

MORPHOLOGICAL CHANGES OF JACK PINE LATEWOOD AND EARLYWOOD FIBERS IN THERMOMECHANICAL PULPING

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The morphological changes of jack pine (*Pinus banksiana*) earlywood (EW) and latewood (LW) in thermomechanical pulping (TMP) were studied by light microscopy and scanning electronic microscopy. The results indicate that: under the mechanical forces in refining, the EW fibres tend to separate in the P/S1 interface, while separation of the LW fibres takes place commonly in the P/S1 and S1/S2 regions. The thick-walled LW fibres exhibit much more external fibrillation than the thin-walled EW. As a result, the LW fines contain more fibrillar component than EW fines. The EW fibers suffer more fiber cutting and splitting than the LW fibers. In addition, the thin-walled EW fibres show higher collapsibility and conformability than the LW counterparts.

Keywords: Jack Pine (*Pinus banksiana*); Earlywood; Latewood; Thermomechanical Pulping; Morphology

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INTRODUCTION

Within a growth ring, the portion of the growth formed in the early season or spring is commonly designated as earlywood (EW), also called springwood, while the denser and hence frequently darker wood produced later in the growing season/summer is termed latewood (LW), or summerwood. EW cells have thinner cell walls when compared with those of LW. As a general rule, the difference between EW and LW in hardwood is not as distinguishable as that of softwood (Panshin et al. 1970). Studies (Larson 1960; Little et al. 1968; Funada et al. 2001) indicated that the presence of different concentrations of auxin (a growth regulator or hormone) in different stages of the growing season was the main factor controlling the formation of EW and LW. Cell division is faster in the early season due to the higher level of hormone in the cambial zone. The fast growth produces cells with large radial diameter, wide lumen, and thin walls. Because the level of hormone decreases later in the growing season, cell division slows down and the cell diameter gradually decreases, while the cell wall increases in thickness. This wood tissue is called LW. Besides the effect of auxin, other studies (Panshin et al. 1970) indicated that environmental factors, e.g., nutrients, water, temperature, and light also have indirect influences on the development of EW and LW.

Due to their morphological discrepancies, EW and LW fibers behave differently in refining, especially in thermomechanical pulping. Previous research indicated that the EW fibres are more flexible (Agut et al. 1987; Luner 1986; Hattula et al. 1988) and collapsible (Hartler et al. 1969; Uhmeier et al. 1996; Mohlin 1975; Smith et al. 1972; Hartler et al. 1970) than those of LW, the latter being stiffer and less collapsible. In

refining, the LW fibres also exhibit a greater reduction in cell wall thickness. What's more, the EW fibres tend to split, especially in the 1st stage of refining (Reme et al. 2001), and they require more refining energy to reach the same freeness compared to LW fibres (Murton et al. 2001). These findings indicate that EW and LW respond differently to mechanical actions. Due to this dissimilarity, the property of the final pulp or paper would depend on the proportion of EW and LW fibres in the furnish.

In spite of these findings, it remains unclear how these two types of wood tissue are transformed from solid wood into individual fibrous elements in refining. Although some progress has been made in understanding the refining mechanism, there is little published information regarding the breakdown mechanism of EW and LW in refining, especially in thermomechanical refining. In this study, the morphological changes of EW and LW fibers in different TMP pulp length fractions were studied by light microscopy, and the fiber cross-section characteristics were examined by scanning electronic microscopy (SEM). With the results obtained from this research, we could gain better understanding of the mechanical failures of these two types of wood tissue and will also help improve the quality of the pulps produced from Jack pine, an under-exploited and less desirable species among the Canadian forest resources. The expected results also have the potential to help us ameliorate refining efficiency.

EXPERIMENTAL

Materials

Logs of freshly felled Jack pine (*Pinus banksiana* Lam.) were used in this work. The Jack pine trees were taken from a 30-year-old plantation in the St. Maurice region of Quebec. The logs were sawn into disks about 2.5 cm thick in a longitudinal direction. The disks were then debarked manually by means of a chisel. Chips were prepared from the sapwood portion, excluding the heartwood to minimize the possible effects of its high extractive content on pulp properties. The EW and LW chips, approximately 2 to 3 cm in width and length, were also prepared manually using a chisel. The thickness of the chips varied depending on the width of the growth rings and the proportion of EW and LW in the growth increment. The separation of EW from LW was based on the difference in colour: LW in Jack pine was broader and much darker than the EW counterpart.

The cross-section features of EW and LW, such as cell wall thickness and lumen area were measured based on the method T263 sp-06 and ImageJ algorithm (National Institute of Health 2011). The fiber length of EW and LW were measured using light microscopy analysis on chemically macerated fibers (Lanouette et al. 2010).

Refining

A Sunds Defibrator 300 CD pilot plant (Metso Paper) was used for refining the chips, with a refining capacity of 2t/day. The models of this refiner rotor and stator plate are R3809BG and R3803, respectively. During the process, in which EW and LW chips were separately refined, the chips were pre-steamed atmospherically for 10 min and then screw-fed into a digester using a 2:1 compression ratio. The refining was carried out in two stages, as indicated in Table 1. The first-stage was pressurized at 160 °C and the

pulps were produced with a freeness of about 500 mL. The Canadian standard freeness (CSF) was measured following Tappi method T227 om-04. The primary pulps were refined atmospherically to a freeness range of 50 to 200 mL and the specific energies were recorded for the pulps at each freeness level. The 1st stage refining consistency is about 20 to 24%, while the 2nd stage is around 10 to 14%. In addition, the refiner plate clearance was different between the two stage refinings: 0.40 to 0.60 mm in the 1st stage refining while 0.25 to 0.50 mm for the 2nd stage refining. After refining, all the pulp samples were disintegrated by means of 90 °C water to remove latency prior to further analysis (Bently et al. 1994).

Table 1. Refining Conditions

	1 st stage	2 nd stage
Refining consistency, %	20-24	10-14
CSF, mL	500-600	50-250
Refiner speed, rpm	2400	2400
Casing pressure, kPa	540	101
Temperature, °C	160	Atmospheric
Refiner plate clearance, mm	0.40 to 0.60	0.25 to 0.50
Specific refining energy, MJ/kg	3.5 to 6.5	3.5 to 9.6

Fractionation of Pulps

The experimental second-stage pulps were fractionated in a Bauer-McNett classifier to obtain 6 fractions denoted as R14, R28, R48, R100, R200, and P200 (fines). The Bauer-McNett fibre classification is a commonly used method to characterize the fibre length distribution of mechanical pulps. The fibres in different Bauer-McNett fractions are morphologically different and have different effects on paper properties. For example, the R14 fraction contains long and stiff fibres, which have poor bonding characteristics. The fines (P200) fraction is comprised of flake-like particles and fibrils, which would strengthen the fibre network. Based on these reasons, it is necessary to fractionate the pulps before characterization.

Pulp Characterization

Each fibre fraction was characterized in terms of cell wall peeling, fibre breakage, fibre wall splitting, external fibrillation, and collapsibility. These analyses were conducted by means of light microscopic analysis. The fiber samples were prepared as follows for the microscopy analysis: The pulp fibers were first stained with Toluidine Blue O (T161, Fisher Scientific Co.), and then mounted on microscope slides for observation using a Zeiss photomicroscope (Carl Zeiss Microscopy, LLC). The scanning electronic microscopy was used to study the fiber surface development and fiber cross-section deformation. The cross-section samples were prepared based on the resin-embedding method as discussed in a previous work by Huang et al. (2008).

Statistical Analysis

In an effort to obtain statistically sound results, at least 300 fibers per sample were measured. The standard error for each analysis was $\pm 5\%$.

RESULTS AND DISCUSSION

Physical Properties

The photomicrograph in Fig. 1 shows the cross-section morphology of jack pine EW and LW. As Table 2 indicates, the EW fibre has a thinner cell wall and larger lumen in comparison to the LW counterpart. The cell wall thickness of LW (4.75 μm) is more than 2 times that of EW (2.12 μm). The EW fibre has a greater outer perimeter and lumen area than LW, while the LW fibre has a larger cell wall area due to its thicker cell wall and smaller lumen. In addition, the LW fibre (3.55 mm) is longer than the EW fibre (3.34 mm). These findings are congruent with those reported earlier (Hatvani et al. 1999).

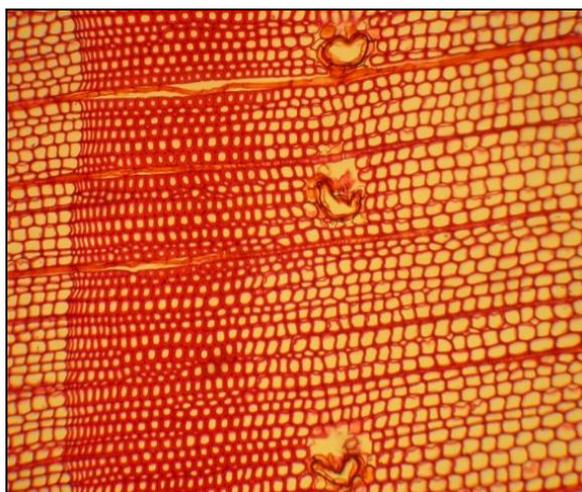


Fig. 1. Cross-section morphology of EW (left) and LW (right) of Jack pine (x200)

Table 2. Basic Properties of Jack Pine EW and LW Fibers

	Fiber length mm	Cell wall thickness μm	Outer perimeter μm	Lumen area μm^2	Cell wall area μm^2
EW	3.34	2.12	130	400	240
LW	3.55	4.75	105	260	350

Refining Energy

Figure 2 shows the pulp freeness as a function of specific refining energy. Each freeness was triplicated, and the average value was reported. Figure 2 clearly shows that refining EW required more energy than refining LW to a given freeness. The EW was defibrated into pulp fibres with relatively little fibrillation when compared to the LW. As a result, EW pulp had higher freeness for a given energy consumption. This finding is in agreement with reports by other researchers (Murton et al. 2001).

Light Microscopy Analysis

Microscopic studies help acquire useful information on fibre development during refining. The major characteristics observed for each Bauer-McNett fraction of EW and LW are presented in a series of photomicrographs, as represented in Figs. 3 to 9. For the convenience of comparison in quality, LW and EW pulps employed in this study were the pulps with a freeness of 150 mL after the second stage refining.

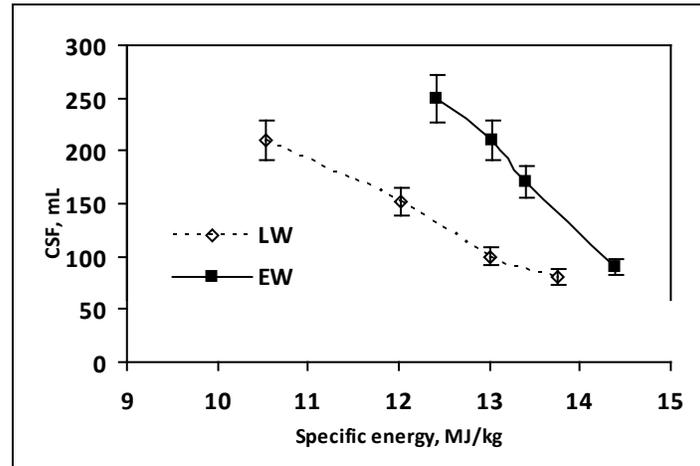


Fig. 2. Freeness as a function of specific refining energy

The nature of the R14 fibres of LW (Fig. 3) ranged from fibre bundles to individual fibres with a large variation of surfaces properties, from a smooth surface to a completely exposed S2 layer. Some fibres have most of their outer layer removed, exposing the S2 layer while the others remained undisrupted.

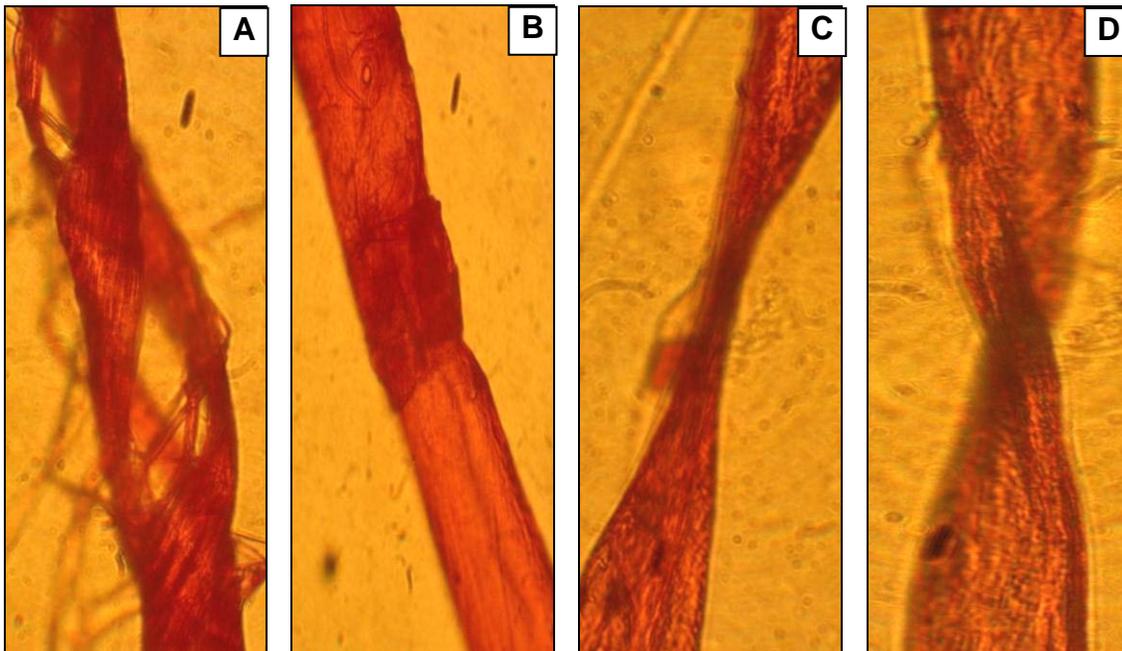


Fig. 3 (A-D). Micrographs showing the surface characteristics of LW fibres in R14 fraction (x400)
 (A): Complete fibrillation of S2;
 (B): A partly peeled fibre (the whole surface has been peeled at different degrees);
 (C): Compressed and twisted zone probably caused by the edges of refiner bars; (D): A twisted fibre;
 (E and F): Localized peeling around the fibre; (G): Fibrillation of S2;
 (H): A non-peeled fibre and completely peeled one.

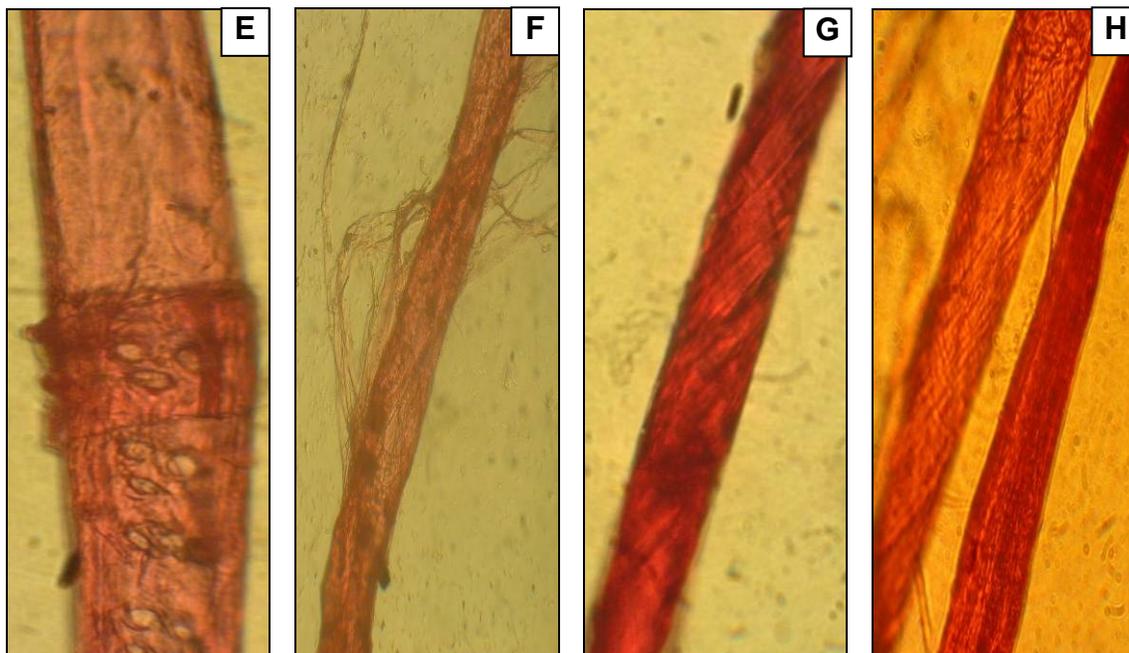


Fig. 3 (E-H). Micrographs showing the surface characteristics of LW fibres in R14 fraction (x400)
 (A): Complete fibrillation of S2;
 (B): A partly peeled fibre (the whole surface has been peeled at different degrees);
 (C): Compressed and twisted zone probably caused by the edges of refiner bars; (D): A twisted fibre;
 (E and F): Localized peeling around the fibre; (G): Fibrillation of S2;
 (H): A non-peeled fibre and completely peeled one.

Peeling of the outer layer or other types of rupture never occurred uniformly along the fibre length. For instance, a fibre could have its outer layer removed over most of its length, while the rest remain undamaged. Moreover, some fibres were compressed and twisted, even completely fibrillated (so-called “sleeve rolling” effect) by the refiner bar. As seen in the R14 fraction of EW (Fig. 4), thin-walled EW fibres were readily ruptured under the refiner forces, split, and broken down into shorter segments. In addition, the fibrillation of EW fibres was not as noticeable as in LW fibres. The diversity of surface structure of LW and EW TMP fibres render fibre characterization complicated. This also reveals that the refining action is not uniform.

The R28 fraction of LW (Fig. 5, A-D) also reveals the complex nature of fibre surface ranging from the “sleeve rolling” effect to complete exposure of S2 with intact or ruptured cell wall. Often, two or more of these characteristics occurred over the fibre length. As for the R28 fraction of EW (Fig. 5, E-H), splitting of fibre wall and fibre cutting were the most evident features, although some fibrillations had also taken place on the fibre surface.

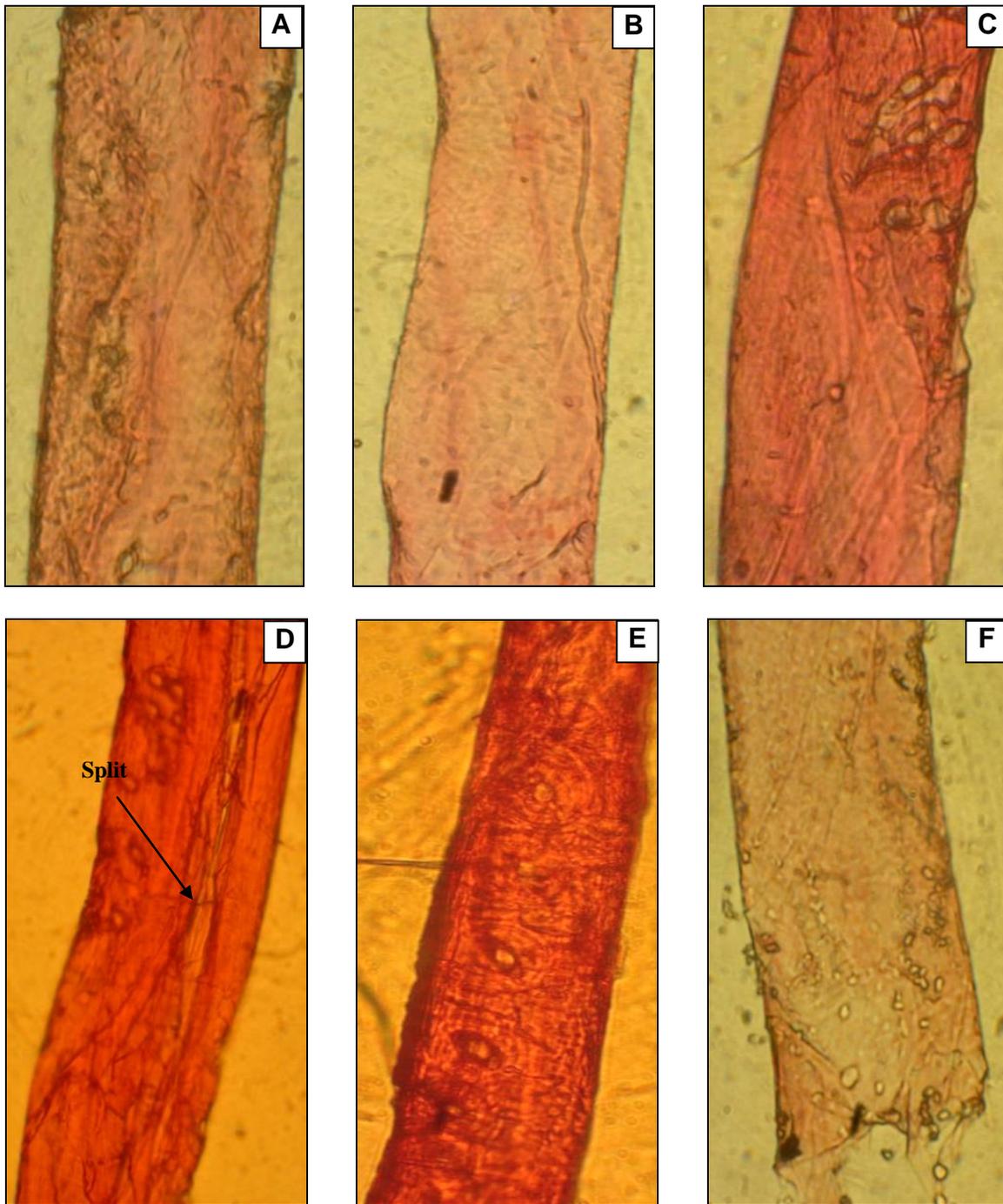


Fig. 4. Micrographs showing the surface characteristics of EW fibres in R14 fraction (x400)

(A-C): unfibrillated fibres, showing smooth surface;

(D): A split fibre;

(E): A fibre with some fibrils on the surface;

(F): Cut fibre end with little fibrillation.

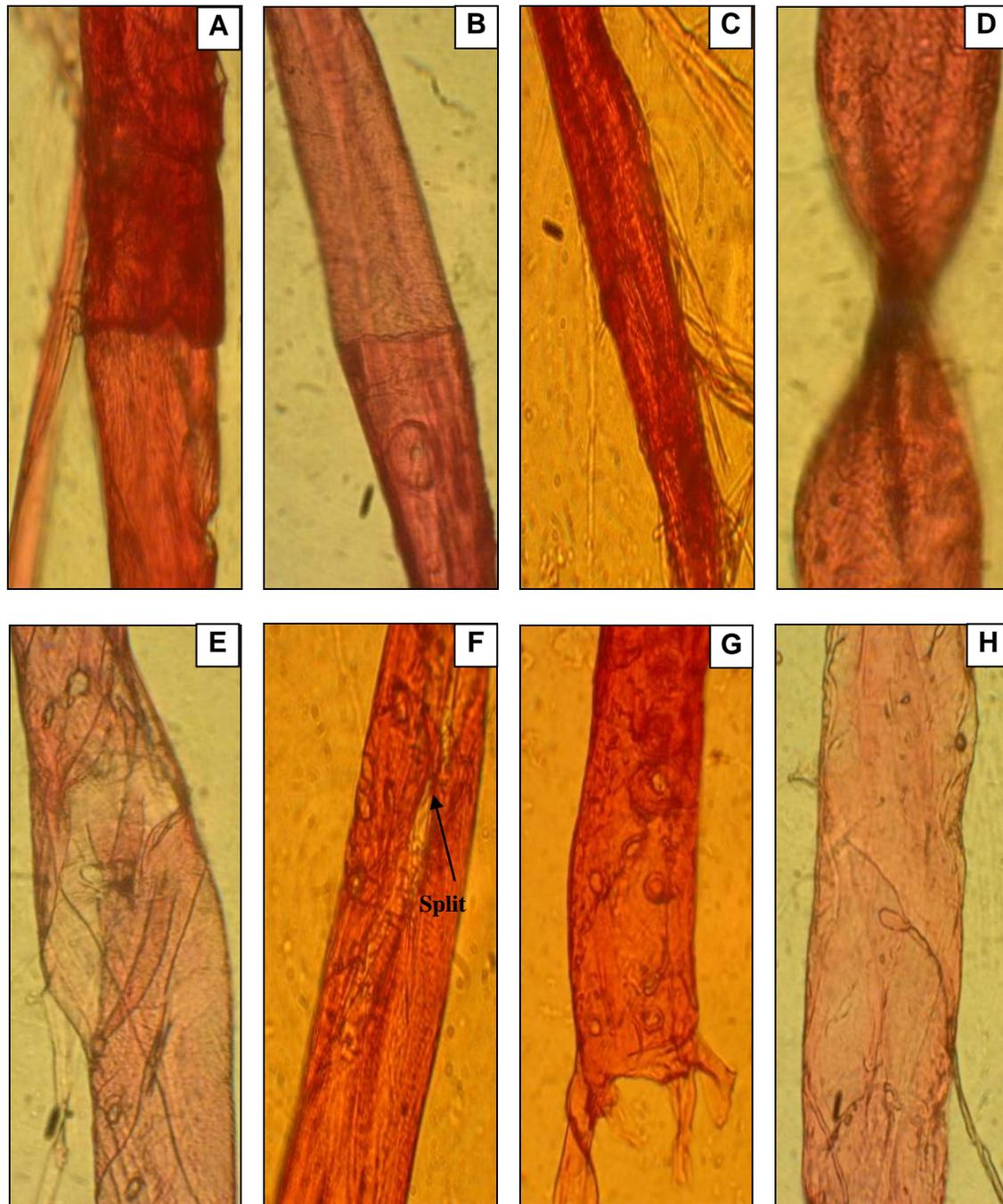


Fig. 5. Micrographs demonstrating the refining effect on R28 fraction of LW and EW fibres (x400)
(A): "Sleeve rolling effect" and fibrillation of S2 on LW fibre;
(B, C) Partly peeled and fibrillated LW fibres;
(D): A twist LW fibre;
(E): Wall fracture in EW fibre;
(F): A split EW fibre;
(G): An EW fibre end with a few fibrils on the surface.

The LW R48 fraction (Fig. 6, A, C, D, E) consisted of well fibrillated and developed fibres. In contrast, the fibre elements of R48 fraction of EW were twisted, split and fractured, as shown in Fig. 6 (B, E, F, and G). Fibrillation was rather limited in these fibres.

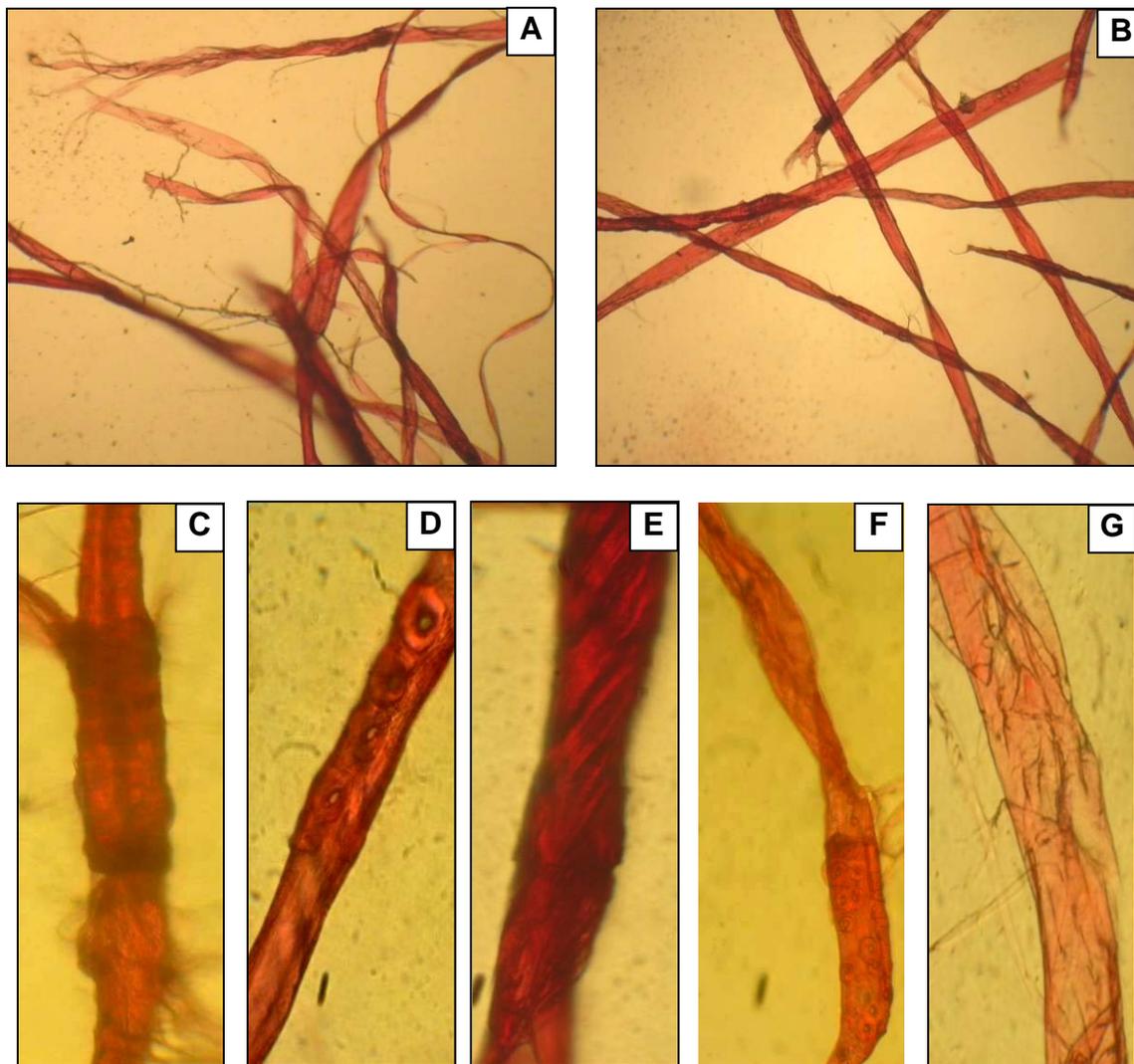


Fig. 6. Micrographs showing the surface characteristics of fibres of LW and EW in R48 fractions (A): LW R48 fibre (x40); (B): EW R48 fibres (x40); (C) “Sleeve rolling” effect on LW fibre (x160); (D) Partly peeled LW fibre (x160); (E): Fibrillated S2 layer in LW fibre, spiral cracks in fibre wall (x160); (F): A twisted and fibrillated EW fibre (x160); (G): Wall-fractured and split EW fibre (x160).

As seen in Fig. 7 (A), the fibre elements of R100 fraction of LW showed fibrillated fibre ends and large bands of fibrils. In the R100 fraction of EW (Fig. 7 (B)), there were many undeveloped and broken fibre segments. Almost all the fibrous elements were unfibrillated.

In order to better understand the morphological changes of EW and LW in refining, a quantitative analysis was carried out for the microscopic images. This analysis was based on two fiber combinations: one was fiber fractions (R14+R28+R48+R100), and another one was fiber fractions (R200+P200), as summarized in Tables 3 and 4. The calculations involved the ratio of number of featured fibers to the number of total measured fibers. The analysis indicated the LW produced more fibrillation, peeling, twisting, but less split and cutting than the EW in the relatively long fiber fractions (R14+R28+R48+R100). In the short fiber fractions ((R200+P200), EW yielded more pits but less fibrils and our-layer fragments from the fiber delamination than the LW.

Table 3. Quantitative Analysis of Fiber Combination (R14+R28+R48+R100)

	EW	LW
"Sleeve rolling" effect and fiber fibrillation	~10	~60
Partly peeled fiber	~13	~65
Twisted fiber	~22	~50
Unfibrillated fiber showing smooth surface	~60	~20
Split fiber	~40	~15
End-cut fiber	~45	~12

Table 4. Quantitative Analysis of Fiber Combination (R100+P200)

	EW	LW
Chunky developed fiber with bands of fibrils	~10	~70
Under-developed fiber with little fibrils	~75	~13
Out-layer from fiber delamination	~20	~60
Entanglement of fibril	~10	~80
Pit border	~45	~20

The characteristics of fibres of the R200 fraction were very complex in terms of the size and shape of the elements. The fibres in the R200 fractions of LW (Fig. 8 (A)) ranged from very fine bundles of fibrils to large fragments of fibre wall, while the elements of the R200 fraction of EW (Fig. 8 (B)) were rather chunky, under-developed, and broken.

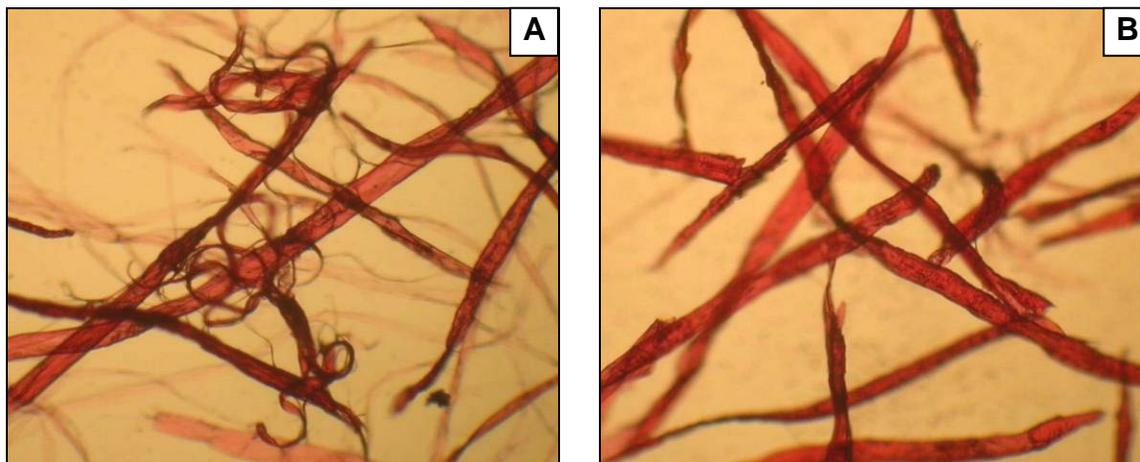


Fig. 7. Micrographs showing the nature of the fibres of R100 fraction of LW and EW: (A): LW R100 fraction showing fibrillated fibre ends and large bands of fibrils (x40); (B): EW R100 fraction showing broken fibre with little fibrils (x40).

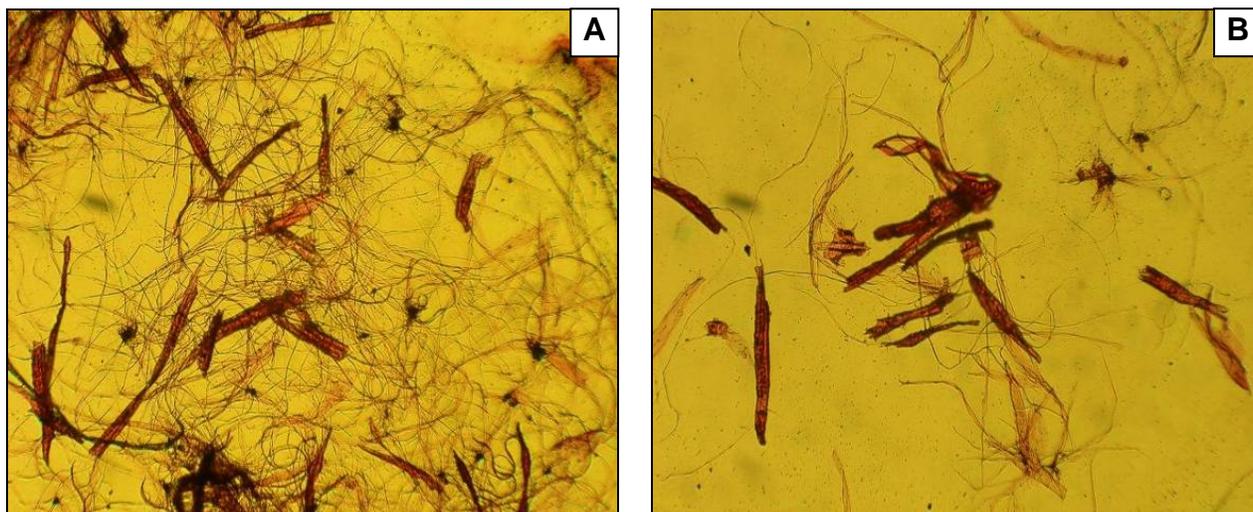
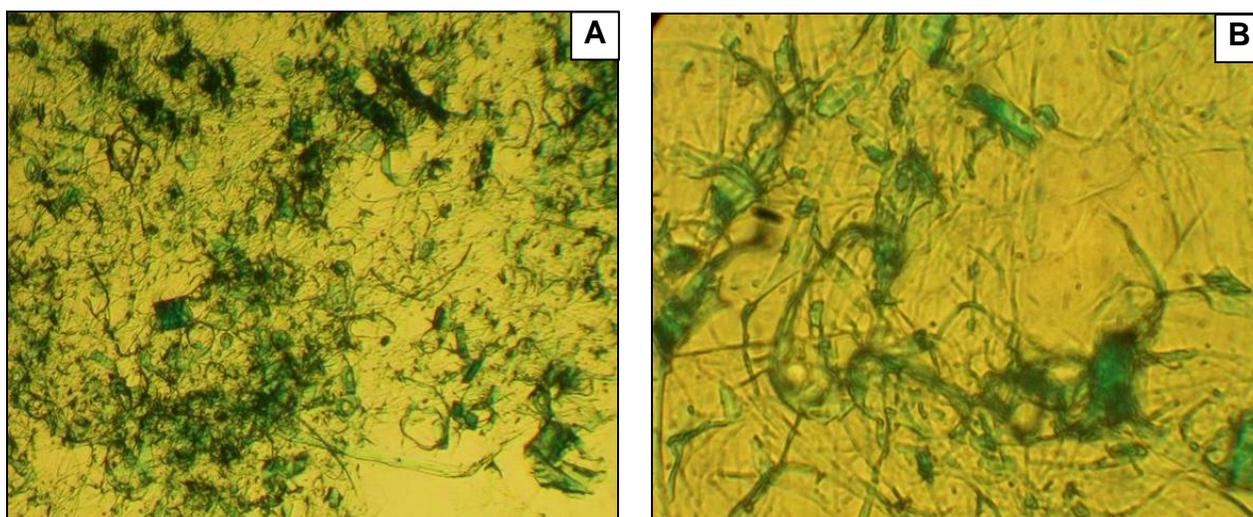


Fig. 8. Micrographs showing the nature fibrous elements of LW and EW fibres in R200 fractions (A): LW R200 fraction showing chunky developed fibre with bands of fibrils (x40); (B): EW R200 fraction showing under-developed fibres and little fibrils (x40).

As shown in Fig. 9, the P200 (fines) fraction was also complex in composition, but with a different nature when compared with the R200. This fine fraction contained bundles of fibrils: flake-like fragments of various size and shape generated from the outer cell wall layer, and ray parenchyma cells. In the LW fines (Fig. 9, A, B), the presence of annular fragments or broken bands of the outer layer indicated the delamination and separation of the outer layer around the secondary wall. Entanglement of fibrils during refining was also observed in this fraction. In addition, some pit borders are occasionally seen in LW fines. In EW fines (Fig. 9, C, D), besides the annular fragments or broken bands of the outer layer, there were many ring-shaped detached pit borders. In comparison with LW fines, EW fines showed little entanglement of fibrils.



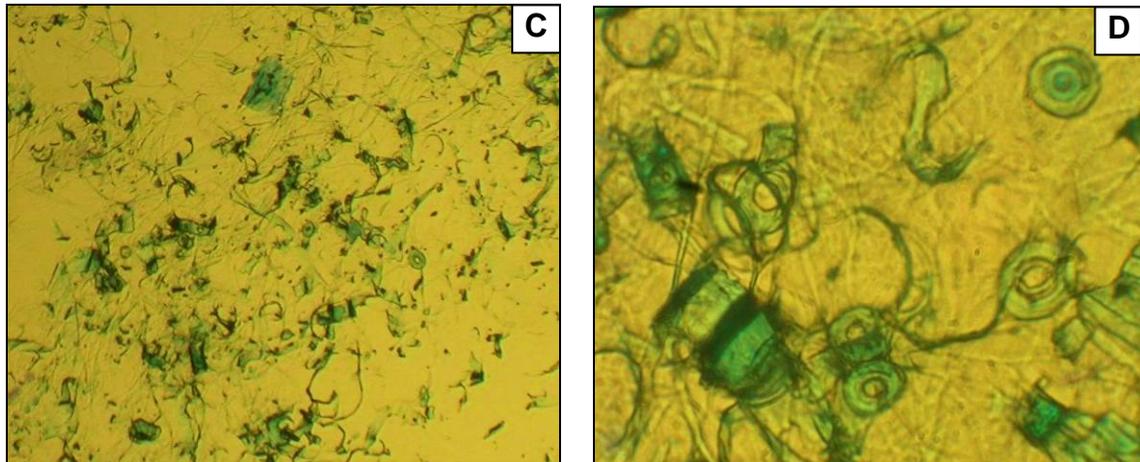


Fig. 9. Micrographs showing the particles of P200 fraction (fines)

[A: LW fines (x40), B: LW fines (x400)]: LW fines show the presence of annular or straitened fragments of outer layer derived from delamination and separation around the secondary wall. The entanglement of fibrils is very noticeable. Some pit borders are occasionally seen in the LW fines;

[C: EW fines (x40), D: EW fines (x400)]: EW fines show some annular fragments of pit borders and their outer rings. There are little fibrils in EW fines.

SEM Analysis

Fiber surface development

After refining, the EW fibres are completely collapsed, diminishing their lumens' volume, as shown in Fig. 10. In fact, the compression and shear forces render EW fibres split and twisted. The majority of the failures took place at the P/S1 interface, especially around the pits. Failures at the S1/S2 interface were also occasionally noticeable. Fibre external fibrillations associated with the shear force are not evident in EW fibres; a few fibrils were occasionally observed.

For LW fibres, the compression force had limited effects on the change in lumen dimension since collapsed fibres are rarely observed (Fig. 11). Most of the LW fibres showed various exposed surfaces such as the P, the S1, and the S2. However, the S1/S2 separation is commonly seen in LW fibres. Fibrils were frequently seen along the cell wall of LW fibres and some long and ribbon-like layers were detached from the cell wall.

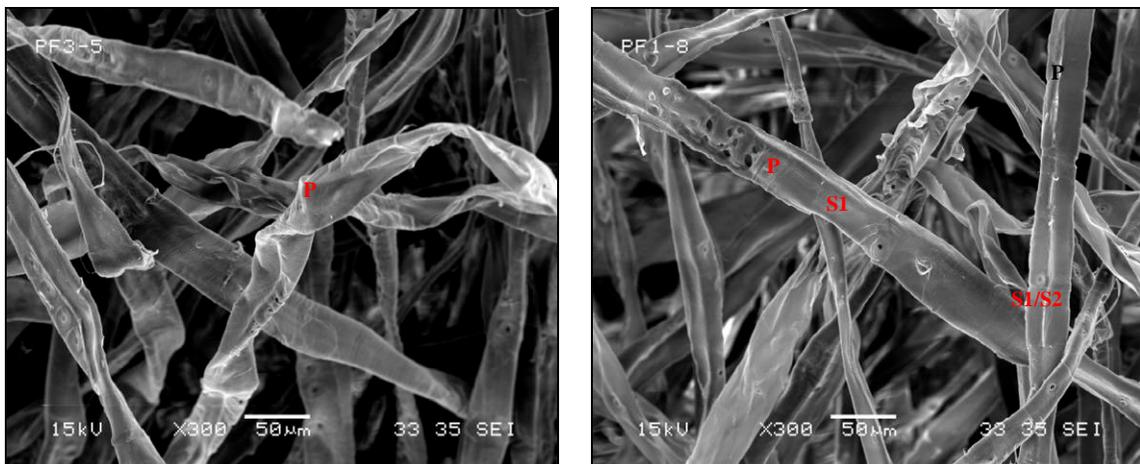


Fig. 10. Surface nature of R28 fraction EW fibers

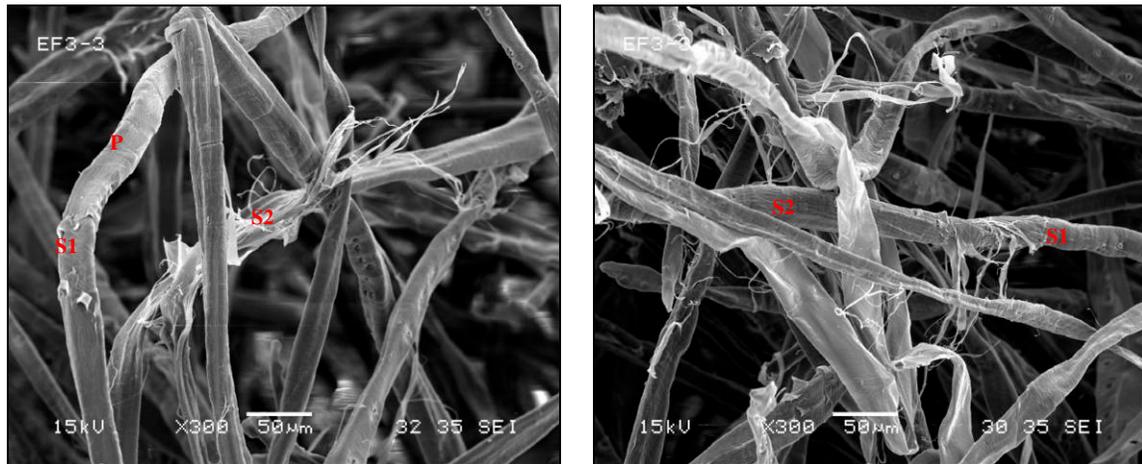


Fig. 11. Surface nature of R28 fraction LW fibers

Internal fibrillation

Figure 12 shows the internal fibrillation of an EW fibre, in which the inner layer, presumably the S3 layer of the secondary cell wall, was completely detached from the S2 layer. Also, some minor cracks are visible, indicating the presence of delamination. Interestingly, the nature of internal fibrillation is quite different for LW fibres, where the S3 layer remained attached to the S2 layer. However, the bulk of S2 layer was fractured to a great extent. The distribution of cracks (delamination) in the S2 layer was uneven. These findings reveal that thin-walled EW fibres are much more fragile under the mechanical forces than the rigid and thick-walled LW. Therefore, EW fibres produce more extensive delamination than LW fibres.

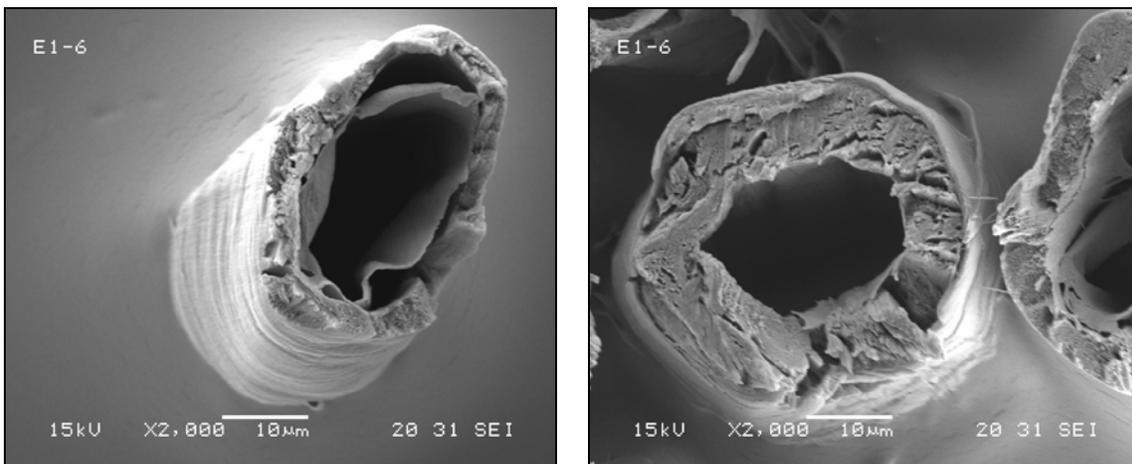


Fig.12. Internal fibrillation of EW (left) and LW (right) fibers

Cross-section deformation

Figure 13 shows the fiber cross-section deformation of EW and LW R28 fraction fibers. It can be observed that under the compression forces in refining, the thin-wall EW fibres are readily deformed and collapsed. Because thick-walled LW fibres are rigid and resistant to mechanical forces, they tend to retain their forms after refining. The SEM

observations reveal that EW fibres are usually collapsed while LW fibres are rarely noticeably deformed.

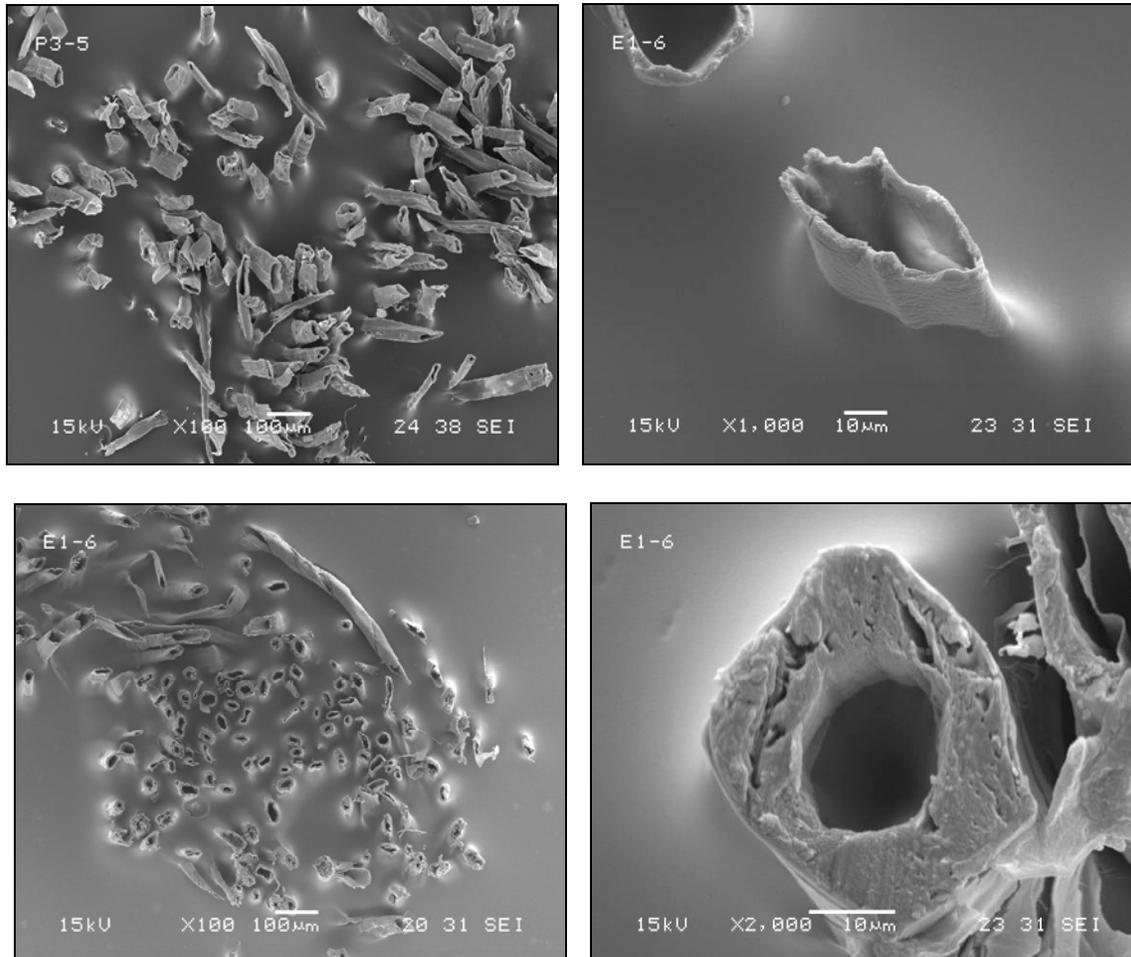


Fig. 13. Cross-section of EW (upper) and LW (lower) R28 fraction fibers

CONCLUSIONS

1. Under the mechanical forces during refining, the earlywood (EW) fibres tend to separate in the P/S1 interface, while the separations of latewood (LW) fibres take place commonly in the P/S1 and S1/S2 regions.
2. The thick-walled LW fibres exhibit much more external fibrillation than the thin-walled EW. As a result, the LW fines contain more fibrillar component than EW fines.
3. The EW fibers suffer more fiber cutting and splitting than the LW fibers.
4. The thin-walled EW fibres show higher collapsibility and conformability than the LW counterparts.

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REFERENCES CITED

- Agut, P., Gouttenoire, P., Michalowicz, G., Robert, A., Choudens, C. D., and Lombardo, G. (1987). "Deconstruction of wood chips in a roller press – morphological aspects and application to kraft and bisulfite cooks," Poster Presentations, Vol. 2, Proc. 4th Intl. Symp. Wood & Pulping Chem. Paris, France, 149-155.
- Bently, R. G., Scudamore, P., and Jack, J. S. (1994). "A comparison between fibre length measurement methods," *Pulp & Paper Canada* 95(4), 41-45.
- Funada, R., Kubo, T., Tabuchi, M., Sugiyama, T., and Fushitani, M. (2001). "Seasonal variations in endogenous indole-3-acetic acid and abscisic acid in the cambial region of pinus densiflora sieb. et zucc., stems in relation to EW-latewood transition and cessation of tracheid production," *Holzforschung* 55(2), 128-134.
- Hartler, N., and Nyren, J. (1969). "Influence of pulp type and post-treatments on the compressive force required for collapse," *TAPPI STAP* No. 8, 265-270, discussion 271-277.
- Hartler, N., and Nyren, J. (1970). "Transverse compressibility of pulp fibers II, influence of cooking method, yield, beating, and drying," *Tappi J.* 53(5), 820-823.
- Hattula, T., and Niemi, H. (1988). "Sulfate pulp fiber flexibility and its effect on sheet strength," *Paperi ja Puu* 70(4), 356-361.
- Hatvani, T. G., Evans, R., Kibblewhite, R. P., and Parker, I. H. (1999), "Relationships between tracheid and kraft pulp fibre transverse dimensions," *Proc. 53rd Appita Ann. Gen. Conf.*, Rotura, New Zealand, 87-94.
- Huang, F., Lanouette, R., Law, K., and Li, K. (2008). "Microscopic analysis of early- and latewood in thermomechanical pulp refining," *Appita J.* 61(6), 445-449.
- Lanouette, R., Law, K., and Huang, F. (2010). "Impact of early- and latewood on thermomechanical pulping," *Appita J.* 63(2), 119-125.
- Larson, A. (1960). "Physiological consideration of springwood summerwood transition in red pine," *Forest Sci.* 6, 110-122.
- Little, C. H. A., and Eidt D. C. (1968). "Effects of abscisic acid on bud-break and transpiration in woody species," *Nature* 220, 498-499.
- Luner, P. (1986). "Wet fiber flexibility as an index of pulp and paper properties, new technologies in refining," Vol. 1, Session 1, Paper 3, *Proc. PIRA Intl. Conf.* Birmingham, England.
- Mohlin, U. B. (1975). "Cellulose fiber bonding. (5) Conformability of pulp fibers," *Svensk Papperstid* 78(11), 412-416.
- Murton, K. D., Richardson, J. D., Corson, S. R., and Duffy, G. G. (2001). "TMP refining of radiata pine earlywood and laterwood fibres," *Proc. Intl. Mech. Pulp. Conf.* Helsinki, Finland, 361-371.
- National Institute of Health, USA, <http://rsb.info.nih.gov/ij/> (consulted on Sept 22, 2011).

- Panshin, A. J., and Zeeuw, C. D. (1970). *Textbook of Wood Technology*, 3rd Edition, Vol.1, McGraw-Hill, New York, USA, 48-51, 91, 151-177.
- Reme, P. A., and Helle, T. (2001). "Quantitative assessment of mechanical fibre dimensions during defibration and fibre development," *J. Pulp Paper Sci.* 27(1), 1-7.
- Smith, W. E., and Byrd, V. L. (1972). "Fiber bonding and tensile stress-strain properties of earlywood and latewood handsheets," U.S. Forest Serv., Res. Paper FPL 193.
- Uhmeier, A., and Salmén, L. (1996). "Repeated large radial compression of heated spruce," *Nordic Pulp Paper Res. J.* 11(3), 171-176.

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