the fibers are recycled. These strength losses may be the result of a loss in bonding potential, either in the strength of the interfiber bonds or in their number (*6*). Sheets made from repulped fiber are also slightly lower in brightness than virgin fiber sheets.

Improving the strength of recycled fiber

Strength loss generally can be regained by refining (*7*). Unfortunately, this usually reduces drainage and production capacity. Increased refining also limits the amount of strength that can be regained by refining in future cycles.

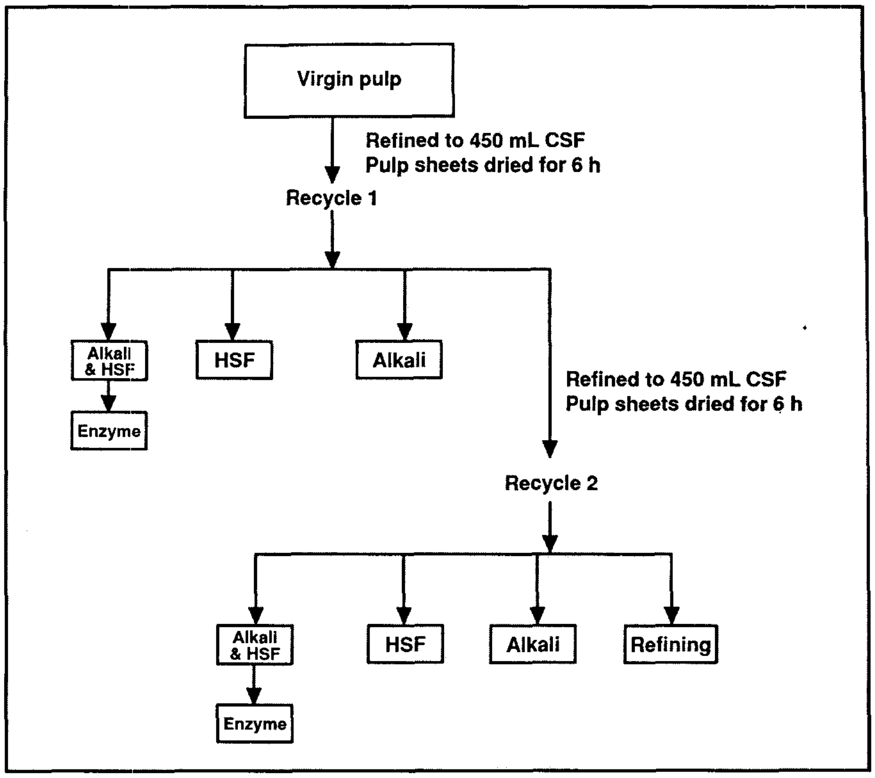
The use of chemical additives, which improve the strength properties without changing the repulping requirement, can provide an alternative method to refining (*8*). Two resins often used are an anionic polymer (*8*), which is capable of facilitating hydrogen bonding, and a cationic polymer, which is capable of forming strong electrostatic bonds between fibers and fines. These resins improve the dry strength of paper by increasing both the strength and the area of the interfiber bonds (*9*).

Treatment of wastepaper with sodium hydroxide increases the freeness and the strength properties of recycled fiber (*10*). Sodium hydroxide treatment promotes fiber swelling, thereby increasing fiber flexibility and surface conformability. Both alkaline treatment and delignification can improve the papermaking potential of recycled fibers.

Oxygen-alkali delignification has recently been studied as a means of improving strength properties in old corrugated container recycled pulp (*11*). The delignification treatment was found to improve bonding and strength characteristics, probably because of softening, swelling, and lignin removal. The strength improvement in the fiber is especially noticeable in the higher burst value and strain-to-failure value at a given drainage rate (*12*).

This paper presents the results of a study that was carried out to evaluate the effects of various mechanical and chemical treatments on the strength properties of handsheets prepared from recycled fibers.

1. Experimental design



Experimental

Pulp preparation

The virgin pulps used in this study included bleached and unbleached kraft softwood pulps. Unbleached kraft pulp of kappa no. 78 with an initial freeness of 758 mL CSF was obtained from an integrated paper mill. The bleached kraft pulp was a once-dried commercial pulp supplied in the form of air-dried sheets. Pulp brightness was 79 and freeness was 689 mL CSF.

The bleached pulp sheets were repulped in a hydrapulper, and the pulp was centrifuged to 30% consistency. The bleached and unbleached pulps were shredded and stored in plastic bags in a cold room at 4°C.

Both the bleached and unbleached kraft softwood pulps were refined to 450 mL CSF in a laboratory beater. Handsheets made from these pulps were tested for strength properties using TAPPI test methods. A freeness of 450 mL CSF was chosen for study because many commercial paper machines operate at or near this level of freeness.

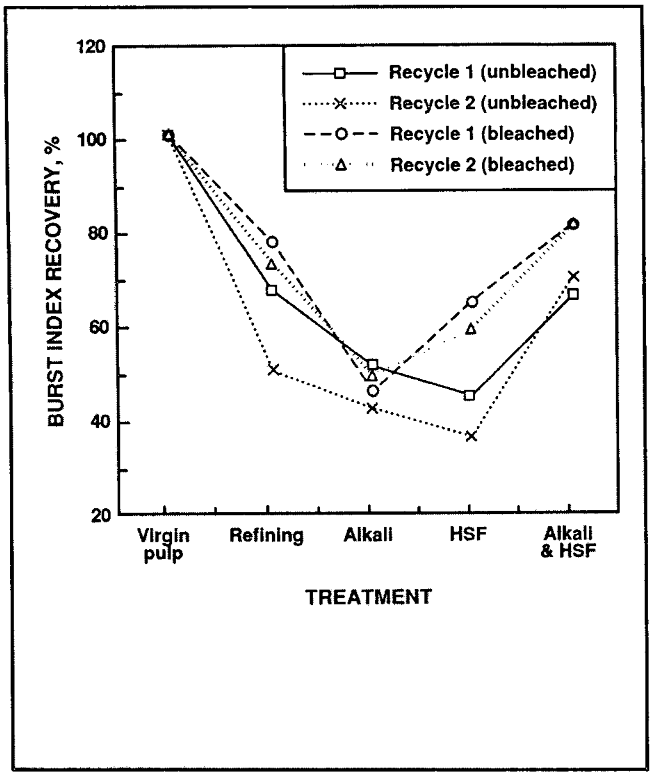
Preparation of recycled pulp

The samples used in this recycling study were obtained by forming handsheets and then repulping the handsheets. With this approach, the pulps could be dried by a controlled and reproducible technique. The severity and degree of drying were held constant for all pulp types and drying cycles. The paper suffered no "use" (supercalendering, printing, folding, or handling) between manufacture and repulping. Recycling by this method made it possible to control precisely the nature of the recycled fiber.

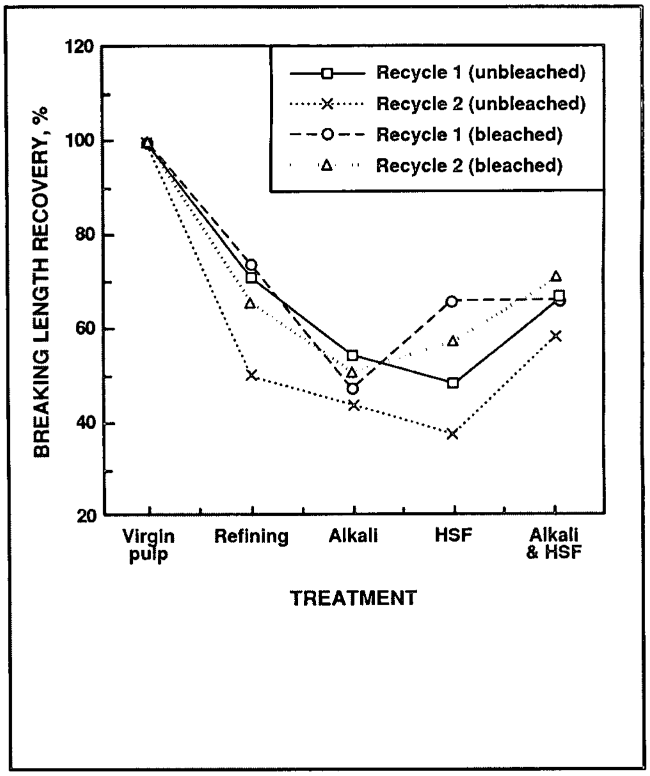
The study was carried out through two stages of recycling, Recycle 1 and Recycle 2. Throughout this discussion, Cycle 0 refers to the first time fibers are processed and dried to make paper. Recycle 1 represents fibers that were repulped for the first time, and Recycle 2 refers to fibers that were repulped for a second time.

For each recycle, pulp was refined to 450 mL CSF in a laboratory beater. The refined pulp was then made into pulp sheets of comparable weight on cheese cloth in a Buchner funnel. The sheet was then pressed at 50 psig and dried in an oven for 6 h at 105°C. Sheet forming, pressing, and drying were all held constant while generating the recycled fiber.

2. Burst index recovery after treatment of recycled pulp (virgin pulp = 100%)



3. Breaking length recovery after treatment of recycled pulp (virgin pulp = 100%)



Recycled pulp treatments

Refining. Pulp was refined to 450 mL CSF in a laboratory beater. Handsheets were made and tested for strength properties using the appropriate TAPPI test methods.

Alkali treatment. Repulped, recycled pulp was mixed with 3% NaOH (o.d. pulp basis) in a plastic bag after adjusting the consistency to 10%. The sealed plastic bag was kept in a water bath at 70°C for 30 min. The pulp was then washed thoroughly. Handsheets were formed and tested for strength properties.

High-shear field treatment. A Quantum Technologies Mark II high-intensity mixer/reactor was used to provide the high-shear-field (HSF) treatment. The recycled pulp was treated in the high-shear mixer bowl at 10% consistency and 70°C. The pulp was given HSF treatment for two min at 2400 rpm. Handsheets were formed and tested for strength properties using the appropriate TAPPI test methods.

Alkali and HSF treatment. Pulps were also treated with a combination of alkali and HSF treatments. The recycled pulp was mixed with 3% NaOH (o.d. pulp basis) in a plastic bag containing pulp at 10% consistency. The sealed bag was kept in a hot water bath at 70°C for 30 min. After 30 min of alkali treatment, the pulp was placed in the Quantum mixer bowl. Mixer temperature was maintained at 70°C. The pulp was subjected to HSF treatment for 2 min at 2400 rpm. The pulp was then washed and handsheets tested for strength properties using the appropriate TAPPI test methods.

Enzyme treatment. Pulps were treated with Liftase A 40, a commercial product that is a blend of enzymes from *Trichoderma longibrachiatum*. The product is composed mainly of cellulase and hemicellulase enzymes (*13*). Both unbleached and bleached kraft softwood pulps were studied in Recycle 1 and Recycle 2 stages. Recycled fibers were first treated with alkali and HSF treatments. The details of the treatment conditions are the same as described previously, except for the time of mixing in the reaction chamber. Pulps were subjected to different levels of HSF treatment to obtain different freeness levels. The alkali-treated pulp was mixed in a Quantum mixer for 30, 60, 80, 100, or 120 s. After the alkali and HSF treatment, the pulp was washed with water to remove the alkali.

The washed pulps were adjusted to pH 5.0 with sulfuric acid and then mixed with the enzyme [0.2% or 0.4% wt./wt. (o.d. pulp basis)] in a plastic bag at 3% consistency. The bag was sealed and kept in a water bath at 45°C with intermittent mixing. After 30 min of reaction time, the pulp was washed thoroughly with hot water at 70°C to denature the enzyme. The freeness of the enzyme-treated pulp was measured and compared with control pulps (no enzyme treatment).

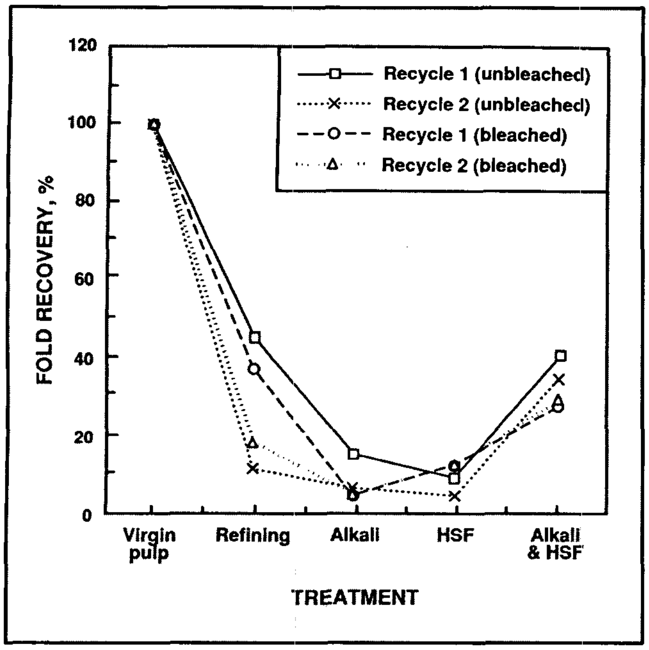
The experimental design is outlined in **Fig. 1**. The strength properties studied were burst index, breaking length, folding endurance, tear index, and ring crush. The Concora flat crush test was also studied for unbleached kraft softwood pulp. The results reported are the means from two duplicate sets of experiments.

Results for treatment of recycled pulps

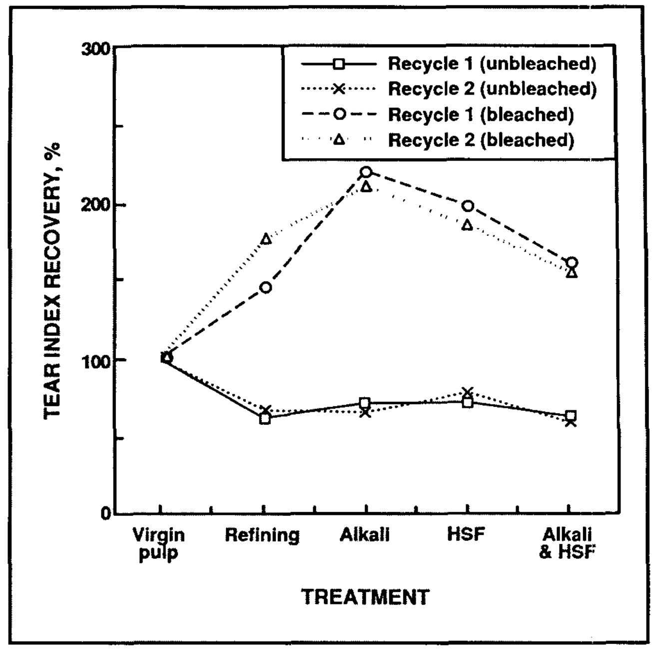
Chemimechanical treatments

The effectiveness of the various chemimechanical treatment methods was evaluated by measuring strength (burst, tear, fold, breaking length, ring crush) following each treatment and

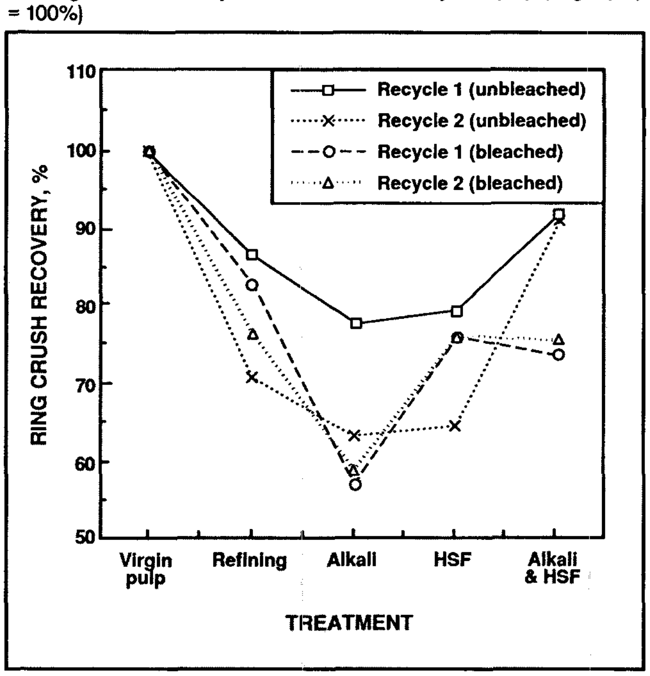
4. Fold recovery after treatment of recycled pulp (virgin pulp = 100%)



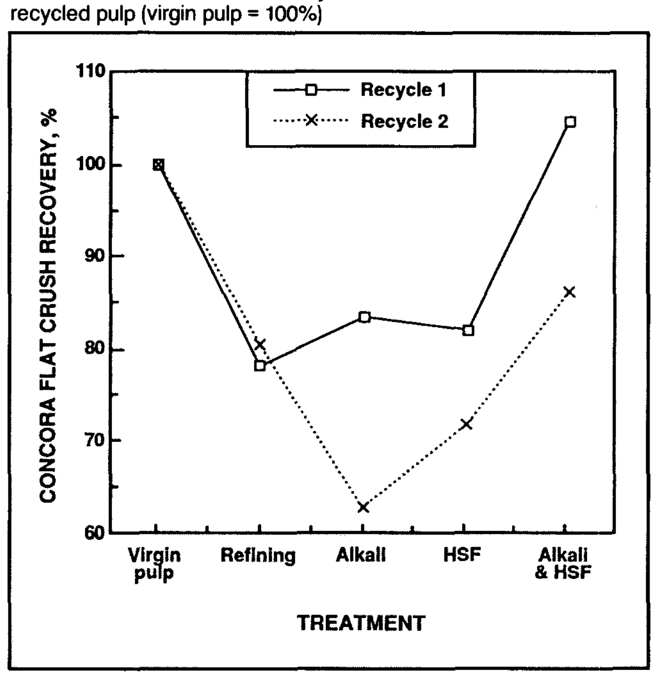
5. Tear index recovery after treatment of recycled pulp (virgin pulp = 100%)



6. Ring crush recovery after treatment of recycled pulp (virgin pulp = 100%)



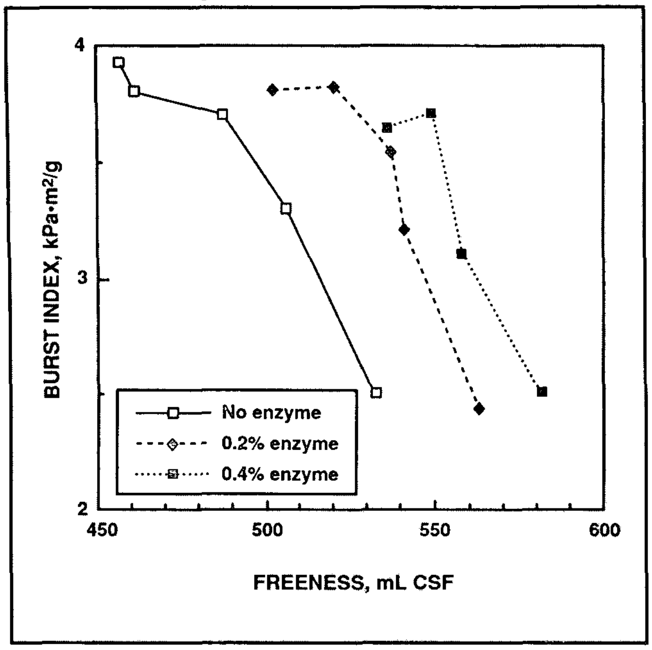
7. Concora flat-crush recovery after treatment of unbleached recycled pulp (virgin pulp = 100%)



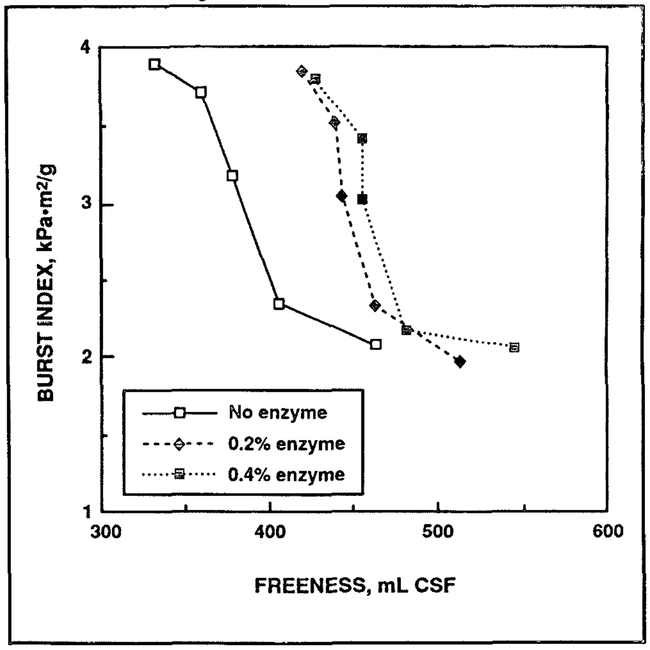
comparing it with the value obtained with the corresponding virgin pulp. However, the Recycle 1 and Recycle 2 pulps were not necessarily at the same freeness level following each treatment, even though all pulps were refined to 450 mL CSF prior to each recycling simulation. Freeness can be considered to be a measure of the drainage rate, which is influenced by the degree of fibrillation. In turn, fibrillation varies depending upon the nature, duration, and intensity of treatment. In this study, each treatment was carried out under constant conditions (reaction time, concentrations) for both Recycle 1 and Recycle 2 fibers. The main goal was to compare the effect of each treatment rather than comparing the strength properties at the same freeness.

Figure 2 shows the burst index recovery following treatment of the recycled pulps. Refining provides greater recovery than the separate alkali and HSF treatments. The combined alkali/HSF treatment provided about the same recovery as refining for the unbleached Recycle 1 pulp. For the other three pulps (un-

8. Burst index as a function of freeness for enzyme-treated unbleached Recycle 1 kraft pulp. Virgin pulp burst index at 450 mL CSF = 5.94 kPa • m²/g.



9. Burst index as a function of freeness for enzyme-treated unbleached Recycle 2 kraft pulp. Virgin pulp burst index at 450 mL CSF = 5.94 kPa • m²/g.



bleached Recycle 2 and bleached Recycle 1 and 2), the combined alkali/HSF treatment provided the greater recovery of burst index. It is interesting to note that burst recovery for the Recycle 2 pulps was equal to or higher than that of the Recycle 1 pulps after combined alkali/HSF treatment. However, burst index for the refined Recycle 2 pulps was lower than for the refined Recycle 1 pulps. This decline in strength for recycled pulps that have previously been refined can be explained by the cutting action in the refiner, which we believe produces more fines than HSF treatment. When refined Recycle 1 handsheets are recycled and refined again (refined Recycle 2), the resultant handsheet is weaker because the Recycle 2 pulp has more fines and fewer long fibers than the Recycle 1 pulp. Burst index for Recycle 2 pulps was retained better by a combination of alkali/HSF treatment. In this treatment, we hypothesize that the swollen fibers are flexed to delaminate and open the cell wall and that the cutting effect is reduced. Strength properties are thus retained or even increased when alkali/HSF-treated fibers are recycled a second time.

Figure 3 presents the tensile strength (breaking length) data for bleached and unbleached Recycle 1 and 2 kraft softwood pulps. Recovery of breaking length shows the same trends as were observed for burst index.

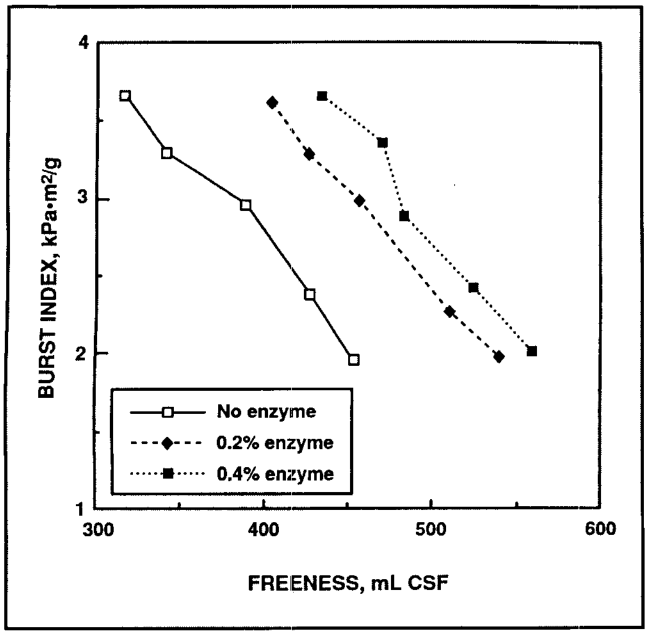
Figure 4 presents the folding endurance recovered by different treatments. Folding endurance attained after either alkali treatment or HSF treatment was lower than that for refining and for the alkali/HSF combination treatment. For the Recycle 1 pulps, folding endurance after the combination treatment was comparable with that for the refining treatment. For the Recycle 2 pulps, however, the folding endurance recovered after the combination treatment was much higher than that for the refining treatment. As in the case of burst index, this phenomenon can be explained by the cutting action in the refiner.

Figure 5 shows the tear index recovered following the different treatments for Recycle 1 and 2 pulps. For unbleached pulps, there was a significant strength loss of the same magnitude regardless of treatment. In the case of bleached pulp, strength was increased, with alkali treatment showing the greatest effect. The alkali treatment was the least effective of the investigated treatments in promoting fiber flexibility and interfiber bonding. Thus the tear strength can be attributed to weakened bonding, in agreement with current theory on tear strength.

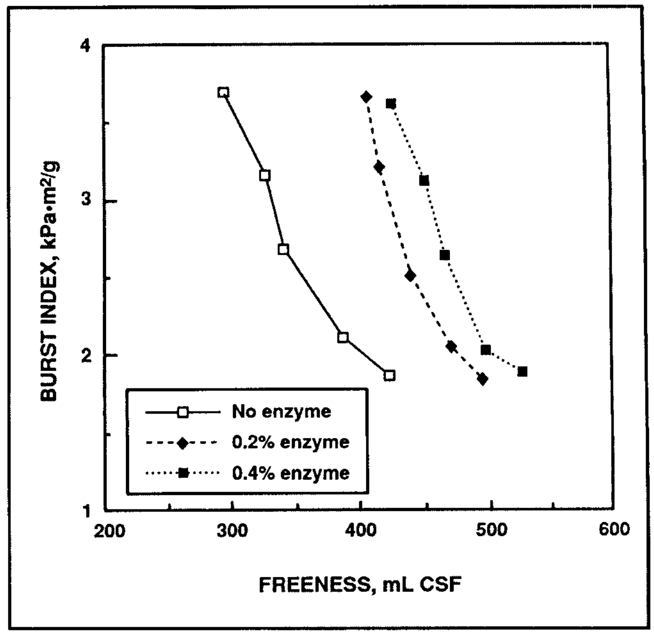
Ring crush attained by different treatments on Recycle 1 and Recycle 2 pulps is shown in **Fig. 6**. Recovery of ring crush following the combination treatment was higher than that for refining in the case of unbleached recycled fiber. For the bleached pulps, recovery was comparable with the refining treatment. The ring crush test is a very important test for container board, since it reflects stacking strength. Because recycled fibers are increasingly used in board manufacturing, the alkali/HSF combination treatment may be a useful alternative to refining.

Concora flat crush was measured for the unbleached kraft softwood pulp. This is an important test for the corrugated container industry. **Figure 7** shows the Concora flat crush recovered with different treatments for Recycle 1 and Recycle 2 pulps. Flat crush attained by the combination treatment is much higher than that for refining in Recycle 1 and even exceeds that for the virgin pulp. In Recycle 2, about 85% of the virgin pulp flat crush value is recovered using the combination treatment. Thus, the combined alkali/HSF treatment appears to be a better alternative for the corrugated container industry, since it would allow manufacturers to maximize the use of secondary fiber.

10. Burst index as a function of freeness for enzyme-treated bleached Recycle 1 softwood pulp. Virgin pulp burst index at 450 mL CSF = 4.8 kPa • m²/g.



11. Burst index as a function of freeness for enzyme-treated bleached Recycle 2 softwood pulp. Virgin pulp burst index at 450 mL CSF = 4.8 kPa • m²/g.



Enzyme treatment

The remaining figures show burst index as a function of pulp freeness for enzyme-treated (0.2% and 0.4%) recycled pulps. The various freeness levels were obtained by varying the HSF treatment in the Quantum mixer from 30 s to 120 s. Each point on the curve represents the freeness obtained for a particular mixing time and after enzymatic treatment.

The results for unbleached Recycle 1 in **Fig. 8** show that the control pulp (no enzyme) has a freeness of about 512 mL CSF corresponding to a burst index of 3 kPa•m²/g. At the same burst index, the 0.2% enzyme treatment increases the freeness to 548 mL CSF, while the 0.4% treatment increases it further to 565 mL CSF. Thus the enzymatic treatment can increase the freeness at the same burst index, in accordance with the findings of Pommier *et al.* (*13*).

Figure 9 shows the results for enzyme treatment of unbleached Recycle 2 pulp. For the control pulp, the burst index corresponding to a freeness of 450 mL CSF is 2.05 kPa•m²/g. At the same freeness, the burst index increases to 2.95 kPa•m²/g for the pulp treated with 0.2% enzyme and to 3.5 kPa•m²/g for the pulp treated with 0.4% enzyme. The results show that the enzyme increases pulp strength without loss of freeness.

Figures 10 and 11 show the burst index for bleached kraft softwood pulps in Recycle 1 and Recycle 2, respectively. Again, these figures show that higher freeness can be obtained without loss of strength properties by treating the pulps with enzyme. Enzyme treatment of bleached and unbleached recycled pulps showed similar results for breaking length, folding endurance, ring crush, and Concora flat crush. (Data not shown.)

The increase in freeness by enzymatic treatment has been explained by Pommier *et al.* (*13*) as follows. The enzymes act on the surface of the fibers, producing a "peeling" effect (*14, 15*). Fiber fines and fibrils are acted on preferentially, since they have an extremely high specific surface area (*16*), although they contribute very little to the hydrogen-bonding potential of the fiber slurry. Thus, with the fines removed, the pulp drains more easily without suffering a loss in mechanical properties.

As observed in Figs. 8–11, the freeness increase is greater for the bleached softwood pulp than for the unbleached softwood pulp. This is probably because the cellulose in the bleached pulp is more accessible to enzymes. The lignin in the unbleached pulp may hinder the attack of enzyme on cellulose, thereby reducing the peeling effect. In any event, the enzyme is less efficient in removing the fines and fiber fibrils in the unbleached pulp, and the increase in freeness is not as emphatic.

The magnitude of the increase in freeness was found to depend on the initial freeness of the pulp. If the freeness is low initially, the freeness increase is greater. A low initial freeness corresponds to more fines in the stock and more cellulose fragments and surface areas accessible to the enzyme. Thus, the rate of freeness increase is higher, which results in a higher freeness gain after a fixed period of time.

The relative gain in freeness is not as large when going from 0.2% to 0.4% enzyme as when going from no enzyme to 0.2% enzyme. Thus the 0.2% enzyme concentration seems to be the most economical way to obtain the desired freeness increase. This suggests that increasing the enzyme concentration will yield diminishing returns in terms of freeness gain. Furthermore, it has been demonstrated that high enzyme concentrations can cause fiber damage and strength loss (*17*).

Summary

Recycled paper loses strength mainly because of the fiber stiffening and hornification that occur when virgin fibers are dried during their initial papermaking cycle. This phenomenon is difficult to reverse. Some of the lost strength can be regained by making the recycled fiber more flexible, thereby increasing the surface area for bonding. Repulping under alkaline conditions and refining are the most commonly used methods to improve the strength of secondary fiber.

Alkali treatment helps to swell the secondary fiber, which increases the surface area available for bonding. A 3% NaOH treatment at 10% pulp consistency for 30 min at 70°C is typical of conditions commonly used. High-shear-field (HSF) treatment, in a pulp consistency range of 10–20%, can be used to produce an effect similar to refining. We hypothesize that the fiber wall structure is modified by the brushing and bending action, which increases the bonding area. The HSF treatment produces less fines than refining, and this results in less freeness loss.

A combination of alkali and HSF treatment may be a better alternative to obtain high product quality from secondary fiber. The strength properties of the recycled paper obtained by the combination alkali/HSF treatment are higher than those obtained by refining and, in some cases, are comparable with the virgin pulp. The combination treatment seems most effective in restoring ring crush and Concora flat crush strength. This treatment offers a potentially valuable, practical method of increasing the use of secondary fiber in boxboard as well as corrugating medium.

Enzymes can be used to increase the freeness of the secondary fiber without affecting the quality of the final product. A preparation of cellulase and hemicellulase at 0.2% enzyme concentration, 30 min, 10% pulp consistency, pH 5, and 45°C was the most economical and practical level examined for the pulps investigated.□

Literature cited

1. Franklin, W. E., Tappi J. 69(2): 28(1986).
2. Ince, P. J., Likely Developments in Pulp and Paper, USDA Forest Products Laboratory, Madison, Wis., 1987.
3. Broeren, L. A., Pulp Paper 64(3): 71(1990).
4. De Ruvo, A., Htun, M., and Ehrnrooth, E., Fundamental aspects on the maintaining properties of paper made from recycled fiber, EuCePa Symposium Proceedings (Warsaw, Poland), EuCePa, Paris, 1978.
5. McKee, R., Paper Trade J. 155(21): 34(1971).
6. Guest, D., and Weston, J., 1986 Pulping Conference Proceedings, TAPPI PRESS, Atlanta, p. 589.
7. Chase, R., Tappi 58(4): 90(1975).
8. Chan, L., Pulp Paper Can. 77(6): 43(1976).
9. Linke, W., Tappi 51(11): 59A(1968).
10. Eastwood, F., and Clarke, B., Paper Technol. 18(5): 156(1977).
11. De Ruvo, A., Farstrand, P., Hagen, N., and Haglund, N., Tappi J. 69(6): 100(1986).
12. Markham, L., and Corchene, C., Tappi J. 71(12): 168(1988).
13. Pommier, J-C., Fuentes, J-L., and Goma, G., Tappi J. 72(6): 187(1989).
14. Lee, S., and Kim, I., Biotechnol. Bioeng. 25(1): 33(1983).
15. Chanzy, H., and Hernissat, B., FEBS Letters 184(2): 285(1985).
16. Paquot, M., Thonart, P., Jacquemin, P., and Rassel, A., Holzforschung 35(2): 87(1981).
17. Oltus, E., Mato, J., Bauer, S., and Farkas, V., Cellulose Chem. Technol. 21(9): 663(1987).

Received for review Jan. 2, 1991.

Accepted Feb. 27, 1991.