

Determination of within-tree variation of kraft pulp yield using near-infrared spectroscopy

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A SUCCESSFUL TREE BREEDING program relies heavily on the assessment of the trait of interest in large numbers of trees. When that trait is kraft pulp yield, the expensive wet chemistry methods involved make large scale assessment unrealistic. Traditional assessment has involved felling selected trees and pulping a bulked sample of chips taken from a number of sampling zones along the tree's stem. Such sampling is time-consuming and destructive. Nondestructive sampling has the advantage of conserving genotypes for later experimentation, research, and tree breeding. It can be achieved by taking cores representative of the whole tree. To facilitate the selection of representative sampling points, a detailed knowledge of pulp yield variation within plantation trees is necessary.

Few studies have examined the within-tree variation of pulp yield for eucalypts. The paucity of work can be related to the time and high costs involved in pulping the necessarily large numbers of samples. Several authors (1-5) have examined the within-tree variation of pulp yield with increasing height for a number of eucalypt species. For each species it was found that pulp yield increased rapidly from the base of the tree to 10% to 20% of tree height, leveled off at approximately 60% height, and then decreased sharply. Each study pulped whole disks taken from several heights but did not study yield variation from pith to bark within the disk.

Crawford *et al.* (6) examined soda pulp yield variation, with increasing height and distance from the pith, for two trees of *Eucalyptus delegatensis* grown in Northern Tasmania. The trees were aged 349 and 120 years. Contour maps were produced that illustrated patterns of pulp yield variation within the tree. Unfortunately, the trees were too old relative to those grown in plantations for reliable comparisons to be made.

Young plantation-grown trees are small and provide few samples for pulping using traditional methods. To obtain an accurate representation of variation, many sampling points are required. An option to pulping is near-infrared spectroscopy (NIRS), which can measure the chemistry of small samples quickly. NIRS analysis involves measuring the NIR spectra of a large number of samples closely related to the unknown samples, developing a model relating the spectra to the property of interest, and then using this model and a new sample's spectrum to predict the concentration of the property (7). NIRS has been used to estimate pulp yield (8-10) and other pulping parameters, including Kappa number (10-12). Only a small sample is required, making sampling at the within-ring level possible.

The aims of this study were to use NIR spectroscopy to measure the within-tree variation of kraft pulp yield in plantation-grown trees of *Eucalyptus nitens*, to model the longitudinal and radial variation, and to locate sampling regions that represent the whole tree.

ABSTRACT

A study involving near-infrared spectral measurements of wood meals was used to measure the within-tree variation of kraft pulp yield in plantation-grown trees of *Eucalyptus nitens*, to model the longitudinal and radial variation, and to locate sampling regions that represent the whole tree. A study of samples from 15 trees resulted in mapping of within-tree variations of pulp yield.

Application:

A knowledge of the within-tree variation of kraft pulp yield enables the identification of representative sampling points and facilitates the nondestructive sampling of standing trees using increment cores.

MATERIALS AND METHODS

Sample origin

Fifteen *E. nitens* trees aged 18 years were used in this study. They were taken from three families (five trees per family) selected for giving high, medium, and low kraft pulp yields. Each tree was sampled at 2, 5, 10, 15, 20, 30, 40, 50, 60, 70, 80, and 90% heights. Disks up to 70% height had the north aspect indicated.

One tree from each family was chosen for a detailed study of kraft pulp yield variation. Tree selection was based on good form; trees with eccentric piths were not used for this part of the study. The trees chosen were T08, T09, and T12. Disks from all heights were sampled. The tree number, kraft pulp yield, and height of each of the trees used in this study are presented in **Table I**.

The disks from tree T08 were further subdivided into north and south hemispheres. This was done to enable comparison between the two hemispheres. One disk (T09, 20% height) was chosen for a study of the radial variation of the NIR spectra.

Tree Number	Kraft Pulp Yield ^a	Height (m)	DUB at 5% ht (mm)
T01	Low	23.0	117
T02	Medium	23.2	90
T03	High	23.0	106
T04	High	21.0	113
T05	Low	17.5	129
T06	Low	17.7	84
T07	Medium	24.0	144
T08 ^b	Medium	21.9	113
T09 ^b	High	24.9	106
T10	High	24.4	124
T11	Medium	26.1	119
T12 ^b	Low	21.0	121
T13	Low	15.4	136
T14	Medium	24.7	116
T15	High	26.1	85

^aBased on prior work on kraft pulp yield from siblings of the same family.
^bDenotes the three trees that were examined at all heights. DUB = diameter under bark.

I. Tree number, kraft pulp yield and height of the 15 trees used in this study

CRC Tree Number	Disc Height (%)	Rings	Kraft Pulp Yield (%)
T08	10	1-5	52.7
T08	30	1-5	52.5
T08	60	1-4	52.5
T09	10	1-5	58.5
T09	30	1-5	58.3
T09	60	1-4	56.6
T12	10	1-2	48.8
T12	30	1-3	48.9
T12	60	1-4	48.8

II. The kraft pulp yield of outer ring samples taken at various heights for trees T08, T09, and T12

The remaining 12 trees were studied in less detail, with samples being taken from the 5, 20, 60, and 80% heights only.

Within-ring sampling

Each disk was debarked and sanded to facilitate the identification of growth rings. The rings were then numbered from bark to pith with the outer growth ring labeled as number one, taking care to ignore any false rings.

Ring widths were measured in four cardinal directions for all trees, except for T08, where ring widths were measured in three directions within the north and south hemispheres. Average ring widths were calculated.

Individual rings were sampled using a drill where possible. In some cases the rings were too narrow, and

two rings had to be sampled together. As many locations as possible were sampled around the ring to provide a representative sample, and at least 2.0 g of sample was obtained.

Sample preparation

All samples examined in this study were ground in a Wiley mill No. 4. Grinding was carried out through a 1.0 mm screen for 1 minute. The meal was then mixed and a sample removed, using a spatula, and packed in a NIR Systems micro sample cup (NR-7076).

Determination of hot water solubles content

Inner and outer heartwood and sapwood samples were removed from the 20% height disk of T09 using a drill. Test samples were milled in accordance with Australian and New

Zealand Standard AS/NZ 1301.002s-91 (13) and were tested for hot water solubles in accordance with Australian and New Zealand Standard AS/NZ 1301.P4m-61 (14).

Measurement of NIR spectra and spectral manipulation

The NIR spectra were measured in diffuse reflectance from samples of meal contained in a spinning sample cup in an NIR Systems Inc. Model 5000 scanning spectrophotometer. The instrument reference was a ceramic standard. The NIR spectra were collected at 2 nm intervals over the wavelength range 1100-2500 nm.

A total of 50 scans was accumulated for each of the duplicate samples and the results averaged. The data were converted before analysis to the second derivative mode by using the instrument's NSAS® software. A segment width of 10 nm and a gap width of 20 nm were used for the conversion.

Minipulping

Nine samples were minipulped to obtain data for the NIRS calibration model. The outer rings of T08, T09, and T12 were sampled at 10, 30, and 60% height. Generally the first four to five growth rings were required to provide enough wood for two minipulping charges (40 g oven dried wood per charge). The samples were minipulped by staff from North Eucalypt Technologies Ltd. using the following conditions. The H-factor was 2865, with times to and at 170°C both being 90 min. The sulfidity was 25% and liquor-to-wood ratio 3.5:1. The active alkali was varied to give kappa no. 18.

Calibration model for kraft pulp yield

A calibration model is required to relate the measured spectra to kraft pulp yield. This model is obtained by Partial Least Squares (PLS) regression of the spectral data versus pulp yield data from similar wood samples (9). Here, the model was developed using NSAS software (version 3.25) and the second derivative spectra of 97 *E. nitens* samples from Northern Tasmania. The

model was developed with four cross validation segments. The final number of factors used was recommended by the NSAS software (15).

Modeling longitudinal and radial kraft pulp yield variation

The radial and longitudinal variation of weighted kraft pulp yield was modeled using Table Curve 2D (version 2.01) (Jandel Scientific). The radial variation of kraft pulp yield was modeled at 5, 20, and 60% of tree height, for all trees.

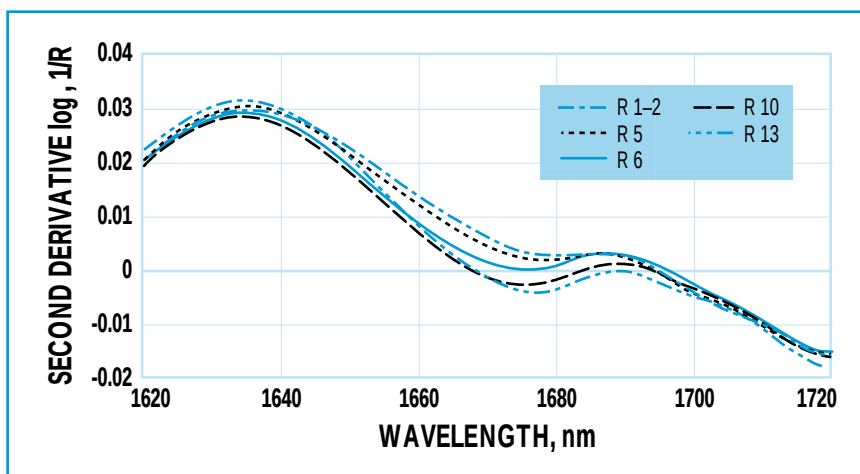
Area-weighted averages were calculated by weighting the kraft pulp yield of each ring by its area. The longitudinal variation of kraft pulp yield was modeled using only the three trees studied in detail. Here, volume-weighted averages were used. Average kraft pulp yields for each ring and section were weighted during modeling, using the respective ring areas and sectional volumes. In all cases, the simplest model was selected.

The development of whole-tree maps showing kraft pulp yield variation

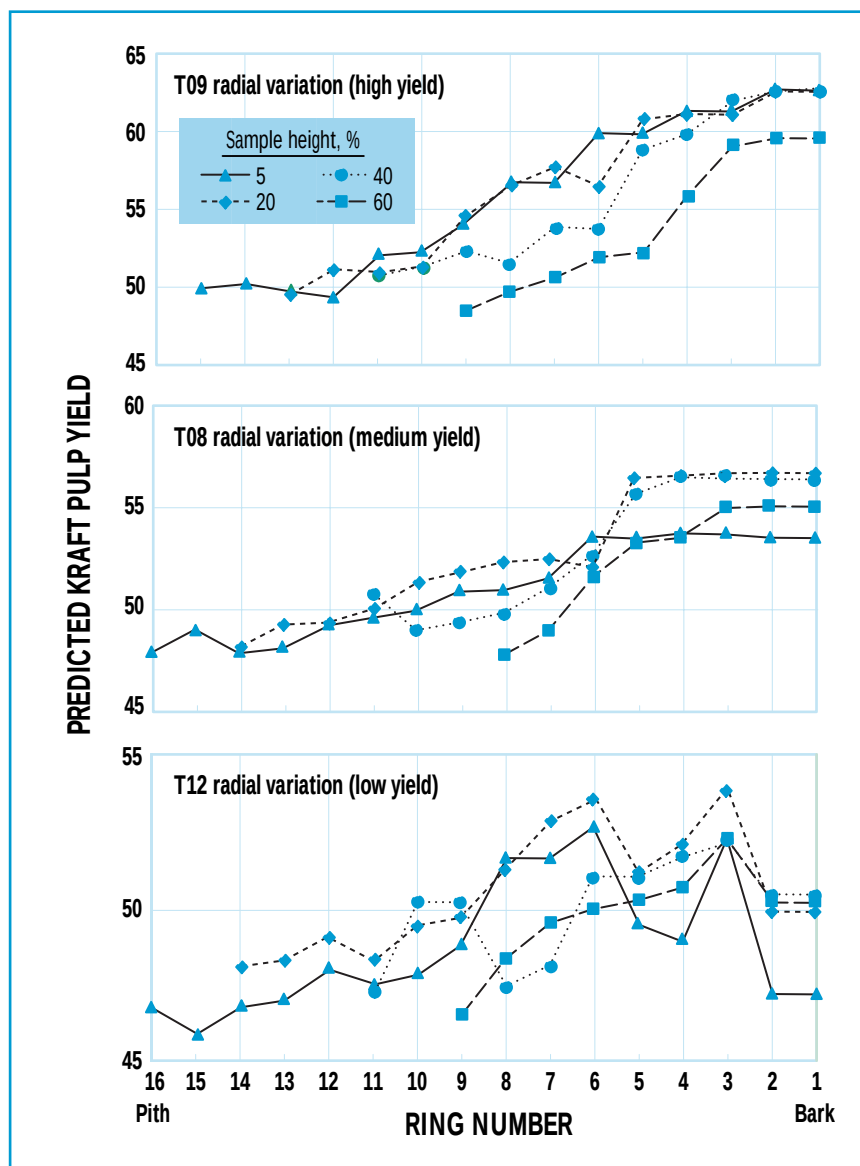
Whole-tree maps demonstrating the within-tree variation of kraft pulp yield were developed for each tree using two computer programs developed by Dr. R. Evans. Together the programs produce a TIF (tagged image file format) grey scale image that illustrates the variation occurring within a tree. Each grey level corresponds to a kraft pulp yield range, which is set by using a step value and maximum and minimum values. A step value of 1.5 was used for the 15 trees.

The information used includes height, ring number and ring width, which identify a region in space; and the kraft pulp yield, which assigns a value to that space. The program creates images by interpolating the yield values between heights for each ring. Values near the pith were obtained by extrapolation.

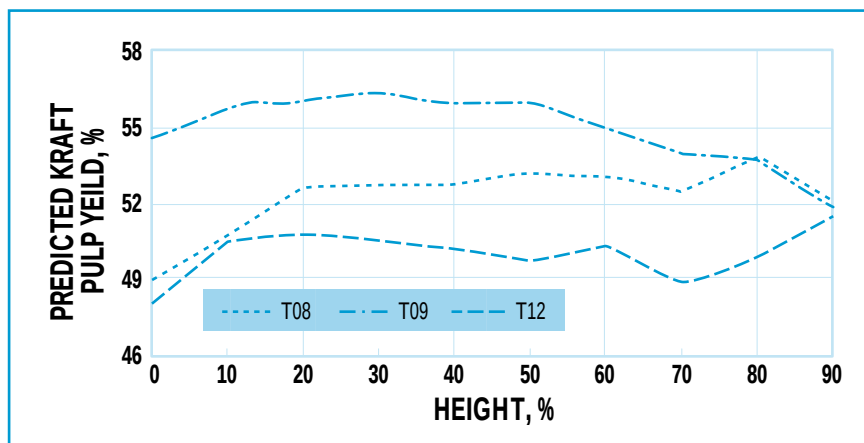
The TIF images were manipulated using ZIP (version 4.1) (Catenary Systems). The mirror and square



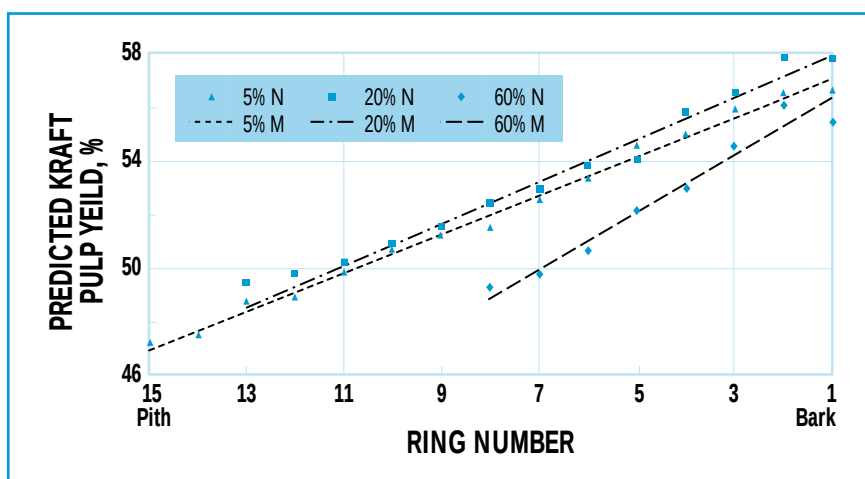
1. Pith to bark NIR spectral variation in the region 1620-1720 nm for T09 at 20% height



2. The radial variation of kraft pulp yield for trees T08, T09, and T12 at 5, 20, 40, and 60% height



3. Longitudinal variation of weighted kraft pulp yield for trees T08, T09, and T12



4. NIRS predicted and modeled radial variation of kraft pulp yield at 5, 20, and 60% height.

Note: M = model predicted yield and N = NIR predicted yield

aspect ratio functions were used. Microsoft PowerPoint (version 4.0a) was used to manipulate the size of the image. Color maps can be produced from the grey scale images.

Two additional maps of the within-tree variation of pulp yield were developed. The first was an average of the three trees studied in detail, and the second was an average of all 15. For the first map, the average kraft pulp yield of each ring was calculated by area weighting based on ring widths for each ring at each height. Ring widths and the height of each disk were also averaged for the three trees. For the second map, yield information of the remaining 12 trees for 5, 20, 60, and 80% heights was included. Again, area-weighted yields were calculated.

Average ring widths were calculated for the four heights only using all 15 trees. The average height of each disk was calculated using the three trees studied in detail only. As yield extremes were not as great, a step value of 1.25 was used.

Correlation of kraft pulp yield at each height with the whole-tree value

Initially, correlations were calculated at all heights for the three trees examined in detail, but it was realized that the method gave erroneously high correlations due to the autocorrelation that existed between the single disk and whole-tree yields. In addition, three trees were too few to determine stable correlations.

As a consequence, the correlation of yield at specific heights (5, 10, 20, 40, 60%) with whole-tree yield was calculated using all trees. A representative sample of each disk was obtained by removing a pith to bark strip. Independent whole-tree samples were obtained by removing representative samples from each disk and combining those samples. Unfortunately, this method was too time consuming to test all trees at all heights.

RESULTS AND DISCUSSION

Calibration model for kraft pulp yield

The kraft pulp yields obtained by manipulating the outer growth rings of three trees at 10, 30, and 60% height are shown in **Table II**. These results were combined with those from kraft pulps from 88 Northern Tasmanian *E. nitens* samples and the requisite NIR spectra to give a PLS model relating the band absorbances to pulp yield. The model had four factors, an R-value of 0.948, and a standard error of calibration (SEC) of 0.74.

Radial NIR spectral variation

The largest variation in the NIR spectra for the pith to bark samples taken from a single disk was in the regions 1620-1720 nm and 2050-2150 nm. The former region is associated with the first overtone of C_{ar}-H stretching vibrations in lignin and polyphenols (16). The radial spectral variation for the region 1620-1720 nm for T09 at 20% height is illustrated in **Fig. 1**. The spectra are in second derivative form, with the peaks pointing downwards. Ring 13 is the pith, and ring 1 is the outermost ring. Several spectra were similar and are omitted for simplification: rings 3-4 (similar to ring 5); rings 7, 8, and 9 (similar to ring 6); and rings 11 and 12 (similar to ring 10).

The greatest spectral variation in the region 1620-1720 nm was noted at 1676 nm (**Fig. 1**). The outer rings have the weakest absorbances, while the inner have the strongest. The peak absorption shifts to higher wavelengths

Variation Modeled	Equation	a	b	c	R ²	Fit Std. Error
Radial 5% Height	$y = a + bx$	46.19	0.72	-	0.99	0.28
Radial 20% Height	$y = a + bx$	47.68	0.79	-	0.98	0.39
Radial 60% Height	$y = a + bx$	47.73	1.08	-	0.92	0.68
Longitudinal	$y = a + bx + cx^{1/2}$	50.79	-8.41	9.36	0.96	0.16

III. A summary of the models fitted to the kraft pulp yield data (y = predicted kraft pulp yield and x = ring number)

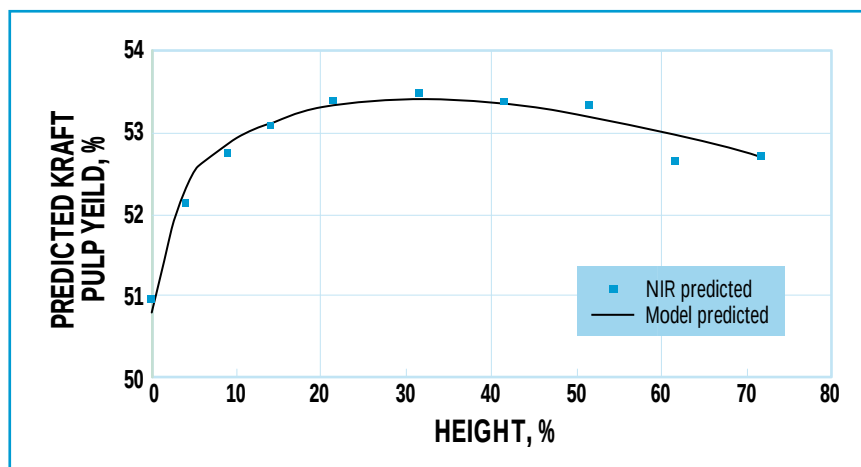
for wood nearer the pith. In the region 2050-2150 nm, differences in the spectra are most apparent at 2082 nm. At this wavelength, the outer rings have the strongest absorbance. The most noticeable difference between the spectra is between rings 5 and 6.

In both regions the spectra form two groups: rings 1 to 5 form one group, and rings 6 to 13 the other. Examination of the disk from which the samples were taken indicates that the heartwood- sapwood boundary, identified by an abrupt change in color from orange-yellow to pale cream, is located between rings 5 and 6. Wood color is influenced by the presence of extractives, and the abrupt change in color corresponds to an abrupt change in extractives content (17). Hot water solubles analysis confirmed that the extractives content of the outer and inner heartwood (5.9% and 4.6%, respectively) was higher than that of the sapwood (3.3%). As a result, the large differences that exist between the spectra of rings 5 and 6 are consistent with the chemical differences between heartwood and sapwood.

Radial kraft pulp yield variation

The NIR predicted radial variation of kraft pulp yield for T08, T09, and T12 at various heights (5, 20, 40, and 60%) is shown in Fig. 2. Heights chosen for study were biased towards the base of the tree as it represents the majority of the tree's volume and is more accessible to nondestructive sampling.

Figure 2 demonstrates that patterns of radial variation vary between trees and also with height within trees. T08 and T09 show a strong trend of increasing pulp yield from pith to bark. Radially, T09 demonstrated a greater rate of increase in yield than T08. An overall trend of increasing pulp yield



5. NIRS predicted and modeled longitudinal variation of kraft pulp yield

from pith to bark is also shown by T12, but the rate of increase is lower and reversion between rings is apparent, particularly between rings 3 and 9.

Few studies have examined radial variation of pulp yield. The scarcity of studies is due to the difficulty and cost of obtaining enough wood for conventional chemical pulping. A study of old growth *E. delegantensis* also found that radial variation of pulp yield is highly variable and that each height had a different pattern of variation (6).

An interesting feature for T08 and, to a lesser extent T09, is a marked change in pulp yield that occurs in the vicinity of the heartwood-sapwood boundary. Heartwood has a much higher extractives content than sapwood and as a result has a lower pulp yield (17,18). A decrease in soda pulp yield due to the higher extractives content of the outer heartwood is also shown in old growth *E. delegantensis* (6).

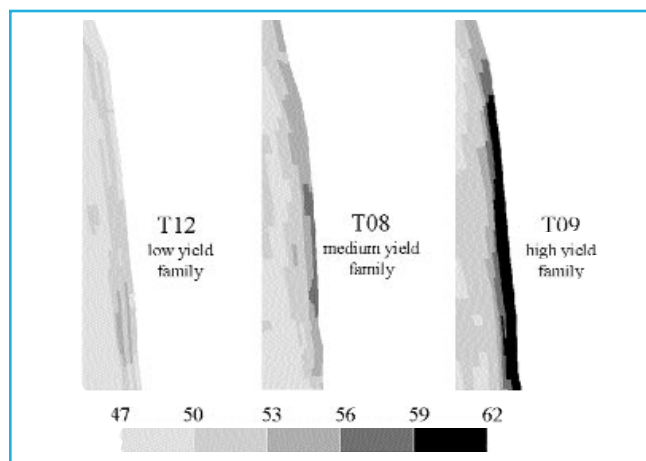
Longitudinal kraft pulp yield variation

The longitudinal variation of kraft pulp yield was examined as part of the

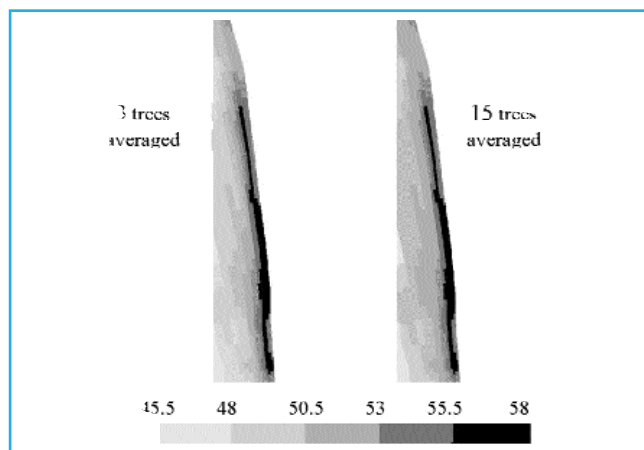
detailed study of the three trees. At each height, the area-weighted kraft pulp yield was calculated for the whole disk. The longitudinal variation of kraft pulp yield for T08, T09, and T12 is shown in Fig. 3.

The trees fit the family classification of high, medium, and low pulp yield (Table I). Each tree exhibited a different pattern of longitudinal variation. An initial increase in pulp yield, tending to a maximum at approximately 30% of total tree height and then decreasing, was shown by each tree. However, T08 then appeared to rise to a local maximum near 80%, while T12 rose to another maximum near 60% with a further rise nearing the top of the tree. This is surprising, but perhaps less so in view of the somewhat erratic results in the radial direction (Fig. 2).

Studies of within-tree variation of kraft pulp yield have mainly concentrated on longitudinal variation. Generally, yield increases sharply from the base of the tree to approximately 10 to 30% of tree height before be-



6. The within-tree variation of kraft pulp yield for trees T08, T09, and T12



7. The average within-tree variation of kraft pulp yield for the three trees studied in detail and all 15 trees

coming constant until the height exceeds 60%, whereafter it declines (1-3, 5). Tree T09 was the only tree that demonstrated longitudinal variation typical of past studies.

Modeling the longitudinal and radial variation of kraft pulp yield

The radial variation of kraft pulp yield at 5, 20, and 60% height, and the curves fitted to the data, are shown in Fig. 4. The radial variation is the weighted average of 15 trees. The longitudinal variation of kraft pulp yield and the curve fitted to the data is shown in Fig. 5. The longitudinal variation is the weighted average of the three trees studied in detail.

The radial and longitudinal variations of kraft pulp yield are much smoother when averaged over several trees. Radial variation at all heights was close to linear, reaching a maximum at the outer rings. Samples from 60% height had the lowest average yields; those from 5% had the highest. The longitudinal variation of kraft pulp yield was not linear. It increased rapidly from 0 to 20% height, reaching a maximum at 30% height, after which it decreased. The variation is similar to

that found by other authors (1-3, 5). The models fitted to the kraft pulp yield data are summarized in Table III.

The models that describe radial variation are all simple, being of the form $y = a + bx$. The radial models at 5 and 20% height fit the data well, with high R^2 values and low standard errors. The data at 60% height were not modeled as well, as indicated by a lower R^2 value and higher standard error. Linear models were fitted for the trees studied here but would be unsuitable for older trees, as demonstrated by the study of Crawford *et al.* (6). The trees in this study were only 18 years old, with kraft pulp yield still increasing radially, but this could not continue indefinitely. An age will be reached when kraft pulp yield will no longer increase, or increase at a much slower rate, and a more complex model will be required to explain the variation.

The model that described longitudinal variation was more complex, being of the form $y = a + bx + cx^{1/2}$. It had a high R^2 value (0.96) and low standard error (0.16).

Whole-tree kraft pulp yield variation

Whole-tree maps of the within-tree variation of kraft pulp yield were developed for each tree. Maps of the three trees examined in detail are shown in Fig. 6. The darker the grey level, the higher the pulp yield.

By mapping kraft pulp yield variation, patterns of variation that other-

wise would have been difficult to discern become apparent. The region of highest yield is the outer three to four rings between 10 and 60% of tree height. In contrast, the lowest yield region is the center of the tree at 2-10% and at 30 to 60 or 70% of tree height. At breast height the majority of wood is of a low pulp yield for all trees.

The existing variation within and between trees makes recognition of a consistent pattern of variation difficult using single trees. Maps representing the average of 3 and 15 trees, respectively, were developed to clarify the variation. The maps of the within-tree variation for three averaged trees and 15 averaged trees are presented in Fig. 7.

Averaging pulp yield over several trees smooths local fluctuations in pulp yield that confuse the real pattern of variation (Fig. 7). The two average maps show that kraft pulp yield increases with increasing distance from the pith. At ring 8 or 9, yield increases rapidly to the outer three or four rings that have the highest yield. The outer rings between 15 and 40% height were the highest yield regions of the tree. Regions of the lowest yield (45.5 to 48.0%) were found in the center of the tree between 2 to 15% height and around 30% height.

Patterns of variation, particularly for the inner rings, appear to be in agreement with the model of a tree that exhibits conical symmetry (19). The high yield outer rings do not ex-

KEYWORDS

Bibliographies, breeding, *Eucalyptus nitens*, growth rings, heart wood, infrared spectroscopy, kraft pulps, models, nondestructive tests, sampling, sap wood, selection, trees, variations, yield.

hibit strict conical symmetry as they do not extend to the base or the top of the tree. The variation is not cylindrically symmetric (19). A number of physical parameters of *Pinus radiata* exhibit cylindrical symmetry (19, 20). Cylindrical symmetry is important because, in theory, cores taken from any height should have the same average properties as those of the tree above or below that height. Therefore the estimation of whole-tree properties in *P. radiata* based on breast height cores can be made with high accuracy. Estimation of the whole-tree kraft pulp yield of *E. nitens* from measurement on breast height cores would be expected to be less accurate.

The paucity of studies of the within-tree variation of pulp yield makes comparison with the above observations difficult. There is only one published study that maps the within-tree variation of pulp yield (6). Unfortunately, tree age, and the fact that the contour maps (6) developed bear little resemblance to the maps developed in this study, make comparison difficult. It can be noted, though, that the center of the tree at ground level is of a very low yield and that the outer rings do not have the highest pulp yields. Recently, whole-tree maps for several chemical parameters including δ -cellulose, extractives, hemicellulose, holocellulose, and lignin have been developed (21-25). Pulp yield was not one of the mapped parameters.

Kraft pulp yield variation within north and south hemispheres

Examination of the north and south hemispheres of T08 found that cardinal direction had little influence on the within-tree variation of pulp yield. The variation in the north hemisphere was almost a mirror image of that in the south, with the size and shape of the yield regions similar.

Correlation of kraft pulp yield at 5, 10, 20, 40, and 60% height with whole-tree yield

The correlation of the weighted kraft pulp yield at 5, 10, 20, 40, and 60%

height with the whole-tree kraft pulp yield for all trees is shown in **Table IV**.

Identification of representative sampling points is very important if nondestructive sampling methods, i.e., cores, are to represent the whole tree accurately. Traditionally, cores have been taken from breast height (1.3 to 1.5 m), but recently several authors have questioned how representative breast height is of the whole tree for eucalypts (26-28). A study of 9-year-old *E. grandis* found that micropulped core samples from 1.3 m were poorly correlated with whole-tree yield (29). A more recent study determined that for the estimation of whole-tree pulp properties and pulpwood productivity, 2.8 m was the most appropriate sampling height for *E. camaldulensis* and *E. globulus* (30). In this study, the correlation of yield at breast height (assumed to be 5% height) with whole-tree yield was found to be high. The highest correlation ($R^2 = 0.82$) was for disks from 10% height which, based on the average height of the 15 trees examined, is 2.2 m.

CONCLUSIONS

The NIR spectral variation between samples taken from different rings was mainly confined to the 1620-1720 nm and 2050-2150 nm regions. NIR spectra of rings 5 and 6 demonstrated the greatest differences between spectra of successive rings. The heartwood-sapwood boundary was located between these two rings, and differences between the heartwood and sapwood, over and above the radial wood chemical variation, might be responsible for the larger spectral variation.

Maps of within-tree variation of pulp yield demonstrated that pulp yield is highly variable within individual trees and between trees of the same family. Maps representing the average of three and 15 trees clarified the variation by averaging local fluctuations in yield. These maps demonstrated that the outer ring region between 15 and 40% of tree height

% Height	R ²
5	0.75
10	0.82
20	0.76
40	0.56
60	0.67

IV. Correlation of weighted kraft pulp yield at 5, 10, 20, 40, and 60% height with whole-tree kraft pulp yield

gave the highest yield.

The influence of cardinal direction on pulp yield was small. The patterns of pulp yield variation were very similar for each hemisphere.

Longitudinal and radial variation (at selected heights) of kraft pulp yield were modeled. Radial variation was adequately described by linear equations, while longitudinal variation was described by an equation of the form $y = a + bx + cx^{1/2}$. The yield of samples from 10% of tree height (approximately 2.2 m) gave the best correlation with whole-tree yield ($R^2 = 0.82$). The correlation of yield at breast height (assumed to be 5% of total tree height) with whole-tree yield was also quite high.

NIRS is well suited to examining the within-tree variation of kraft pulp yield for plantation-grown eucalypts. One big advantage is its ability to predict the kraft pulp yield of very small samples, making possible studies at the within-ring level and thus enabling very detailed maps of within-tree variation to be created. The detail of the maps is superior to that obtained previously for pulp yield (6) or any other chemical component of wood (21-25) and would have been prohibitively laborious and expensive using micropulping methods. While NIRS was only used to predict kraft pulp yield, it could have been used to predict other chemical components (31), as a number of analyses can be performed simultaneously using NIRS (32,33). As well as pulp yield, cellulose, xylan, and lignin content, for example, could all have been predicted, provided that

suitable models based on wet chemistry data existed. Whole-tree maps illustrating how each of these components varied could be produced.

The greatest limitation of the method is the time and cost of the wet chemical analysis. Samples from the trees being studied must be pulped and included in the calibration set to

obtain an adequate NIRS calibration model. TJ

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