Estimation of whole-tree wood quality traits using near infrared spectra from increment cores

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SUMMARY

There is worldwide interest in the potential of eucalypts for fibre production. Selection of the most suitable stock for propagation is important and many wood quality traits related to pulp production are measured. Effective evaluation requires rapid, inexpensive and preferably non-destructive techniques. Near infrared (NIR) spectroscopy provides a rapid method for evaluating a range of wood quality traits but studies are generally limited to the evaluation of whole-tree properties requiring destructive sampling. In this study we compare calibrations for wood quality traits obtained using whole-tree data and NIR spectra obtained from milled whole-tree composite chip samples, and 5 mm diameter increment cores non-destructively sampled from 0.65 m and 1.30 m. Increment core and whole-tree composite calibrations provided similar results indicating that calibrations based on increment cores can be used to estimate wood quality traits on a whole-tree basis. Of the two heights investigated 1.30 m was identified as the most suitable height for nondestructive sampling.

Keywords
Eucalypts, near infrared spectroscopy, nondestructive sampling, tree improvement, tree properties

INTRODUCTION

Globally there is interest in the potential of eucalypts for fibre production. Eucalypt pulps are renowned for their ability to produce high quality printing and writing papers and tissue products. Eucalypt fibres are relatively short, slender and thin-walled (*1,2*) and these properties lead to excellent sheet formation and a sheet that has high bulk, excellent surface properties and good density, stiffness and optical properties (*2*).

Aracruz Celulose S.A. is the largest producer of bleached eucalypt market pulp in the world. Tree improvement began at Aracruz Celulose S.A. in 1973 (*3*) by selecting for various growth and wood properties and using vegetative propagation (*4*). Dramatic improvements in growth (*4*), productivity and wood quality (*3*) have been reported. Tree improvement has mainly concentrated on *Eucalyptus grandis* W. Hill ex Maiden x *Eucalyptus urophylla* S. T. Blake hybrids that have been selected for their growth and pulp properties. Selection of the most suitable hybrids for propagation is important and many wood quality traits related to pulp production are measured. For these characteristics to be effectively considered in the selection process, operational techniques that permit rapid, inexpensive and preferably non-destructive evaluation of a large number of individuals in the field is important.

Several studies have demonstrated that near infrared (NIR) spectroscopy can be used to determine several wood quality traits related to pulp production including cellulose content, extractives, lignin content and pulp yield (*5-17*). Generally these studies have involved the analysis of a single species and rarely have multiple species or hybrids been included in wood property calibrations. An exception is the study by Garbutt et al. (*10*) who included 13 eucalypt species and a hybrid in their cellulose and lignin calibrations. At Aracruz Celulose S.A. many species and hybrids are evaluated and the development of multiple species wood property calibrations is desirable.

An NIR spectrum (700-2500 nm) consists of overtone and combination bands of the fundamental stretching vibrations of O-H, N-H and C-H functional groups (*18*). The estimation of any wood property by NIR spectroscopy depends on the development of a calibration using a set of samples about which something is known, for example pulp yield. The calibration is then used to predict the pulp yield of additional samples based on their NIR spectra. A major advantage of NIR spectroscopy that is of particular importance to tree improvement programs, is that it allows the assessment of a number of traits using a single NIR spectrum, provided the appropriate calibrations exist (*19*).

An important objective in tree improvement programs is the estimation of whole-tree properties. Many of the NIR studies noted have examined relationships between whole-tree data and NIR spectra obtained from milled chips that represent the whole-tree, i.e. a composite sample comprised of chipped billets or discs from several heights. The reported calibrations can only be applied, with confidence, to other whole-tree composite samples requiring further destructive sampling. To facilitate nondestructive estimation of whole-tree properties calibrations could be based on whole-tree data from destructively sampled trees and NIR spectra from increment cores removed at an easily accessible sampling height. While destructive sampling is necessary to obtain whole-tree data, once a calibration has been built using increment core spectra, it could be applied to increment cores non-destructively sampled from standing trees. This approach has not been reported in the literature.

The aims of this study were:

a. to compare calibrations for a range important pulp properties obtained using NIR spectra from whole-tree composite chip samples and 5 mm increment cores removed from two heights (0.65 and 1.30 m); and
b. to investigate the development of calibrations using several species and hybrids demonstrating wide genetic variation.

MATERIALS AND METHODS

Sample origin

One hundred selected trees (various species and hybrids) that demonstrated wide genetic variation were destructively sampled and chipped to give whole-tree composites. Increment cores (5 mm in diameter) were also removed from these trees at sampling heights of 0.65 and

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Table 1
Number of trees sampled and summary statistics for each eucalypt species and hybrid.

Species	No. of samples	Basic density (kg/m^3)	NaOH charge (%)	Pentosans (%)	Pulp yield (%)	Specific cons (%)	Total lignin (%)
Eucalyptus citriodora Hook.	4	646.0 - 710.0 (30.3, 675.8)	12.5 - 13.0 (0.3, 12.8)	18.6 - 21.2 (1.1, 19.6)	51.7 - 54.6 (1.3, 53.3)	2.7 - 3.0 (0.2, 2.8)	24.3 - 25.8 (0.7, 24.9)
Eucalyptus dunnii Maiden	3	466.0 - 536.0 (32.2, 489.3)	13.5 - 14.6 (0.6, 14.0)	18.8 - 19.2 (0.2, 19.0)	52.0 - 55.7 (1.9, 53.7)	3.7 - 4.0 (0.2, 3.9)	25.9 - 28.2 (1.2, 27.2)
Eucalyptus globulus (globulus) Labill.	5	467.0 - 484.0 (7.5, 477.2)	13.5 - 14.5 (0.4, 13.9)	20.1 - 21.9 (0.7, 20.9)	51.0 - 54.6 (1.3, 53.2)	3.9 - 4.3 (0.2, 4.0)	26.6 - 27.5 (0.4, 27.0)
Eucalyptus grandis W. Hill ex Maiden	15	331.0 - 500.0 (49.1, 416.5)	13.0 - 15.0 (0.6, 13.8)	14.7 - 18.8 (1.1, 17.3)	49.4 - 54.4 (1.4, 52.2)	3.8 - 6.0 (0.6, 4.7)	27.6 - 31.4 (1.1, 29.6)
Eucalyptus pellita F. Muell.	1	445.0	15.0	14.8	48.5	4.7	33.3
Eucalyptus saligna Sm.	5	376.0 - 496.0 (51.6, 443.2)	12.8 - 15.0 (0.8, 14.0)	14.8 - 17.8 (1.2, 16.3)	49.5 - 54.3 (2.2, 51.5)	3.8 - 5.5 (0.7, 4.5)	28.7 - 32.6 (1.6, 30.4)
Eucalyptus urophylla S. T. Blake	19	394.0 - 484.0 (26.8, 442.0)	12.7 - 14.5 (0.5, 13.7)	14.7 - 17.8 (0.8, 15.9)	49.5 - 54.4 (1.4, 52.0)	3.9 - 5.0 (0.3, 4.5)	28.1 - 32.2 (1.1, 30.1)
Hybrids							
E. grandis x *E. globulus*	2	455.0 - 466.0 (7.8, 460.5)	13.1 - 13.2 (0.1, 13.2)	16.7 - 17.8 (0.8, 17.3)	53.0 - 53.3 (0.2, 53.2)	4.1 - 4.2 (0.1, 4.1)	26.5 - 27.7 (0.9, 27.1)
E. grandis x *E. pellita*	2	419.0 - 573.0 (108.9, 496.0)	12.8 - 13.3 (0.4, 13.1)	15.4 - 15.9 (0.4, 15.7)	52.3 - 52.8 (0.4, 52.6)	3.4 - 4.6 (0.9, 4.0)	30.0 - 30.7 (0.5, 30.4)
E. grandis x *E. urophylla*	32	327.0 - 494.0 (32.8, 435.0)	12.5 - 15.5 (0.7, 13.8)	14.6 - 18.9 (1.0, 16.2)	46.9 - 55.94 (1.6, 52.2)	3.9 - 6.6 (0.5, 4.5)	28.1 - 31.2 (1.0, 29.6)
E. urophylla x *E. globulus*	8	400.0 - 486.0 (30.2, 454.9)	12.7 - 13.6 (0.4, 13.1)	16.3 - 18.5 (0.7, 17.5)	52.1 - 54.3 (0.7, 52.8)	3.8 - 4.9 (0.3, 4.2)	27.0 - 29.2 (0.8, 28.6)
E. urophylla x *E. pellita*	2	460.0 - 473.0 (9.2, 466.5)	15.0 - 15.2 (0.1, 15.1)	15.0 - 15.6 (0.4, 15.3)	49.8 - 50.4 (0.4, 50.1)	4.1 - 4.2 (0.1, 4.1)	26.5 - 27.7 (0.9, 27.1)

1.30 m. The trees were sampled at 3 locations Aracruz (Long. 40°07', Lat. 19°42'), Bahia (Long. 39°36', Lat. 17°52') and São Mateus (Long. 39°48', Lat. 18°40') and were aged between 2 and 3 years (early selection age at Aracruz). The number of trees sampled and summary statistics for each eucalypt species and hybrid is given in Table 1.

Sample preparation

All samples were air-dried. A subsample of each whole-tree composite and the increment cores (0.65 and 1.30 m) were milled in a Wiley mill and then in an Udy mill. The Udy mill was used to maximize sample recovery from the 5 mm cores.

Determination of wood quality traits

Aracruz staff determined several wood quality traits on the whole-tree chip samples using published standard methods where possible. Traits included basic density (BD) (*20*), screened pulp yield (PY) (500 g of chips pulped to Kappa number 18 ± 1), soda (NaOH) charge, specific consumption (SC), which is a reverse function of basic density and pulp yield, that determines the wood volume required to produce one ton of pulp (m^3/air dry ton), total lignin (*21*) and pentosans (*22*).

Near infrared spectroscopy

The NIR spectra were measured in diffuse reflectance mode from samples held in a spinning sample holder in a NIRSystems Inc. Model 5000 scanning spectrophotometer. The spectra were collected at 2 nm intervals over the wavelength range 1100-2500 nm. The instrument reference was a ceramic standard. Fifty scans were accumulated for each sample and the results averaged. After the spectrum had been obtained, the sample cup was emptied, repacked and a duplicate spectrum obtained. The duplicate spectra were averaged and converted to the second derivative using Vision® software (version 3.1). A segment width of 10 nm and a gap width of 20 nm were used for the conversion.

PLS calibrations for wood quality traits

Calibrations were developed for the whole-tree composites and 0.65 m and 1.5 m core samples using Partial Least Squares (PLS) regression and second derivative spectra. A detailed description of PLS regression is given elsewhere (*23*). The calibrations were obtained using Vision® software, with full cross validation (i.e. leave-one-out) and a maximum of ten factors. A total of 60 samples (selected by the Vision® software) were used for calibration, while 38 samples

Table 2
Range of each parameter for the calibration and prediction sets.

Trait	Calibration set (60 samples) Minimum	Maximum	Av.	Std. dev.	Prediction set (38 samples) Minimum	Maximum	Av.	Std. dev.
Basic density (kg/m^3)	327	710.0	453.0	67.8	376.0	655.0	449.6	48.1
NaOH charge (%)	12.5	15.2	13.6	0.6	12.7	15.5	13.8	0.7
Pentosans (%)	14.7	21.9	17.1	1.7	14.6	20.1	16.5	1.4
Pulp yield (%)	46.9	55.9	52.3	1.6	48.5	54.4	52.2	1.4
Specific consumption (m^3/adt)	2.7	6.6	4.4	0.7	3.0	5.5	4.4	0.4
Total lignin (%)	24.3	32.2	29.0	1.7	25.8	33.3	29.6	1.5

were used to test the predictive performance of the calibrations (2 samples were omitted from the set as outliers owing to their large residuals). The 60 calibration samples were identified using the whole-tree sample set and the samples in the 0.65 m and 1.30 m sample sets were selected to match those of the whole-tree sets. Table 2 provides a statistical summary of the calibration and prediction sets. To facilitate the comparison of different calibrations between sample sets (whole-tree, 0.65 m and 1.30 m) the number of factors used for a given trait was held constant. For all properties (aside from pulp yield) the number of factors used was the number recommended by the software for the whole-tree calibrations. In the case of the pulp yield calibrations 4 factors were recommended for the 0.65 m and 1.30 m calibrations while only one factor was recommended for the whole-tree pulp yield calibration, consequently 4 factors were used.

The Standard Error of Calibration (SEC) (determined from the residuals of the final calibration), the Standard Error of Cross Validation (SECV) (determined from the residuals of each cross validation phase), the coefficient of determination (R^2), and the ratio of performance to deviation (RPD_c) (*24*), calculated as the ratio of the standard deviation of the reference data to the SECV were used to assess calibration performance.

The Standard Error of Prediction (SEP) (determined from the residuals of the predictions) was calculated and gives a measure of how well a calibration predicts parameters of interest for a set of samples not included in the calibration set. The predictive ability of the calibrations was also assessed by calculating the R_p^2 (defined as the proportion of variation in the independent prediction set that was explained by the calibration) and the RPD_p (which is similar to the RPD_c) but uses the standard deviation of the prediction set reference data and the SEP. Determination of RPD allows comparison of calibrations for different properties that have differing data ranges and units, the higher the RPD the more accurate the data is described by the calibration. Though an RPD_p of greater than 2.5 is considered satisfactory for screening, it has been shown that calibrations with an RPD_p of approximately 1.5 can be useful for initial sample screening (*25*).

RESULTS

PLS calibrations for each trait

Calibrations for basic density (2 factors), NaOH charge (4 factors), pentosans (4 factors), pulp yield (4 factors), specific consumption (2 factors) and lignin (4 factors) were created using whole-tree data and the NIR spectra of the milled whole-tree composite chips, and the milled wood of the 0.65 and the 1.30 m cores. Figure 1 provides a comparison of the coefficients of determination (R^2) obtained for each calibration for each set while Figure 2 provides a comparison of the RPD_c obtained for each calibration.

The pentosans calibrations had the strongest R^2 (0.81 to 0.83) and the highest RPD_c values (1.95 to 2.09). R^2 and RPD_c values for pentosans were similar regardless of which sample set was used with the 0.65 m calibration giving the best results. Total lignin also gave strong calibration statistics, R^2 ranged from 0.79 (whole-tree chips) to 0.77 (0.65 m cores) and RPD_c ranged from 1.90 (whole-tree chips) to 1.77 (0.65 m cores). Calibrations obtained using NIR spectra from whole-tree composite samples and whole-tree data for (a) pentosans, (b) pulp yield, and (c) total lignin are shown in Figure 3.

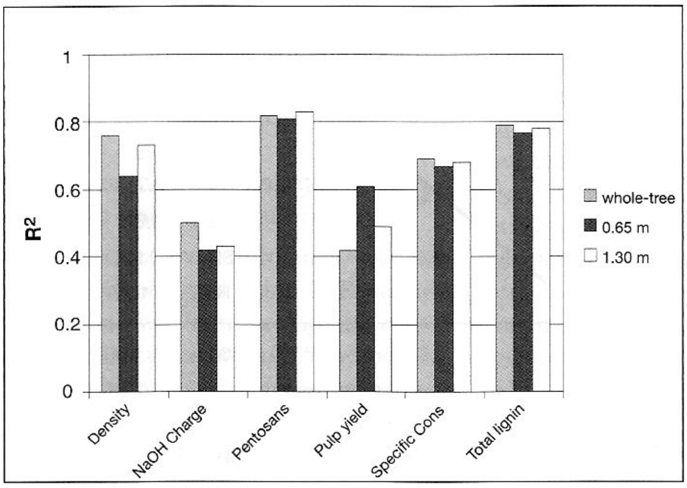

Fig. 1 **R^2 for each trait for calibrations developed using milled whole-tree chips, 0.65 m cores and 1.30 m cores.**

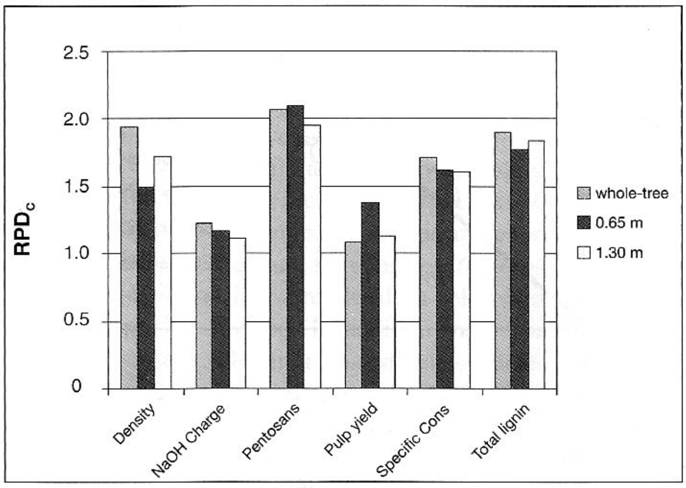

Fig. 2 **RPD_c values for each trait for calibrations developed using milled whole-tree chips, 0.65 m cores and 1.30 m cores.**

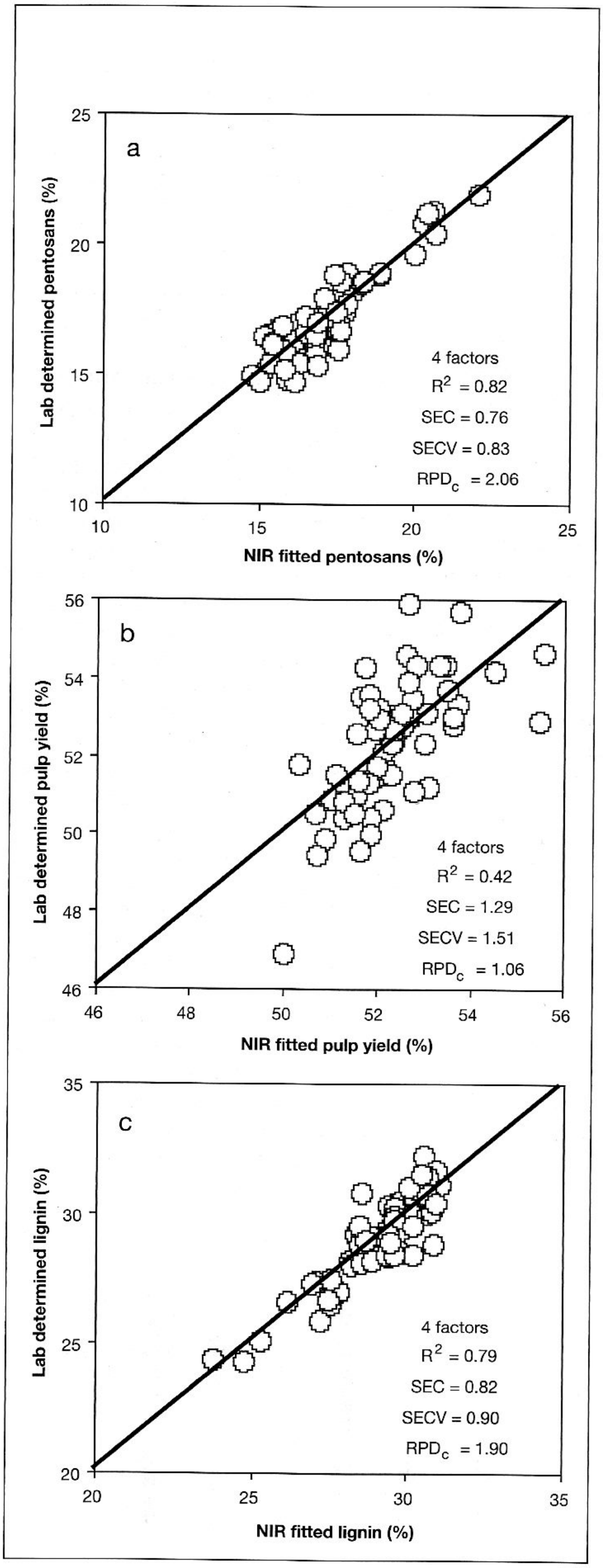

Fig. 3 Relationships between measured values and NIR-estimated values for (a) pentosans, (b) pulp yield, and (c) total lignin. Sixty NIR spectra from milled whole-tree composite chips were used. Note that the regression line has been plotted.

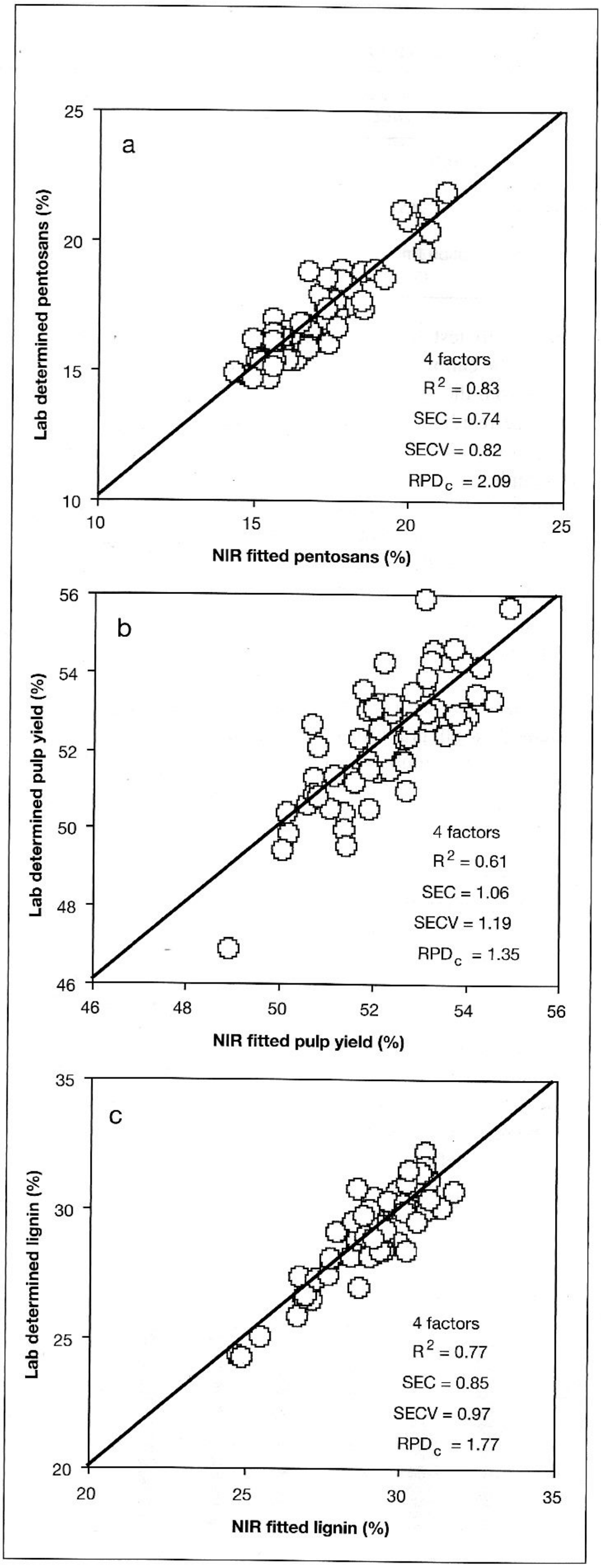

Fig. 4 Relationships between measured values and NIR-estimated values for (a) pentosans, (b) pulp yield, and (c) total lignin. Sixty NIR spectra from milled 5 mm increment cores removed at a height of 0.65 m were used. Note that the regression line has been plotted.

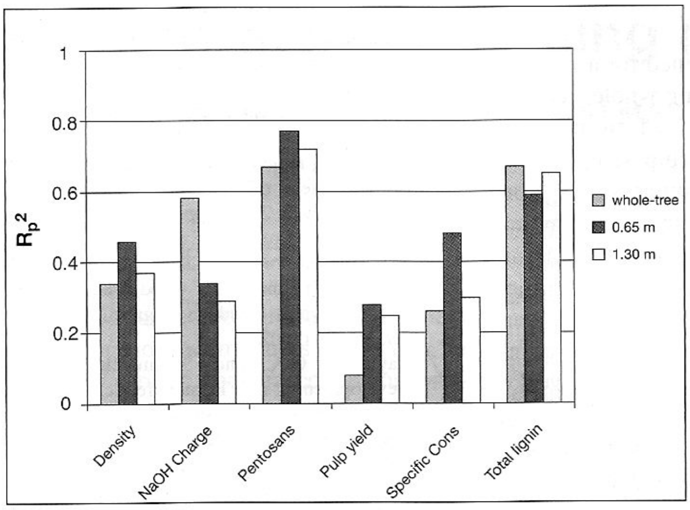

Fig. 5 **R_p^2 for each trait for calibrations obtained using milled whole-tree chips, 0.65 m cores and 1.30 m cores. Note one sample was removed as an outlier from the prediction set for pentosans and total lignin.**

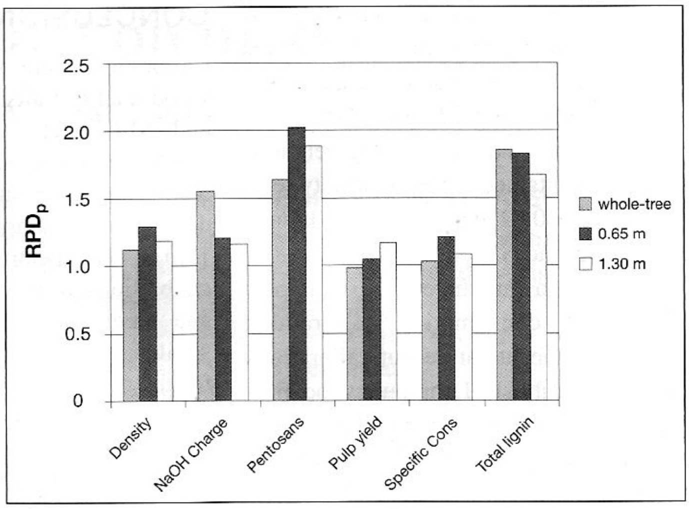

Fig. 6 **RPD_p values for each trait for calibrations obtained using milled whole-tree chips, 0.65 m cores and 1.30 m cores. Note one sample was removed as an outlier from the prediction set for pentosans and total lignin.**

For comparison (a) pentosans, (b) pulp yield, and (c) total lignin calibrations obtained using NIR spectra from 0.65 m cores and whole-tree data are shown in Figure 4. Both sets of figures show that the lignin and pentosan data were well fitted by the respective calibrations and that there is very little difference between calibrations obtained using whole-tree composite chip spectra and 0.65 m core spectra. For pulp yield the data was fitted moderately well by the 0.65 m calibration but poorly by the whole-tree calibration.

Calibrations for basic density and specific consumption were not as good as those for pentosans and total lignin. The basic density calibrations had R^2 that were variable ranging from 0.76 (whole-tree chips) to 0.64 (0.65 m cores) while R^2 were similar for the specific consumption calibrations (R^2 ranged from 0.67 to 0.69). The calibrations for pulp yield and NaOH charge were weak with the 0.65 m core pulp yield calibration having the highest R^2 (0.61), noticeably higher than the R^2 obtained for the whole-tree composite chips and 1.30 m cores.

Prediction of traits using whole-tree chip and core calibrations

The predictive performance of the calibrations obtained using NIR spectra from milled whole-tree composite chips, and the milled wood of the 0.65 and the 1.30 m cores, was evaluated using the separate test set (38 samples). R_p^2 (proportion of variation in the prediction set for each trait explained by the calibration) is shown in Figure 5. A comparison of RPD_p values obtained for each calibration is provided in Figure 6.

Predictions of pentosans and total lignin were consistently good regardless of which set of NIR spectra were used. Predictions of pentosans were strongest using the 0.65 m cores ($R_p^2 = 0.77$), while the whole-tree set pentosans calibration gave the weakest R_p^2 (0.67). R_p^2 for predicted lignin were lower than those obtained for pentosans (0.59 to 0.67) with the whole-tree calibration giving the strongest relationship and the 0.65 m cores the weakest. RPD_p values ranged from 1.63 (whole-tree) to 2.03 (0.65 m) for pentosans and 1.86 (whole-tree) to 1.67 (1.30 m) for total lignin. Predictions of the remaining properties were poor. The whole-tree NaOH calibration was the only calibration to give predictions with R_p^2 greater than 0.50 (0.58).

DISCUSSION

Pentosans and total lignin calibrations obtained using whole-tree data and NIR spectra obtained from the milled wood of whole-tree composite chips, and milled 0.65 and 1.30 m increment cores showed similar calibration and prediction statistics. The similarity between calibrations indicates that calibrations for the estimation of whole-tree properties can be obtained using increment cores obtained from a height that is easily accessible from the ground with little loss of predictive accuracy. RPD_p values for pentosans and total lignin ranged from 1.63 to 2.03 indicating that the calibrations would not be suitable for making accurate estimations of these properties, though the predictions would be suitable for ranking purposes.

For the nondestructive sampling of standing trees it is important that a suitable sampling height be identified. In this study two sampling heights were examined (0.65 and 1.30 m) and both gave similar results to calibrations obtained using whole-tree composite chip samples. Consideration of which of the two heights would be the most suitable for sampling is important. Generally the calibrations obtained using cores from 0.65 m gave slightly better calibration and prediction statistics than those for cores from 1.30 m but the differences were small, indicating that both heights could be used with confidence. Of the two heights 1.30 m is by far the most practical.

The basic density and specific consumption calibrations provided reasonable calibration statistics for all sets but performed poorly in prediction. The poor results of the calibrations when applied to the separate test set may be a consequence of the skewed distribution (towards lower density) of the samples. As basic density is an important factor in the determination of specific consumption this trait displayed similar behaviour. The correlation coefficient (r) between basic density and specific consumption was -0.87. Several strong relationships are reported in the literature for density (*26-29*) but these studies were based on solid wood not milled wood. In addition these studies did not attempt to estimate whole-tree values. Schimleck et al. (*30*) examined the use of NIR spectroscopy for the estimation of basic density of milled increment cores obtained from eight-year-old, plantation-grown *Eucalyptus globulus* Labill. subspecies *globulus* (Tasmanian blue gum) but found that predictive errors were too large for NIR spectroscopy to be used for this purpose. In a later study based on the milled wood of clonal *E. globulus* whole-tree composite samples, strong relationships were obtained for basic density (calibration $R^2 = 0.82$ and SEC = 22.9 kg/m^3, $R_p^2 = 0.75$ and SEP = 28.8 kg/m^3) (*31*). As far we are aware there are no examples of specific consumption calibrations reported in the literature but calibrations have been published for pulpwood productivity (where pulpwood productivity is equal to pulp yield multiplied by basic density), a comparable trait, and higher R^2 were obtained (*31*).

The pulp yield calibrations were disappointing, with statistics inferior to those reported in other studies (*8,12,13*). The pulp yield data used in this study had variable Kappa numbers (range = 16.7 to 18.8) and as a consequence the pulp yield data is not directly comparable between samples, possibly explaining the poor results for pulp yield. An attempt was made to adjust the pulp yield data to Kappa 18 but calibration statistics were not improved using adjusted data. This was hardly surprising as the samples used demonstrated wide genetic variation and would not demonstrate similar pulping behaviour. More work is required to investigate the development of whole-tree pulp yield calibrations using NIR spectra from increment cores.

The development of wood property calibrations using several different species and hybrids is important to Aracruz Celulose S.A. from an operational perspective because the evaluation of a wide range of genetic material is ongoing. If strong calibration statistics can be obtained for a diverse genetic population representing a wide range of sites then it may not be necessary to develop a new calibration when samples from a new species or different site are tested. While several studies have demonstrated that near infrared (NIR) spectroscopy can be used to determine several wood quality traits related to pulp production, rarely have multiple species or hybrids been included in the calibration set. This study indicates that multiple species or hybrids can be included in calibrations for wood properties, but the accuracy of the calibrations are sufficient only for ranking.

CONCLUSIONS

Calibrations were obtained for a range of wood quality traits using whole-tree data and NIR spectra obtained from milled whole-tree composite chip samples, and 5 mm diameter increment cores non-destructively sampled from 0.65 m and 1.30 m. The pentosans and total lignin calibrations displayed strong calibration and prediction statistics and were very similar for all sample sets. Calibrations for the remaining properties were weaker but similar, regardless of the set, indicating that calibrations based on increment cores can be used to estimate wood quality traits on a whole-tree basis.

Of the two heights used to obtain increment cores, 1.30 m was identified as the most suitable height for nondestructive sampling.

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