Fiber characterization using confocal microscopy—the effects of recycling

Ho Fan Jang, Roger C. Howard, and Rajinder S. Seth

ABSTRACT: The effects of laboratory recycling on the transverse dimensions of fibers of three softwood pulps—a TMP, a CTMP, and a kraft—have been determined with confocal microscopy and image analysis. From the decrease in lumen area and increase in the degree of collapse, the mechanical pulp fibers appear to flatten upon recycling. However, this did not occur for the chemical pulp fibers as they were mostly collapsed to start with. These results agree with previous findings on these samples that recycled mechanical pulp fibers, in contrast to chemical pulps, tend to give a denser and stronger sheet. Recycling decreased fiber wall cross-sectional area. Although fiber wall thickness also decreased, it was significant only for TMP.

KEYWORDS: Chemical pulps, chemithermomechanical pulps, fibers, image analysis, kraft pulps, lumens, microscopy, reclaimed fibers, recycling, softwood pulps, thermomechanical pulps.

The effects of recycling on the properties of papermaking pulps have been investigated extensively *(1–3)*. The increasing use of recycled fibers in newsprint furnishes, which contain mostly mechanical or chemimechanical pulps, has generated interest in the effect of recycling on these pulp fibers. In a recent study on the physical properties of laboratory recycled pulps *(1)*, it has been shown that handsheets made of recycled mechanical pulps have a higher density, a lower scattering coefficient, and improved tensile and bursting strengths. These changes were attributed to mechanical pulp fibers becoming progressively flattened and more flexible during recycling. Although fiber flattening upon recycling has been observed in micrographs of handsheet cross-sections *(1)*, these changes in fiber transverse dimensions have not been quantified. Confocal laser scanning microscopy (CLSM) is a powerful new technique for rapidly and nondestructively generating cross-sectional images of wood pulp fibers *(4)*. Combined with image analysis, the technique can accurately determine fiber transverse dimensions. In this report, we have used this technique to examine the effects of recycling on fiber collapse and transverse dimensions for the fibers of a previous investigation *(1)*.

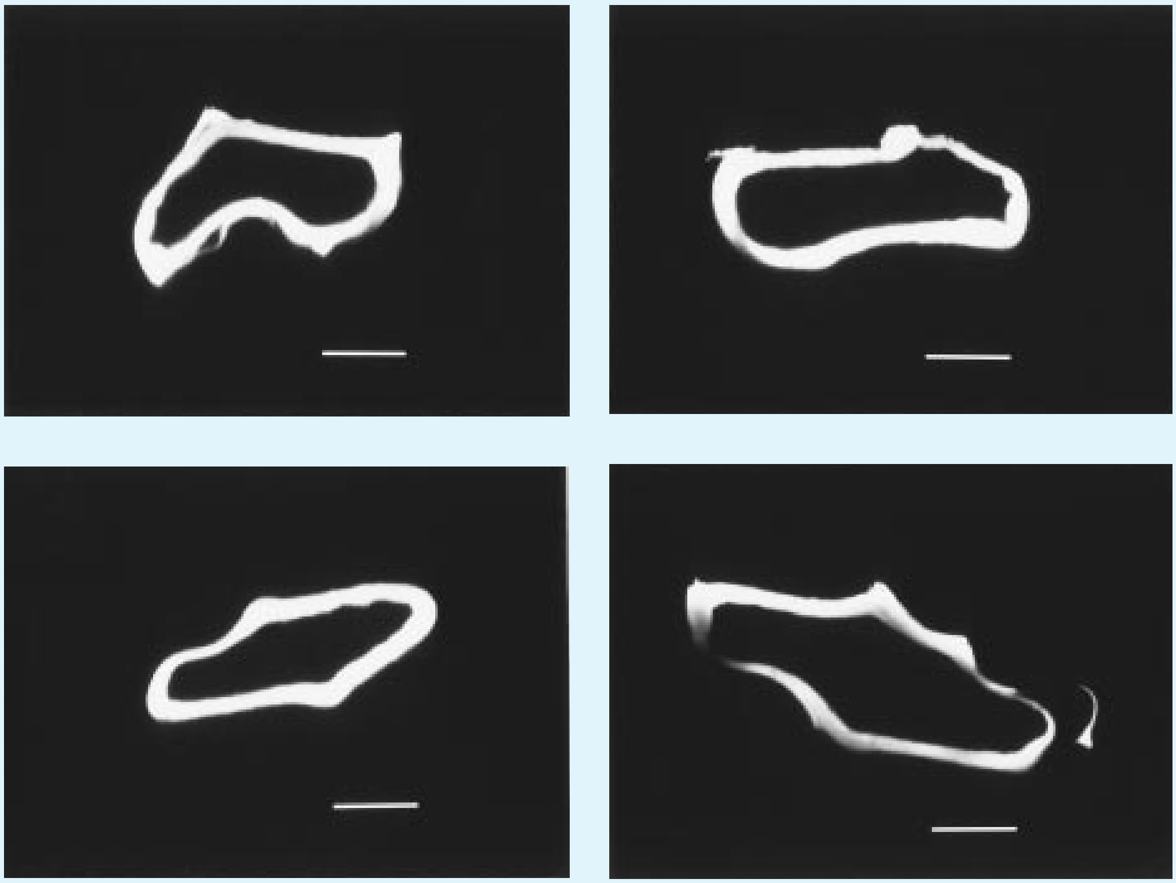
Experimental

Three commercial softwood pulps were examined: a thermomechanical pulp (TMP), a chemithermomechanical pulp (CTMP), and a western Canadian beaten bleached softwood kraft pulp (HS). Fibers recycled once and five times, denoted by symbols 1 and 5 (e.g., TMP-1 and TMP-5), were used. The origin of these pulps, the recycling procedures which involved reslushing, handsheet making and drying, and handsheet physical properties have been reported earlier *(1)*.

A Bio-Rad MRC-500 confocal laser scanning system attached to a Zeiss Axiophot microscope operated in epifluorescence mode was used to generate fiber cross-sectional images. Fibers were lightly dyed with a fluorochrome dye and air-dried on microscope slides. During measurements, each fiber was oriented perpendicular to the scanning direction, and an optical section through its thickness was obtained. **Figure 1** shows typical cross-sectional images of TMP fibers obtained with the CLSM. Only well-defined fiber

Jang, Howard, and Seth are scientists at the Pulp and Paper Research Institute of Canada, 3800 Wesbrook Mall, Vancouver, BC V6S 2L9, Canada.

1. Epifluorescence cross-sectional images of airdried TMP fibers. The scale bar is 10 µm.



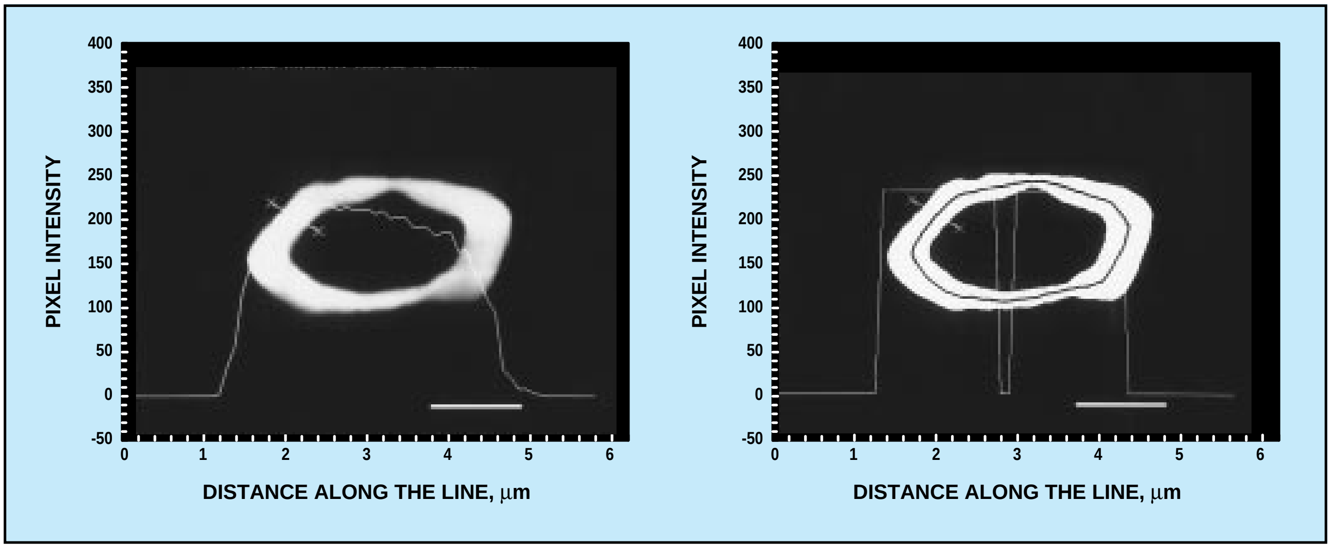
cross-sections were counted. That is, severely damaged fibers and fiber fragments were ignored. Image analysis was used to quantify these images. **Figure 2** shows an original confocal image and its processed binary image with the center-line perimeter drawn. The details of confocal microscopy and image analysis techniques have been reported earlier *(5)*. Fiber transverse dimensions such as cross-sectional area (*A*), center-line perimeter (*P*), lumen area (*LA*), and lumen perimeter were measured as shown in **Fig. 3**. The fiber wall thickness (*T*) was calculated as *A/P*. The longest (*D*max) and shortest (*D*min) Feret diameters, which can be used to define fiber width and fiber thickness, were also determined.

During pulping and papermaking processes, fibers are exposed to external forces which can change their native cross-sectional shape. The volume or cross-sectional area of the lumen is usually reduced or even eliminated. Such fiber flattening or collapse can be characterized and estimated in different ways *(6–8)*. For example, the aspect ratio (*AR*) defined by the ratio of *D*min/*D*max in Fig. 3 can indicate the degree of fiber flattening or collapse *(6)*; a smaller ratio implies a greater collapse. Based on the decrease in fiber lumen volume or cross-sectional area, we have defined a collapse index (*CI*) as follows:

$$\text{Collapse Index } (CI) = 1 - (LA/LA_0) \quad (1)$$

where *LA* is the lumen area obtained from the cross-sectional image of a fiber (after treatments that may have caused flattening or collapse), and LA_0 is the lumen cross-sectional area of the same fiber, as if it had not been subjected to any external forces, and therefore had not collapsed. *LA* at any point along the length of a fiber can be measured from its con-

2. Original CLSM image (A) and image after processing (B). The cross-sectional area and center-line perimeter were 197 μm^2 and 54.4 µm, respectively. The calculated mean wall thickness was 3.6 µm. The scale bar is 10 µm. Pixel intensity profiles vs. length along line x–x are also shown.



I. Cross-section dimensions and their differences for TMP-1 and TMP-5

Parameters	*TMP-1 Mean ± SD[a] n = 553*	*TMP-5 Mean ± SD n = 481*	*Difference ± 95% CI[b]*	*Relative difference, %*
Cross-sectional area (*A*), μm^2	129 ± 3	119 ± 3	-10 ± 8	-8.3
Perimeter (*P*), µm	60.0 ± 0.9	59.5 ± 1.0	-0.7 ± 2.6	-1.2
Wall thickness (*T*), µm	2.22 ± 0.04	2.05 ± 0.05	-0.17 ± 0.13	-7.9
Lumen area (*LA*), μm^2	147 ± 6	128 ± 5	-20 ± 15	-14
Longest diameter (D_{max}), µm	28.3 ± 0.4	28.5 ± 0.4	+0.1 ± 1.1	+0.5
Shortest diameter (D_{min}), µm	13.2 ± 0.2	11.8 ± 0.2	-1.4 ± 0.6	-11
Aspect ratio (*AR*)	0.48 ± 0.01	0.43 ± 0.01	-0.04 ± 0.02	-9.6
Collapse index (*CI*)	0.21 ± 0.01	0.26 ± 0.02	+0.05 ± 0.04	+21

[a]SD = Estimated standard deviation of the mean
[b] CI = Confidence interval
[n] = number of fibers

focal cross-sectional image. LA_0, being not known, is determined as follows. The native lumen cross-section was assumed to be rectangular. LA_0 was calculated from the measured lumen perimeter and its "estimated" uncollapsed lumen aspect ratio *(9)*. This collapse index is more representative of the collapse behavior of a fiber as it measures the fractional change in the lumen cross-sectional area with respect to the uncollapsed one. For example, it does not depend on the shape of collapsed fiber, as does the degree of collapse indicated by *D*min/*D*max. *CI* is equal to 1 if a fiber is totally collapsed, whereas it is zero for an uncollapsed fiber. This *CI* can be used for comparing the degree of flattening or collapse of a pulp as it is processed or of different pulps. Fiber collapse is an important property because it affects sheet structure and, therefore, the physical properties of the sheet.

Results and discussion

Figure 4 shows frequency distributions of *A*, *P*, *T*, and *LA* for TMP-1 and TMP-5 fibers. The mean values, the estimated uncertainties of the means, and the differences of the means for these parameters are shown in **Table I**. The frequency distributions show that, relative to TMP-1, TMP-5 had a higher proportion of fibers with *A* less than 120 μm^2, *T* less than 2.5 µm, and *LA* less than 100 μm^2. The mean values for *A*, *T*, and *LA* decreased by 8%, 8%, and 14%, respectively, with the additional four recycles (Table I). However, the small decrease in *P* was not significant. The increase in mean *D*max was 0.5%, but the decrease in mean *D*min was 11%. This means that fiber width was unchanged, but fiber thickness decreased because of fiber flattening. The mean aspect ratio decreased about 10% and the mean

II. Cross-section dimensions and their differences for CTMP-1 and CTMP-5

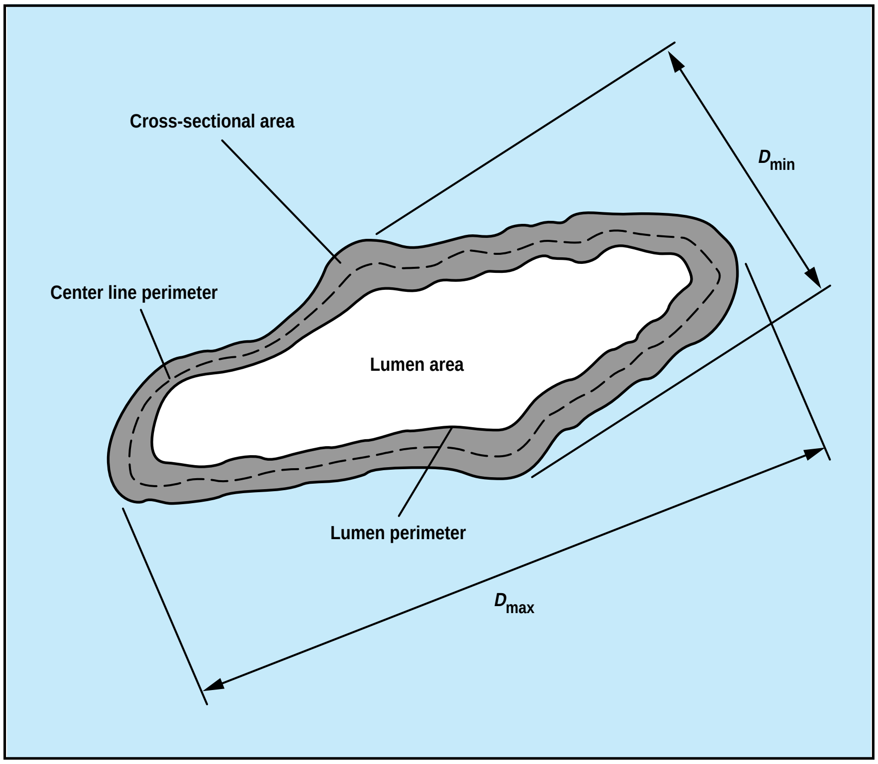
Parameters	CTMP-1 Mean ± SD[a] n = 95	CTMP-5 Mean ± SD n= 91	Difference ± 95% CI[b]	Relative difference, %
Cross-sectional area (A), μm²	155 ± 8	143 ± 7	-12 ± 20	-7.9
Perimeter (P), μm	69.4 ± 2.3	67.9 ± 2.4	-1.5 ± 6.6	-2.2
Wall thickness (T), μm	2.35 ± 0.13	2.24 ± 0.12	-0.12 ± 0.34	-5.0
Lumen area (LA), μm²	153 ± 14	111 ± 12	-41 ± 37	-31
Longest diameter (D_{max}), μm	32.2 ± 1.0	32.3 ± 1.2	+0.0 ± 3.0	+0.07
Shortest diameter (D_{min}), μm	13.6 ± 0.5	12.0 ± 0.6	-1.6 ± 1.5	-13
Aspect ratio (AR)	0.45 ± 0.02	0.41 ± 0.02	-0.04 ± 0.06	-9.6
Collapse index (CI)	0.34 ± 0.03	0.41 ± 0.04	+0.06 ± 0.11	+17

[a]SD = Estimated standard deviation of the mean
[b]CI = Confidence interval
[η] = number of fibers

collapse index (CI) increased by 21%. The substantial decreases in lumen area, Dmin, and aspect ratio, and increase in collapse index (CI) from TMP-1 to TMP-5 strongly suggests that fibers in TMP-5 were more flattened than in TMP- 1. These results are consistent with the previous hypothesis that the degree of collapse of mechanical pulp fibers increases due to repeated mechanical treatment and drying during recycling *(1)*.

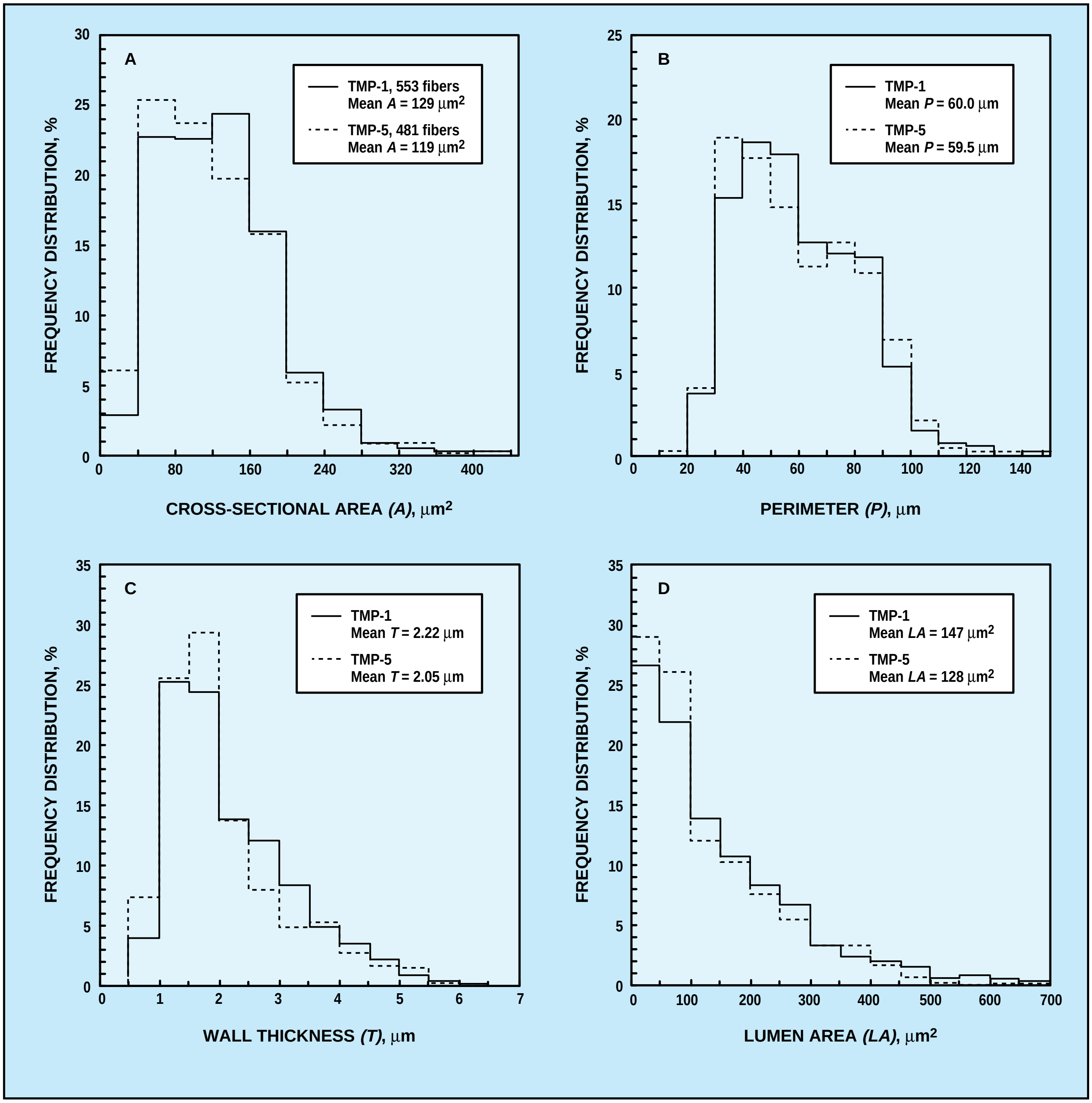
Two mechanisms can be suggested for the flattening of TMP fibers upon recycling: (a) decrease in cell wall transverse rigidity, and (b) decrease in fiber wall thickness. Lumen area (LA) and collapse index (CI) are plotted against fiber wall thickness in **Figure 5A and B,** respectively. Both TMP-1 and TMP-5 fibers of a given wall thickness were found to have, on the average, similar perimeters. Figure 5A shows that TMP-5 fibers have a smaller lumen area than TMP-1 fibers (except perhaps for very thick-walled fibers). Therefore, the smaller lumen area of TMP-5 suggests that these recycled fibers were more flattened or collapsed. This is further illustrated in Fig. 5B where collapse index (CI) is plotted against fiber wall thickness; for all wall thicknesses, recycled fibers had a higher collapse

3. Various cross-sectional dimensions of a fiber



index. The resistance of fibers to collapse is a function of the rigidity of the fiber wall in its transverse plane in addition to the physical dimensions such as wall thickness *(8)*. Since the decrease in lumen area and increase in collapse index upon recycling have occurred for all wall thicknesses, this suggests that the recycling process has decreased the rigidity of the fiber wall, supporting the first mechanism. However, previous work has shown that simple rewetting and drying high yield fibers does not change the elastic modulus of the wet fiber wall *(10)*. Since in our recycling procedure, fibers were given mechanical treatment through repeated pulp disintegration and handsheet press-

4. Frequency distributions of (A) cross-sectional area, (B) perimeter, (C) wall thickness, and (D) lumen area for airdried TMP-1 and TMP-5 fibers



ing, it is likely that this incidental treatment may have caused the decrease in the fiber wall rigidity.

Figure 5B, which shows the relationship between fiber collapse and wall thickness for TMP fibers, is similar to that previously shown for chemical pulps *(8)*, and, as expected, indicates that thin-walled fibers collapse more readily than fibers with thicker walls. Since the walls of TMP-5 fibers were about 8% thinner, on the average, than TMP-1, they should flatten more easily. This supports the second suggested mechanism. It is possible, therefore, that decreases in both fiber wall thickness and the rigidity of the cell wall material were responsible for the flattening of mechanical pulp fibers upon recycling. The sheets made from such fibers would be expected to be denser and better bonded, supporting previous findings that these recycled mechanical pulp fibers give denser and stronger sheets *(1)*.

The effects of recycling on CTMP fibers are given in **Table II**. Although *A*, *P*, and *T* decreased upon recy-

III. Cross-section dimensions and their differences for HS-1 and HS-5

Parameters	CTMP-1 Mean ± SD[a] n = 333	CTMP-5 Mean ± SD n= 374	Difference ± 95% CI[b]	Relative difference, %
Cross-sectional area (*A*), µm^2	143 ± 3	133 ± 3	-11 ± 8.4	-7.7
Perimeter (*P*), µm	65.9 ± 1.3	63.9 ± 1.3	-1.9 ± 3.6	-3.0
Wall thickness (*T*), µm	2.41 ± 0.06	2.33 ± 0.06	-0.09 ± 0.18	-3.6
Lumen area (*LA*), µm^2	1.4 ± 0.4	0.9 ± 0.2	-0.53 ± 0.87	-47
Longest diameter (D_{max}), µm	34.6 ± 0.6	33.9 ± 0.6	-0.70 ± 1.7	+2.1
Shortest diameter (D_{min}), µm	6.5 ± 0.1	6.0 ± 0.1	-0.50 ± 0.35	-8.1
Aspect ratio (*AR*)	0.22 ± 0.01	0.21 ± 0.01	-0.01 ± 0.02	-3.9
Collapse index (*CI*)	0.99 ± 0.01	0.98 ± 0.01	-0.01 ± 0.02	-0.8

[a]SD = Estimated standard deviation of the mean

[b] CI = Confidence interval

[η] = number of fibers

cling, the decreases were not significant. Also, similar to TMP, CTMP tended to flatten upon recycling, as indicated by the 17% increase in collapse index, and the decrease in *LA*, *D*min, and *AR*. It is of interest to note that the collapse index for CTMP-1 was higher than that of TMP-1, and this trend continued; the collapse index of CTMP-5 was higher than that of TMP- 5. This probably reflects the lower cell wall rigidity of CTMP fibers, resulting from the chemical pretreatment given prior to mechanical pulping.

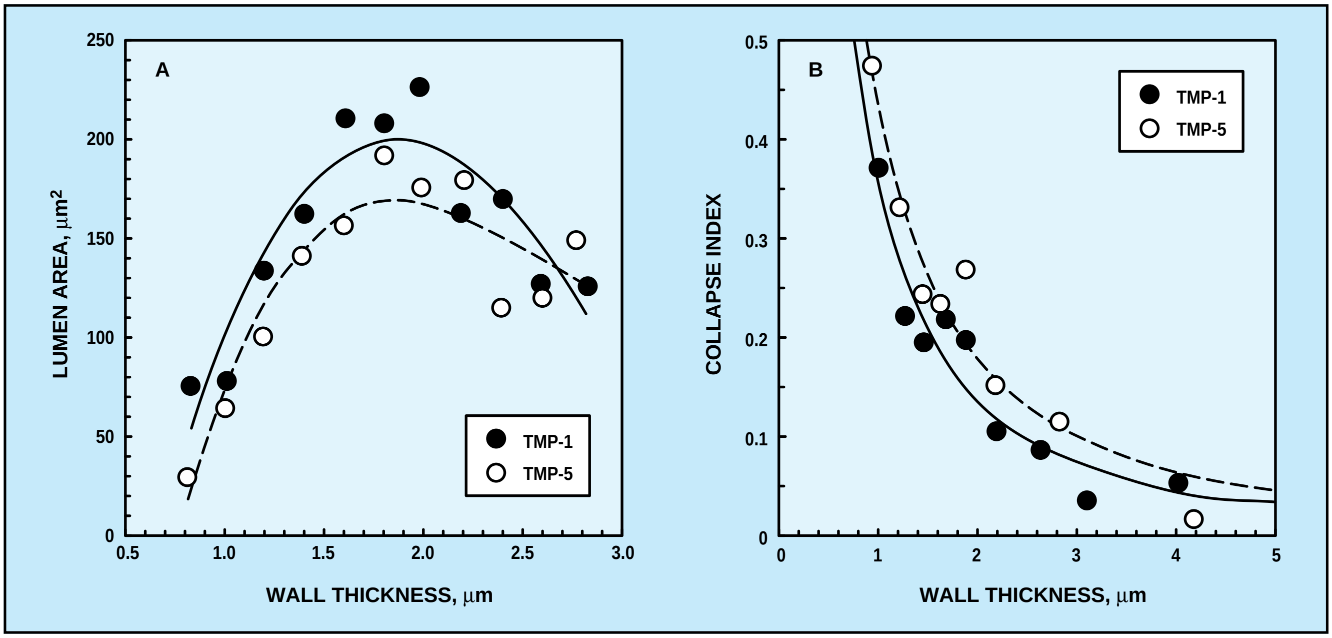
For a given pulp type, the fiber width, *D*max, remained relatively constant for all pulps; however, recycling caused more than a 10% decrease in fiber thickness, *D*min, for both TMP and CTMP fibers.

Table III shows results for the beaten chemical pulp fibers HS-1 and HS-5. As with mechanical pulps, general decreases occurred in *A*, *P*, and *T* upon recycling. However, only the decrease in *A* was significant. The low mean value of *LA* and the high mean value of *CI* for HS-1, 1.4 µm^2 and 0.99, respectively, indicate that these pulp fibers were mainly collapsed at the outset. The changes in lumen area and the degree of collapse upon recycling were therefore not expected to be significant. This conclusion is also consistent with previous findings that after the first recycle, the density of sheets made from this chemical pulp changed only slightly *(1)*.

For all pulps in this investigation, the decrease in the mean wall cross-sectional area was about 8% after recycling four times. The decrease in mean perimeter was less than 3% and only weakly significant. Because of the decrease in *P*, the decrease in wall thickness was not proportional to and was less than the percentage decrease in *A*. The decrease in *T* was significant only for TMP fibers (Table I).

We are unable to explain, at this time, why the fiber wall cross-sectional area decreases about 8% upon recycling, but can offer some possible reasons. The first is dissolution of the fiber wall material during repeated reslushing. However, this may not be large enough to account for the entire loss *(11)*. The second is detachment of the outer wall material. It has been suggested that peeling of the outer fiber layers and the consequent decrease in fiber coarseness occurs during mechanical pulping *(12, 13)*. However, we do not know if the gentle mechanical treatment during reslushing could cause such peeling. The third is that our fiber selection procedures may bias the analysis somewhat. Since cross-sectional areas were measured only on undamaged fibers, it is possible that some fibers with large cross-sections, which might conceivably have been more prone to damage during sheetmaking and recycling, were left out. This could result in a bias toward fibers of smaller cross-sectional areas. Whatever the explanation, it does not alter the main observation that at a given wall thickness, recycled mechanical pulp fibers are flatter. Our results represent quantitative evidence in support of what has hitherto been only observed qualitatively *(1)*.

5. Lumen area (A) and collapse index (B) versus wall thickness for TMP-1 and TMP-5 fibers. The degree of collapse decreased with increasing fiber wall thickness. The total numbers of fibers characterized are given in Table I. In Fig. 5A, each point represents fibers whose wall thicknesses fell within a 0.2 µm wide class. In Fig. 5B, each point represents 50 fibers. The curves in Fig. 5B are fits of a function, $CI = 1 - \exp(-\alpha/T^{\beta})$, where α and ß are constants of the fit *(9).*



Concluding remarks

Confocal microscopy combined with image analysis was used to examine the changes in transverse dimensions of fibers upon laboratory recycling. The recycling procedure involved repeated reslushing, handsheet making, and drying. This decreased fiber wall cross-sectional area. The decrease in fiber wall thickness was significant for TMP fibers. From the decrease in the lumen area and the increased fiber collapse, mechanical pulp fibers tended to flatten upon recycling. The increased collapse was ascribed to the reduced rigidity of the fiber wall material in addition to the effect of decreased fiber wall thickness. For the chemical pulp, the changes in lumen area and fiber collapse were not significant.

These findings agree with previous results that mechanical pulp fibers, recycled in the laboratory, give a denser and stronger sheet. We would anticipate that mechanical pulp fibers which have been through a paper machine before recycling, rather than a handsheet machine, would experience even more flattening because of the heavier pressing and calendering. This explains the common observation that recycled newsprint is usually less bulky than virgin newsprint.

Literature cited

1. Howard, R. C. and Bichard, W., *J. Pulp Paper Sci*. 18(4): J151(1992); 19(2): J57(1993).
2. Chatterjee, A., Roy, D. N., and Whiting, P., *78th CPPA Annual Meeting Preprints,* TS CPPA, Montreal, 1992, p. A277.
3. Laivins, G. V. and Scallan, A. M., Trans. of the Tenth Fundamental Research Symposium, Oxford, UK, 1993, p. 1235.
4. Jang, H. F., Robertson, A. G., and Seth, R. S., *Tappi J.* 74 (10): 217(1991).
5. Jang, H. F., Robertson, A. G. and Seth, R. S., *J. Mater. Sci.* 27: 6391(1992).
6. Kibblewhite, R. P. and Bawden, A. D., PIRA conference on "Current and future technologies of refining," Birmingham, UK, December 1991.
7. Amiri, R., Gorres, J., Grondin, M., *et al.*, Trans. of the Tenth Fundamental Research Symposium, Oxford, UK, 1993, p. 285.
8. Page, D. H., *Tappi* 50 (9): 449(1967).
9. Jang, H. F. and Seth, R. S., "Fiber characterization using confocal microscopy—The collapse behaviour of papermaking fibers," to be submitted.
10. Scallan, A. M. and Tigerström, A. C., *J. Pulp Paper Sci*. 18(5): J188(1992).
11. Bouchard, J. and Douek, M., *J. Pulp Paper Sci.* 20(5): J131(1994).
12. Karnis, A., *J. Pulp Paper Sci.* 20(10): J280(1994).
13. Jang, H. F., Amiri, A., Seth, R. S., *et al.*, *81st CPPA Annual Meeting Preprints,* TS CPPA, Montreal, 1995, B147.

We thank Vindhya Amarasinghe of the University of British Columbia (UBC) for generating CLSM images, Ben Chan for image analysis, Gordon Robertson for helpful discussions, John Carlson (UBC) for use of the CLSM, and the Science Council of British Columbia for its postdoctoral fellowship to H. F. Jang.

Received for review Sept. 2, 1994.

Accepted May 30, 1995.