

# CHARACTERIZATION OF THE REINFORCEMENT POTENTIAL OF DIFFERENT SOFTWOOD KRAFT FIBERS IN SOFTWOOD/HARDWOOD PULP MIXTURES

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## ABSTRACT

Two morphologically different market kraft pulps, a New Zealand radiata pine medium grade and a benchmark northern hemisphere spruce pulp from interior British Columbia, were evaluated and compared for their reinforcement potential following supplementation at different levels to a Eucalyptus market kraft pulp. A full factorial experimental design including softwood pulps types, three levels of both wet-pressing and refining, and six different softwood proportions in softwood/eucalypt furnish mixtures (0, 5, 10, 15, 20, and 100%), was employed to generate two independent regression models using SAS General Linear Model procedure, from data obtained from handsheet evaluation. It was conclusively shown, with statistical significance, that the reinforcement potential of softwood market kraft pulps in softwood/hardwood mixtures can readily be characterized and predicted through the measurement of fracture energy index of handsheets made from 100% softwood fiber.

*Keywords:* Fiber morphology, fiber dimensions, fiber flexibility, microfibril angle, refining, wet-pressing, pulp blends, reinforcement strength, handsheet properties, fracture energy.

## INTRODUCTION

Globally, there is a trend towards continually increasing paper machine speeds and a drive for reduced resource consumption (paper of lower

grammage). Consequently, a number of paper grades, including newsprint and higher-value mechanical papers, such as supercalendered grade A (SCA) and light weight coated (LWC), require the addition of softwood kraft fiber to act as a reinforcement pulp. Supplementation with softwood kraft fiber alleviates problems related to web breaks, especially at lower basis weights,

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and maintains sufficient tensile strength in the sheet for machine and pressroom runnability (Fairbank and Detrick 2000). Additionally, in coated papers the introduction of reinforcing fibers ensures that the sheets withstand the coating process and supercalendering, without detrimental and costly sheet deformations.

Inherent fiber characteristics including wood species, origin, age, and chemical composition have been shown to significantly influence the final properties of paper (Lumiainen and Oy 1997). Similarly, the relationship between reinforcement fiber characteristics and the resulting paper properties has been studied (Seth and Page 1988; Seth 1996; Kärenlampi and Yu 1997; Kärenlampi 1998; Wanigaratne et al. 2002). However, only the importance of fiber length is well understood, while the significance of the other fiber attributes remains unclear in the role of reinforcing fibers. It is recognized that interactions between fiber properties are difficult to avoid when studying natural fibers, particularly the relationship between fiber length and coarseness, which are commonly used as reference attributes when comparing pulp fibers for their influence on final paper properties.

Furthermore, the role of mechanical treatment (refining) of fibers is an important step in the development of pulp furnishes for papermaking (Baker 1995). The extent to which pulps are refined, as well as the type and sequence of refining (independent or simultaneous refining of mixed fiber blends), significantly influences the resultant fiber properties, and consequently the end products (Kibblewhite 1993; Hiltunen et al. 2000; Mansfield and Kibblewhite 2000). The refining process itself has a critical effect on the formation and runnability of the paper machine. In general, the refining process produces a number of modifications to fiber morphology, such as fiber breakage or cutting, external fibrillation, secondary wall delamination, and altered collapsibility and flexibility; however, the extent of each is different depending on the starting furnish. For example, the refining process effectively causes fibers consisting of thin cell walls to readily collapse during pressing and drying, and therefore are consequently highly suitable

for fine paper production. In contrast, thick-walled fibers (relative to their perimeter) are more difficult to develop, and consequently retain their intrinsic strength and are more appropriate for some packaging grade materials and specialty products, such as fiber cement boards.

In addition to refining, wet-pressing has been shown to have a profound effect on the papermaking process. While refining modifies the structure of the fibers prior to formation, wet-pressing removes water and improves wet-sheet consolidation prior to drying (Waterhouse 1993). Consequently, many of the strength properties of the paper sheet are enhanced. It has been suggested that some grades of paper may be made with little or no refining if the water removal and consolidation process could be improved (Bither and Waterhouse 1992).

Canadian and Scandinavian softwood resources enjoy the perceived advantages of more than one tree species to provide variability in softwood pulp characteristics, and hence end-use applicability. In New Zealand, on the other hand, radiata pine represents the only softwood resource available for pulp and papermaking. This radiata pine is, however, fast grown with a natural variation in wood characteristics that have been recognized to ensure market kraft pulp uniformity and to optimize end-use applicability. Considerable research and commercial attention have thus been directed in New Zealand at the characterization of the radiata pine wood and fiber resource (Kibblewhite 1993; Kibblewhite and Bawden 1993; Mansfield and Kibblewhite 2000).

New Zealand radiata pine is grown in plantations over a wide range of geographical sites and locations, and is subjected to a range of silvicultural regimes and management practices. As such, the inherent within-tree variability results in considerable differences in the characteristics of the wood, the wood fibers, and the pulps produced, depending on the origin of wood and the position from within the tree where the pulp fiber is generated. This variability within the radiata pine tree and resource has been recognized in New Zealand, and several classes of market pulp have been developed, namely High,

Medium, Low, and Ultralow (Kibblewhite 1987, 1993; Wahjudi et al 1998; Mansfield and Kibblewhite 2000). These classes of pulp cover a range of fiber qualities and have potential usage in a wide range of product types. The objective of pulp grade differentiation is to maximize the potential of radiata pine fibers for specific end uses through the development of pulp uniformity and the selection of specific fiber qualities. Market kraft pulp manufacturers in New Zealand are able to control pulp uniformity and fiber quality through the segregation and monitoring of chips in the wood yard before they enter pulp digesters (Williams 1994).

Radiata pine market kraft pulps can have fibers of similar length, high coarseness, and low numbers per gram compared to those from eastern Canada and the interior region of British Columbia (Kibblewhite 1987, 1993; Mansfield and Kibblewhite 2000). Furthermore, the radiata pine pulps require more refining to a given tensile strength, can have similar or very different tear-tensile strength relationships depending on fiber length, and lower optical properties. However, these differences rapidly decrease with reducing proportions of softwood fiber included in eucalypt/softwood and mixed hardwood/softwood pulp blends (Kibblewhite 1993; Brindley and Kibblewhite 1996; Mansfield and Kibblewhite 2000). For example, for 80:20 eucalypt/softwood blends, refining requirements, tear/tensile strength relationships, and optical properties are similar when the softwood component consists of either the Canadian, or the Medium or Low radiata pine pulp.

The aim of the present research initiative was to characterize the reinforcement potential of softwood market kraft pulps with significantly different fiber properties. A full factorial experimental design including softwood pulp types, three levels of both wet-pressing and refining, and six different softwood proportions in softwood/eucalypt furnish mixtures (0, 5, 10, 15, 20, and 100%) was employed to elucidate the reinforcing potential of different softwood fibers using fracture energy index as well as conventional handsheet tests. In addition, detailed assessments of a range of fiber and chemical properties were made on each of the

five softwood Market pulps (the Canadian, and radiata pine High, Medium, Low, and Ultralow categories).

## EXPERIMENTAL

### *Market pulp samples*

Four radiata pine bleached market kraft pulps (High, Medium, Low, and Ultralow categories), differing primarily by fiber length and secondly by fiber coarseness, were supplied by the Carter Holt Harvey, Tasman Mill, Kawerau, New Zealand. This 1994 set of radiata pine standard pulps (Wahjudi et al. 1998) exhibits slightly different fiber dimensions from those originally characterized in 1988, and commonly referred to as New Zealand High, Medium, Low, or Ultralow market kraft pulps (Kibblewhite 1987, 1993). This discrepancy is explained by a difference in tree-age at time of felling.

The fully bleached kraft softwood pulp from the interior region of British Columbia was supplied by the McKenzie mill of Fletcher Challenge, Canada. Pulp species composition was determined as 88:12 spruce:lodgepole pine. The McKenzie pulp is used as the benchmark for radiata pine kraft since it is recognized by papermakers to be a leading softwood market pulp.

Fully bleached eucalypt kraft pulp obtained from Brazil was reference material 8496 supplied by Aracruz Cellulose, South America and distributed by National Institute of Standards and Technology, Standard Reference Materials Program, Building 202, Room 205, Gaithersburg, Maryland 20899.

### *Pulp processing and evaluation*

The market kraft lap pulps were re-slushed by tearing lap pulp samples into thin pieces of less than 10 cm<sup>2</sup> and soaking them overnight in distilled water. In accordance with standard procedures, cut edges of whole-lap samples were excluded from the material used. The soaked pieces were disintegrated at 1.2% consistency, for 75,000 revolutions using a standard British pulp disintegrator. The softwood-derived re-slushed pulps were refined, at 10% stock consis-

tency for 500, 2000, and 4000 revolutions, in a PFI mill in accordance with standard procedures, employing an applied refining load of 3.4 N/mm, while the hardwood kraft pulp was refined at 10% consistency for 1000 revolutions only at a refining load of 1.8 N/mm. The independently refined pulps were then mixed on a weight-to-weight basis to produce blends of eucalypt:softwood kraft pulps ranging from 100:0, 95:5, 90:10, 85:15, 80:20, and 0:100.

Nominal 40 gsm handsheet sets were prepared from the different pulp blends, and subsequently individually wet-pressed at 50, 75, and 100 psi. Pulp freeness measurements were made on the softwood:hardwood blends prior to sheetmaking. Physical handsheet evaluations were made in accordance with standard procedures. Physical evaluation data are reported on oven-dried basis.

#### *Fracture energy index methods*

Fracture energy index was determined following a modified version of Tryding and Gustafsson (2000). In short, fracture energy was calculated by employing the stiffness values determined on an Instron 5500 model Universal Testing Machine, equipped with a 10-kN load cell and specifically designed clamps to hold a 15-mm  $\times$  10-mm test strip at a constant rate of elongation (0.5-mm/min).

#### *Fiber properties*

*Fiber length.*—Weighted average fiber length was determined using a Kajaani FS-200 instrument following TAPPI standard method T271 pm-91.

*Microfibril angle.*—The microfibril angle of the kraft pulp fibers was determined as previously described (Donaldson 1991). Briefly, fiber suspensions were mounted in glycerol on glass slides, and the microfibril angles were obtained using polarized light by determining the maximum extinction position using a first-order red retardation plate. In order to ensure that measurements were on single cell-wall thickness

samples, observations were made through either bordered pit or cross-field pit apertures. A minimum of 400 measurements were obtained for each kraft fiber sample.

*Curl.*—Pulp (~50 mg) was diluted to 1% consistency with distilled water and the fibers were dyed by the addition of 20 mL of crystal violet solution (25 g/L). After gentle stirring, the suspension was allowed to stand for 5 h and then diluted to ~10 liters total volume. The suspension was very gently agitated and a 150-mL sample was filtered slowly through a 15-cm filter paper on a Buchner funnel. Clean slides (24) were placed on the filter paper and then sandwiched between wet and dry blotters. The sample was then pressed in a standard handsheet press to 50 psi pressure for 30 s. The slides were removed from the filter paper and allowed to dry for 30 min before permanent mounting using Eukitt resin and a large cover slip. After calibrating the image-processing computer, each of the 24 slides was analyzed until 600 fiber measurements had been taken. The contoured (c) and projected (p) lengths of each fiber were recorded, and the curl index was calculated using the following formula  $[(c/p)-1]*100$ .

*Fiber dimensions.*—Cross-section fiber dimensions of thickness, width, wall area, and wall thickness were measured using image-processing procedures as described previously (Kibblewhite and Bailey 1988). All fiber dimension measurements were made on unrefined fibers rewetted from handsheets. Two hundred fibers were measured for each pulp sample.

*Fiber flexibility.*—Effective fiber flexibility of the papermaking fibers was determined according to the method of Steadman and Luner (1985). Briefly, representative pulp fiber samples (2 o.d. grams) were disintegrated for 50,000 revolutions at 0.2% consistency. The disintegrated pulp suspensions were then diluted to 0.03% consistency (stock dilution). Approximately 20–50 mL of the stock dilution was then added to the sheet-former, mixed, and drained onto Whatman #4 (coarse, fast-draining) filter paper, depositing a light network of fibers onto the filter paper surface. Glass slides (5  $\times$  5 cm), which had been thoroughly

washed and wrapped with a 25- $\mu\text{m}$  diameter stainless steel wire producing parallel lines approximately 3–4 mm apart, were placed on top of the filter paper with their wires sides orientated downwards (one face of the slides had the wrapped wire removed). The slides were then pressed in a standard handsheet press for 2.5 min at 50 psi. After pressing, the fiber-coated slides were allowed to dry in a desiccator.

A minimum of 300 fibers were analyzed for each treatment by image analysis, under reflected illumination at 100 $\times$  on an Axioskop Light Microscope (Zeiss, Germany). Fibers that cross the wire perpendicularly ( $90^\circ \pm 15^\circ$ ) were selected for analysis. The span length (defined as the distance between the wires and contact point with slide) and width of the corresponding fibers were determined by image analysis using a JAVA package (Jandel Scientific).

*Pulp chemical composition.*—The lignin and carbohydrate composition of the softwood pulps was determined using standard Klason lignin analysis (TAPPI Method T249 cm-85). Each hydrolysate was filtered using a sintered-glass filter of medium coarseness for the gravimetric determination of Klason lignin (acid-insoluble lignin), and its absorbance at 205 nm was measured for the quantification of acid-soluble lignin (TAPPI Useful Method UM250 1991). The monosaccharide constituents were quantified by anion-exchange chromatography on a CarboPac PA-1 column using a Dionex HPLC system (Dionex, Sunnyvale, Calif.).

### Statistical analysis

Two independent regression models were calculated using SAS General Linear Model procedure, employing data obtained from the handsheet tests. The classification effects in the models were softwood market pulp type, softwood PFI mill refining revolutions, and wet-pressing level. In the analysis, softwood kraft refining revolutions and wet-pressing level were treated as qualitative classification variables, not quantitative variables. Percentage softwood was treated as a quantitative covariate.

Model specifications employed:

#### Model specification 1

$$\text{Property} = \text{wet-press} \times \text{psw} \quad (1)$$

$$\text{psw} \times \text{wet-press} \text{ psw} \times \text{pfirevs} \quad (2)$$

$$\text{psw} \times \text{swpulp} \quad (3)$$

$$\text{psw} \times \text{pfirevs} \times \text{swpulp} \quad (3)$$

#### Model specification 2

$$\text{Property} = \text{wet-press} \text{ psw} \times \text{swpulp} \times \text{pfirevs (no intercept)} \quad (4)$$

where, wet-press represents handsheet wet-pressing level (50, 75, or 100 psi), psw represents percentage softwood (0, 5, 10, 15, or 20%), pfirevs represents softwood kraft PFI mill refining revolutions (500, 2000, or 4000 revs), and swpulp represents softwood pulp type (Canadian or New Zealand Medium)

The solution model for both specifications is identical. The advantage with model specification 2 is that the parameter estimates (Table 2) can be readily interpreted as the different intercepts for each wet-pressing level and the different slopes for each combination of softwood market pulp and softwood refining energy level. Additionally, the statistical significance effects (Table 1) can be used to test differences between wet-pressing levels, and slopes for each market pulp, slopes for each softwood refining energy level, and slopes for combinations of softwood market pulp and softwood refining energy level. The statistical significance of the  $\text{psw} \times \text{swpulp} \times \text{pfirevs}$  interaction term is used to test if the effects of softwood refining level on slope are different for the two softwood market pulps.

The only fixed effect in the model that is not continuous-by-class is wet-pressing. This constrains the model to having an intercept that depends only on the wet-pressing level. Therefore, at a given wet-pressing level and 0% softwood (100% hardwood), the model predicted value of a handsheet property is the same for all softwood refining levels and both market pulps. The other model terms (percent softwood and percent softwood interactions) give a model solution with a different predicted slope for each softwood mar-



ket pulp and softwood refining energy combination.

## RESULTS AND DISCUSSION

### *Market kraft fiber and chemical properties*

The fiber and chemical properties of the bleached market kraft pulps used in the softwood/hardwood blends are listed in Table 1, together with those for corresponding High, Low, and Ultralow radiata pine pulps generated routinely in New Zealand. Compared to the radiata pine Medium category pulp, fibers in the Canadian kraft pulp are significantly longer, thin-walled, of lower perimeter (Fig. 1) and coarseness (as indicated by wall area), more resistant to collapse, and demonstrate a greater numbers per unit mass due to their inherent morphological attributes. Furthermore, the Canadian pulp is composed of fibers of lower microfibril angle, and higher curl index, and they are rich in

hemicelluloses compared to the radiata pine Medium fiber (Table 1).

Both fiber flexibility and fiber collapse (as indicated by the width/thickness ratio) increase through the radiata pine High, Medium, Low, to Ultralow category range (Table 1). The situation is completely different for the Canadian fibers since they show a low level of collapse, but are highly flexible (Fig. 2). While the fibers in the Canadian pulp are less collapsed than those in radiata pine High (3.04 compared to 3.12), they are almost as flexible as those in the radiata pine Ultralow pulp (5.98 compared to 6.11). These data suggest that fiber flexibility may be directly related to cell-wall thickness and/or hemicellulose content (Table 1). Perhaps this suggests different fiber conformabilities in paper webs for the two species when compared at the same level of fiber collapse.

Curl indices were relatively low for the five softwood pulps, indicating that the fibers were quite straight, and not excessively damaged dur-

TABLE 1. *Fiber and chemical properties of bleached market kraft pulps.*

|  | Bleached market kraft categories |          |              |        |        |          | LSD*  |
|--|----------------------------------|----------|--------------|--------|--------|----------|-------|
|  | Eucalypt                         | Canadian | Radiata pine |        |        |          |       |
|  |                                  |          | High         | Medium | Low    | Ultralow |       |
| <b>Fiber dimensions</b>                                      |                                  |          |              |        |        |          |       |
| Length mm  | 0.74                             | 2.49     | 2.60         | 2.24   | 2.08   | 1.88     | 0.05  |
| Wall area $\mu\text{m}^2$                                    | 58                               | 130      | 202          | 177    | 174    | 169      | 20    |
| Relative number  | 924                              | 123      | 76           | 100    | 110    | 125      | 18    |
| Perimeter $\mu\text{m}$                                      | 39.4                             | 68       | 81.8         | 78.6   | 79.4   | 78.8     | 2.7   |
| Wall thickness $\mu\text{m}$                                 | 2.27                             | 2.57     | 3.57         | 3.11   | 3.05   | 2.96     | 0.33  |
| Width/thickness  | 2.05                             | 3.04     | 3.12         | 3.48   | 3.56   | 3.63     | 0.33  |
| <b>Fiber properties</b>                                      |                                  |          |              |        |        |          |       |
| Microfibril angle ( $^\circ$ )                               | ND                               | 14.85    | 18.24        | 20.70  | 21.24  | 22.72    | 0.31  |
| Curl   | ND                               | 0.231    | 0.238        | 0.208  | 0.203  | 0.182    | 0.020 |
| Flexibility ( $\times 10^{11} \text{ N}^{-1}\text{m}^{-2}$ ) | ND                               | 5.98     | 3.02         | 3.99   | 4.87   | 6.11     | —     |
| Log Flexibility  | ND                               | 11.791   | 11.489       | 11.648 | 11.733 | 11.790   | 0.073 |
| <b>Pulp chemistry</b>  |                                  |          |              |        |        |          |       |
| Glucose %  | ND                               | 80.73    | 83.67        | 83.21  | 83.82  | 83.37    |       |
| Xylose %   | ND                               | 6.80     | 5.24         | 5.43   | 5.56   | 5.55     |       |
| Mannose %  | ND                               | 7.08     | 5.43         | 5.51   | 5.63   | 5.55     |       |
| Galactose %  | ND                               | 0.08     | —            | —      | 0.03   | 0.06     |       |
| Arabinose %  | ND                               | —        | —            | —      | —      | —        |       |

\* Least significant difference (5% level) for comparing softwood market pulps.

ND = Not determined

TABLE 2. General linear model: Probabilities of F-statistics (Type III) and fit statistics

| Source*                | Density<br>(g/cm <sup>3</sup> ) | Tear<br>index<br>(mNm <sup>2</sup> /g) | Fracture<br>energy<br>index<br>(Jm/kg) | Tensile<br>index<br>(Nm/g) | Stretch<br>(%) | T.E.A.<br>index<br>(J/kg) | Scott<br>internal<br>bond<br>(kJ/m <sup>2</sup> ) | Light<br>scatt.<br>coeff.<br>(m <sup>2</sup> /kg) |
|------------------------|---------------------------------|--|--|----------------------------|----------------|---------------------------|---|---|
| Wet-press              | <0.0001                         | 0.8777                                 | <0.0001                                | <0.0001                    | 0.0001         | <0.0001                   | <0.0001   | <0.0001   |
| psw                    | <0.0001                         | <0.0001                                | <0.0001                                | <0.0001                    | 0.0013         | <0.0001                   | <0.0001   | <0.0001   |
| psw × swpulp           | 0.0009                          | 0.0130                                 | <0.0001                                | <0.0001                    | <0.0001        | <0.0001                   | 0.0045  | 0.0644  |
| psw × pfirevs          | <0.0001                         | 0.3909                                 | 0.1435                                 | <0.0001                    | 0.0001         | <0.0001                   | 0.0010  | <0.0001   |
| psw × swpulp × pfirevs | 0.3538                          | 0.2058                                 | 0.0445                                 | 0.1854                     | 0.4064         | 0.9949                    | 0.7113  | 0.0465  |
| R <sup>2</sup>         | 0.92                            | 0.82                                   | 0.92                                   | 0.89                       | 0.64           | 0.81                      | 0.60  | 0.90  |
| Root MSE               | 0.00765                         | 0.613                                  | 0.443                                  | 1.17                       | 0.0847         | 5.35                      | 0.315   | 0.761   |
| Mean                   | 0.594                           | 8.10                                   | 7.28                                   | 46.4                       | 2.36           | 795                       | 3.23  | 38.9  |

\* where, wet-press represents handsheet wet-pressing level (50, 75, or 100 psi), psw represents percentage softwood (0, 5, 10, 15, or 20 %), pfirevs represents softwood kraft PFI mill refining revolutions (500, 2000, or 4000 revs), and swpulp represents softwood pulp type (Canadian or Medium).

ing pulping and pulp processing operations. However, there was a very strong correlation between the fiber length and the propensity for fibers to curl ( $R^2 = 0.98$ ). This observed relationship included both the radiata pine and spruce derived fibers. The fiber and chemical property trends of the four radiata pine categories are consistent with the High being rich in outerwood fibers, and the Medium-to-Low-to-Ultralow categories containing decreasing proportions of toplog fibers, and increasing proportions of thinnings rich in juvenile wood fibers (Kibblewhite 1999a, 1999b). The premium papermaking qualities of softwood kraft pulps from the Interior region of British Columbia and Eastern Canada can be related mainly to their large numbers of long slender fibers of low coarseness (Kibblewhite 1987; Kibblewhite and Shelbourne 1997; Seth 1990a; Seth 1990b), and secondly to high

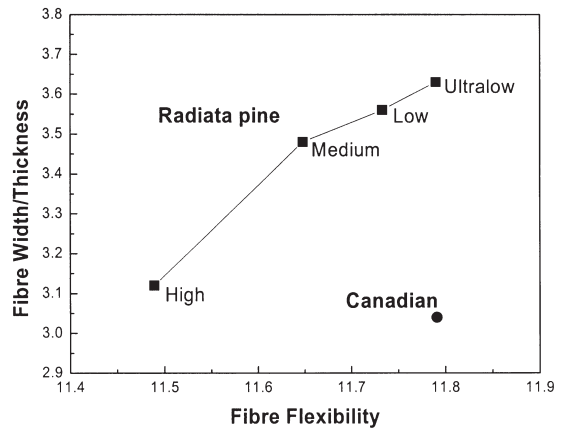
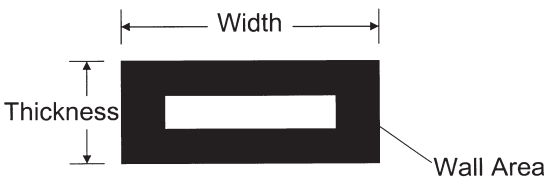


FIG. 2. Fiber collapse (width/thickness ratio) versus fiber longitudinal flexibility for the Canadian and the four radiata pine market kraft pulps. Fiber flexibility represents the log of mean fiber flexibility as measured by the Steadman and Lunar (1985) method, and is dimensionless.



- Coarseness  $\propto$  Wall Area
- Collapse = Width/Thickness
- Number  $\propto$  1/(Wall Area x Length)

FIG. 1. Cross-section dimensions for dried and rewetted fibers.

hemicellulose contents (Cottrall 1950; Thompson et al. 1953) and low microfibril angles (Watson and Dadswell 1964; Kibblewhite 1999a, 1999b).

*Softwood market kraft reinforcement properties*

It has previously been demonstrated that fibers that are longer and coarser contain fewer fibers per unit mass, and therefore affect the quality and strength of the paper produced (Kibblewhite and Bawden 1993; Kibblewhite and Shelbourne 1997). Additionally, inherent characteristics, such

as fiber stiffness (flexibility) and collapsibility have been shown to be essential fiber characteristics as they influence sheet compaction and inter-fiber bonding. While it is clear that there is a distinct difference in the quality of paper derived from Canadian and radiata pine softwood kraft fibers (Kibblewhite 1987, 1993), the reinforcement behavior of these fibers when supplemented to mechanical or hardwood kraft fibers remains unclear. The current trend in the paper industry is the production of paper that maintains the required functional properties with as small an amount of raw material as possible. As a result, the basis weight (grammage) of paper has been continually decreasing. Additionally, there is an effort to produce more fine paper containing wood pulp (mechanical fibers), which represents a higher yield product from the starting raw material. The reduction in basis weight and the use of mechanical pulp-derived fibers in fine paper products causes deterioration in nearly all paper strength properties, as well as machine runnability. In an attempt to circumvent some of these deleterious effects, softwood chemical fiber (normally kraft) is supplemented to hardwood kraft or mechanical fibers in various proportions, depending on the grade of manufacture, to act as a reinforcing furnish to help meet the required physical specifications.

Although fiber length has been suggested to be the most important fiber attribute in determin-

ing web reinforcement and runnability (Seth 1990a; Seth and Page 1988), a number of other characteristics, including coarseness, conformability, bonding capacity, fiber strength, and furnish compatibility, may also be influential in determining the quality of both reinforcement and the end-product quality (Ting et al. 2001; Yu et al. 2001). In an attempt to address this question, we evaluated standard handsheet physical and optical properties, as well as fracture energy index, of the Canadian and Medium radiata pine pulps when blended with eucalypt kraft pulp at six levels of softwood supplementation (0, 5, 10, 15, 20, and 100%), three levels of softwood refining (500, 2000, and 4000 PFI mill revolutions) and three levels of wet-pressing pressure (50, 75, and 100 psi).

Using a full factorial experimental design, together with the chosen softwood proportions (0–20%), has increased the level of understanding of the effects of softwood fiber types on softwood/eucalypt mixture properties. For example, handsheet tensile index, stretch and T.E.A. index predictive models for the Canadian/eucalypt and Medium/eucalypt mixtures are significantly different (<0.001 level) from one another (Tables 2, 3). Furthermore, the effects of “wet-pressing” (wet-press), and of the “percent softwood (psw) × softwood type (swpulp)” and “psw × PFI mill revolutions (pfirevs)” interactions, are also

TABLE 3. General linear model: Model solution parameters.

| Parameter*                      | Density<br>(g/cm <sup>3</sup> ) | Tear<br>index<br>(mNm <sup>2</sup> /g) | Fracture<br>energy<br>index<br>(Jm/kg) | Tensile<br>index<br>(Nm/g) | Stretch<br>(%) | T.E.A.<br>index<br>(J/kg) | Scott<br>internal<br>bond<br>(kJ/m <sup>2</sup> ) | Light<br>scatt.<br>coeff.<br>(m <sup>2</sup> /kg) |
|---------------------------------|---------------------------------|--|--|----------------------------|----------------|---------------------------|---|---|
| wet-press                       |                                 |  |  |                            |                |                           |   |   |
| 50 intercept                    | 0.557                           | 6.35                                   | 5.07                                   | 41.5                       | 2.24           | 665                       | 2.64  | 42.8  |
| 75 intercept                    | 0.593                           | 6.39                                   | 5.55                                   | 45.0                       | 2.35           | 770                       | 3.10  | 40.5  |
| 100 intercept                   | 0.614                           | 6.43                                   | 5.68                                   | 46.4                       | 2.38           | 804                       | 3.34  | 39.0  |
| psw × swpulp × pfirevs (slopes) |                                 |  |  |                            |                |                           |   |   |
| Canadian 500                    | 0.00049                         | 0.177                                  | 0.201                                  | 0.180                      | 0.0049         | 4.33                      | 0.0131  | -0.138  |
| Canadian 2000                   | 0.00100                         | 0.207                                  | 0.235                                  | 0.397                      | 0.0064         | 8.54                      | 0.0275  | -0.199  |
| Canadian 4000                   | 0.00109                         | 0.169                                  | 0.210                                  | 0.369                      | 0.0141         | 10.79                     | 0.0438  | -0.199  |
| Medium 500                      | -0.00014                        | 0.153                                  | 0.147                                  | -0.026                     | -0.0040        | -1.67                     | 0.0011  | -0.153  |
| Medium 2000                     | 0.00041                         | 0.155                                  | 0.147                                  | 0.141                      | 0.0004         | 2.39                      | 0.0141  | -0.189  |
| Medium 4000                     | 0.00091                         | 0.164                                  | 0.168                                  | 0.204                      | 0.0034         | 4.63                      | 0.0217  | -0.268  |

\* where, wet-press represents handsheet wet-pressing level (50, 75, or 100 psi), psw represents percentage softwood (0, 5, 10, 15, or 20 %), pfirevs represents softwood kraft PFI mill refining revolutions (500, 2000, or 4000 revs), and swpulp represents softwood pulp type (Canadian or Medium).



highly significant. This information is illustrated graphically for tensile and T.E.A. indices in Figs. 3 and 4. The greater effectiveness of the Canadian softwood, compared to Medium, in increasing the tensile properties of the furnish mixtures is clearly evident. Also, refining requirements of the Canadian softwood are shown to be low compared to the Medium. For example, with 500 rev PFI mill refining, the tensile properties of the furnish mixtures are markedly increased with the Canadian kraft pulp, but essentially unchanged with the Medium softwood component. Also, the development of tensile index with refining is close to the maximum achievable at 2000 rev for the Canadian, whereas with the Medium tensile index is increased with additional refining (in this case 4000 rev).

Handsheet properties that change with pulp refining (or increased bonding), such as apparent density, Scott bond, and light-scattering coefficient, show trends similar to those obtained for tensile index, stretch, and T.E.A. index (Ta-

bles 2, 3 and Figs. 3, 4). Trends for light-scattering coefficient are of interest since they show a significant (0.05 level) three-term interaction effect –  $psw \times swpulps \times pfirevs$  (Table 2). Light-scattering coefficient prediction models are also very different for the Canadian/eucalypt and Medium/eucalypt mixtures, since the mixture response to refining the softwood component is not the same for the two softwood types (Fig. 5).

Tear index increases with increasing proportions of softwood fibers in the softwood/eucalypt mixtures, with the Canadian fiber-type showing the greatest effect (Fig. 6, Tables 2, 3). Both effects are highly significant at  $<0.0001$  for percent softwood ( $psw$ ), and 0.013 for fiber type ( $psw \times swpulps$ ). However, the effect(s) of wet-pressing are not significant, nor are the effects of refining revolutions and fiber type. Furthermore, the levels of variation are high for tear index as indicated by a high root mean square

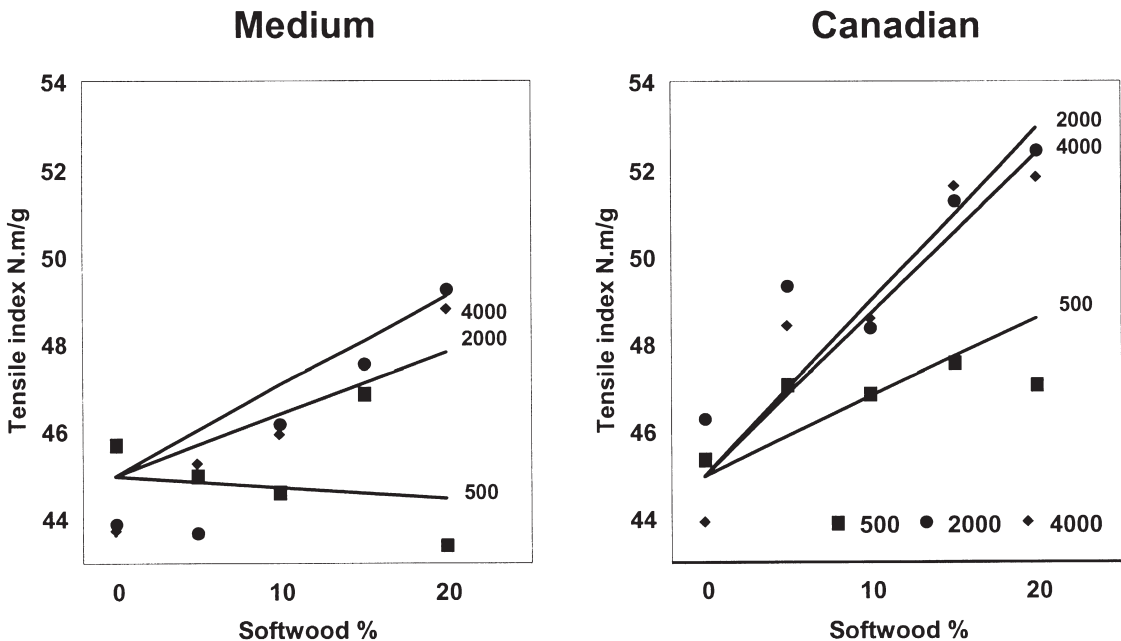


FIG. 3. Handsheet tensile index versus percent softwood showing actual values (points) at one wet-pressing level (75 psi) and predicted models (lines) based on data of three wet-pressing levels (50, 75, and 100 psi). Furnish mixtures consist of eucalypt market kraft supplemented with 0 to 20 percent radiata pine (Medium) or spruce-rich fiber from the interior region of British Columbia (Canadian). The softwood component of each blend was refined in a PFI mill at 500, 2000, and 4000 rev.

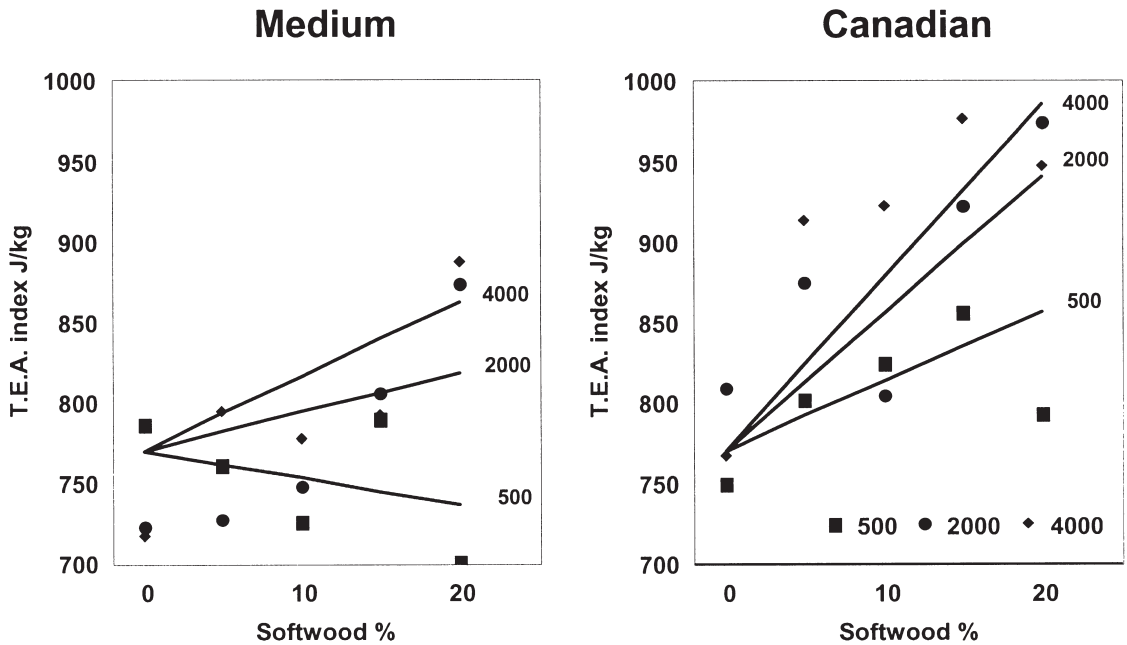


FIG. 4. Handsheet T.E.A. index versus percent softwood showing actual values (points) at one wet-pressing level (75 psi) and predicted models (lines) based on data of three wet-pressing levels (50, 75 and 100 psi). Furnish mixtures consist of eucalypt market kraft supplemented with 0 to 20 percent radiata pine (Medium) or spruce-rich fiber from the interior region of British Columbia (Canadian). The softwood component of each blend was refined in a PFI mill at 500, 2000, and 4000 rev.

error, which can be addressed by some cautionary commentary on tear index:

1. The absence of a significant wet-pressing effect is explained by a high analytical error associated with the Elmendorf tear test which is exaggerated by the low tear values of the zero and low softwood content handsheets made of low grammage (40 gsm). The variation in tear index values at 75 psi of wet-pressing, and three levels of refining, is shown in Fig. 6.
2. Separation of effects of softwood refining levels is small, as shown in Fig. 6.
3. The lower tear index obtained with the Medium fiber-type is to be expected because of its shorter fiber length (0.26 mm) as indicated in Table 1. Previous comparisons of the Canadian pulp and a radiata pine pulp of equivalent length developed similar handsheet tear indices when either in softwood/eucalypt mixtures or separately processed (Kibblewhite 1987, 1993; Mansfield and Kibblewhite 2000).

Handsheet fracture energy index increases with increasing softwood proportions (0–20%), with the greatest increase occurring with the Canadian fiber-type (Fig. 7). The fracture energy index also demonstrated statistically significant (0.0001 level) increases with increasing softwood proportions (psw) (Tables 2, 3). Additionally, the  $psw \times swpuls \times pfirevs$  interaction indicates that the blend fracture energy index response to softwood refining is different for the two softwood types (0.05 level). The high fracture energy index developed with the Canadian fiber-type at 2000 rev refining is an example of this three-way interaction effect (Fig. 7). The model solution coefficients (Table 3) demonstrate the substantially higher rate of increase in fracture energy with softwood percent for the Canadian/eucalypt mixtures than for the Medium/eucalypt mixtures. The differences between softwood refining levels in fracture energy index versus percent softwood slopes are small compared to the difference between the two softwood types (Table 3, Fig. 7).

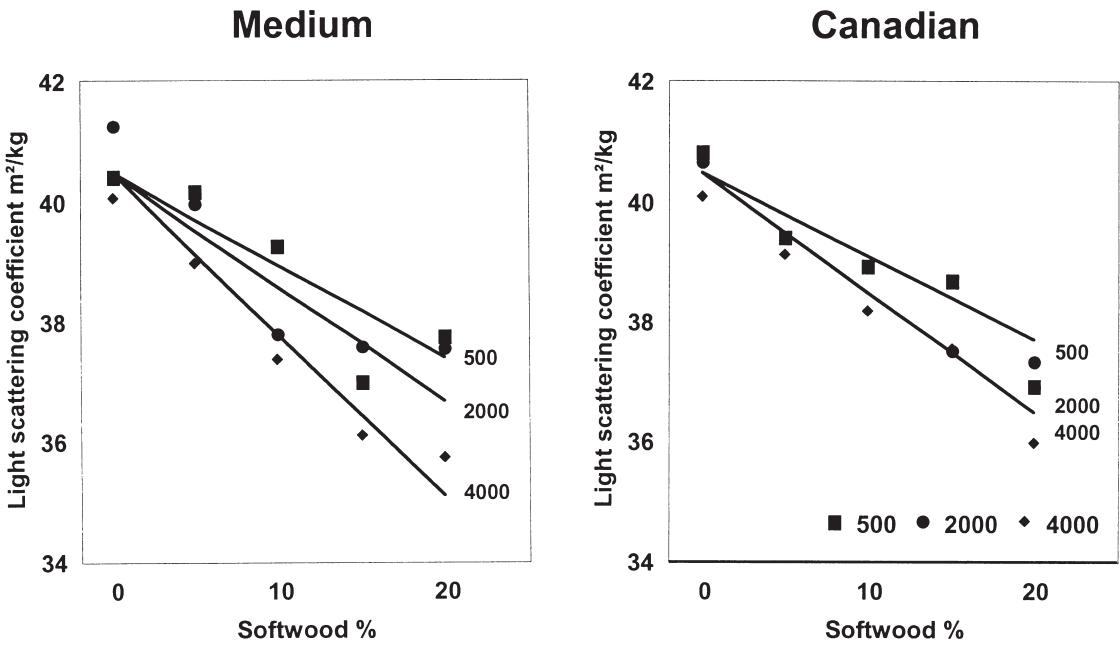


FIG. 5. Handsheet light scattering coefficient versus percent softwood showing actual values (points) at one wet-pressing level (75 psi) and predicted models (lines) based on data of three wet-pressing levels (50, 75, and 100 psi). Furnish mixtures consist of eucalypt market kraft supplemented with 0 to 20 percent radiata pine (Medium) or spruce-rich fiber from the interior region of British Columbia (Canadian). The softwood component of each blend was refined in a PFI mill at 500, 2000, and 4000 rev.

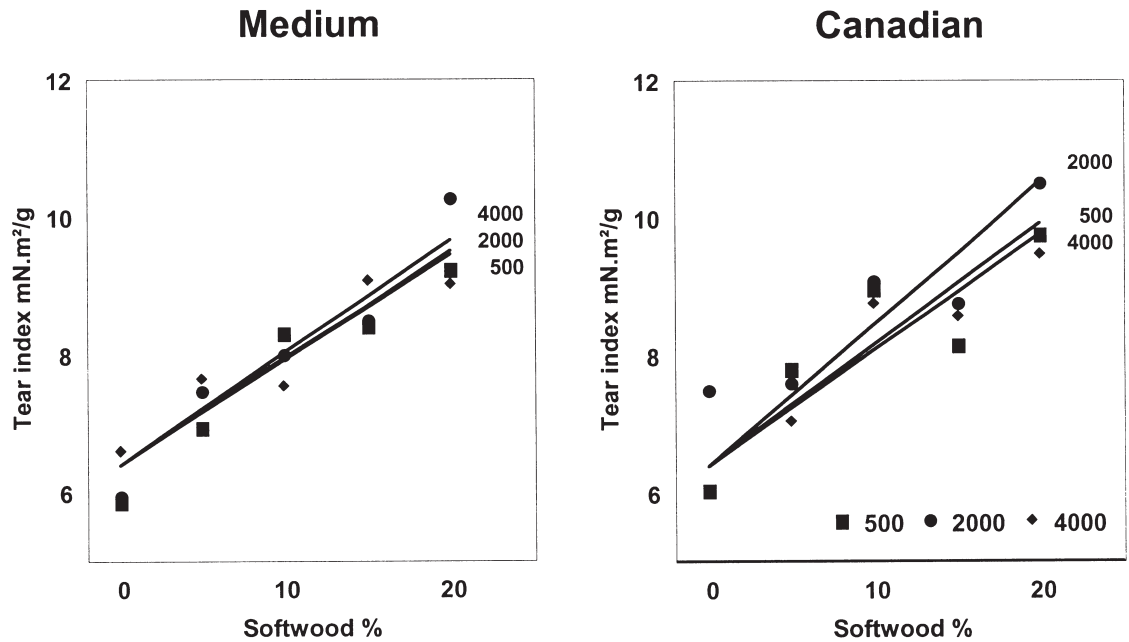


FIG. 6. Handsheet tear index versus percent softwood showing actual values (points) at one wet-pressing level (75 psi) and predicted models (lines) based on data of three wet-pressing levels (50, 75, and 100 psi). Furnish mixtures consist of eucalypt market kraft supplemented with 0 to 20 percent radiata pine (Medium) or spruce-rich fiber from the interior region of British Columbia (Canadian). The softwood component of each blend was refined in a PFI mill at 500, 2000, and 4000 rev.

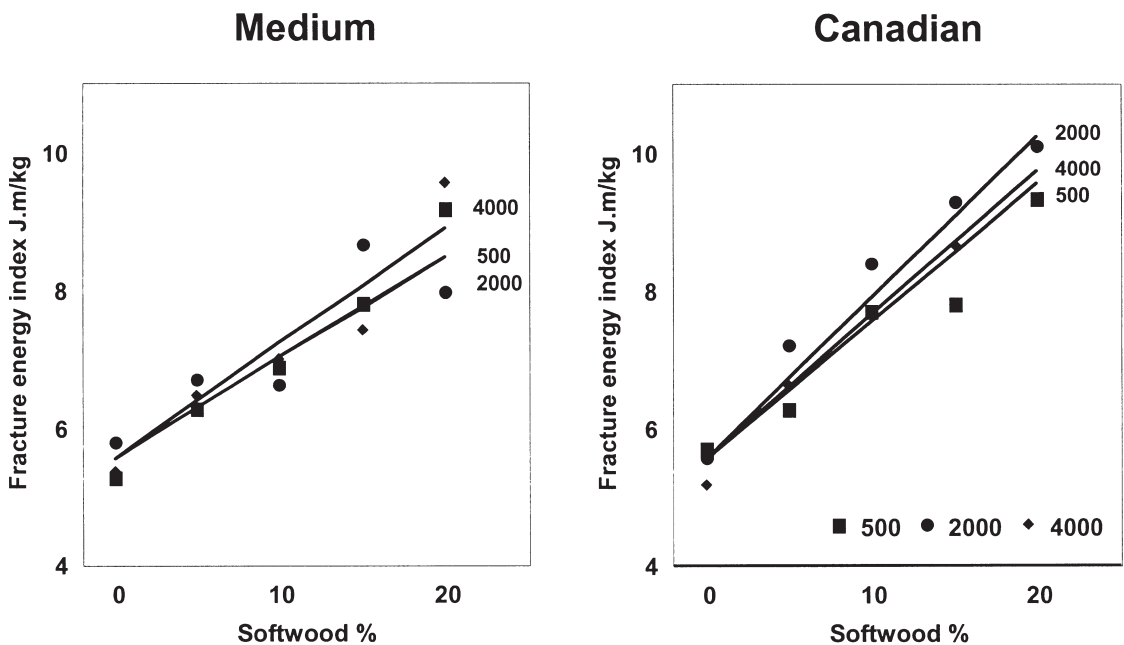


FIG. 7. Handsheet fracture energy index versus percent softwood showing actual values (points) at one wet-pressing level (75 psi) and predicted models (lines) based on data of three wet-pressing levels (50, 75, and 100 psi). Furnish mixtures consist of eucalypt market kraft supplemented with 0 to 20 percent radiata pine (Medium) or spruce-rich fiber from the interior region of British Columbia (Canadian). The softwood component of each blend was refined in a PFI mill at 500, 2000, and 4000 rev.

It is of interest to consider fracture energy index relationships among handsheets made from 100% softwood fiber and the softwood/eucalypt mixtures. Fracture energy indices of handsheets made from softwood fiber only are higher for the Canadian than for the Medium fiber-type, and increase with increasing wet-pressing at 500 rev refining with inconsistent trends at 2000 and 4000 rev (Table 4). Furthermore, fracture energy index trends with pulp refining are strong being lowest at 500 revs for both fiber-types, increasing with increasing refining for Medium, and similar at 2000 and 4000 revolutions for the Canadian fiber-type.

Fracture energy index values for the softwood/eucalypt mixtures obey the linear rule of mixtures at 2000 and 4000 revolutions of refining based on values at 100% softwood and 100% eucalypt. That is, the Canadian/eucalypt mixtures are of higher fracture energy index than the Medium blends, as predicted by the higher value of the 100% Canadian softwood fiber-type. The

magnitude of the difference between the Canadian and Medium blends is well predicted by the linear rule of mixtures where the softwood component is refined at 2000 and 4000 revolutions (Table 5). In contrast, where the softwood component received 500 revolutions of refining, fracture energy index values of the softwood/eucalypt mixtures are considerably higher than predicted by the linear rule of mixtures (Fig. 8, Table 5). The lower fracture energy index attained with the Canadian pulp at 4000 compared to 2000 revolutions refining is mirrored by other handsheet properties (Figs. 2, 4, 5), and could potentially be explained by the pulp being "over-refined."

#### *Reinforcement capacity of kraft fibers*

There is a need to be able to describe softwood fiber-types according to their reinforcement or carrier capacity when in wet webs and/or papers made from softwood/hardwood

TABLE 4. Handsheet fracture energy index of Canadian and radiata pine Medium softwood pulps.

| Wet-pressing (psi) | Canadian |          |          | Medium  |          |          |
|--------------------|----------|----------|----------|---------|----------|----------|
|                    | 500 rev  | 2000 rev | 4000 rev | 500 rev | 2000 rev | 4000 rev |
| 50                 | 17.0     | 26.5     | 28.8     | 13.1    | 21.1     | 23.3     |
| 75                 | 19.6     | 31.5     | 28.3     | 14.0    | 20.9     | 22.9     |
| 100                | 21.4     | 28.2     | 28.9     | 14.2    | 19.8     | 23.3     |
| Mean               | 19.3     | 28.7     | 28.7     | 13.8    | 20.6     | 23.2     |

TABLE 5. Differences between observed fracture energy index and linear rule of mixtures predicted fracture energy index for each softwood and refining level (averaged over all wet-pressing levels and percent softwood levels).

| Softwood fiber-type | PFI mill (revs) | Mean difference* | n  | T-test | P value |
|---------------------|-----------------|------------------|----|--------|---------|
| Canadian            | 500             | 0.61             | 15 | 3.73   | 0.0022  |
|                     | 2000            | 0.15             | 15 | 1.32   | 0.2083  |
|                     | 4000            | -0.27            | 15 | -1.90  | 0.0773  |
| Medium              | 500             | 0.62             | 15 | 3.92   | 0.0015  |
|                     | 2000            | -0.04            | 15 | -0.36  | 0.7210  |
|                     | 4000            | -0.09            | 15 | -0.77  | 0.4544  |

\* actual minus linear-rule-of-mixtures predicted

fiber mixtures. As such, it is fair to say that tear index, or tear-tensile relationships, are generally unable to distinguish between softwood fiber types of similar length. However, different numbers per unit mass when considered by themselves or when in softwood/hardwood mixtures may be a good indicator to differentiate this property (Kibblewhite 1993; Mansfield and Kibblewhite 2000). It is unfortunate that the mean fiber lengths of the Canadian and Medium pulps of this study differ by 0.26 mm (Table 1). De-

spite this setback, fracture energy index has clear advantages over other handsheet parameters, particularly tear index, for the characterization of softwood fiber reinforcement potential:

1. Levels of significance of the softwood-type effects are higher for fracture energy index ( $P < 0.0001$ ) than for tear index ( $P = 0.013$ ).

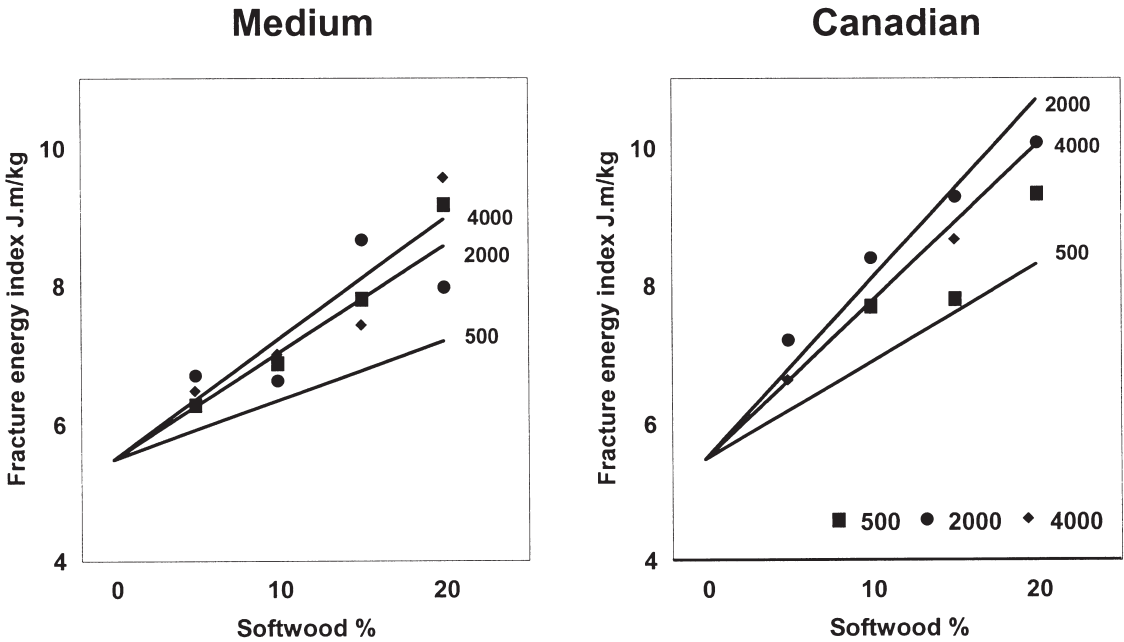


FIG. 8. Handsheet fracture energy index versus percent softwood showing actual values (points) and predicted models using the linear rule of mixtures (lines). Both data points and predicted models are for the 75 psi wet-pressing level. Furnish mixtures consist of eucalypt market kraft supplemented with 0 to 20%, and 100% radiata pine (Medium) or spruce-rich fiber from the interior region of British Columbia (Canadian). The softwood component of each blend was refined in a PFI mill at 500, 2000, and 4000 rev.

2. Levels of variation among test results are slightly lower for fracture energy index (6.1%) than for tear index (7.6%), as indicated by the error coefficient of variation ( $100 \times \text{root means square error} / \text{mean}$  (all data)) (Table 2). It should be noted that the handsheets tested were purposely all of lower-than-normal grammage (40 gsm), which could inflate testing analytical error compared to standard handsheets.
3. Fracture energy index of softwood/eucalypt mixtures (at 0–20 % softwood) are able to be predicted using handsheets made from 100% softwood fiber (refined at 2000 or 4000 revolutions).

### CONCLUSIONS

It can be concluded from this study that the reinforcement potential of softwood market kraft pulps in softwood/hardwood mixtures can readily be characterized through the measurement of fracture energy index of handsheets made from 100 % softwood fiber. These findings support and further the claims that fracture energy is a good indicator of reinforcing potential of kraft pulp fibers (Kärenlampi et al. 1998; Niskanen et al. 1999; Kettunen and Niskanen 2000; McDermid 2000). However, further research is warranted in identifying inherent fiber attributes contributing to the development of fracture energy index, including perimeter, wall thickness, length and microfibril angle, pulp chemistry, all of which could play an integral role in sheet development. Fiber wall area (or coarseness) is excluded from this list since it is described by perimeter and wall thickness in combination (wall area = perimeter  $\times$  wall thickness).

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