# Near infrared spectroscopy for the nondestructive estimation of clear wood properties of *Pinus taeda* L. from the southern United States

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**Abstract**

The estimation of specific gravity (SG), modulus of elasticity (MOE), and modulus of rupture (MOR) of loblolly pine (*Pinus taeda* L.) clear wood samples from a diverse range of sites across the southern United States was investigated using near infrared (NIR) spectroscopy. NIR spectra were obtained from the radial and cross sectional (original, rough, and sanded) surfaces of blocks cut from the ends of 280 clear wood samples (140 matching juvenile and mature wood). Calibrations based only on juvenile or mature wood samples had weak calibration statistics and failed to perform well when applied to a separate test set. Calibrations developed using both juvenile and mature wood NIR spectra provided good relationships for all properties with coefficients of determination ($r^2$) ranging from 0.82 (MOE, radial face) to 0.90 (SG, radial face) demonstrating that it is possible to obtain multi-site calibrations for SG, MOE, and MOR estimation. Prediction $r^2$ ranged from 0.77 (MOE, radial face and SG, original cross-sectional face) to 0.86 (MOR, sanded cross-sectional face). Though differences between surfaces were small, on average the sanded cross-sectional surface provided the best calibration and prediction statistics.

The Southern United States produces more timber products than any other single nation in the world. The South produces 60 percent of the wood used in the United States and 15 percent of the wood consumed globally (Wear and Greis 2002). An increasing proportion of this production is from plantation-grown loblolly pine (*Pinus taeda* L.), which has been favored over other *Pinus* species native to the South because of its ability to grow well on a wide variety of sites. Owing to its importance as a plantation species, loblolly pine has been subjected to genetic improvement that has greatly improved the growth and yield of plantation-grown trees. Li et al. (1999) reported that loblolly pine trees grown from seeds obtained from first-generation seed orchards have produced 7 to 12 percent more volume per acre at harvest, and from second-generation seed orchards, it is estimated that gains in volume will be 13 to 21 percent more than trees grown from wild seed.

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*Forest Products Society Member.
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Forest Prod. J. 55(12):21-28.

The southern United States encompasses a wide range of geographic regions and studies have shown that wood properties differ between them (Zobel and McElwee 1958, Talbert and Jett 1981). The southern pine wood industry is interested in knowing how important wood properties, such as wood specific gravity (SG), modulus of elasticity (MOE), and modulus of rupture (MOR), vary between geographic regions. Currently, the determination of MOE and MOR are based on tests that require destructive sampling and extensive sample preparation; the industry would benefit from employing a more rapid, nondestructive technique for the estimation of these properties.

An option for the estimation of these wood properties is near infrared (NIR) spectroscopy. Several studies (Hoffmeyer and Pedersen 1995; Gindl et al. 2001; Schimleck et al. 2001, 2002a; Thumm and Meder 2001; Via et al. 2003; Kelley et al. 2004) have demonstrated that it is possible to estimate the wood properties of clear wood samples. Hoffmeyer and Pedersen (1995) examined the ability of NIR spectroscopy to nondestructively determine several Norway spruce (*Picea abies* (L.) Karst) wood properties (moisture content [MC], SG, compression and bending strength, and chemical and biological degradation) using NIR spectra collected from the cross-sectional surface of clear wood samples. Their results showed that NIR spectroscopy is an excellent nondestructive method for the estimation of many wood properties. Gindl et al. (2001) based their study on European larch (*Larix decidua* Miller) samples (cross-section 18 by 18 mm, 250 mm longitudinally) cut from boards purchased from a commercial supplier. NIR spectra were collected from the sanded radial surface of each sample at three points using a fiber optic probe (spot size 4 mm). Strong calibrations ($r^2$ ranged from 0.93 to 0.97 for 51 samples) were obtained for basic density, bending strength, MOE, and compressive strength. It was also found that the properties of compression wood samples were well modelled. Kelley et al. (2004) also used a fiber optic probe to collect NIR spectra from the surface of 989 clear wood samples from six softwood species. Correlation coefficients ($r$-values) varied depending on the sample set used, i.e., single species or mixed species, but were generally greater than 0.8 for both MOE and MOR. Meder and Thumm (2001) also utilized a large sample set (406 samples for calibration and 80 for prediction) for the development of a radiata pine (*Pinus radiata* D. Don) MOE calibration. NIR spectra were collected from the radial surface of samples in motion (moving past the NIR detector at a rate of 900 mm/min). A prediction $r^2$ of 0.74 was reported when first derivative spectra were used. Studies by Schimleck et al. (2001, 2002a) used small strips (2 mm tangentially, 7 mm longitudinally, ~20 mm radially) cut from the end of larger alpine ash (*Eucalyptus delegatensis* R.T. Baker) and radiata pine clear wood samples for NIR analysis. Strong calibrations were developed for density, MOE, microfibril angle, and MOR. In a later study (Schimleck et al. 2002b), it was shown that the alpine ash and radiata pine samples could be combined to give a single broad calibration for MOE. Recently Via et al. (2003) developed whole tree density, MOE and MOR calibrations using NIR spectra obtained from the radial face of strips cut from discs (five different heights) from 10 mature longleaf pine (*Pinus palustris* Mill.) trees. $r^2$ ranged from 0.71 to 0.89 depending on the statistical method used to develop the calibration. When NIR spectra collected from pith wood only were used only calibrations for density provided strong $r^2$ (0.69 to 0.87).

Despite the positive results from these studies several questions remain before NIR spectroscopy can be developed sufficiently to replace some or all of the standard destructive test methods. For example, is it possible to develop strong calibrations with wood of the same species having diverse genetic backgrounds and grown a wide range of sites? Do spectra collected from the radial or cross-sectional surface provide better calibration statistics? Is there any benefit in sanding the cross-sectional surface of a sample prior to NIR analysis? Is it possible to develop strong calibrations with only juvenile wood or only mature wood that are predictive? This study addresses these important questions utilizing loblolly pine, the most commercially important tree species in the United States.

## Materials and methods

### Sample origin

Two hundred and eighty clear wood samples obtained from trees growing on 81 plantations across the southern United States were utilized in this study. The location of the plantations sampled is shown in **Figure 1**. **Table 1** provides growth and age information, by physiographic region, for the sampled trees. The 280 samples represented 140 trees that had been sampled to yield matching juvenile and mature wood samples. Three trees were selected from each plantation for destructive sampling. The selected trees were harvested, and a 600-mm bolt was cut at 2.4 m above ground. A 50-mm-thick slab was cut from bark to bark through the pith for processing into static bending samples. The static bending slabs were kiln-dried to 12 percent equilibrium moisture content (EMC). After drying the slab, two clear static bending samples (25.4 by 25.4 by 406 mm) were cut from juvenile wood and two from mature wood. Juvenile wood samples were cut from rings 2 through 4 (to avoid sampling transition wood), and mature wood samples were cut next to the bark. The static bending samples were conditioned at 12 percent EMC before testing.

### Determination of wood properties

The 25.4- by 25.4- by 406-mm clear static bending samples were tested at 12 percent EMC over a 355.6-mm span with center loading and pith up on a Tinius Olsen static bending machine following the procedures for alternate sample size under ASTM D 143 (ASTM 1980). A continuous load was applied at

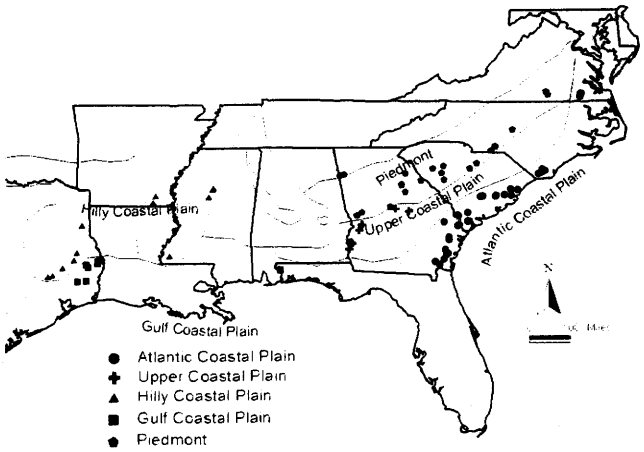


*Figure 1. — Location of P. taeda plantations sampled for this study.*

*Table 1. — Average characteristics of trees sampled by physiographic region.*

| Region | No. of trees sampled | Diameter at breast height | | Total height | | Age | |
|---|---|---|---|---|---|---|---|
| | | Mean | Range | Mean | Range | Mean | Range |
| | | (cm) | | (m) | | (yr) | |
| South Atlantic Coastal Plain | 33 | 24.4 | 18.0 to 33.5 | 20.7 | 15.8 to 25.3 | 23 | 21 to 25 |
| North Atlantic Coastal Plain | 28 | 25.9 | 20.6 to 36.6 | 21.0 | 18.0 to 24.7 | 22 | 20 to 25 |
| Upper Coastal Plain | 14 | 25.9 | 20.6 to 33.0 | 20.7 | 16.2 to 27.1 | 23 | 20 to 26 |
| Piedmont | 29 | 25.9 | 20.8 to 32.0 | 18.9 | 15.9 to 23.2 | 23 | 20 to 26 |
| Gulf Coastal Plain | 9 | 22.6 | 19.1 to 29.0 | 20.4 | 16.5 to 22.6 | 21 | 18 to 22 |
| Hilly Coastal Plain | 27 | 24.4 | 19.3 to 30.2 | 19.5 | 14.3 to 23.2 | 22 | 20 to 25 |
| All regions combined | 140 | 25.1 | 18.0 to 36.6 | 20.1 | 14.3 to 27.1 | 22 | 18 to 26 |

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

| Wood property | Calibration set | | | | Prediction set | | | |
|---|---|---|---|---|---|---|---|---|
| | Minimum | Maximum | Average | SD | Minimum | Maximum | Average | SD |
| Juvenile plus mature | | | | | | | | |
| Specific gravity | 0.33 | 0.71 | 0.49 | 0.09 | 0.34 | 0.64 | 0.48 | 0.08 |
| MOE (GPa) | 2.54 | 14.84 | 7.41 | 3.43 | 2.32 | 14.69 | 7.25 | 3.15 |
| MOR (MPa) | 31.64 | 126.17 | 73.48 | 24.42 | 32.79 | 115.69 | 72.00 | 21.94 |
| Juvenile | | | | | | | | |
| Specific gravity | 0.33 | 0.48 | 0.42 | 0.03 | 0.34 | 0.48 | 0.42 | 0.03 |
| MOE (GPa) | 2.50 | 8.94 | 4.59 | 1.45 | 2.32 | 8.80 | 4.62 | 1.37 |
| MOR (MPa) | 31.64 | 80.67 | 52.79 | 10.13 | 32.79 | 78.74 | 52.89 | 8.91 |
| Mature | | | | | | | | |
| Specific gravity | 0.47 | 0.71 | 0.57 | 0.05 | 0.45 | 0.64 | 0.55 | 0.04 |
| MOE (GPa) | 4.24 | 14.84 | 10.05 | 2.49 | 4.94 | 14.69 | 9.88 | 2.02 |
| MOR (MPa) | 56.77 | 126.17 | 93.36 | 15.60 | 61.59 | 115.69 | 91.11 | 12.18 |

SD = standard deviation.

a head speed of 1.78 mm per minute, rather than 1.29 mm per minute to reduce test time. Preliminary tests showed specimens failed primarily in compression with no defined break or tension failure. After testing, each sample was ovendried at 103°C, and SG was calculated based on specimen dimensions at 12 percent EMC and ovendry weight. MOE and MOR were calculated using procedures outlined in ASTM D 143 (ASTM 1980).

## NIR spectroscopy

After static bending tests were completed, a small block (25.4 mm radially, 25.4 mm tangentially, and approx. 25.4 mm longitudinally) was cut from each end of one juvenile and one mature clear wood sample for each tree. To analyze the importance of surface roughness on calibration statistics, the cross-sectional surface was tested in its original state (very rough and highly variable), after being cut with a bandsaw (rough) and after being sanded with 300-grit sandpaper for approximately 25 seconds (sanded). Diffuse reflectance NIR spectra were collected from the radial and cross-sectional surface of each block using a NIRSystems Inc. Model 5000 scanning spectrometer. Samples were held in a custom-made holder similar to that described in Schimleck et al. (2001). A mask with a 5- by 12.5-mm window was used to ensure an area of constant size was analyzed. Two spectra were collected from adjacent 12.5-mm windows for each surface; for the cross-sectional surface, the long axis of the window was perpendicular (approximately) to the growth rings. As both ends from a clear wood sample were tested, a total of four spectra were obtained per sample to give a total of 1,120 spectra per surface (4 spectra per sample multiplied by 280 samples). The spectra were collected at 2-nm intervals over the wavelength range 1100 to 2500 nm. The instrument reference was a ceramic standard.

## Partial least squares calibrations for the prediction of SG, MOE, and MOR

For the calibration set, matching samples from an individual tree from each site were randomly selected for calibration. As there were a total of 81 sites, 162 samples were used for calibration; the remaining 118 samples were used as a separate test set. To examine juvenile and mature wood relationships, the two sets were split into their juvenile and mature wood halves giving a total of 81 samples for the calibration set and 59 samples for the prediction set. **Table 2** contains the summary statistics for the different sets.

All calibrations were created using the Unscrambler® (version 8.0) software package (Camo AS, Norway) and second derivative spectra (obtained from the untreated spectra using the Savtizky-Golay approach, with left and right gaps of 8 nm).

Partial least squares (PLS) regression was used for the calibrations with four cross-validation segments and a maximum of 10 factors. The Unscrambler® software recommended the final number of factors to use for each calibration.

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 (SD) of the reference data to the SECV were used to assess calibration performance. $RPD_c$ allows comparison of calibrations for different wood properties that have differing data ranges and units, the higher the $RPD_c$ the more accurate the data is described by the calibration.

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 SD prediction set reference data and the SEP.

## Results

### SG, MOE, and MOR calibrations: Juvenile plus mature wood

Calibrations for each wood property were created using PLS regression and NIR spectra obtained in 12.5-mm sections from the radial and cross-sectional (original, rough, and sanded) surfaces of blocks cut from the ends of 162 *P. taeda* clear wood samples. The calibrations were then applied to a separate test set of 118 NIR spectra. **Table 3** provides summary statistics of each calibration.

SG, MOE, and MOR calibrations all gave strong relationships regardless of whether the cross-sectional or radial surface was very rough, rough, or sanded (**Table 3**). The coefficients of determination ($r^2$) ranged from 0.82 for the original cross-sectional surface and radial surface MOE calibrations to 0.90 for the radial surface SG calibration. $RPD_c$ values were generally good ranging from 2.19 for the radial surface MOE calibration to 2.88 for the radial surface SG calibration. On average the SG calibrations gave the highest $RPD_c$ values (2.67), but it should also be noted that fewer factors were generally recommended for the MOE and MOR calibrations. Relationships between measured values and NIR-estimated values for SG, MOE, and MOR are shown in **Figure 2** (the results shown are for NIR spectra obtained from the sanded cross-sectional surface).

The calibrations were applied to the separate test set. Strong to moderate relationships were obtained for all properties with $R_p^2$ ranging from 0.77 for MOE predicted using the original cross-sectional and radial surface calibrations to 0.86 for MOR predicted using the sanded cross-sectional surface calibration. SEP values were very similar to the SEC and SECV values reported for each calibration. $RPD_p$ values ranged from 2.06 to 2.67 and were lower than $RPD_c$ values. On average predicted SG gave the highest $RPD_p$ values (2.42), followed by predicted MOR (2.37) and predicted MOE (2.23).

For each of the four surfaces tested, the $RPD_c$ and $RPD_p$ values for each property were averaged, to determine which surface gave the best overall calibration and prediction statistics. The sanded cross-sectional surface gave the highest average $RPD_c$ (2.67) followed by the rough cross-sectional surface (2.57), and the radial surface as well as the original cross-

*Table 3. — Summary of calibrations obtained for SG, MOE, and MOR using NIR spectra collected from juvenile and mature wood samples.*

| | Calibration set | | | | | Prediction set | | |
|---|---|---|---|---|---|---|---|---|
| Wood property | # factors | $r^2$ | SEC | SECV | $RPD_c$ | $R_p^2$ | SEP | $RPD_p$ |
| Original | | | | | | | | |
| Specific gravity | 4 | 0.89 | 0.03 | 0.03 | 2.60 | 0.84 | 0.03 | 2.31 |
| MOE (GPa) | 2 | 0.82 | 1.46 | 1.48 | 2.32 | 0.77 | 1.53 | 2.06 |
| MOR (MPa) | 2 | 0.88 | 8.63 | 9.53 | 2.56 | 0.82 | 9.58 | 2.25 |
| Rough | | | | | | | | |
| Specific gravity | 3 | 0.86 | 0.03 | 0.04 | 2.44 | 0.82 | 0.03 | 2.30 |
| MOE (GPa) | 1 | 0.87 | 1.24 | 1.25 | 2.74 | 0.82 | 1.37 | 2.30 |
| MOR (MPa) | 1 | 0.84 | 9.55 | 9.65 | 2.53 | 0.81 | 9.69 | 2.23 |
| Sanded | | | | | | | | |
| Specific gravity | 2 | 0.88 | 0.03 | 0.03 | 2.77 | 0.84 | 0.03 | 2.47 |
| MOE (GPa) | 1 | 0.85 | 1.33 | 1.34 | 2.55 | 0.84 | 1.29 | 2.45 |
| MOR (MPa) | 2 | 0.87 | 8.60 | 9.08 | 2.69 | 0.86 | 8.09 | 2.67 |
| Radial | | | | | | | | |
| Specific gravity | 3 | 0.90 | 0.03 | 0.03 | 2.88 | 0.85 | 0.03 | 2.61 |
| MOE (GPa) | 3 | 0.82 | 1.45 | 1.57 | 2.19 | 0.77 | 1.51 | 2.09 |
| MOR (MPa) | 3 | 0.84 | 9.61 | 10.16 | 2.40 | 0.82 | 9.34 | 2.31 |

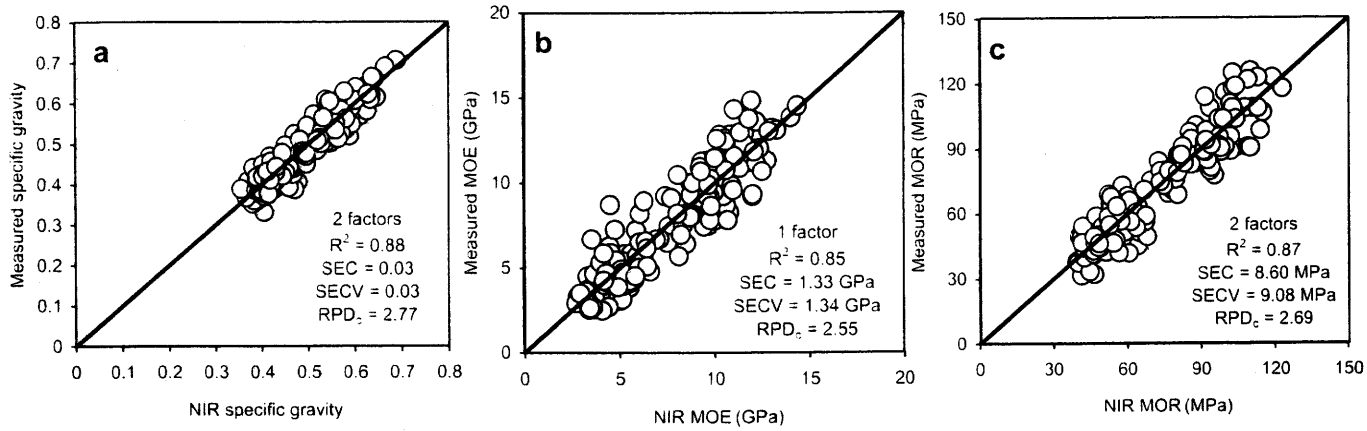


*Figure 2. — Relationships between measured values and NIR-estimated values for (a) SG, (b) MOE, and (c) MOR. Calibrations were developed using 162 NIR spectra collected from the sanded cross-sectional surface of blocks cut from the ends of short clear samples. Note that the regression line has been plotted through the origin.*

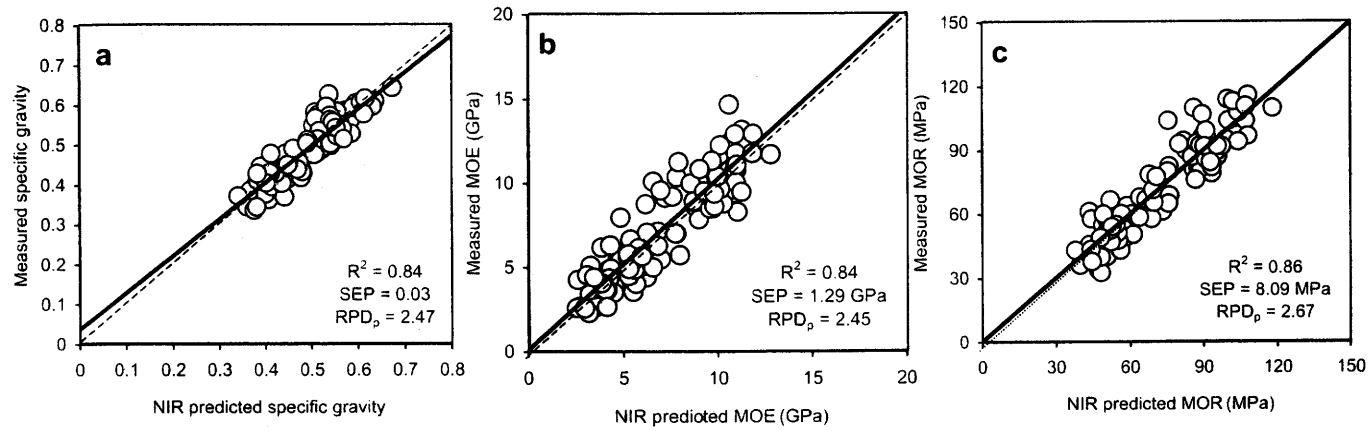


*Figure 3. — Relationships between measured values and NIR-predicted values for (a) SG, (b) MOE, and (c) MOR. Predictions were made on a set of 118 NIR spectra collected from the sanded cross-sectional surface of blocks cut from the ends of short clear samples. Note that the regression line has been plotted through the origin and that the thin broken line represents the line of equivalence.*

sectional surface both gave average $RPD_c$ values of 2.49. For the prediction set, the sanded cross-sectional surface clearly gave the highest average $RPD_p$ (2.54), followed by the radial surface (2.35), the rough cross-sectional surface (2.28), and the original cross-sectional surface (2.22). In addition to having the highest average $RPD_c$ and $RPD_p$ values, the sanded cross-sectional surface calibrations were also obtained using the least number of factors, two for SG and MOR and one for MOE (**Table 3**). Relationships between measured values and NIR-predicted values for SG, MOE, and MOR are shown in **Figure 3** (the results shown are for NIR spectra obtained from the sanded cross-sectional surface).

## SG, MOE, and MOR calibrations: Juvenile wood

Juvenile wood calibrations for each property were developed using PLS regression and NIR spectra obtained from the radial and cross-sectional (original, rough, and sanded) surfaces of blocks cut from the ends of 81 juvenile clear wood samples. The calibrations were then applied to a separate test set of 59 NIR spectra (also from juvenile wood samples). **Table 4** provides summary statistics of each calibration. The SG, MOE, and MOR calibrations gave variable relationships with $r^2$ ranging from 0.43 (rough cross-sectional surface SG calibration) to 0.86 (original cross-sectional surface SG calibration). Nine factors were recommended for the original cross-sectional surface SG calibration, which could be considered excessive. The next strongest $r^2$ (0.77) was for the sanded cross-sectional surface SG calibration. $RPD_c$ values (1.18 to 1.36) were lower than found for the juvenile plus mature wood calibrations. Each property had similar $RPD_c$ values (1.25 to 1.28) when averaged over the four surfaces.

When applied to the separate test set, the calibrations generally performed poorly. The SG prediction, using the sanded cross-sectional surface calibration gave the strongest $R_p^2$ (0.61), while the next best $R_p^2$ was 0.49 (SG predicted using the radial surface calibration). $RPD_p$ values ranged from 0.96 to 1.56, with predictions based on NIR spectra obtained from the sanded cross-sectional surface giving the highest $RPD_p$ values for each property. When averaged over the four surfaces pre-

*Table 4. — Summary of calibrations obtained for SG, MOE, and MOR using NIR spectra collected from juvenile wood samples.*

| | Calibration set | | | | | Prediction set | | |
|---|---|---|---|---|---|---|---|---|
| Wood property | # factors | $r^2$ | SEC | SECV | $RPD_c$ | $R_p^2$ | SEP | $RPD_p$ |
| Original | | | | | | | | |
| Specific gravity | 9 | 0.86 | 0.01 | 0.03 | 1.26 | 0.34 | 0.03 | 1.15 |
| MOE (GPa) | 4 | 0.61 | 0.89 | 1.10 | 1.32 | 0.23 | 1.26 | 1.09 |
| MOR (MPa) | 3 | 0.58 | 6.53 | 7.80 | 1.29 | 0.11 | 9.38 | 0.96 |
| Rough | | | | | | | | |
| Specific gravity | 3 | 0.43 | 0.02 | 0.03 | 1.18 | 0.35 | 0.03 | 1.28 |
| MOE (GPa) | 1 | 0.49 | 1.03 | 1.07 | 1.36 | 0.27 | 1.13 | 1.21 |
| MOR (MPa) | 2 | 0.49 | 7.28 | 7.81 | 1.29 | 0.26 | 7.92 | 1.14 |
| Sanded | | | | | | | | |
| Specific gravity | 5 | 0.77 | 0.02 | 0.02 | 1.34 | 0.61 | 0.02 | 1.56 |
| MOE (GPa) | 2 | 0.50 | 1.02 | 1.16 | 1.26 | 0.47 | 0.99 | 1.38 |
| MOR (MPa) | 3 | 0.50 | 7.15 | 8.17 | 1.23 | 0.26 | 7.77 | 1.16 |
| Radial | | | | | | | | |
| Specific gravity | 3 | 0.60 | 0.02 | 0.02 | 1.36 | 0.49 | 0.02 | 1.40 |
| MOE (GPa) | 6 | 0.69 | 0.79 | 1.23 | 1.18 | 0.21 | 1.33 | 1.03 |
| MOR (MPa) | 4 | 0.54 | 6.81 | 8.43 | 1.19 | 0.13 | 9.13 | 0.99 |

dicted SG gave the highest $RPD_p$ (1.35), followed by predicted MOE (1.18) and predicted MOR (1.06).

When average $RPD_c$ and $RPD_p$ values were determined for each of the four surfaces, it was found that $RPD_c$ values were very similar (1.24 to 1.30) regardless of the surface used. The highest average $RPD_c$ was for the original cross-sectional surface but more factors were used for these calibrations than for the other surfaces. For the prediction set, the sanded cross-sectional surface gave the highest average $RPD_p$ (1.37), followed by the rough cross-sectional surface (1.21), the radial surface (1.14), and the original cross-sectional surface (1.07).

## SG, MOE, and MOR calibrations: Mature wood

Mature wood calibrations for each property were created using PLS regression and NIR spectra obtained from the radial and cross-sectional (original, rough, and sanded) surfaces of blocks cut from the ends of 81 mature clear wood samples. The calibrations were then applied to a separate test set of 59 NIR spectra (also from mature wood samples). **Table 5** provides summary statistics of each calibration. The SG, MOE, and MOR mature wood calibrations gave moderate relationships with $r^2$ ranging from 0.57 to 0.80 for the sanded cross-sectional surface SG calibration. $r^2$ were generally stronger than reported for juvenile wood but weaker than reported for the juvenile plus mature wood calibrations. $RPD_c$ values were better than those obtained for the juvenile wood calibrations ranging from 1.30 to 1.85. When averaged over the four surfaces, MOE gave the highest $RPD_c$ (1.58) followed by SG (1.55) and MOR (1.47).

When applied to the separate test set, the calibrations performed poorly with $R_p^2$ ranging from (0.35 to 0.57). $RPD_p$ values ranged from 1.11 to 1.54, similar to those obtained for juvenile wood. Predictions based on NIR spectra obtained from the sanded cross-sectional surface gave the highest $RPD_p$ values for MOE and MOR. On average predicted SG gave the highest $RPD_p$ values (1.34), followed by predicted MOR (1.31), and predicted MOE (1.29).

When average $RPD_c$ and $RPD_p$ values were determined for each of the four surfaces, it was found that the sanded cross-sectional surface gave the highest $RPD_c$ and $RPD_p$ values (1.68 and 1.41, respectively). The radial surface gave the next highest average $RPD_p$ (1.36) but had the lowest average $RPD_c$ (1.43). The original cross-sectional surface gave the lowest average $RPD_p$ (1.18).

## Discussion

The SG, MOE, and MOR calibrations reported in this study were developed through correlations to traditional low resolution, destructive measures. These strong calibrations demonstrate that the properties of loblolly pine clear wood samples can be estimated by NIR spectroscopy provided that both juvenile and mature wood samples are included in the calibration set. Limiting the calibration set to juvenile or mature wood samples gave calibrations with weaker statistics that failed to perform well when applied to a separate test set. The failure of the juvenile wood calibrations could be expected as the variation in wood properties was small being approximately half of what it was for the combined juvenile/mature wood set (**Table 2**). The mature wood calibration set was more variable than the juvenile wood set; for example, variation of the mature MOE data was 86 percent of the combined juvenile/mature wood set, and while some reasonable relationships were obtained for calibrations, the strongest $R_p^2$ was only 0.57.

The SG, MOE, and MOR calibrations reported in this study were developed using clear wood samples obtained from trees growing in 81 plantations spread across the southern United States. Owing to geographic and genetic differences, the variation included in the calibration set should be considerable and quite possibly representative of much of the wood property variation present in plantation-grown loblolly pine. The ability

*Table 5. — Summary of calibrations obtained for SG, MOE, and MOR using NIR spectra collected from mature wood samples.*

| | Calibration set | | | | | Prediction set | | |
|---|---|---|---|---|---|---|---|---|
| Wood property | # factors | $r^2$ | SEC | SECV | $RPD_c$ | $R_p^2$ | SEP | $RPD_p$ |
| Original | | | | | | | | |
| Specific gravity | 3 | 0.66 | 0.03 | 0.04 | 1.42 | 0.35 | 0.04 | 1.11 |
| MOE (GPa) | 2 | 0.57 | 1.61 | 1.67 | 1.49 | 0.38 | 1.70 | 1.19 |
| MOR (MPa) | 3 | 0.69 | 8.64 | 10.03 | 1.56 | 0.45 | 9.70 | 1.23 |
| Rough | | | | | | | | |
| Specific gravity | 5 | 0.78 | 0.02 | 0.04 | 1.41 | 0.52 | 0.03 | 1.36 |
| MOE (GPa) | 1 | 0.70 | 1.36 | 1.43 | 1.74 | 0.48 | 1.54 | 1.31 |
| MOR (MPa) | 1 | 0.57 | 10.30 | 10.66 | 1.46 | 0.36 | 9.88 | 1.21 |
| Sanded | | | | | | | | |
| Specific gravity | 4 | 0.80 | 0.02 | 0.03 | 1.85 | 0.55 | 0.03 | 1.36 |
| MOE (GPa) | 1 | 0.66 | 1.45 | 1.52 | 1.64 | 0.52 | 1.47 | 1.37 |
| MOR (MPa) | 3 | 0.70 | 8.52 | 10.02 | 1.56 | 0.57 | 7.97 | 1.50 |
| Radial | | | | | | | | |
| Specific gravity | 3 | 0.71 | 0.03 | 0.03 | 1.52 | 0.57 | 0.03 | 1.54 |
| MOE (GPa) | 4 | 0.70 | 1.33 | 1.70 | 1.46 | 0.44 | 1.60 | 1.27 |
| MOR (MPa) | 4 | 0.64 | 9.30 | 12.04 | 1.30 | 0.45 | 9.34 | 1.28 |

to develop calibrations across many different sites and encompassing wide variation is important as it provides calibrations that are more robust (Berzaghi et al. 2002). Generally studies that have utilized NIR for the estimation of wood properties have not included a wide variety of sites. In a recent study, Jones et al. (2005) was able to obtain calibrations for SilviScan measured air-dry density (Evans 1994, 1997), microfibril angle, and MOE (determined using x-ray densitometry and x-ray diffraction data) using a large set of samples from sites representing the Lower Atlantic Coastal Plain, Upper Atlantic Coastal Plain, and Piedmont physiographic regions in Georgia. Standard errors were larger than those reported for calibrations based on a set of radiata pine samples (Schimleck and Evans 2002a, 2002b, 2003) from a single site but this could be expected as the multiple-site set utilized by Jones et al. (2005) encompassed far greater variation. Hence the calibrations reported here do not have the excellent calibration statistics reported by Gindl et al. (2001), for example, but it is probable that they would provide more robust predictions of wood properties from trees in breeding populations and from trees grown on a wide variety of sites.

When NIR spectra are obtained from clear wood samples, three surfaces are available for analysis: cross-sectional, radial, and tangential. In this study the tangential surface was not examined because it does not represent all of the wood property variation present in a short clear wood sample. Thumm and Meder (2001) compared results for spectra collected from the radial and tangential surface and found that the tangential surface provided inferior results to the radial surface. Thumm and Meder (2001) also noted that a NIR spectrum collected from the radial surface better represents the whole sample. Potentially NIR spectra collected from the cross-sectional surface could represent as much variation as NIR spectra collected from the radial surface. The samples used in this study were 25.4 by 25.4 mm. By using a 12.5-mm window to collect two adjacent spectra from either the radial or cross-sectional surface of samples cut from both ends of short clear wood samples, four spectra per sample were collected that when averaged represented the sample very well. This approach differed from that of Thumm and Meder (2001), who collected spectra from the radial and tangential surfaces of moving samples, and Gindl et al. (2001) who collected three spectra from three different locations on the radial surface of their samples. Owing to the straight grain of the samples analyzed in this study, it was not thought necessary to collect additional spectra from the radial surface in an attempt to better represent it. Both the radial and cross-sectional surfaces provided good results for all properties when the juvenile/mature wood sample set was used. NIR spectra obtained from the radial surface provided marginally better calibration and prediction results for SG, while NIR spectra collected from the sanded cross-sectional surface provided the best predictions of MOE and MOR. When $RPD_c$ and $RPD_p$ values for each wood property were averaged for each surface, it was found that the sanded cross-sectional surface provided the best overall results, particularly in prediction.

The influence of surface roughness on calibration and prediction results was examined by collecting spectra from the original cross-sectional surface, which was very rough and also had resin bleeding from resin canals for many samples, a fresh cross-sectional surface (referred to as rough) produced when blocks were cut from the ends of the clear wood samples using a bandsaw and a sanded cross-sectional surface. Each surface provided similar calibrations and predictions for each wood property but overall the sanded cross-sectional face gave the best results (based on average $RPD_c$ and $RPD_p$ values). The poor condition of the original cross-sectional surface had a small negative impact on the quality of NIR spectra collected from its surface. This finding is in agreement with those of

Hoffmeyer and Pederson (1995) and Schimleck et al. (2003) who reported a only a small negative difference between calibrations using NIR spectra collected from rough and smooth surfaces.

## Conclusions

NIR spectroscopy can be used to estimate SG, MOE, and MOR of loblolly pine clear wood samples from a wide range of sites provided that both juvenile and mature wood samples are included in the calibration set.

Calibrations based solely on juvenile or mature wood samples had weaker calibration statistics, compared to the juvenile/mature wood calibrations and failed to perform well when applied to a separate test set.

Though differences between results with the sanded and rough cross-sectional surfaces were small as were differences between the sanded cross-sectional and radial faces, NIR spectra obtained from the sanded cross-sectional surface provided the best overall calibration and prediction statistics.

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