The effect of fibre fibril angle on some handsheet mechanical properties

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Segments of three eucalypt trees from the same site, two Eucalyptus nitens and one E. globulus, were selectively chipped. Segments at 10%, 30% and 70% of the tree height were taken. Wood chips coming from the inner and outer parts of each segment were then separated from each other, producing six chip samples per tree. The average fibril angle of each sample was determined using confocal microscopy. After kraft pulping, fibre and handsheet properties were measured.

For most samples there was a statistically significant reduction in fibril angle moving from the inner to the outer segment. However, the reduction in fibril angle from inner to outer segment was generally small (~5^{o} for most samples). The average fibril angle determined for each eucalypt sample lay within the range 0 to 13^{o}. As the measured change in fibril angle for the eucalypts was so small, it was concluded that the fibril angle had had a negligible effect on fibre and sheet mechanical properties.

Keywords

Microfibril angle, Eucalyptus nitens, Eucalyptus globulus, tensile strength, tearing resistance, zero-span tensile strength, pulp yield, fibre length, fibre property distribution.

Paper is a network of cellulose fibres. The mechanical properties of paper are a function of both the network characteristics and the mechanical properties of the individual fibres. Fibres have a layered structure, of which the most important layer is the S2 layer that makes up about 80% of the fibre wall. The S2 layer consists of helically wound cellulose microfibrils, with the angle of the helix with respect to the fibre axis known as the fibril angle. The fibril angle has a major influence on both the fibre's tensile strength and elastic modulus *(1,2)*. Fibres with small fibril angles are stronger and stiffer. Other fibre morphological properties that strongly influence sheet properties include fibre length, fibre wall thickness and fibre diameter.

New papermaking technology allows papermakers the use of a myriad of fibre combinations to produce paper products. These include recycled fibre and fractionated fibres. In very recent times the production of multilayered sheets of copy paper and other fine paper grades has become a reality. These developments will continue to place pressure on the fibre supplier to deliver more uniform fibres as well as fibres of specific qualities for specific end uses. A full understanding of the distribution of fibre characteristics, both within and between trees, and their influence on paper properties, will be important to ensure the fibre supplier meets these challenges and that customers can fully utilise fibre differences to produce their various papers at the highest quality.

From studies on softwoods, it has been found that the fibril angle varies strongly with position in the tree. In particular, the juvenile wood in the pith generally has a much higher fibril angle than mature wood. Similar data have not been available for eucalypts.

As there is such a strong dependence of the mechanical properties of the fibre on the fibril angle, there is interest in selectively chipping trees to produce wood chip stocks with different average fibril angles. These could then be sold into the mills on the basis of their expected end use. Thus the low fibril angle fibres from a tree could be sold, for example, to make containerboard grades where strong sheets are required.

Variation of fibre properties within eucalypts

There is a wide variation in the properties of fibres from different parts of a tree and between different species of eucalypts. There have been numerous studies done which have been summarised in a recently published book on sampling eucalypt plantations *(3)*. Some general trends for plantation grown eucalypts *(3)* include:

- Fibre length increases from pith to bark, tending to plateau in the mature wood.
- As a function of height in the tree and starting at the base, fibre length initially increases or stays constant before steadily falling.
- Fibre coarseness decreases with height up the tree.

Muneri *(4)* reported results from a study into within tree variation in fibre coarseness of *E. grandis* grown in Zimbabwe and found that coarseness increased from pith to bark and decreased with tree height.

There seems to be no published data on the radial variation of fibril angle within eucalypt trees, although it is believed that similarly to softwoods the fibril angle decreases moving from the juvenile wood in the pith to the mature wood on the outside of the tree. Also there is no data available on the variation of fibril angle with height in eucalypts.

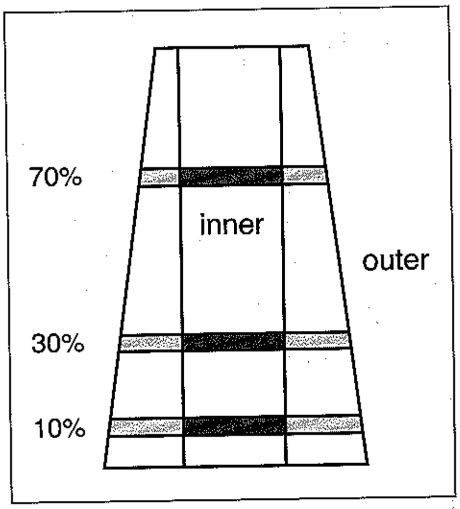

Fig. 1 Schematic diagram of the six sampling locations (shaded) from each tree. Disc cut from each location were segmented radially into inner and outer parts.

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Fibril angle measurements

Ideally, to assess the potential usefulness of fibres from different parts of the tree, or from different species, we would directly measure the distributions in all relevant single fibre properties. However, while fibre length, diameter and wall thickness distributions can be easily determined, single fibre strength and elastic modulus measurements remain far too time consuming for routine use. Thus it is essential to have fast and accurate methods for measuring the fibril angle, as this is the single factor that most strongly determines the breaking stress and elastic modulus of the fibres.

Fibril angles have either been measured using crossed Polaroids techniques *(5,6)* or X-ray diffraction techniques *(7,8)*. Recently a technique has been published *(9)* for measuring the fibril angle on single fibres and solid wood samples. The technique is a variant on the classical crossed Polaroids method but uses the z-depth resolution capabilities of a confocal microscope to optically isolate light reflected from a single cell wall, eliminating the need for extensive sample preparation. The technique is better suited than XRD techniques to the measurement of very low fibril angles typically found in the mature wood of eucalypts.

EXPERIMENTAL METHOD

Selective chipping

Two 15-year-old *E. nitens* (*E. nitens #*1 and *E. nitens #*2) and one 15-year-old *E. globulus* tree were chosen from the same site for selective chipping. Wood discs were cut at three sampling locations: 10%, 30% and 70% of merchantable height. The discs were air-dried. Prior to chipping in a disc chipper, an inner circular core at the centre of each disc was dyed. The diameter of this inner core was chosen so as to give ~50% of the total tree volume. After chipping, the chips were sorted into three groupings, chips with fully dyed ends, partially dyed ends and undyed ends. The partially dyed chips were not processed further. Thus for each tree, six samples were produced (Fig. 1).

Confocal fibril angle measurements

Fibril angle measurements were made using the new confocal technique described by Batchelor et al.*(9)*. Fibril angle measurements were carried out on laboratory chipped samples with no sample preparation. The wood chip surfaces were smooth enough for use with the limited working distance of the confocal microscope's 40x air objective lens. The wavelength of the laser light was 488 nm.

A single fibril angle measurement was made on each of ten chips sampled from different radial (inner and outer) and height (10, 30 and 70%) locations and from the three different trees, with the exception of the 70% outer location of *E. nitens* #1 where only seven chips were sampled. A total of 177 fibril angle measurements were made. Extra measurements were also made to determine the variability of fibril angles within a single chip. The measured fibril angle for a chip was an average of all fibres (typically 1 to 6 fibres) that were in the field of view.

Handsheet preparation and mechanical testing

The chips were kraft pulped (25% sulfidity) using a variable active alkali charge to achieve a target Kappa number of 18. Yield was determined for all of the samples. A full pulp evaluation for the six samples from *E. nitens* #1, and the inner and outer segments at 30 % height for the other two trees, was then performed. For each full pulp evaluation, beating curves were established using a PFI mill operating at a beating load of 1.77 N per mm of bar length. The following pulp and handsheet properties were then measured for each point on the beating curve: freeness, bulk, burst index, tear index, tensile index, stretch, wet zero-span tensile index, scattering coefficient and Sheffield air permeance. Fibre coarseness and length were measured using a Kajaani FS200 for samples from *E. nitens* # 1 only.

The zero-span tensile index measurements were made on rewetted samples with no gap between the jaws of the instrument. The measured values were not corrected for the residual span.

Australian and New Zealand standard methods (AS 1301), where available, were used in the testing.

RESULTS

Fibril angle results

The measured fibril angles from the six different locations on the three trees are shown in Figure 2 (*E. nitens* #1), Figure 3

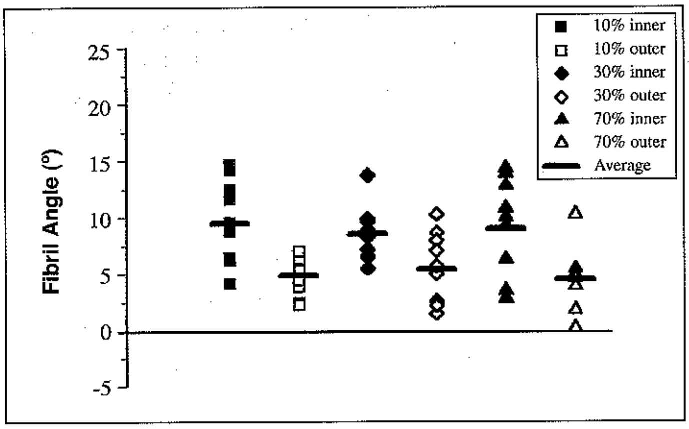

Fig. 2 Fibril angles for E. nitens #1. The 10, 30 and 70% height samples are shown as squares, diamonds and triangles respectively. The inner and outer samples are shown as filled and hollow symbols respectively. A horizontal bar represents the average fibril angle measurements for each location.

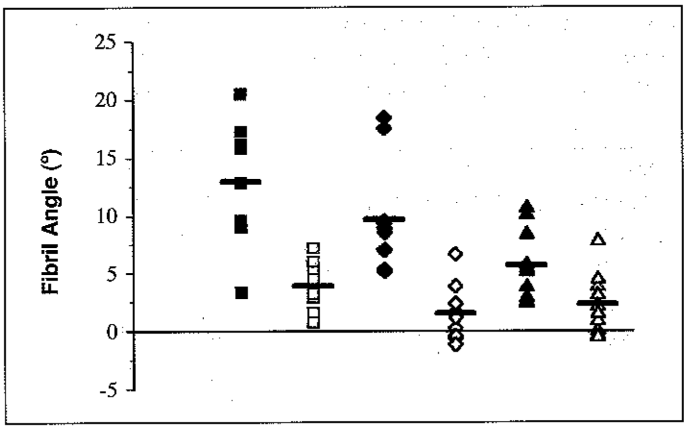

Fig. 3 Fibril angle measurements for E. nitens #2 tree. Symbols as per the legend for Figure 2.

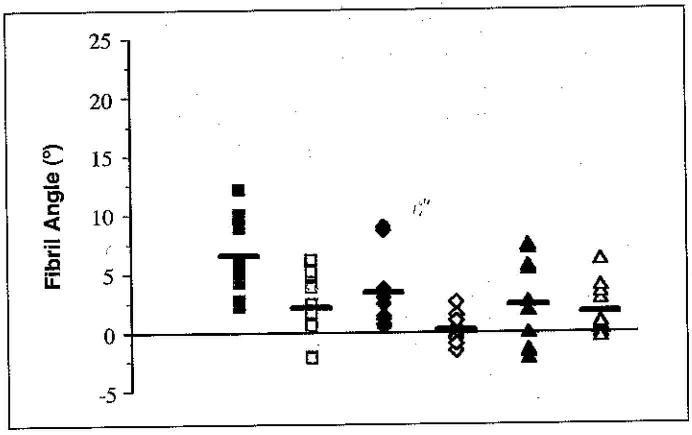

Fig. 4 **Fibril angles measured on E. globulus #2. Legend given in Figure 2.**

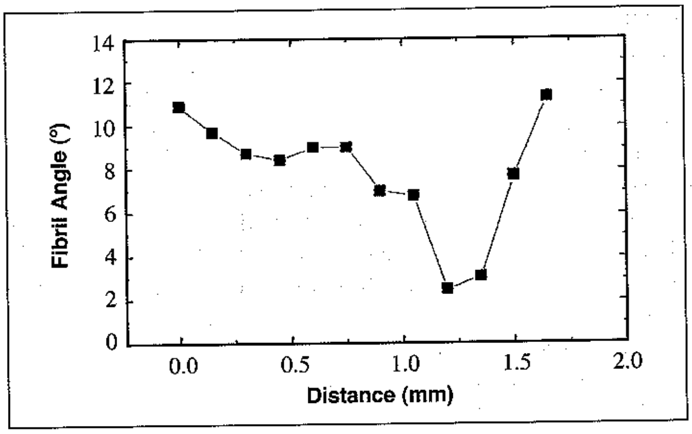

Fig. 5 **Variation in fibril angle with distance along a chip (E. nitens #1, 10% inner).**

(*E nitens* #2) and Figure 4 (*E. globulus* #1). The legend given in Figure 1 applies to all the Figures. The horizontal bars shown for each data set represent the averages for the set and are summarised in Table 1. In this table, the error quoted is the Standard Error (standard deviation divided by the square root of the number of samples).

In Figures 2, 3 and 4, some small negative fibril angles are apparent, the most negative being at -2°. The estimated error for measuring the fibril angle on chips is slightly greater than that for single fibres as there are usually several fibres contributing to the confocal image. An average reference fibre axis must be estimated from which the fibril angle is calculated. The error associated with each fibril angle measurement was estimated to be of the order of ± 3° to 4°. All the observed negative values are consistent, within experimental error, with small positive fibril angles.

In all cases the inner radial sample had a higher mean fibril angle than the outer sample. This difference in mean fibril angle was statistically significant at the 95% confidence level for all trees and heights except the 70% *E. globulus* sample (using t-tests assuming unequal variances).

A reduction in mean inner fibril angle with height for both the *E. nitens #2* and *E. globulus* trees is also apparent. Analysis of variance showed these trends to be significant at the 95% confidence level. No significant trend as a function of height was observed with the *E. nitens* #1 tree inner samples. A comparison of eucalypt species shows that the mean fibril angle of both *E. nitens* trees at each of the six sampling positions was higher than that of the corresponding locations in the *E. globulus* tree. In general the differences between inner and outer radial locations was also greater in the *E. nitens* trees than in the *E. globulus*.

The variation of fibril angle within a single chip was investigated. An example of this variation is shown in Figure 5 for a 10% inner *E. nitens* #1 chip. In this figure, fibril angle is plotted as a function of distance along the surface of the chip. In less than 2mm the fibril angle varies between 11° and 3°. The range observed from measurements over the entire set of 10 chips was 4° to 15°. Thus most of the fibril angles sampled from this location can be found within a 2mm distance on a single chip.

Handsheet preparation and mechanical testing

Pulp yields: A complete set of pulp and handsheet property data was not obtained for all samples. The data that is available is a complete evaluation for *E. nitens* #1 and complete evaluations for the samples taken from the 30% heights of the other two trees.

Table 2 shows the pulping data for all samples as well as the basic density of the wood from which the pulp was made. In each case, the pulp yield has been corrected to a Kappa number of 18. The major trend for all three trees is that the pulp yield increases from the inner segments of the tree to the outer segments of the tree. It can also be seen that the measured yield for the inner segment of each tree increases with height. These trends both imply that the yield of the juvenile wood is significantly lower than for the mature wood for all three trees.

Table 1
Average fibril angles measured on chips taken from six different positions within two E. nitens and one E. globulus trees.

Tree Segment	E. nitens #1 fibril angle	E. nitens #2 fibril angle	E. globulus fibril angle
10% Inner	9.5 ± 1.2	13.0 ± 1.6	6.6 ± 1.0
10% Outer	4.9 ± 0.5	3.9 ± 0.6	2.2 ± 0.9
30% Inner	8.6 ± 0.7	9.8 ± 1.5	3.5 ± 1.0
30% Outer	5.5 ± 1.0	1.6 ± 0.7	0.3 ± 0.4
70% Inner	9.1 ± 1.3	5.8 ± 0.9	2.5 ± 1.2
70% Outer	4.6 ± 1.2	2.4 ± 0.8	1.9 ± 0.7

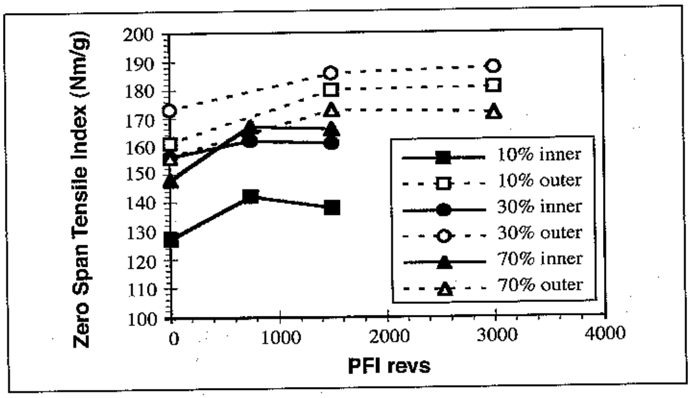

Fig. 6 **Development of wet zero-span tensile strength with beating for E. nitens #1.**

Table 2
Pulp yield and basic density variation with tree and location.

	E. nitens #1	E. nitens #2		E. globulus	
Tree Segment	Pulp Yield (%)	Pulp Yield (%)	Basic Density (kg/m^3)	Pulp Yield (%)	Basic Density (kg/m^3)
10% Inner	52.4	51.8	436	51.2	456
10% Outer	57.8	58.2	432	52.4	503
30% Inner	53.8	53.8	413	52.8	459
30% Outer	59.1	59.1	437	55.9	502
70% Inner	54.7	55.0	475	53.3	487
70% Outer	58.1	59.3	495	55.8	503

In comparing the data for the three trees, it can be seen that pulp yield data for the two *E. nitens* trees are almost identical but that there are significant differences between the results from the *E. globulus* tree and the two *E. nitens* trees. In general the differences between inner and outer yields are smaller for the *E. globulus* tree than for the *two E. nitens* trees, implying that the change in pulp yield moving from the juvenile to the mature wood is smaller for the *E. globulus* tree.

One should always be wary, however, if attributing differences between trees to 'species' rather than to 'environment'. Even though *E. nitens* and *E. globulus* are taxonomically very close, the optimal growing environs for these two species differ in many respects. The trees studied here were all grown in one environment.

When the results for pulp yield are compared with the basic density data, it can be seen that there is very little correlation between the two. One explanation would be that differences in the lignin content, rather than the morphology of the wood are producing the changes in pulp yield. This is consistent with NIR spectroscopy results given in Chapter 4 of *(3)* in which the estimated lignin content varied strongly with radial position and vertical position in *E. nitens* trees.

From the data in Table 2, separate pulping of wood from different segments of an *E. nitens* tree will be very advantageous as the pulping conditions can be set individually. When pulping all the wood together, the cooking conditions are set on the average lignin content of the wood. Thus chips with a lignin content lower than the average will tend to be overcooked, while chips with a lignin content higher than the average could be undercooked.

Zero-span tensile index: Figure 6 shows a plot of the wet zero-span tensile index against PFI mill revolutions for *E. nitens* #1. It can be seen that the wet zero-span tensile index improves slightly with beating for all six data sets. The wet zero-span tensile index is often taken as a direct measure of fibre strength, but in fact is also heavily influenced by other factors such as fibre curl and fibre length *(10)*. The observed increases with beating are probably due to the straightening of fibres that occurs during beating.

In comparing the wet zero-span values at zero PFI revs, a range between 127 Nm/g (10% inner) and 172 Nm/g (30% outer), is observed, with the highest measurement being 35% larger than the smallest. The measured average fibril angles of the two samples were 9.5° and 5.5° for the 10% inner and the 30% outer samples, respectively. From the theoretical curve of single fibre breaking strength with fibril angle *(1)*, it is expected that fibre strength will only increase by ~5% for this change in fibril angle. Thus the observed change in fibril angle does not account for the measured change in wet zero-span tensile strength.

Figures 7 and 8 show the wet zero-span tensile strength plotted against the fibril angle and the length weighted fibre length, respectively. Three points have been plotted for each sample using the results from the beating curve. The results from Figure 7 agree with the discussion given above. There is no strong dependence of the measured wet zero-span tensile strength on the fibril angle. However, it can be seen from Figure 8 that there is an apparent linear relationship between the wet zero-span tensile strength and the fibre length.

The fibre length heavily influences the measured wet zero-span strength because of the residual span. To be a true measure of fibre strength, all of the fibres crossing

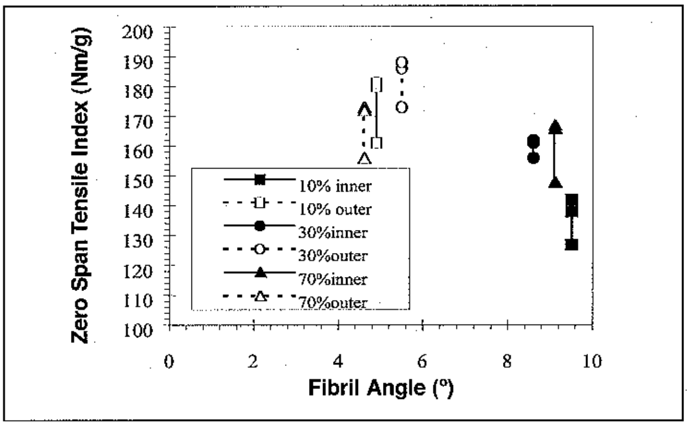

Fig. 7 **Wet zero-span strength as a function of fibril angle for E. nitens #1. The lines connect points on the PFI mill beating curve.**

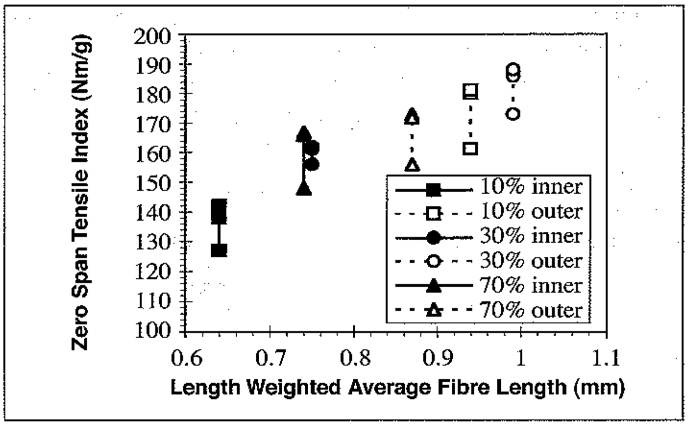

Fig. 8 **Wet zero-span tensile strength as a function of the length-weighted average fibre length for E. nitens #1.**

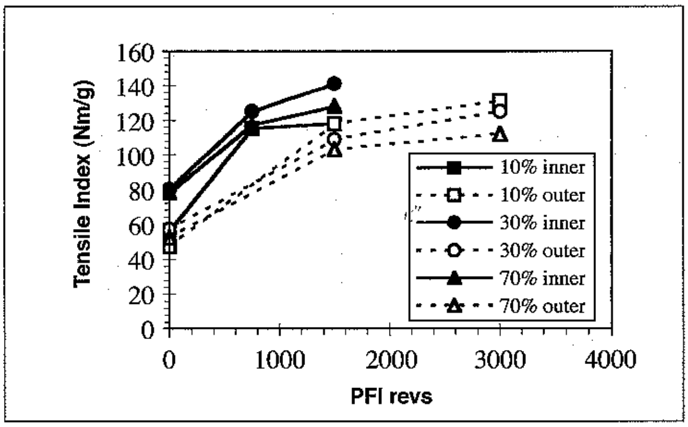

Fig. 9 **Development of tensile strength for E. nitens #1 as a function of PFI revolutions.**

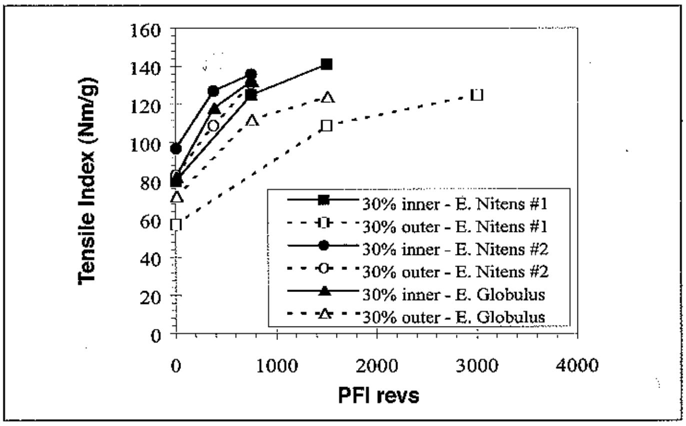

Fig. 10 **Development of tensile strength as a function of the number of PFI revolutions for inner and outer samples taken at 30% height for three eucalypt trees.**

the jaw lines must be broken. A residual span occurs because the jaws on the wet zero-span instrument require a finite distance before the force on the fibres is sufficient to hold them without slipping *(11,12)*. The difference between the true zero-span strength (no effect of residual span) and the measured wet zero-span strength will depend on the fibre length, since this determines how rapidly the measured strength changes with span *(12)*.

Beating curves: Figures 9 and 10 show the results of tensile index as a function of the number of PFI mill revolutions. The data from *E nitens* #1 are given in Figure 9, while the comparative data between the three trees at 30% height are given in Figure 10. It can be seen that similar general trends can be observed comparing the tensile strength development between the inner and outer segments. In general, the initial tensile strength for the pulp from the inner segments is higher than for the outer segments. Even after pulp from the outer segments was refined for twice the number of PFI revolutions, the pulp has somewhat lower strength. While complete beating curves were not obtained it is not likely that tensile strength will increase a great deal further given that the strength of the sheet is approaching that of the zero-span strength.

The type of action of a PFI mill cannot be compared to a commercial scale refiner. However, some conclusions are possible about the differing refining treatments that can be given to the fibres from the inner and the outer segments. In examining the coarseness data for *E. nitens* # 1 (Table 3) it can be seen that the fibres from the outer segments have consistently higher coarseness than the fibres from the inner segments. While the coarseness data should be combined with measurements of the fibre cross-sectional thickness to provide the complete story, it is intuitively reasonable that these fibres will be more resistant to collapse and therefore be poorer bonding, producing a bulkier, weaker sheet.

Thus the fibres from the outer segment of the tree will require more refining than fibres from the inner segment. As the fibres from the outer segment of the tree are coarser and longer they are likely to be able to withstand a more intense refining treatment. This may be significant because the manufacture of refiner plates to produce a very low intensity treatment of eucalypt pulps is difficult, with the resultant plates having very low lifetimes because they have very narrow width bars.

If the wood could be separated from the different parts of the tree, then the different refining treatments required could produce either significant savings in refining costs or optimisation of the quality of the refined pulp.

Figures 11 shows the development of the tensile strength as a function of freeness for all of the *E. nitens* # 1 samples, while in Figure 12, the results for the six samples from the three trees sampled at 30% height are given for comparison. On both graphs the lines between different points indicate the different points on the beating curve for the pulps. Thus the farthest right point on each curve gives the freeness of the unbeaten pulp. It can be seen on both graphs that the tensile strength is linearly related to the freeness. There is no observable trend difference between the inner and outer segments on either graph. However, as the freeness of the unbeaten pulp from the outer segments is always higher than for the pulp from the corresponding inner segments, less beating energy would be required to beat the inner pulp to a given level of freeness.

To further emphasise that the small differences in fibril angle seem to have had negligible impact on the mechanical properties of the sheet, results from the tearing resistance tests for the pulps from *E. nitens* #1 are presented in Figures 13, 14 and 15. The first of these figures shows the development of tearing resistance as a function of beating. It can be seen that

Table 3
Fibre length and coarseness values for E. nitens #1.

Sample	Length weighted fibre length (mm)	Fibre coarseness (mg/100m)
10% Inner	0.64	5.4
10% Outer	0.94	5.5
30% Inner	0.75	5.3
30% Outer	0.99	6.0
70% Inner	0.74	5.3
70% Outer	0.87	6.2

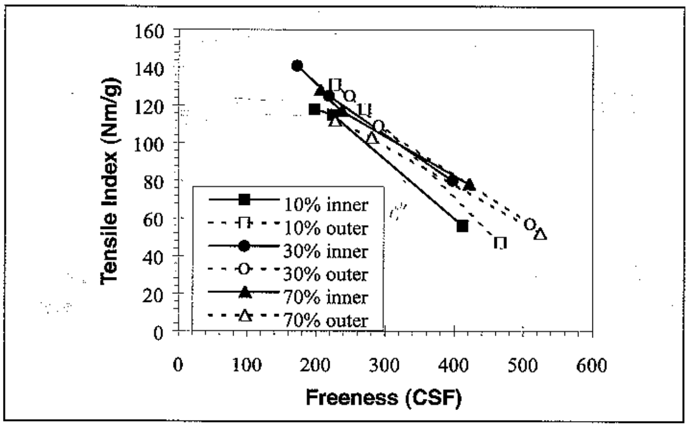

Fig. 11 Development of tensile strength as a function of freeness for E. nitens #1.

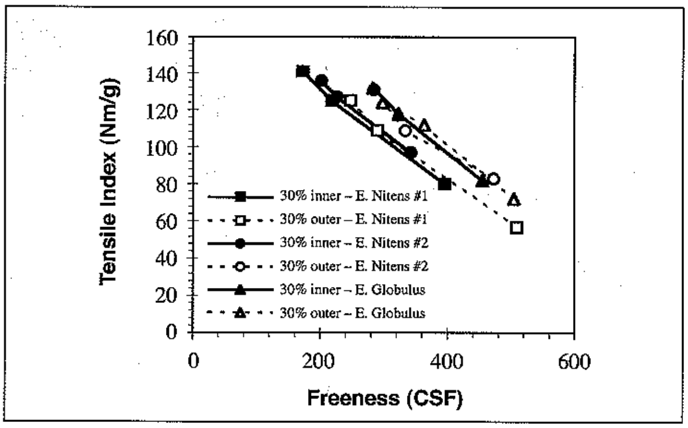

Fig. 12 Development of tensile strength as a function of freeness at 30% tree height.

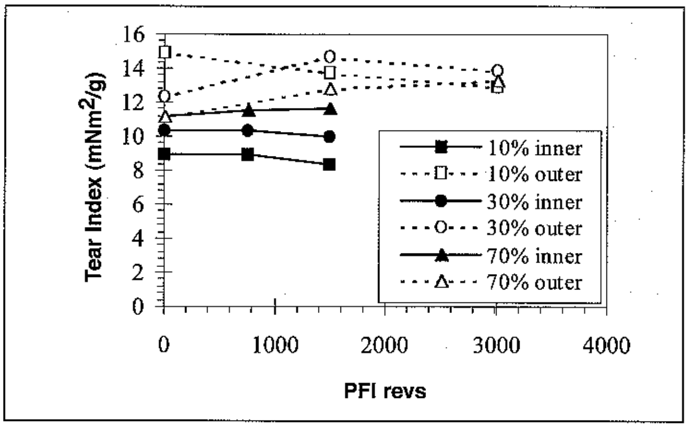

Fig. 13 Development of tearing resistance with beating for E. nitens #1.

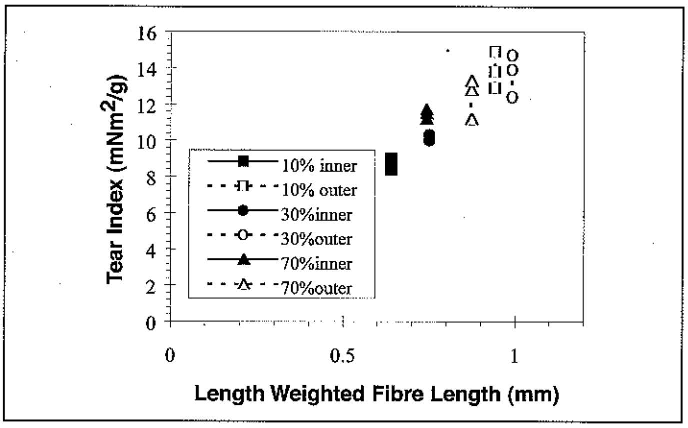

Fig. 14 Tear index as a function of length weighted fibre length for E. nitens #1.

there are clear differences between the tearing resistance for the inner and outer segments at all heights up the tree, with the tearing resistance of the outer segments being generally higher than that of the inner. The one exception to this is for the inner and outer segments of the tree at 70% height, which have very similar tearing resistances with the outer having slightly higher tearing resistance. In Figures 14 and 15, tear index has been plotted against fibre length and fibril angle, respectively. It can be seen that while there is an approximately linear correlation between fibre length and tear index, it appears that the correlation is not as good when plotted against fibril angle. The apparent linear correlation between fibre length and tear index is consistent with theory *(13)*. Given that fibre length, coarseness and strength (as indicated by the fibril angle) are all changing from sample to sample it is not possible to definitively extract, from this data, any effect of fibril angle on tear index.

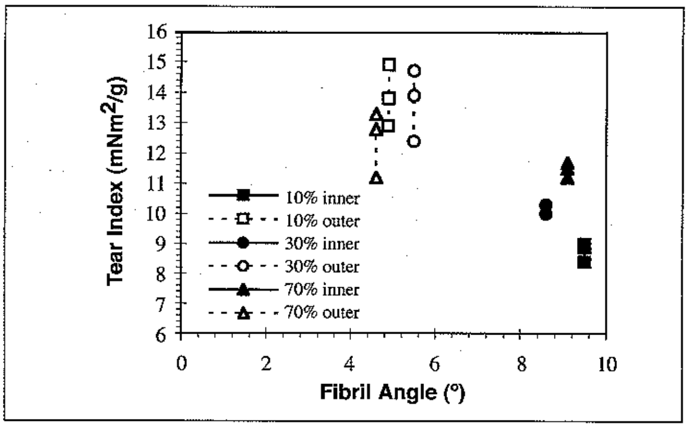

Fig. 15 Tear index as a function of fibril angle for samples from E. nitens #1.

CONCLUSIONS

Three eucalypt trees from the same site, two *E. nitens* and one *E. globulus*, were selected. For each tree, inner and outer segments at 10%, 30% and 70% of the tree height were taken and chipped. The average fibril angle of each sample of chips was determined using confocal microscopy. For most samples there was a statistically significant reduction in fibril angle moving from the inner to the outer segment. However, the reduction in fibril angle from inner to outer segment was generally small (~5° for most samples). The average fibril angle determined for each eucalypt sample lay within the range 0 to 13°.

Wood chips from each sample were pulped to a Kappa number of 18 and evaluated for yield. It was found that the yield was higher in the outer segment than the inner segment and increased with height in the tree. The differences

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The same trends were observed for slotted baskets fitted with the 'Bump' rotor (Fig. 18). The two fine slot baskets had similar thickening characteristics and efficiency capabilities over a relatively wide range of values. The lesser-contoured screen tended towards higher values for both. Wider slots could be used to reduce thickening but at the expense of efficiency.

Figure 19 shows the effect of rotor type on the same relationships. By using the VF rotor the thickening range could be extended, but with a similar overall relationship between thickening and efficiency.

Inter-relationships between capacity, separation and thickening were also examined. Generally, efficient separation and, by association, high thickening, were characterised by low capacity. As might be expected, there was a trade-off between these three important performance measures confirming that screen selection must always be based on seeking an appropriate compromise.

CONCLUSIONS

Both small-hole and fine-slot baskets can be used to fractionate a softwood kraft stock efficiently (based on fibre length), but there is a trade-off with other performance measures. With single stage screening, the smooth-surface medium hole baskets (1.60mm to 2.00mm∅) appear to provide the best compromise in that reasonable efficiency could be achieved under acceptable operating conditions. The more vigorous rotors enhanced runnability but with significant loss in efficiency. Surface profiling of the screen surface was detrimental to fibre separation for the 1.40mm hole basket as was an increase in contour height for the fine slot baskets. Altering rotor speed and aperture velocity could extend the operating range for each of the basket/rotor combinations but these were not as influential on screen performance as design features such as aperture size.

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between inner and outer segment pulp yields were larger for the *E. nitens* trees than for the *E. globulus*. Samples were selected for a full evaluation of handsheet properties. It was concluded that the changes in wet zero-span tensile strength and tearing resistance were satisfactorily explained by differences in fibre length and that the fibril angle probably had a negligible effect because the differences in fibril angle between inner and outer segments were only relatively small.

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