DETERMINATION OF IMPORTANT PULP PROPERTIES OF HYBRID POPLAR BY NEAR INFRARED SPECTROSCOPY

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ABSTRACT

Hybrid poplars are widely grown in the northwestern United States for manufacturing short fiber market pulp. Improvement of whole-tree basic density and pulp yield, important variables in the economics of pulp production, is an objective of tree breeding programs; but the number of trees analyzed is limited by expensive analytical methods. Near infrared (NIR) spectroscopy provides a rapid alternative, and in this study we investigate its ability to estimate poplar pulpwood properties. Whole-tree cellulose content and pulp yield calibrations, based on 3- and 6-year-old clones, were generally strong, while relationships were weaker for basic density. Breast height cores from 6-year-old clones gave a strong core cellulose content calibration. Cellulose content and pulp yield calibrations based on NIR spectra from milled increment cores and whole-tree data gave strong relationships for 6-year-old clones, indicating that the prediction of these properties, on a whole-tree basis, using breast height increment cores may be possible.

Keywords: Basic density, cellulose content, hybrid poplars, near infrared spectroscopy, pulp yield.

INTRODUCTION

Hybrid poplars are widely grown in the northwestern United States for the manufacture of short fiber market pulp and for an emerging solid wood market (Stanton et al. 2002). Boise Cascade Corporation (Boise) manages one of the largest plantation areas (currently 6,210 ha on three locations in the region), having established intensively managed fiber farms in eastern Oregon and Washington in 1991. Plantations are maintained in a stress-free condition including irrigation, pest and weed control, and fertilization. Rotation length is 6–7 years and harvest activities are ongoing year round. On average, 1,000 ha are planted each year. The trees are utilized to provide high quality, short fiber furnish to Boise Cascade Corporation's Wallula, Washington, Pulp and Paper Mill, where the chips are used in the manufacture of uncoated freesheet. The fiber farm poplar tree improvement program was started in 1993. Tree breeding programs, in general, have aimed to improve important pulpwood properties such as whole-tree basic density and pulp yield, both of which are important variables in the economics of pulp production (Borralho et al. 1993; Greaves et al. 1997; Dinus et al. 2001).

The determination of basic density and, in particular, pulp yield of whole-tree composite samples is extremely time-consuming and costly. Consequently, the number of samples that can be analyzed using traditional methods is limited making it impossible to screen large numbers of trees as is required for breeding programs. Several studies have demonstrated that near infrared (NIR) spectroscopy has the potential to provide a rapid, inexpensive method for estimating the pulp yield of plantation eucalypts and pines (Wright et al. 1990; Michell 1995; Olsson et al. 1995). Other studies have demonstrated that NIR spectroscopy can also be used to estimate basic density, though most studies have been based on solid wood or shavings (Thygesen 1994; Hoffmeyer and Pedersen 1995; Schimleck et al. 1999, 2001). Schimleck et al. (1999), used milled *Eucalyptus globulus* increment cores and found that the relatively high error of basic density estimates indicated that NIR spectroscopy was unsuitable for the accurate estimation of increment core basic density. No work is reported in the literature regarding NIR spectroscopy and the estimation of wood properties of clonal poplars.

Another important wood property is cellulose content, which is known to have a strong relationship with pulp yield (Dillner et al. 1971; Wallis et al. 1996; Kube and Raymond 2002) and has been used as a secondary standard for pulp yield in breeding programs. Several studies have shown that NIR spectroscopy can be used to predict cellulose content (Wright et al. 1990; Garbutt et al. 1992; Schimleck et al. 1997; Raymond and Schimleck 2002), and once a robust calibration has been established, a large number of samples can be quickly analyzed (Raymond and Schimleck 2002).

Bands in NIR spectra of wood arise from vibrations of chemical bonds in wood components such as cellulose and lignin. As the bands arise from wood constituents, changes in bands reflect changes in the chemistry of the wood. Spectra that occur in the NIR region (700–2500 nm) consist of overtone and combination bands of the fundamental stretching vibrations of O–H, N–H, and C–H functional groups (Osborne et al. 1993). NIR analysis relies on creating a calibration that relates NIR spectra of a large number of samples to a known constituent. The calibration is then used to predict the constituent of further samples based on their NIR spectra.

Another important consideration for tree breeding is nondestructive sampling. Often a breast height increment core is removed from a standing tree and used for the measurement of wood properties. Nondestructive sampling is particularly important as it preserves the sampled tree for future research if required, but it is important that the core adequately represents the whole tree (Evans et al. 2000). In a recent study based on eucalypt clones grown in Brazil (Schimleck et al. 2005), it was shown that NIR spectra from milled increment cores showed very strong relationships with whole-tree properties, indicating that NIR spectra collected from increment cores could be used to predict whole-tree wood properties. Therefore the aims of this study were to develop:

a. Whole-tree basic density, cellulose, and pulp yield calibrations for poplar clones (aged 3 and 6 years) using NIR spectra from milled whole-tree composite chips;
b. A cellulose calibration for poplar clones (aged 6 years) using NIR spectra from milled increment cores;
c. Whole-tree basic density, cellulose, and pulp yield calibrations for poplar clones (aged 6 years) using NIR spectra from milled increment cores; and
d. Whole-tree basic density, cellulose, and pulp yield calibrations for poplar clones using NIR spectra from milled whole-tree composite chips (aged 3 years) and data from 6-year-old clones.

METHODS

Poplar samples

The following samples were used in this study:

1. Fifteen poplar clones aged 3 years;
2. Twenty poplar clones aged 6 years (the same clones as the 3-year-olds, sampled 3 years later). Note: chip samples from 5 3-year-old clones were not available for this study; and
3. Twenty breast height 12-mm increment cores from the 6-year-old clones.

For each clone sampled, a whole-tree composite chip sample was obtained. From each existing whole-tree composite sample, a subsample (approximately 250 g) was removed for NIR analysis. Table 1 provides a statistical summary of basic density, cellulose, and pulp yield for each set.

Sample preparation

All samples were milled in a Wiley mill through a 1-mm screen (approximately an 18-mesh screen) for 3 min.

Determination of cellulose content

Cellulose content (g cellulose per g o.d. wood) was determined for all samples using the method of Wallis et al. (1997). Non-cellulosic compounds were solubilized by digestion in diglyme (10 mL) and hydrochloric acid (2 mL, 10 M) for 1 h on a shaker table in a water bath at 90°C. The residue was collected by filtration, washed, dried, and weighed to determine the mass of crude cellulose.

TABLE 1. *Summary statistics for basic density, cellulose content, and pulp yield for the 3- and 6-year-old whole-tree composite samples and the 6-year-old breast height cores.*

Wood property	Max.	Min.	Av.	Std. dev.
3-year-old (whole-tree)				
Basic density (kg/m^3)	346	287	317.4	12.6
Cellulose (%)	44.9	41.8	43.3	0.9
Pulp yield (%)	56.2	51.8	54.3	1.3
5-year-old (whole-tree)				
Basic density (kg/m^3)	325	266	296.8	15.6
Cellulose (%)	47.6	44.0	45.9	1.1
Pulp yield (%)	57.7	52.7	55.5	1.4
6-year-old (cores)				
Cellulose (%)	47.7	43.1	45.3	1.4

Determination of pulp yield

The following two series of cooks were performed on whole-tree composite samples obtained from each poplar clone:

1. 14% active alkali on wood with an H-factor of 900 at 170°C (maximum cooking temperature); and
2. 15% active alkali on wood with an H-factor of 625 at 170°C (maximum cooking temperature).

Total liquid to wood ratio was 4/1 with 30% sulfidity on active alkali for all cooks performed. Pulp yields were adjusted to a target Kappa number of 18 using a 0.2% yield correction per Kappa unit (R. Lowe, Econotech, pers. comm. 2004). The total pulp yield at an H-factor of 900 was fairly similar to that from the 625 H-factor cooks.

Determination of basic density

Basic wood density was measured in duplicate on screened composite chip samples using Tappi method T 258. Obvious knots were removed before analysis.

Measurement of NIR spectra and spectral manipulation

Each milled sample was mixed and a sample removed and placed in a NIRSystems large sample cup (NR-7070). 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 and repacked and a duplicate spectrum was obtained. The duplicate spectra were averaged and converted to the second derivative mode using NSAS® software (version 4.52) (NIRSystems, Silver Spring, MD). A segment width of 10 nm and a gap width of 20 nm were used for the conversion.

Calibration development

All calibrations were developed using NSAS® (version 4.52) and second derivative spectra. Partial Least Squares (PLS) regression was used for the calibrations with full cross validation (i.e. leave-one-out) and a maximum of ten factors. The software recommended the final number of factors for each calibration unless otherwise indicated. Owing to the small number of samples in individual sets, calibrations were initially created using all samples and not tested in prediction. The following calibrations were created:

1. Basic density, cellulose, and pulp yield (3-year-old whole-tree composite chips);
2. Basic density, cellulose, and pulp yield (6-year-old whole-tree composite chips);
3. Cellulose (increment cores from 6-year-old clones);
4. Basic density, cellulose, and pulp yield (3-year-old whole-tree composite chips) and whole-tree wood property data from 6-year-old clones; and
5. Basic density, cellulose, and pulp yield (increment cores from 6-year-old clones) and whole-tree wood property data from 6-year-old clones.

To investigate the predictive ability of basic density, cellulose, and pulp yield calibrations for clonal poplars, the 3- and 6-year old whole-tree samples were combined to give a total of 35 samples. The samples were then split at random into a calibration (23 samples) and prediction set (12 samples).

Calibration statistics

The Standard Error of Calibration (SEC) (determined from the residuals of the final calibration) and the coefficient of determination (R^2) were used to assess calibration performance. The SEC is given by the following formula:

$$\mathrm{SEC} = \sqrt{\frac{\sum_{i=1}^{NC}(\hat{y}_i - y_i)^2}{(NC - k - 1)}} \qquad (1)$$

where $\hat{y}_i$ is the value of the constituent of interest for validation sample i estimated using the calibration, y_i is the known value of the constituent of interest of sample i, NC is the number of samples used for calibration, and k is the number of factors for calibration (NIRSystems Inc. 1990; Workman 1992).

The Standard Error of Prediction (SEP) was used to give a measure of how well a calibration predicts the parameter of interest for a set of unknown samples different from the calibration set. The SEP is given by:

$$\mathrm{SEP} = \sqrt{\frac{\sum_{i=1}^{NP}(\hat{y}_i - y_i)^2}{(NP - 1)}}, \qquad (2)$$

where $\hat{y}_i$ is the value of the constituent of interest for sample i predicted by the calibration, y_i is the known value of the constituent of interest for sample i, and NP is the number of samples in the prediction set (NIRSystems Inc. 1990; Workman 1992).

RESULTS AND DISCUSSION

Cellulose and pulp yield relationships

The relationship between whole-tree pulp yield and whole-tree cellulose was good for the 3-year-old clones (coefficient of determination (R^2) = 0.75). The 6-year-old clones demonstrated a stronger relationship between whole-tree pulp yield and cellulose (R^2 = 0.82) (Fig. 1) than the 3-year-old clones.

NIR Spectroscopy of poplar clones

Variation in the second derivative NIR spectra of whole-tree 6-year-old clonal poplar samples

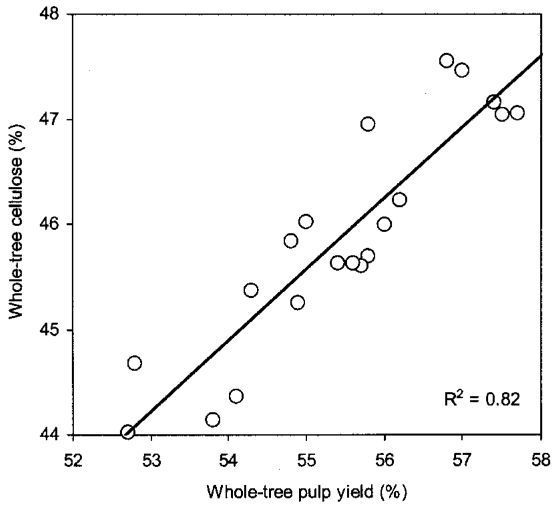

FIG. 1. Relationship between whole-tree cellulose content and whole-tree pulp yield for 6-year-old clonal poplar.

having different pulp yields (53.8% and 57.7%) is shown in Fig. 2. The wavelength range was limited to 1350 to 2150 nm to aid interpretation. Minor variation, owing to small differences in wood chemistry (Table 1) can be observed in the spectra. In the region shown, several bands have been assigned to cellulose (1490, 1780, 1820, 1900, 1930, and 2100 nm) and lignin and extractives (1668, 1685, 2132 nm) (Shenk et al. 1992; Osborne et al. 1993; Michell and Schimleck 1996).

Standard linear regression was used to examine the relationship between the measured properties and second derivative absorbance at individual NIR wavelengths for the 3- and 6-year-old whole-tree samples and the 6-year-old breast height cores. Cellulose content of the 6-year-old cores had strong relationships with bands at 1492 nm (r = –0.92), 1688 nm (r = 0.90), 2106 nm (r = 0.90) and 2358 nm (r = –0.93). For the 6-year-old whole-tree chip samples, cellulose had its strongest relationships at 1688 nm (r = 0.89) and 1608 nm (r = –0.86). Pulp yield had relationships similar to cellulose, with the band at 1690 nm (r = 0.88) giving the strongest correlation coefficient. For basic density, the band at 1890 nm gave the strongest relationship (r = 0.65). For the 3-year-old whole-tree chip samples, the strongest correlation for cellulose was at 1604 nm (r = –0.82), pulp yield at 1248 nm (r = –0.84) and basic density at 1208 nm (r = –0.79).

NIR diffuse reflectance spectra (second derivative) of the same clone when aged 3 years and 6 years were also examined. The NIR spectrum of a 3-year-old clone was different from that of a 6-year-old clone indicating a change in wood chemistry between the ages of 3 and 6 years, i.e. an increase in cellulose content. Most of the variation was limited to the regions from 1620–1720 nm and 2050–2150 nm.

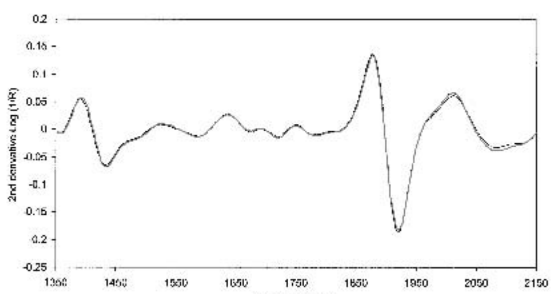

FIG. 2. Second derivative NIR diffuse reflectance spectra of two clonal hybrid poplar samples over the wavelength range 1350–2150 nm. Samples having relatively high (thick, light line) and low (thin, dark line) pulp yields are shown.

Basic density, cellulose, and pulp yield PLS calibrations

PLS calibrations for each sample set (3- and 6-year-old whole-tree and 6-year-old increment core) are summarized in Table 2. Cellulose gave the strongest calibrations (R^2 = 0.86) for the 3-year-old clones. The R^2 was moderate for pulp yield (0.62), while the R^2 for basic density (0.48) was weak. The 6-year-old whole-tree samples gave excellent calibrations for cellulose (Fig. 3a) and pulp yield (Fig. 3b). The basic density calibration was also improved with an R^2 of 0.64 (3 factors). The cellulose calibration for breast height increment cores from 6-year-old clones also gave very good calibration statistics (R^2 = 0.96, SEC = 0.31).

TABLE 2. *Summary statistics for the basic density, cellulose, and pulp yield PLS calibrations obtained using 3- and 6-year-old whole-tree clonal poplar samples and the 6-year-old clonal poplar breast height cores.*

Sample Set	Parameter	No. factors	R^2	SEC
Whole-tree	Basic density (kg/m^3)	2	0.48	9.8
3-years-old	Cellulose (%)	4	0.86	0.40
	Pulp yield (%)	2	0.62	0.83
Whole-tree	Basic density (kg/m^3)	3	0.64	10.2
6-years-old	Cellulose (%)	5	0.94	0.31
	Pulp yield (%)	5	0.96	0.35
Cores (6 yrs)	Cellulose (%)	5	0.96	0.31

Prediction of basic density, cellulose, and pulp yield

To investigate the predictive ability of clonal poplar calibrations, the 3- and 6-year old whole-tree chip samples were combined to give a total of 35 samples. The samples were then split at random into a calibration (23 samples) and prediction set (12 samples). Calibration and prediction results for basic density, cellulose, and pulp yield are reported in Table 3. Strong calibration statistics were obtained for cellulose and pulp yield while they were poor for basic density. When used to predict properties of prediction set samples, the cellulose and pulp yield calibrations provided moderate relationships. Prediction errors were higher than calibration errors, particularly for pulp yield.

Calibrations for estimating 6-year-old wood properties based on 3-year-old samples

PLS calibrations for each wood property were developed using NIR spectra from 3-year-old whole-tree samples and data obtained from the same clones when 6-years-old (Table 4). Moderate relationships were obtained for cellulose (R^2 = 0.61) and pulp yield (R^2 = 0.74) indicating that it may be possible to estimate wood properties at age 6 using wood from 3-year-old clones. The relationship for basic density was poor.

Calibrations for the estimation of whole-tree properties based on increment cores

PLS calibrations for 6-year-old whole-tree basic density, cellulose, and pulp yield were developed using NIR spectra from increment cores of 6-year-old clones (Table 5). The cellulose content and pulp yield calibrations are plotted in Figs. 4a and 4b. Strong statistics were obtained for all properties (R^2 ranged from 0.84 to 0.90). The cellulose and pulp yield calibrations had weaker statistics than those reported for the whole-tree composite samples (Table 2). This could be expected considering the relationship (R^2 = 0.65) that exists between core cellulose and whole-tree cellulose (Fig. 5) and that the two sample sets are inherently different, i.e. one set represents the whole-tree, while the other represents only a small amount of wood taken from breast height. The basic density calibration based on increment core NIR spectra and whole-tree data had stronger statistics than the corresponding calibration reported in Table 2. Six factors were used for the basic density calibration (Table 5), if 3 factors were used (equivalent to the Table 2 basic density calibration), an R^2 of 0.34 and a SEC = 13.8 kg/m^3 was obtained.

Cellulose and pulp yield calibrations obtained using NIR diffuse reflectance spectra from the milled wood of whole-tree clonal poplar provided strong statistics, while calibrations for basic density were weaker. Breast height cores from 6-year-old clones also gave a strong cellulose calibration. These findings are in agreement with other studies that have reported strong calibration statistics for cellulose content (Wright et al. 1990; Garbutt et al. 1992; Schimleck et al. 1997; Raymond and Schimleck 2002) and pulp yield (Wright et al. 1990; Michell 1995; Olsson et al. 1995) and weaker results for basic density using milled wood (Schimleck et al. 1999). The strength of the whole-tree cellulose and pulp

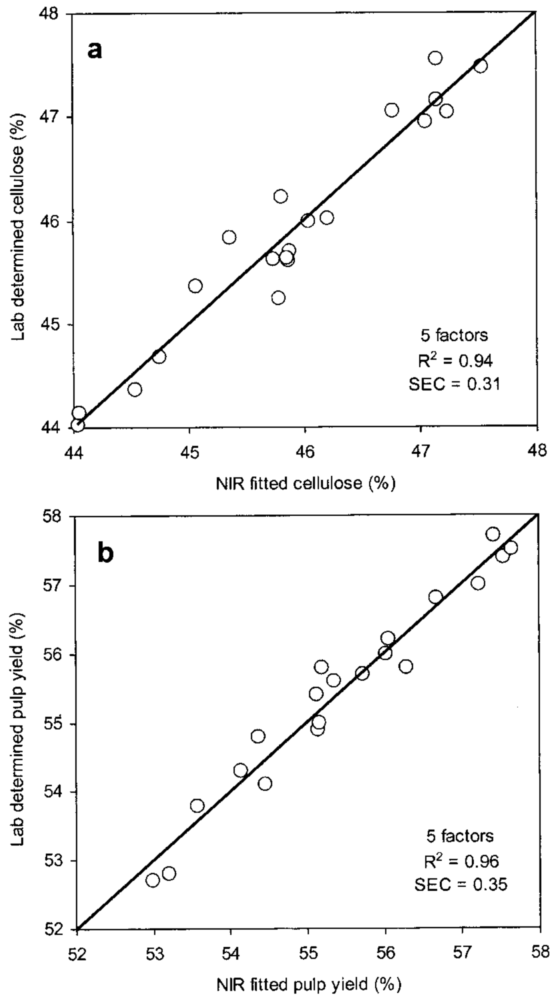

FIG. 3. Relationships between laboratory-determined whole-tree values and NIR fitted whole-tree values for (a) cellulose content and (b) pulp yield. The relationships shown are for 6-year-old clonal poplar samples. The regression line has been plotted through the origin in both figures.

yield calibrations indicates that NIR spectroscopy could be used for the rapid estimation of cellulose content and pulp yield in poplar breeding programs, but the use of larger sample sets of a given age is required to fully assess the ability of NIR spectroscopy to estimate wood properties of clonal poplars.

Cellulose content was included in this study as a possible alternative to kraft pulp yield because the two properties are closely related (Dillner et al. 1971; Wallis et al. 1996; Kube and Raymond 2002). For a breeding program that aims to increase pulp yield, using cellulose as an alternative presents several advantages including; lower cost, more trees can be sampled, and samples can be taken without falling the tree (Kube and Raymond 2002). In this study it was found that the relationships between whole-tree cellulose and whole-tree pulp yield were good for clonal poplars aged 3 and 6 years. The strong relationships indicate that poplar clones having relatively high cellulose content will also have relatively high pulp yields. Therefore whole-tree cellulose content has the potential to be used as a rapid, inexpensive surrogate for pulp yield in poplar clones aged 3 or 6 years. It is probable that the diglyme method underestimated the cellulose content of the poplar samples analyzed in

TABLE 3. *Summary statistics for the basic density, cellulose, and pulp yield PLS calibrations obtained using the 3- and 6-year-old whole-tree clonal poplar sample sets combined. Calibrations were created using 23 samples and tested on the remaining 12 samples.*

Parameter	# of factors	R^2	SEC	R_p^2	SEP
Basic density	1	0.24	15.5	0.21	17.2
Cellulose	3	0.84	0.73	0.64	0.96
Pulp yield	5	0.93	0.47	0.56	0.90

TABLE 4. *Summary statistics for the basic density, cellulose, and pulp yield PLS calibrations obtained using 3-year-old whole-tree clonal poplar samples and 6-year-old data determined using whole-tree composite chips.*

Parameter	No. factors	R^2	SEC
Basic density (kg/m^3)	1	0.06	17.4
Cellulose (%)	3	0.61	0.71
Pulp yield (%)	3	0.74	0.81

TABLE 5. *Summary statistics for the basic density, cellulose, and pulp yield PLS calibrations obtained using 6-year-old increment core clonal poplar samples and 6-year-old data determined using whole-tree composite chips.*

Parameter	No. factors	R^2	SEC
Basic density (kg/m^3)	6*	0.84	7.7
Cellulose (%)	5	0.89	0.41
Pulp yield (%)	6	0.90	0.55

* Note the NSAS® software recommended 8 factors for the basic density calibration but this was considered excessive.

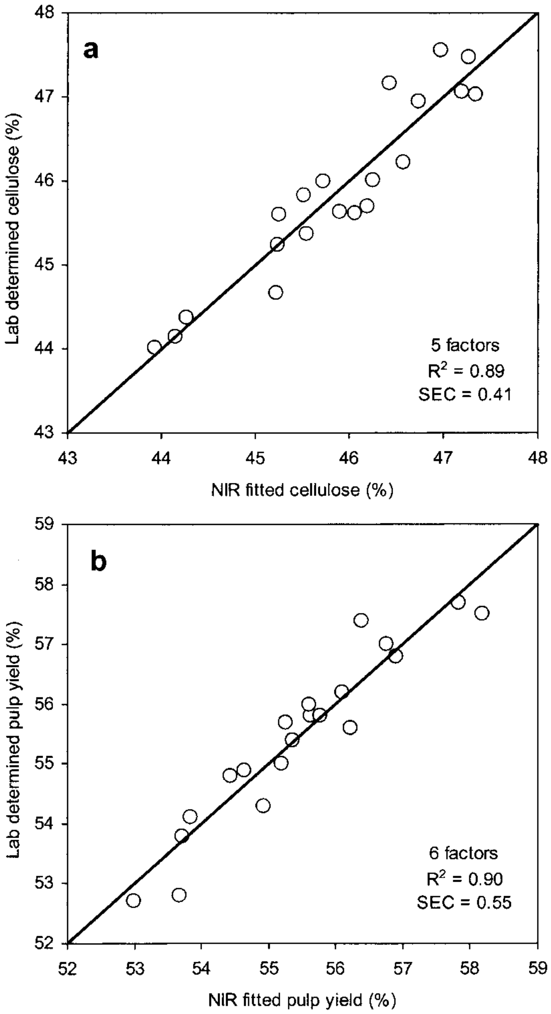

FIG. 4. Relationship between laboratory determined whole-tree values and NIR fitted whole-tree values for (a) cellulose content and (b) pulp yield. Breast height cores were used for calibration. The regression line has been plotted through the origin in both figures.

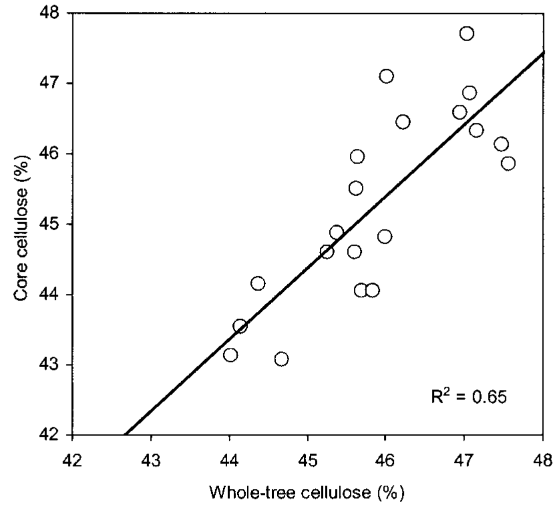

FIG. 5. Relationship between core cellulose content and whole-tree cellulose content for 6-year-old clonal poplar.

this study. For an absolute measure of cellulose content, we recommend that high performance anion exchange chromatography (HPAEC) be used.

An important aspect of a clonal breeding program is the age at which clones can be analyzed for selection purposes. If clones are selected at too young an age, it is possible that by the time trees reach maturity they will no longer have the most desirable properties. Studies of age-age correlations in poplar indicate that early selection should be possible (Chantre and Cahalan 2001). A potential application of NIR spectroscopy, therefore, could be to test clonal material when young with the aim of estimating the wood properties of the clone when older. The samples used in this study provided an opportunity to investigate such calibrations. Using NIR spectra from 3-year-old whole-tree samples and data obtained from the same clones when 6-year-old calibrations were obtained for basic density, cellulose, and pulp yield. Cellulose and pulp yield calibrations provided moderate relationships indicating that it may be possible to estimate these wood properties at age 6 using wood from 3-year-old clones. The relationship for basic density was poor. The success of the calibrations depends on relationships between the 3- and 6-year-old data. The relationships between basic density, cellulose, and pulp yield were weak to moderate (R^2 = 0.34, 0.61, and 0.64, respectively). The weak relationship for basic density may explain why it had poor calibration statistics.

Another important aspect of tree breeding programs is nondestructive sampling of standing trees. Increment cores are often removed from breast height for this purpose, but it is important that the core represents the whole-tree. For clonal poplars, the relationship between core cellulose content and whole-tree cellulose content at age 6 was good ($R^2 = 0.65$), while the relationship between core cellulose content and whole-tree pulp yield was weaker ($R^2 = 0.58$). One sample had a large negative influence on the core cellulose whole-tree pulp yield relationship and if removed the R^2 improved to 0.76. Removal of this sample also improved the core cellulose whole-tree cellulose relationship ($R^2 = 0.71$).

Potentially whole-tree properties could be estimated with calibrations developed using NIR spectra collected from increment cores and whole-tree data (Schimleck et al. 2005). If whole-tree properties could be estimated in this way, it would negate the need to destructively sample large numbers of trees. When this was investigated in this study, strong calibration statistics were obtained for cellulose and pulp yield (Table 5) indicating that it may be possible to predict these properties on a whole-tree basis in clonal poplar using breast height increment cores.

CONCLUSIONS

Whole-tree cellulose content and pulp yield calibrations, based on NIR diffuse reflectance spectra from the milled wood of 3- and 6-year-old clones, were generally strong, while relationships were weaker for basic density. Breast height cores from 6-year-old clones gave a strong calibration for core cellulose content.

The relationship between whole-tree pulp yield and whole-tree cellulose content was good for the 3- and 6-year old clones.

Whole-tree cellulose and pulp yield calibrations obtained with NIR spectra from milled breast height cores gave strong relationships. Relationships between core cellulose content, whole-tree cellulose content, and whole-tree pulp yield were moderate.

Whole-tree cellulose and pulp yield calibrations obtained with NIR spectra from milled whole-tree chips (3-years-old) and 6-year-old data gave moderate relationships.

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