

# Nondestructive estimation of *Pinus taeda* L. wood properties for samples from a wide range of sites in Georgia

P.D. Jones, L.R. Schimleck, G.F. Peter, R.F. Daniels, and A. Clark III

**Abstract:** Preliminary studies based on small sample sets show that near infrared (NIR) spectroscopy has the potential for rapidly estimating many important wood properties. However, if NIR is to be used operationally, then calibrations using several hundred samples from a wide variety of growing conditions need to be developed and their performance tested on samples from new populations. In this study, 120 *Pinus taeda* L. (loblolly pine) radial strips (cut from increment cores) representing 15 different sites from three physiographic regions in Georgia (USA) were characterized in terms of air-dry density, microfibril angle (MFA), and stiffness. NIR spectra were collected in 10-mm increments from the radial longitudinal surface of each strip and split into calibration (nine sites, 729 spectra) and prediction sets (six sites, 225 spectra). Calibrations were developed using untreated and mathematically treated (first and second derivative and multiplicative scatter correction) spectra. Strong correlations were obtained for all properties, the strongest  $R^2$  values being 0.83 (density), 0.90 (MFA), and 0.93 (stiffness). When applied to the test set, good relationships were obtained ( $R_p^2$  ranged from 0.80 to 0.90), but the accuracy of predictions varied depending on math treatment. The addition of a small number of cores from the prediction set (one core per new site) to the calibration set improved the accuracy of predictions and importantly minimized the differences obtained with the various math treatments. These results suggest that density, MFA, and stiffness can be estimated by NIR with sufficient accuracy to be used in operational settings.

**Résumé :** Des études préliminaires basées sur de petits groupes d'échantillons montrent que la spectroscopie dans le proche infrarouge offre la possibilité d'estimer rapidement plusieurs propriétés importantes du bois. Cependant, pour utiliser cette méthode sur une base opérationnelle, il faut établir des courbes de calibration à l'aide de plusieurs centaines d'échantillons représentatifs d'une grande variété de conditions de croissance et tester leur performance avec des échantillons provenant de nouvelles populations. Dans cette étude, 120 bandes radiales (coupées à partir de carottes) de *Pinus taeda* L. (pin à encens) provenant de 15 sites différents dans trois régions physiographiques de la Georgie (É.-U.) ont été caractérisées sur la base de la masse volumique séchée à l'air, de l'angle des microfibrilles (AMF) et de la rigidité. Les spectres dans le proche infrarouge ont été obtenus à tous les 10 mm le long de la surface radiale de chaque bande et divisés en deux groupes : un groupe pour établir les courbes de calibration (neuf sites, 729 spectres) et un autre groupe pour tester les prédictions (six sites, 225 spectres). Les courbes de calibration ont été élaborées avec des spectres non traités ou traités mathématiquement (première et seconde dérivée et correction de dispersion multiplicative). De fortes corrélations ont été obtenues pour toutes les propriétés, les  $R^2$  les plus élevés atteignant 0,83 (masse volumique), 0,90 (AMF) et 0,93 (rigidité). Les auteurs ont obtenu de bonnes relations ( $R_p^2$  variait de 0,80 à 0,90) avec le groupe de spectres destinés à tester les prédictions mais l'exactitude des prédictions variait selon le traitement mathématique. L'ajout d'un petit nombre de carottes provenant du groupe de spectres destinés à tester les prédictions (une carotte par nouveau site) au groupe de spectres utilisés pour établir les courbes de calibration a amélioré l'exactitude des prédictions et passablement minimisé les différences obtenues avec les divers traitements mathématiques. Ces résultats indiquent que la spectroscopie dans le proche infrarouge permet d'estimer la masse volumique, l'AMF et la rigidité avec suffisamment d'exactitude pour être utilisée de façon opérationnelle.

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## Introduction

Plantation-grown *Pinus taeda* L. (loblolly pine) is the major source of wood fiber and solid wood products in the southern United States. The deployment of genetically improved planting stock coupled with intensive silvicultural management has greatly improved the growth and yields of *P. taeda*. Li et al. (1999) report that *P. taeda* trees grown from seeds obtained from first-generation seed orchards produced 7%–12% more volume per acre at harvest than trees grown from wild seed, while for second-generation seed orchards it is estimated that gains in volume would be 13%–21%. It is expected that the inclusion of quality traits such as stem straightness and wood quality in *P. taeda* breeding programs will greatly increase genetic gains and the value of the trees (Li et al. 1999). However, traditional methods of measuring wood properties are slow and cost prohibitive for measuring these properties in the large number of trees needed for breeding programs. Also, the measurement of modulus of elasticity and modulus of rupture are destructive, so if a tree is determined to have traits that are desirable for inclusion in future generations, the tree has already been destroyed and is therefore no longer available for crossing. Thus, rapid, nondestructive methods for measuring wood properties must be developed.

Near infrared (NIR) spectroscopy has the potential to provide the forestry industry with a rapid tool for the nondestructive estimation of wood properties. Several studies (Birkett and Gambino 1988; Wright et al. 1990; Garbutt et al. 1992; Michell 1995; Schimleck et al. 2000; Raymond and Schimleck 2002) have used NIR spectroscopy to estimate chemical properties of wood. Studies have also shown that NIR spectroscopy can be used to estimate the physical-mechanical properties of wood, including density and stiffness (Thygesen 1994; Hoffmeyer and Pedersen 1995; Schimleck et al. 1999; Gindl et al. 2001; Thumm and Meder 2001; Meder et al. 2003). Recently, it has been shown that reasonable calibrations can be created for green *Pinus taeda* L. (loblolly pine) samples for air-dry density, microfibril angle (MFA), and stiffness (Schimleck et al. 2003).

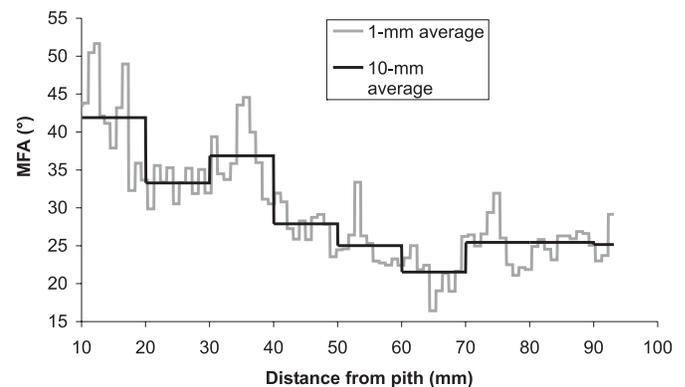
The successful application of NIR spectroscopy relies on the provision of accurate calibration data for large numbers of samples. Several recent NIR spectroscopic studies (Schimleck et al. 2002; Schimleck and Evans 2002a, 2002b, 2003; Schimleck et al. 2003) have used data provided by the SilviScan instruments for calibration purposes. The SilviScan instruments, developed by CSIRO's Forestry and Forest Products Division, use X-ray densitometry, diffractometry, and image analysis to accurately measure several wood properties at high spatial resolution.

Strong relationships between SilviScan and NIR estimates for a range of wood properties including density, MFA, and stiffness have been obtained for *Eucalyptus delegatensis* R.T. Baker (alpine ash) and *Pinus radiata* D. Don (radiata pine) samples (Schimleck et al. 2001; Schimleck et al. 2002). Studies based on radial wooden strips cut from selected *P. radiata* increment cores have shown promising results for air-dry density, MFA, stiffness (determined using SilviScan-2 diffractometric data and measured density), and several tracheid morphological characteristics (Schimleck and Evans

**Table 1.** Stand descriptions for samples used in the calibration and prediction sets including physiographic region, site index ( $SI_{25}$ ), age, latitude (lat.), and longitude (long.).

Region	$SI_{25}$	Age (years)	Lat. (N)	Long. (W)
<b>Calibration set</b>				
Upper Atlantic Coastal Plain	67	24	32°33'	84°60'
Upper Atlantic Coastal Plain	69	25	32°78'	82°97'
Upper Atlantic Coastal Plain	78	22	31°62'	84°99'
Lower Atlantic Coastal Plain	76	25	32°26'	81°27'
Lower Atlantic Coastal Plain	79	25	31°31'	81°60'
Lower Atlantic Coastal Plain	84	25	31°11'	81°81'
Piedmont	54	25	33°70'	83°22'
Piedmont	55	21	34°09'	85°22'
Piedmont	61	26	32°96'	83°62'
<b>Prediction set</b>				
Upper Atlantic Coastal Plain	55	22	32°13'	84°70'
Upper Atlantic Coastal Plain	67	24	32°34'	84°60'
Lower Atlantic Coastal Plain	80	25	31°42'	81°68'
Lower Atlantic Coastal Plain	81	24	31°79'	81°57'
Piedmont	54	24	32°77'	84°61'
Piedmont	66	26	33°46'	83°10'

**Fig. 1.** SilviScan microfibril angle (MFA) data for a single sample from the calibration data set at both the original 1-mm resolution and at 10-mm resolution (averaged).



2002a, 2002b, 2003, 2004). Calibrations performed well when applied to a separate test set of two cores that were from the same population as the calibration samples. However, in general, the studies showing the utility of NIR to estimate wood properties have been based on small sample sets with limited variation. As with any statistical procedure, it is expected that with a larger sample size more of the population variation will be captured within the calibration, but the difficulty arises in determining whether enough variation has been captured by the selected samples. The use of large sample sizes in agricultural applications indicates that calibrations based on large sample sets are more robust (Berzaghi et al. 2002).

*Pinus taeda* is grown on a wide variety of sites in the southern United States, and it has been shown that geographic regions within the southern United States have differing wood properties (Zobel and McElwee 1958; Talbert and Jett 1981). In Georgia, *P. taeda* is grown within three distinct

**Table 2.** Summary of wood of the variation in wood properties of the samples used for the calibration and prediction sets.

Wood property	Calibration set (729 spectra)				Prediction set (225 samples)			
	Min.	Max.	Avg.	SD	Min.	Max.	Avg.	SD
Air-dry density (kg·m <sup>-3</sup> )	337.47	832.55	577.16	98.73	378.02	811.21	575.95	104.39
MFA (°)	10.98	45.21	26.64	7.42	9.67	39.58	24.40	7.32
SilviScan stiffness (GPa)	2.35	23.04	9.60	4.89	2.70	23.21	10.14	5.38

**Table 3.** Summary of density, microfibril angle (MFA), and stiffness calibrations developed using untreated and math-treated near infrared spectra collected from the radial longitudinal face of radial wooden strips included in the calibration set.

Wood property	Math treatment	Calibration				
		Factors	R <sup>2</sup>	SEC	SECV	RPD <sub>c</sub>
Density (kg·m <sup>-3</sup> )	Raw	6	0.82	42.33	43.26	2.28
	MSC	5	0.83	41.22	42.94	2.30
	First derivative	5	0.82	42.05	43.26	2.28
	Second derivative	2	0.78	46.01	46.89	2.11
MFA (°)	Raw	8	0.89	2.43	2.51	2.95
	MSC	8	0.90	2.33	2.38	3.11
	First derivative	5	0.89	2.42	2.48	3.00
	Second derivative	6	0.89	2.42	2.63	2.83
Stiffness (GPa)	Raw	8	0.93	1.27	1.34	3.65
	MSC	5	0.91	1.48	1.53	3.19
	First derivative	5	0.92	1.36	1.40	3.50
	Second derivative	6	0.92	1.39	1.52	3.22

**Note:** Calibration statistics: R<sup>2</sup>, coefficient of determination; SEC, standard error of calibration; SECV, standard error of cross-validation; RPD<sub>c</sub>, ratio of performance to standard deviation. MSC, multiplicative scatter correction.

regions (Upper and Lower Coastal Plain, and Piedmont). Ongoing research conducted by the Wood Quality Consortium (WQC) at the University of Georgia (Athens, Georgia, USA) is investigating how the wood properties of each geographic region differ. Thus, an important test of NIR for wood-property estimation is how well general calibrations developed from wood samples obtained from trees growing in each geographic region predict wood properties from these regions. Therefore the objectives of this study are (i) to create wood-property calibrations (air-dry density, MFA, stiffness) using samples drawn from a wide variety of sites chosen to represent the three physiographic regions where *P. taeda* is grown; (ii) to examine the performance of the wood-property calibrations when applied to samples from sites not included in the calibration; (iii) to investigate methods for improving the applicability of wood property calibrations to samples from sites not initially included in the calibration set.

## Materials and methods

### Sample origin

Samples were collected from *P. taeda* plantations located in Georgia, USA. For each of the three physiographic regions in Georgia (Lower and Upper Atlantic Coastal Plain, and Piedmont) five plantations, ranging in age from 21 to 26 years, with a range of site indices were sampled, to represent trees presently being harvested as mature. The sites are summarized in Table 1. The calibration set comprised 90 breast-height (1.37 m) increment cores sampled from three

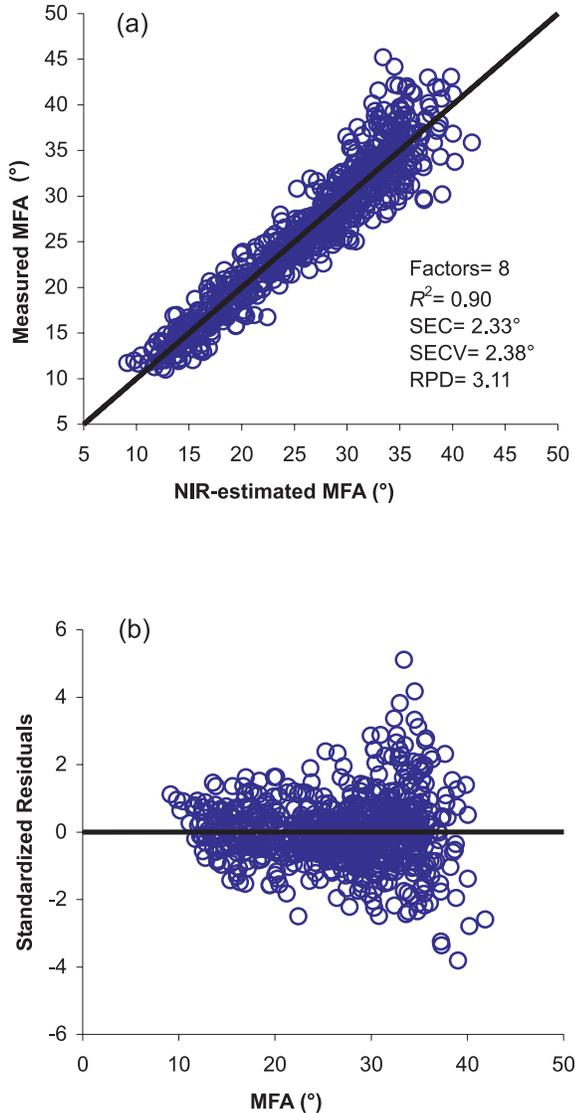
plantations within each region; 10 increment cores per site were taken. One increment core developed blue stain and was unavailable for analysis because of discoloration, leaving 89 cores for analysis. The prediction set comprised 30 breast-height increment cores (5 cores per plantation, 2 plantations per region).

### SilviScan analysis

A total of 119 radial wooden strips were available for SilviScan analysis; these strips were cut from increment cores using a twin-blade saw. Strip dimensions were 2 mm tangentially and 7 mm longitudinally; radial length was determined by the pith-to-bark length of the sample. The radial strips were extracted in warm acetone for 24 h prior to SilviScan analysis.

Wood properties were measured using SilviScan-1 and -2 (Evans 1994, 1997, 1999). Air-dry density (subsequently referred to as density) was measured in 50- $\mu$ m steps using X-ray densitometry. MFA was averaged over 1-mm intervals on SilviScan-2 using scanning X-ray diffractometry. An estimate of wood stiffness (same resolution as MFA) was obtained by combining X-ray densitometry and X-ray diffraction data. All measurements were made in a controlled environment of 40% relative humidity and a temperature of 20 °C. The high-resolution SilviScan data were averaged over 10-mm sections from pith to bark for correlation with the NIR spectra. The original data at 1-mm resolution SilviScan and the 10-mm averaged data are illustrated in Fig. 1 for a single radial strip used in the calibration. While the decrease in resolution

**Fig. 2.** Relationship between measured microfibril angle (MFA) and near infrared (NIR)-estimated MFA (a) and standardized residuals (b). Multiplicative scatter correction treated spectra were used for calibration. SEC, standard error of calibration; SECV, standard error of cross-validation;  $RPD_c$ , ratio of performance to standard deviation.



from 1 mm to 10 mm does not show MFA variation within rings, the 10-mm data follows the pith-to-bark trend.

#### Near infrared spectroscopy

Diffuse reflectance NIR spectra were collected from the radial longitudinal face of each strip using a NIRSystems Inc. Model 5000 scanning spectrometer. All measurements were made in a controlled environment of 40% relative humidity and a temperature of 20 °C. Samples were conditioned in this environment for no less than 1 week prior to scanning. Samples were held in a custom-made holder (Schimleck et al. 2001). A 5 mm × 10 mm mask was used to ensure that an area of constant size was analyzed. The spectra were collected at 2-nm intervals over the wavelength range 1100–2500 nm. The instrument reference was a ceramic standard.

**Table 4.** Summary statistics for density, microfibril angle (MFA), and stiffness, predicted using the original calibration set.

Wood property	Math treatment	Prediction		
		$R_p^2$	SEP	$RPD_p$
Density ( $\text{kg}\cdot\text{m}^{-3}$ )	Raw	0.82	55.15	1.89
	MSC	0.83	119.26	0.88
	First derivative	0.82	45.82	2.28
	Second derivative	0.81	50.80	2.05
MFA (°)	Raw	0.81	4.54	1.61
	MSC	0.84	3.12	2.34
	First derivative	0.83	7.22	1.01
	Second derivative	0.80	5.27	1.39
Stiffness (GPa)	Raw	0.90	2.47	2.18
	MSC	0.89	4.58	1.18
	First derivative	0.90	4.61	1.17
	Second derivative	0.87	2.45	2.20

**Note:** Prediction statistics:  $R_p^2$ , coefficient of determination; SEP, standard error of prediction;  $RPD_p$ , ratio of performance to deviation. MSC, multiplicative scatter correction.

Fifty scans were accumulated for each 10-mm section; these scans were averaged to give a single spectrum per section. A total of 729 spectra were collected from the 89 radial strips representing the calibration set, and 225 spectra were collected from the 30 radial strips representing the prediction set. Table 2 contains the summary statistics for the two data sets.

#### Wood-property calibrations

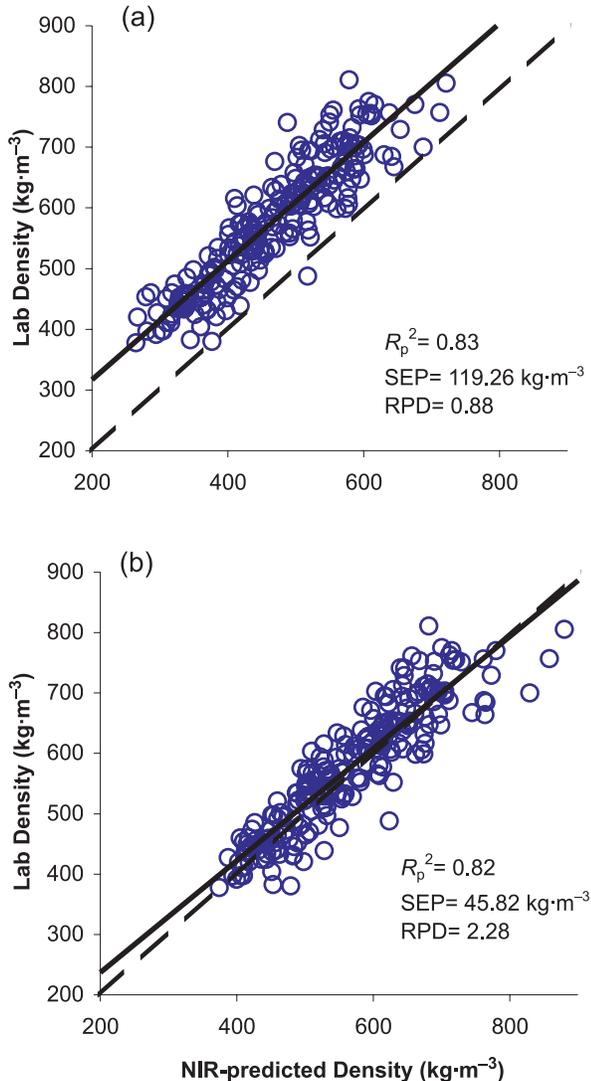
Wood property calibrations were developed using the Unscrambler (version 8.0) software package (Camo AS, Norway). Three math treatments, along with the raw spectral data, were used to create the calibrations using partial least square (PLS) regression. The math treatments were first and second derivatives (obtained from the untreated spectra using the Savitzky–Golay approach, with left and right gaps of 8 nm) and multiplicative scatter correction (MSC) (Næs et al. 2002). All three methods are known to reduce noise that occurs within spectral data (Næs et al. 2002). Calibrations were developed with four cross-validation segments.

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 ( $RPD_c$ ) (Williams and Sobering 1993), calculated as the ratio of the standard deviation of the reference data to the SECV, were used to assess calibration performance. Determination of RPD allows comparison of calibrations developed for different wood properties that have differing data ranges and units; the higher the  $RPD_c$  the more accurately the data are described by the calibration.

#### Prediction of wood properties

To examine the performance of the calibrations, they were used to predict the wood properties (air-dry density, MFA, stiffness) of the test set samples. The standard error of prediction (SEP) (determined from the residuals of the predictions) was calculated and gives a measure of how well a

**Fig. 3.** Relationship between measured density and near infrared (NIR)-predicted density for (a) MSC treated NIR spectra and (b) first derivative treated NIR spectra. The regression line is plotted (thick, dark line) in both figures; the thin broken line indicates a 1:1 relationship between measured and predicted density. SEP, standard error of prediction;  $RPD_p$ , ratio of performance to deviation.



calibration predicts parameters of interest for a set of samples not included in the calibration set. The predictive ability of the calibrations was assessed by calculating the  $RPD_p$ , which is similar to the  $RPD_c$  but uses the standard deviation of the prediction set reference data and the SEP.

## Results

### Wood-property calibrations

Table 3 summarizes the calibration results for all math treatments. Density calibrations had coefficients of determination ( $R^2$ ) that ranged from 0.78 (second derivative) to 0.83 (MSC) and  $RPD_c$  values that ranged from 2.11 (second derivative) to 2.30 (MSC). The number of factors varied between math treatments from two (second derivative) to six (raw data). Calibrations for MFA had  $R^2$  between 0.89 (first

derivative) and 0.90 (MSC). Based on  $RPD_c$  and  $R^2$  values, the MSC math treatment gave the strongest calibration statistics. Figure 2 shows the plots for both the MFA calibration (Fig. 2a) using the MSC math treatment and the standardized residual plot (Fig. 2b) for the same calibration. It is apparent that MFA variation increases at higher MFA values. Several transformations were examined, but did not reduce the variation observed for high MFA values. The stiffness calibration based on raw spectral data gave the highest  $R^2$  value (0.93) and the highest  $RPD_c$  (3.65); however, it was developed using eight factors, while stiffness calibrations developed using math-treated spectra required fewer factors (five or six) and gave similar statistics. The MSC treatment gave the best calibration results overall for density, MFA, and stiffness.

### Wood-property predictions

The true test of a calibration is to use it to predict values for a set of samples unrelated from those used to develop the calibration. Table 4 summarizes results for predictions made on the separate test set. A prediction  $R^2$  ( $R_p^2$ ) was calculated as the proportion of variation in the independent prediction set that was explained by the calibration. In general, the wood-property calibrations developed using different math treatments gave similar  $R_p^2$ , but the SEP and consequently the  $RPD_p$  varied greatly between math treatments. The  $R_p^2$  for predicted density ranged from 0.81 (second derivative) to 0.83 (MSC), while the  $RPD_p$  ranged from 0.88 (MSC) to 2.28 (first derivative); these extremes are shown in Fig. 3. Predicted density had a strong relationship with measured density, as indicated by the strong  $R_p^2$ , but the predictions of density obtained using the MSC-treated spectra were shifted upward from the line of equivalence, that is, NIR underestimated density, causing a large error (SEP = 119.3 kg·m<sup>-3</sup>). The first-derivative predictions were closer to the true values, giving a smaller SEP (45.8 kg·m<sup>-3</sup>) and  $RPD_p$  (2.05). Predicted MFA also had little variation in  $R_p^2$ , ranging from 0.80 (second derivative) to 0.84 (MSC), while  $RPD_p$  varied from 1.01 (first derivative) to 2.34 (MSC). The  $RPD_p$  for stiffness had the lowest range between math treatments, ranging from 1.17 (first derivative) to 2.20 (second derivative), but the  $R_p^2$  range was the same as for density, only with slightly higher overall values ranging from 0.87 (second derivative) to 0.90 (first derivative). None of the math treatments used consistently provided the best predictive ability; while the first derivative worked well for density, it performed less efficiently for MFA and stiffness. Overall, the second-derivative treatment provided the best predictions, giving the best results for stiffness and reasonable results for density and MFA.

### Improvement of the wood-property calibrations

Guthrie and Walsh (2002), while assessing soluble solids in mandarin fruit, found that their general calibration did not predict soluble solids well in samples from new sites owing to the specific variation of a particular site. Their solution was to add a single sample from each of the new sites, which was sufficient to decrease the error associated with the predictions to acceptable levels. Based on the findings of Guthrie and Walsh (2002), we created a new calibration set

**Table 5.** Summary of density, microfibril angle (MFA), and stiffness calibrations developed using untreated and math-treated near infrared spectra collected from the radial longitudinal face of radial wooden strips included in the expanded calibration set.

Wood property	Math treatment	Calibration				
		Factors	$R^2$	SEC	SECV	RPD <sub>c</sub>
Density (kg·m <sup>-3</sup> )	Raw	7	0.82	42.26	43.48	2.29
	MSC	6	0.82	41.67	42.49	2.34
	First derivative	6	0.83	41.19	42.67	2.33
	Second derivative	3	0.79	45.50	46.22	2.15
MFA (degrees)	Raw	9	0.89	2.50	2.55	2.93
	MSC	8	0.90	2.38	2.44	3.07
	First derivative	5	0.90	2.42	2.49	3.00
	Second derivative	7	0.90	2.39	2.58	2.90
Stiffness (GPa)	Raw	8	0.92	1.40	1.44	3.45
	MSC	8	0.93	1.31	1.34	3.70
	First derivative	5	0.92	1.40	1.45	3.43
	Second derivative	7	0.92	1.38	1.51	3.29

**Note:** Calibration statistics:  $R^2$ , coefficient of determination; SEC, standard error of calibration; SECV, standard error of cross-validation; RPD<sub>c</sub>, ratio of performance to standard deviation. MSC, multiplicative scatter correction.

**Table 6.** Summary statistics for density, microfibril angle (MFA), and stiffness, predicted using the expanded calibration set.

Wood property	Math treatment	Prediction		
		$R_p^2$	SEP	RPD <sub>p</sub>
Density (kg·m <sup>-3</sup> )	Raw	0.85	41.65	2.47
	MSC	0.86	39.70	2.59
	First derivative	0.86	38.25	2.69
	Second derivative	0.81	49.99	2.05
MFA (degrees)	Raw	0.80	3.61	1.99
	MSC	0.85	2.99	2.40
	First derivative	0.82	3.25	2.21
	Second derivative	0.83	3.09	2.33
Stiffness (GPa)	Raw	0.90	1.84	2.85
	MSC	0.91	1.56	3.35
	First derivative	0.91	1.86	2.82
	Second derivative	0.88	1.84	2.85

**Note:** Prediction statistics:  $R_p^2$ , coefficient of determination; SEP, standard error of prediction; RPD<sub>p</sub>, ratio of performance to deviation. MSC, multiplicative scatter correction.

by including NIR spectra collected from one randomly selected core from each new site in the prediction set for inclusion in the calibration set.

Table 5 summarizes the results for the wood-property calibrations obtained after NIR spectra from a single core from each of the sites in the prediction set had been added to the calibration set. Each core was randomly selected and increased the number of spectra in the calibration set to 771 (95 increment cores). There were only slight increases in  $R^2$  and RPD<sub>c</sub> values, but when applied to the prediction sets, SEP and RPD<sub>p</sub> values were improved for all wood properties (Table 6). In addition, the calibrations developed using the different math treatments all gave similar predictive results.

The RPD<sub>p</sub> for density increased for all math treatments except for the second derivative, which was not altered. MFA had a range of increased values for RPD<sub>p</sub> from 1.99 (raw

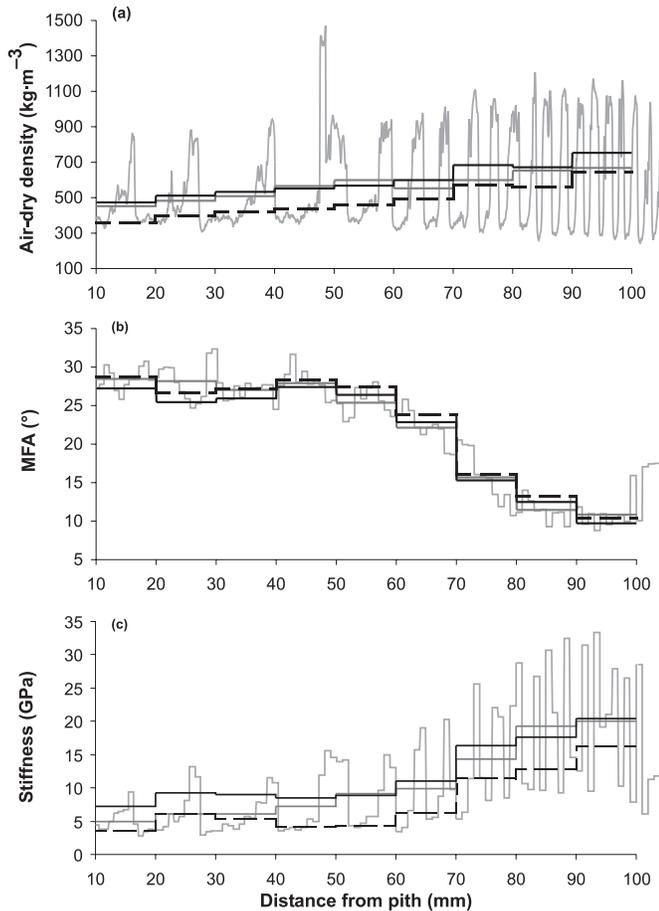
data) to 2.40 (MSC), and  $R_p^2$  values ranged from 0.80 (raw) to 0.85 (MSC). The largest overall improvement occurred for the MSC-treated spectra for stiffness: RPD<sub>p</sub> increased from 1.18 for the original predictions (Table 4) to 3.35 for the expanded predictions, an increase of 2.18. MSC-treated spectra clearly gave the best results for MFA and stiffness, while for density the best results were obtained with first-derivative spectra. In Fig. 4, the data for density (Fig. 4a), MFA (Fig. 4b), and stiffness (Fig. 4c) are shown for a single sample (selected at random) from the prediction set. The figure includes SilviScan data at the original resolution, the SilviScan data averaged at 10 mm, predicted wood properties at 10 mm using the original MSC calibrations, and predicted wood properties at 10 mm using the expanded MSC calibrations.

## Discussion

The purpose of this study was to examine the use of NIR spectroscopy to estimate wood properties of increment cores that had been nondestructively removed from standing trees. Predicted wood properties of increment cores from sites not originally included in the calibration gave estimates that would be good for classification of wood properties, but which had undesirable levels of error. The addition of a single core from each stand used in the prediction set decreased the error of the wood-property predictions, resulting in significantly more accurate estimates. Using a large and diverse population of wood cores increased the applicability of the calibrations for predicting *P. taeda* wood properties and increased the amount of work associated with creating the calibrations.

Development of calibrations using a large population size will give the best representation of the population of interest, which in this study was *P. taeda* grown in Georgia (USA). This study demonstrates that it is possible to create calibrations for density, MFA, and stiffness using a large population that represented a diverse range of sites. In a series of

**Fig. 4.** Plot of pith-to-bark radial variation for (a) density, (b) microfibril angle (MFA), and (c) stiffness for a randomly selected *Pinus taeda* radial strip. The solid thin gray line represents the original SilviScan data, the solid thick gray line represents SilviScan data averaged over 10 mm, the broken black line represents values predicted by the original calibrations, and the solid black line represents values predicted data using the expanded calibrations.



studies by Schimleck and Evans using similar techniques on a small *P. radiata* sample set (8 strips, 119 spectra), second-derivative calibrations gave SEC values of 23.5 kg·m<sup>-3</sup> for density, 1.8° for MFA, and 0.9 GPa for stiffness (Schimleck and Evans 2002a, 2002b, 2003). When these calibrations were used to predict the properties of two separate samples, the resulting SEP values were lower for one core and higher for the other than those reported here. While their work demonstrated that NIR spectroscopy could predict some wood properties of *P. radiata* (and possibly other *Pinus* species), they did not have sufficient samples to make the calibrations applicable to samples from new populations (or sites). The development of calibrations using large populations to create calibrations has been hindered by statistical software packages that could not cope with large numbers of spectra and limited by computer processing power. Berzaghi et al. (2002) successfully created calibrations using a large sample size for the chemical analysis of forage crops, showing that with recent technological advancements large sample sets were manageable for use in creating calibrations. The only fore-

seeable problem with using large sample sets, as used in the present study, is the matching of measured data (SilviScan) that have been averaged over a given distance, 10-mm in this study, with the spectral data that are associated with a given region of a strip. This problem can be overcome by having stringent data-compiling techniques in place.

While the nonexpanded calibrations resulted in higher prediction errors, the overall goodness of fit allows for the predicted values to be useful in classification of wood properties. The use of nonexpanded calibrations would allow a researcher to identify when the transition from less desirable properties to more desirable properties occurs, for example when MFA starts to plateau. Nonexpanded calibrations could be used to identify trees whose properties require additional research, for example, a tree that has an extremely low juvenile wood MFA, which would reduce the cost of analysis. Samples from sites already in the calibration could be tested with a lower amount of associated error.

With the enhancement of calibrations by adding a single strip from the stand of interest, the prediction error is lowered substantially, giving more accurate predictions. The associated SEP for predicted stiffness dropped from 4.58 to 1.56 GPa using the expanded calibration created using the MSC math treatment. In all cases the addition of samples from the sites of interest decreased the associated prediction error. It is believed that this enhancement occurs not because of the increase in variability of the wood properties in the calibration, but because of slight variability in the spectra from each stand that could be expected owing to the unique growing conditions at each site. Each time a new site is sampled, a single sample must be added to the current calibration data set, and then the calibrations must be recalculated for the enhancement to occur.

Using a large population to create calibrations for the non-destructive estimation of wood properties is now possible with NIR spectroscopy. Handling of data for large sample sets requires protocols that allow for a minimum chance for errors when compiling calibration data and NIR spectra. Whether the research requires a calibration for classification (nonexpanded) or accurate measurements (expanded), NIR spectroscopy is capable of providing both for selected wood properties. With an increase in resolution (i.e., spectra collected at 5 mm), the data sets will become larger and more complicated, but will allow for a greater understanding of wood properties, growth, and age-dependent transitions in these properties and possibly within ring estimation of wood properties, using NIR spectroscopy.

## Conclusions

This study demonstrates that large data sets that include a wide range of environmental and genetic variation can establish good calibrations for density, MFA, and stiffness based on NIR spectra obtained in 10-mm sections from the radial longitudinal face of *P. taeda* strips.

The calibrations gave strong relationships when used to predict the wood properties of a separate set of cores from sites not included in the calibration set, but the accuracy of predictions varied depending on the math treatment employed.

The addition of a small number of cores from the prediction set (one core per site) to the calibration set greatly improved the accuracy of predictions and minimized the difference between mathematical processing treatments.

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