

## genetics &amp; tree improvement

# Estimating the Basic Density and Mechanical Properties of Elite Loblolly Pine Families with Near Infrared Spectroscopy

Gifty E. Acquah, Charles Essien, Brian K. Via, Nedret Billor, and Lori G. Eckhardt

Near infrared spectroscopy coupled with partial least squares regression was utilized as a high throughput tool in assessing the density, modulus of rupture, and modulus of elasticity of elite loblolly pine families (*Pinus taeda* L.). These properties dictate wood quality for structural applications. PLS models were calibrated and validated with 260 samples processed from loblolly pine families and commercially acquired southern pine lumber. Developed models having  $R^2$ s greater than 0.7 and RPDs meeting the 1.5 screening criteria were used to predict the wood traits of 351 live trees representing 14 elite families planted on two sites. Two-way ANOVA testing the effect of family, site, and family  $\times$  site interaction was significant for family for all traits. Within a family, site also affected the density, but to a lower extent. With the significant family  $\times$  site interaction term on density and MOR, landowners and tree breeders would have to keep in mind that desired traits of the families might be unstable on different sites. Notwithstanding, four out of the 14 screened families had consistently high density, MOR, and MOE values irrespective of site. This knowledge can be incorporated in tree breeding programs to further improve wood quality.

**Keywords:** *Pinus taeda*, near infrared spectroscopy, wood density, modulus, high throughput techniques

## Introduction

As the wood basket of the nation, the southern United States accounts for 64% of the total timber harvested in the country (Smith et al. 2001). This produces 60% of the wood consumed nationally and contributes 18% to global wood supply (Wear and Greis 2002). *Pinus taeda* L. (loblolly pine) is an important timber species in the nation. The species dominates on approximately 13.4 million ha throughout the southeastern forests and accounts for over 50% of the standing pine volume of this region (Schultz 1997). Economically it generates some 110, 000 direct and indirect jobs and contributes approximately \$30 billion to the economy of the region (Schultz 1999).

Unfortunately, Southern Pine Decline (SPD) (previously known as Loblolly Pine Decline or Pine Decline), which was first observed in the Talladega National Forest, Alabama, back in 1959, has been associated with this species (Brown and McDowell 1968, Hess et al.

2002, Eckhardt et al. 2007) According to Eckhardt et al. (2010), SPD has been observed across the southeastern United States; from the Atlantic and East Gulf Coastal Plains and Piedmont Province, as well as in the Sandhills that overlap these regions. Symptoms of SPD include a reduction of radial growth, thinning of foliage, deterioration of lateral roots, and a heavy production of cones just before premature mortality. SPD has been reported on, especially, mature loblolly pine stands on public lands (Menard 2007), as well as in forests managed by nonindustrial private landowners and forest industries (Eckhardt et al. 2007). In a bid to control this disease complex, stakeholders would like to select and deploy elite loblolly pine families that are currently being screened for tolerance against root fungi associated with SPD. The volume, stem quality, and resistance to fusiform rust of these elite families have been improved through tree breeding programs.

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Since the elite loblolly pine families are essentially a new feedstock, the southern pine industry is interested in knowing such important properties as the density, modulus of rupture (MOR), and modulus of elasticity (MOE) of this material. Furthermore, the stakeholders would like to incorporate this information back into tree breeding programs that aim to further improve wood quality.

The basic density of wood is defined as the ratio of its oven-dry mass to its green volume. It is considered the most important physical property of wood due to its effect on other wood attributes, such as the strength properties, the yield of pulp per unit volume, and the shrinkage and swelling behavior of wood (Haygreen and Bowyer 1989). The porosity of wood (i.e., the ratio of cell wall material to lumen) is the main contributing factor to the basic density. Just like density, the strength properties of MOE and MOR dictate the quality of wood for structural applications. MOE (also known as the stiffness) is the resistance to deformation or distortion. It is the linear relationship between stress applied in bending and the resulting strain. The MOR (also known as ultimate strength) is the ultimate resistance of wood to applied loads. It is a measure of the maximum load-carrying capacity of wood in bending (Haygreen and Bowyer 1989, Ritter 1990, Green et al. 1999). Apart from their importance in structural/dimensional timber, the MOE and MOR are valuable traits in standing trees. These properties reduce mortalities resulting from the failure of stems and uprooting of trees during inclement weather (Lachenbruch et al. 2011).

Currently, test methods used to determine the MOE, MOR, and density require extensive sample preparation and destructive testing. With the large number of trees that have to be sampled in tree breeding programs, these conventional methods will not be practical and feasible. The industry would thus benefit from rapid, nondestructive, and cost-effective alternatives.

Near infrared spectroscopy (NIR) has evolved over the years as a reliable and rapid technique in the nondestructive assessment of wood and other forest products. A good number of studies have reported on the application of NIR to predict the density, MOE, and MOR of wood. Kothiyal and Raturi (2011) related NIR spectra collected from the radial and tangential surfaces of five-year old *Eucalyptus tereticornis* Sm. to its specific gravity, MOE, and MOR. They reported that PLS regression models from these two wood surfaces performed equally well in property prediction, with  $R^2$  values ranging from 0.58 to 0.77. Cooper et al. (2011) have, however, pointed out that surface roughness of the material can affect the spectra. The researchers thus recommended that NIR spectra should be collected on similar surfaces that have ideally been planed. Furthermore, they suggested that a large area should be illuminated especially when scanning is being done on the tangential surface so as to ensure a better representation of both earlywood and latewood. In an earlier study, Via et al. (2003) modeled the density, MOE, and MOR of *Pinus palustris* mature wood, juvenile wood, and pith wood. The authors attributed the poor performance of the developed models for predicting the strength properties of the pith wood to the narrow range of the MOE and MOR of the material used in their study. Another explanation they gave was that the high concentration of extractives in the pith area, although it had a positive correlation with density, did not contribute to the strength. Several other researchers have employed NIR to estimate the density (Gindl et al. 2001, Hein et al. 2009, Alves et al. 2012), as well as the MOE and MOR, of solid wood and composite wood

products (Thumm and Meder 2001, Rials et al. 2002, Adedipe and Dawson-Andoh 2008).

Even though a good number of studies have used NIR to estimate the density and strength properties of wood and wood-based products, the current study focuses on elite loblolly pine families, thus an essentially new resource. The objectives of this study were 1) to develop NIR-based PLS models to rapidly predict the density, MOE, and MOR of loblolly pine families, and 2) to screen out these elite loblolly pine families based on the understudied wood traits.

## Materials and Methods

### Materials

Loblolly pine were acquired from two genetic research plantations established in 1998. Study Site 1 was located near Nahunta, Brantley County, Georgia (N 31° 12'16" W 81° 58'56"), and Site 2 was located near Yulee, Nassau County, Florida (N 30° 63' W 81° 57'). The Georgia site had poorly drained fine sandy loam that was generally poor in nutrients, whereas the Florida site had poorly drained loamy clay soil that formed from marine sediments. The mean annual precipitation for the two sites were respectively 1315 mm and 1350 mm, according to NOAA. The same planting design, silvicultural treatments, and seedlots of genetically improved loblolly pine families were administered on both sites to enable the comparison of growth and stability of the families involved. Each site was partitioned into 15 blocks. Eighty trees representing 80 half-sib families were planted on each block. Fifteen of these elite families were selected to be used, thus a total of 450 trees (i.e., 15 families with one replication on each block per site) were earmarked for the study. In order to maintain anonymity, a unique code was assigned to each of the families for data analysis and reporting.

First, 5 mm increment cores were sampled at breast height from 13-year old trees during the spring and summer of 2011. Three hundred fifty-one tree cores were obtained because some trees were dead at the time of sampling. The cores were stored in a walk-in freezer (temperature: 4°C) until time for further processing.

The second set of material comprised whole trees that were harvested from the selected families in 2014 and 2015. One tree per family was destructively sampled from each of the sites, thus a total of 30 trees. The dbh of the trees ranged from 11.5 cm to 23.4 cm. The

### Management and Policy Implications

This study employed NIR spectroscopy as a high throughput tool to rapidly determine the density, stiffness, and ultimate strength of elite loblolly pine families. Furthermore, developed NIR models were applied in the screening of 351 live trees representing 14 families that are currently on trial on genetic research plantations. The effects of family, site, and family by site interaction were investigated. Results from this study are relevant to the forest products industry because they have provided knowledge about some physical and mechanical properties of the 14 elite families used; these are essentially a new feedstock. In addition, we demonstrated that NIR spectroscopy can be used as a tool to exploit the variation in traits of standing trees. It was for instance determined that the family x site interaction was significant for density and ultimate strength. Landowners and tree breeders would thus have to be mindful that desired traits of the elite families might be inconsistent across sites. This information can also be incorporated in tree breeding programs to further improve wood quality.

mean dbh of trees on the Georgia site was 15.6 cm (SD = 2.5 cm), while that for the Florida site was 19.2 cm (SD = 2.8 cm). Trees were crosscut into 1.5 m lengths along the bole, from which 50 cm bolts were cut from the basal end. Three bolts representative of the butt, mid, and top sections of each tree were selected to be further processed. For smaller diameter logs, two bolts representing a section had to be processed. To hinder the rate of drying, the ends of bolts were coated with wax (Anchorseal Green Wood Sealer, U.C. Coatings Corp. Buffalo, NY, USA) in the field before they were transported to the laboratory. They were also stored in a walk-in freezer (temperature: 4°C) until time of processing.

The last set of material was nominal 2 x 4 in. No. 2 southern yellow pine lumber acquired from West Fraser Inc., a commercial sawmill located in Opelika, AL. This is representative of material that is currently being processed and sold as lumber in the region.

Test samples that were processed from the harvested loblolly pine families and the commercial southern pine boards were used in conventional laboratory testing, as well as NIR model calibration and validation. Validated NIR models were then applied to predict the wood properties and screening of 351 live trees.

### Specimen Preparation and Determination of Wood Properties

Loblolly pine bolts and the commercial southern pine material were processed for three-point bending tests as specified in ASTM D143. Clear wood test specimens were cut and planed to final dimensions of 2.5 x 2.5 x 41 cm and conditioned to an average moisture content (MC) of 9% in a control chamber (temp: 22°C; relative humidity: 55%). Prior to destructive testing, the mass of a sample was measured and the dimensions obtained with calipers. This was used to compute the basic density as the ratio of the mass of a test specimen to its volume. Test samples were then loaded into a Zwick-Roell load frame equipped with 10KN load cell and a computer-controlled screw-drive crosshead, and force applied at 1.3 mm/min on the tangential face. The span for the testing was 36 cm. The MOE (i.e., stiffness) was computed as the slope of the linear portion of the load-deflection curve. The MOR was calculated as

$$\text{MOR} = \frac{1.5 P \cdot L}{(b \cdot h^3)}$$

where P is the ultimate load, L is the span, b is the width, and h is the height of the test specimen. Experiments were run in triplicates for density, MOE, and MOR.

After destructive testing, each specimen was sawed into smaller blocks. Two of these cut pieces were used to determine the moisture content of the sample. The remaining materials were stored in airtight zip-lock bags in a conditioning chamber until they were needed for further analysis.

### Near Infrared Spectroscopy (NIR)

Wood blocks saved after the destructive strength testing were first chipped, and then ground in a Wiley mill (Thomas Scientific, model 3383-L10, Swedesboro, NJ, USA) to pass an 80-mesh screen. In addition, the increment cores that were sampled from the standing trees were also ground in a Wiley mill to pass an 80-mesh screen after being dried in a conditioning chamber (temp: 22°C; relative humidity: 55%) until the average moisture content (MC)

was 9%. NIR spectra of test samples were collected on this milled material with a PerkinElmer Spectrum Model 400 NIR spectrometer (Waltham, MA, USA). The wave-number range of the instrument was from 10000 cm<sup>-1</sup> to 4000 cm<sup>-1</sup>. A sample was scanned 32 times at a resolution of 4 cm<sup>-1</sup> and averaged into one spectrum for analysis. Spectrum of a Spectralon (Labsphere Inc., North Sutton, NH, USA) standard was taken as the background reference sample every 20 minutes to correct for potential drifts with time.

### Multivariate Data Analysis

PerkinElmer Spectrum Quant+ software (Waltham, MA, USA) was used to develop Partial Least Squares regression (PLS) models. The PLS procedure used the NIPALS algorithm to extract successive linear combinations (i.e., factors or components) of the predictors such that variations in both responses and predictors were optimally explained. A dataset comprising 260 samples was split into a calibration set (i.e., 70%) and an independent test set (i.e., 30%). Samples were randomly assigned as either calibration or test set. Using the first derivatives of NIR spectra as the X-matrix and the laboratory-measured density, MOR, or MOE as the Y-matrix, PLS models were built using the leave-one-out (LOO) cross-validation technique. In this method,  $n - 1$  samples are used in training a model that is validated with the held-out sample. This is iterated until each observation has been used as validation data.

The performances of validated models were evaluated using the standard error of calibration (SEC), standard error of cross-validation, standard error of prediction (SEP), coefficient of determination ( $R^2$ ), and the residual predictive deviation/ratio of performance to deviation (RPD). The SEC is an evaluation of how precisely the regression line fits the data, whereas SECV is used to evaluate how well a model predicts samples that were not included in the calibration. SEP measures the precision of a model's predicting ability corrected for bias, which detects any systematic difference between the calibration set and prediction set.  $R^2$  measures the total variance between measured and predicted that can be modeled linearly. Finally, RPD is the ratio of SEP to standard deviation of the reference data (Esbensen 2002). The optimized models that had the best statistics (i.e., lowest error values) were selected, validated with the independent test set, and then used to predict the basic density, MOE, and MOR of the elite loblolly pine families.

To further corroborate the robustness of developed NIR-based models, the acoustic-MOE (MOE<sub>ac</sub>) of the elite loblolly pine families were computed for comparison with NIR-predicted MOE. Acoustics is a well-established and widely accepted technique used by the wood industry for log segregation and product grading among others (Wang et al. 2007). Acoustic data was collected with a FAKOPP Microsecond Timer (Fakopp Enterprise, Agfalva, Hungary), which operates based on the Time-of-Flight (ToF) principle. The transmitter and receiver probes were positioned 120 cm apart on the same side of the tree, ensuring that the midway of these probes is at breast height. A stress wave was then generated with the strike of a steel hammer at a steady force (Wang et al. 2007, Essien et al. 2016). The tree velocity (VT) was computed as the ratio of the distance (m) between the probes (in m) and the time (s) it takes the stress wave to travel from the transmitter to the receiver. The MOE<sub>ac</sub> was calculated as:

$$\text{MOE}_{ac} = (\text{VT})^2 \times \text{density}.$$



Finally, the PROC GLM procedure in SAS (SAS Institute Inc. Cary, NC, USA) was used in the analysis of variance (ANOVA) for the studied properties of the elite loblolly families. Tukey-HSD tests with alpha set to 0.05 were conducted when needed to further investigate pairwise comparison between the treatments. All graphics and tables were produced with MS Excel (Microsoft Corp., Redmond, WA, USA).

## Results and Discussion

### NIR Model Calibration and Evaluation

Descriptive statistics of the density and strength properties of samples used in model training and validation are presented in Table 1. For the total sample set, the MOE ranged from 2380 MPa to 17300 MPa. The highest MOE measured for loblolly pine used in this study is, however, lower than what has been reported in the literature (So et al. 2002). Minimum and maximum values were respectively 25 MPa and 148 MPa for MOR and 0.37 g/cm<sup>3</sup> and 0.79 g/cm<sup>3</sup> for the density. Wide ranges observed in the dataset help improve the robustness of models and in their predicting properties of future unknowns (Via 2003, Haartveit and Flæte 2006). Good overlaps were noted between the means of the calibration and independent validation dataset for all three properties. The average MOE was 8923 MPa (SD = 2534 MPa) for the training set and 8643 MPa (SD = 3759 MPa) for the test set.

Results obtained via the conventional methods were used as the response variables (i.e., Y-variables), whereas NIR spectra was used as the predictor variables (i.e., X-variables) in PLS modeling of properties.

NIR is able to model non-chemistry secondary traits such as density and strength properties of wood because the chemical composition affects these traits. For instance, the fiber tracheid of wood, which is responsible for the strength of wood, is made up of cellulose, hemicelluloses, and lignin. The linear orientation and high degree of polymerization of cellulose makes it the primary contributor to strength, whereas lignin binds the fibers together and also serves as a stiffening agent. Hemicelluloses, on the other hand, act as a matrix for cellulose as well as link the fibrous cellulose to the amorphous lignin. In the case of density, the ratio of cell wall material (i.e., cellulose, hemicelluloses, and lignin) to lumen is a major determinant (Winandy and Rowell 1984, Haygreen and Bowyer 1989).

Results from PLS models that correlated first-derivative NIR spectra to a single property are presented in Table 2. Four latent

variables (LVs) were used in the construction of the optimum model for MOE and MOR, and three were used in developing the density model. The standard error of cross-validation (i.e., SECV), which estimates the errors that will be associated with a model's ability of predicting future unknown samples, was 1267 MPa, 11 MPa, and 0.04 g/cm<sup>3</sup> for MOE, MOR, and density, respectively. The coefficient of determination for the cross-validated models was lowest for density (i.e., R<sup>2</sup> = 0.70) and highest for MOE (i.e., R<sup>2</sup> = 0.75). The RPD, a measure of robustness, was used to evaluate the predictive accuracy of cross-validated models. The relatively lower errors of PLS models compared to the standard deviation of the reference data produced models with good RPDs. For all the three properties, the RPD of models was greater than the 1.5 criteria required in order for a model to be considered as a preliminary screening tool (Hein et al. 2009). Schimleck et al. (2005) conducted a similar study using NIR to estimate the density, MOE, and MOR of loblolly pine (mean age of 22) obtained from 81 plantations across the southern United States. The SEC and SECV obtained by the researchers were very similar to what was determined in this study (i.e., SEC = 0.03 g/cm<sup>3</sup>, 1460 MPa, and 8.6 MPa; and SECV = 0.03 g/cm<sup>3</sup>, 1480 MPa, and 9.5 MPa for density, MOE, and MOR, respectively). The authors, however, reported higher R<sup>2</sup> (0.82–0.89) and RPD values (2.3–2.6) for their cross-validated models.

When the optimum cross-validated models were used in predicting the three properties for an independent test set ( $n = 70$ ), the larger errors consequently reduced the coefficients of determination to 0.19 for density, 0.41 for MOR, and 0.45 for MOE. The SEP values were 0.07 g/cm<sup>3</sup>, 19 MPa, and 2011 MPa for density, MOR, and MOE, respectively. Errors that are associated with the prediction of an independent dataset are usually larger since they factor in how much worse a model performs when applied to this test set not originally used in model training. Even though the calibration and test sets were split to ensure overlapping ranges, and the same set of samples were used for all three properties, the sets of density seemed especially different because in addition to the density model performing poorer, there was also more variation in the density of the different sections of wood along the stem compared to the strength properties. Furthermore, the generally poor predictive statistics could be attributed to the diverse genetic composition of the samples in the test set. As such, the test set was separated out into individual families and one-way ANOVA ( $\alpha = 0.05$ ) conducted to test the equality of property means between NIR

**Table 1. Descriptive statistics of the basic density, MOE, and MOR of southern pine wood.**

Dataset	Property	Mean	SD	Min	Max	SE
Total set $n = 260$	MOE (MPa)	8848	2909	2380	17300	180
	MOR (MPa)	82	25	25	148	2
	Density (g/cm <sup>3</sup> )	0.52	0.09	0.37	0.79	0.01
Training set $n = 190$	MOE (MPa)	8923	2534	2380	15100	184
	MOR (MPa)	82	21	26	132	2
	Density (g/cm <sup>3</sup> )	0.52	0.08	0.37	0.75	0.01
Test set $n = 70$	MOE (MPa)	8643	3759	2540	17300	449
	MOR (MPa)	81	33	25	148	4
	Density (g/cm <sup>3</sup> )	0.55	0.12	0.39	0.79	0.01
Loblolly pine families $n = 180$	MOE (MPa)	8433	3128	2380	17300	180
	MOR (MPa)	82	28	35	148	2
	Density (g/cm <sup>3</sup> )	0.54	0.09	0.37	0.79	0.01
Commercial lumber $n = 80$	MOE (MPa)	9782	2084	5780	14300	233
	MOR (MPa)	81	15	41	112	2
	Density (g/cm <sup>3</sup> )	0.49	0.06	0.39	0.63	0.01

**Table 2. Calibration and prediction statistics of NIR-based PLS models.**

Fit statistics	Density (g/cm <sup>3</sup> )	MOR (MPa)	MOE (MPa)
Number of LVs	3	4	4
SEC	0.036	9.59	1100
SECV	0.042	11.33	1267
R <sup>2</sup>	0.7	0.71	0.75
RPD <sub>cv</sub>	1.81	1.87	2
SEP <sub>iv</sub>	0.065	19.4	2011
R <sup>2</sup> <sub>iv</sub>	0.19	0.41	0.45

Note: Subscript cv means cross-validation; iv means independent validation.

predicted and conventionally measured. Results from these tests showed no significant differences between the measurement techniques within each family. Regression plots relating the measured and NIR-predicted properties are shown in Figure 1.

## Prediction and Screening of Loblolly Pines Families Density

The mean densities of the families ranged from a low of 0.38 g/cm<sup>3</sup> (SD = 0.02 g/cm<sup>3</sup>) to a high of 0.50 g/cm<sup>3</sup> (SD = 0.07 g/cm<sup>3</sup>) on the Georgia site. For the Florida site, the range was from 0.37 g/cm<sup>3</sup> (SD = 0.03 g/cm<sup>3</sup>) to 0.49 g/cm<sup>3</sup> (SD = 0.06 g/cm<sup>3</sup>). The range of density predicted by NIR is comparable to the 0.44 g/cm<sup>3</sup> to 0.51 g/cm<sup>3</sup> reported for 10-year-old loblolly pine trees by Belonger et al. (1997) using X-ray densitometry, as well as the 0.39 g/cm<sup>3</sup> to 0.56 g/cm<sup>3</sup> determined for 12-year-old loblolly pine trees by Jones et al. (2008). A two-way ANOVA ( $\alpha = 0.05$ ) showed that the families differed significantly in their densities on the two sites. In addition, the interaction term had a *P*-value of less than 0.05 (Table 3).

From Figure 2, families A9 and A26 had the highest densities on the Georgia site. These were statistically higher than the densities of families A37, A5, A13, and A33. On the Florida site, families with higher densities included A1, A9, A10, A2, and A26. The densities of A1, A9, A10, and A2 were statistically higher than the densities predicted for A33, F17, A21, F23, and A13.

The elite loblolly pine families did not necessarily rank the same on both sites, with the interaction term being significant (Table 3). The most extreme case was for F23, whose density was among the highest on the Georgia site but among the lowest on the Florida site.

The density of wood has been shown to vary both within a single tree and among trees of a species. Within a tree, the ratio of cell wall material to lumen is known to be a major contributor to density. This overarching trait is due to several factors, such as the ratio of earlywood to latewood, cell size, and wall thickness, and the percentage of the various cell types such as fiber tracheids, ray cells, and vessel elements. Furthermore, the occurrence of extractives can have a pronounced impact on density (Zobel and van Buijtenen 1989). Among softwoods, the density generally decreases farther up the tree and increases outward from the pith toward the bark (Zobel and Sprague 1998). Within a species, density has been reported to vary based on such factors as site conditions and genetic sources (Haygreen and Bowyer 1989). As Zobel and van Buijtenen (1989) pointed out, the properties of wood are a result of the interaction between the genetic potential of the tree with its growing environment.

The different densities noted between the individual families on a particular site are due to the genetic variation (*P*-value < 0.0001) (Table 3). On the other hand, differences noted within an individual family on the two forest sites are a function of the environment, as also observed by Jordan et al. (2008). According to the authors, the differences in wood properties are probably due to the duration of juvenile wood production, which increases as one moves northwest from the South Atlantic region. Also, the occurrence of moisture, especially in the summer, promotes the production of latewood, thus higher density. This was, however, not the case for all the families investigated in this study.

Even though there was a site effect on density and most families did not rank the same on the two sites, some families including A9, A1, A26, and A2 did consistently well on both sites. On the flip side, the densities of families A13, A33, F17, and A37 were low on the different sites.

## Modulus of Rupture (Bending Strength)

The range of predicted MOR values for the elite families was narrower on the Georgia site (i.e., 47 MPa to 143 MPa) compared to the Florida site (i.e., 34 MPa to 150 MPa). According to the ANOVA results in Table 3, there were significant differences in the MOR of families tested. Furthermore, the family  $\times$  site interaction was significant. However, the effect of site was not statistically significant.

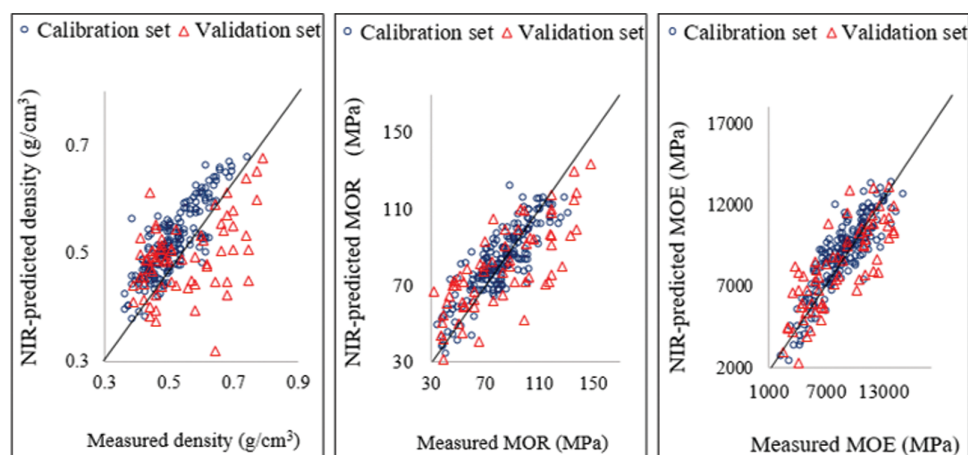
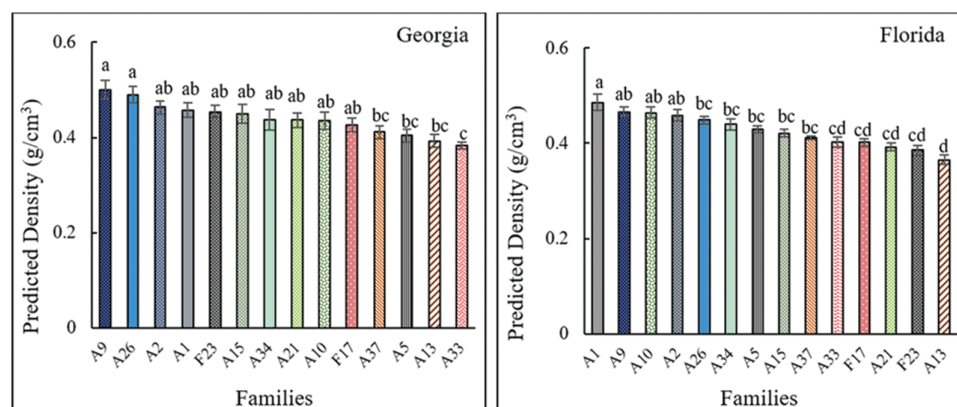


Figure 1. Relationship between measured and NIR-predicted property.

**Table 3. ANOVA F-test values and *P*-values of density, MOR, and MOE by treatment.**

Treatment	df	Density (F-value)	Density ( <i>P</i> -value)	MOR (F-value)	MOR ( <i>P</i> -value)	MOE (F-value)	MOE ( <i>P</i> -value)
Family	13	9.24	< 0.0001	5.7	< 0.0001	3.45	<0.0001
Site	1	4.79	0.0294	0.79	0.3747	0.34	0.5625
Family x site	13	2.34	0.0055	2.94	0.0005	0.93	0.5198



**Figure 2. Rank of loblolly pine families for density on the two sites. \*Bars with different letters are significantly different at 95% confidence level (Tukey's HSD test).**

In spite of the significant interaction effect, the MOR of A1, A2, A9, and A26 ranked high on the two sites, whereas A33, A21, and A5 were among the least strong on both sites (Figure 3).

Rankings of families based on MOR generally followed the trend noted for the density. For instance, on the Georgia site, A9 had the highest density and MOR, with A33 having the lowest values for the two properties. A regression plot of density versus MOR showed the former accounting for 41% of the variation noted in MOR (Figure 4). This result is similar to what has been reported in the literature about the correlation of density with MOR (Hein et al. 2013).

Mean MOR values determined for the elite loblolly pine families of this study were comparable to what has been reported by Schimleck et al. (2005) for older loblolly pine trees with a mean age of 22 years. The higher MOR for these relatively younger trees is due in part to their relatively higher density. MOR increases with increasing density because more stress is required to cause the failure of more material. In Table 1, it was noted that the mean density of the families was higher (0.54 g/cm<sup>3</sup>) than the mean density of the lumber (0.49 g/cm<sup>3</sup>) acquired from the commercial sawmill. Pearson and Ross (1984) reported similar results whereby the density to 15-year-old loblolly pine was slightly higher (0.52 g/cm<sup>3</sup>) than that of 25-year-old trees (0.5 g/cm<sup>3</sup>). The saturation of wood near the pith with extractives has been known to increase the density of the juvenile wood without improving the strength (Larson et al. 2001, Via 2003). However, since higher density of the pine families corresponded to an increase in MOR, the higher density values could be attributed to the cell characteristics rather than extraneous depositions. Further studies of the anatomical features of the elite families will, however, be required in ascertaining this occurrence.

### Modulus of Elasticity (Stiffness)

ANOVA testing of the effect of family, site, and the interaction of family x site on the stiffness was only significant for family

(Table 3). As such, replicates of families from the two sites were pooled together for further analysis.

The range of MOE values predicted for the pine families was from 2981 MPa to 15830 MPa. The mean MOE for the elite families was highest for A9—10946 MPa (SD = 2703)—and lowest for A33—7782 MPa (SD = 2237). NIR-predicted MOE of loblolly pine in this study was comparable to the 2500 MPa–15327 MPa range reported by Kelley et al. (2004). However, the maximum MOE for the elite families was lower than the 23000 MPa determined for loblolly pine aged between 21 to 26 years (Jones et al. 2005). The upward shift of MOE with age is due to the fact that the proportion of juvenile wood (which has thin-walled shorter cells, as well as a higher proportion of earlywood to latewood) is inversely related to age. For instance, Zobel and Blair (1976) stated that 15-year-old loblolly pine has about 85% of its wood as juvenile wood but this proportion decreased drastically to 19% by age 40. Apart from the presence of juvenile wood adversely affecting the density, it also has large microfibril angles and excessive spiral grain.

Compared to the MOR, density accounted for less of the variation in MOE ( $R^2 = 0.2$ ) (Figure 5). Similar results were reported by Burdon et al. (2001) when they investigated the relation of density to the MOR and MOE of *Pinus radiata* D Don. Several studies have shown that the microfibril angle (MFA) has a greater influence on the MOE than density (Tsehaye et al. 1998, Evans and Ilic 2001, Ivkovic et al. 2009). Also, it has been reported that the MFA correlates to the MOR to a lesser extent than it does to MOE (Hein et al. 2013).

Thus, the MFA of the families, which is under moderate to strong genetic control, could have been the major contributor to the differences noted in the MOE of elite families. In terms of ranking, the MOE of families A9, F23, A1, A26, A10, A2, and F17 was all statistically higher than the 7782 MPa predicted for A33 (Figure 6).

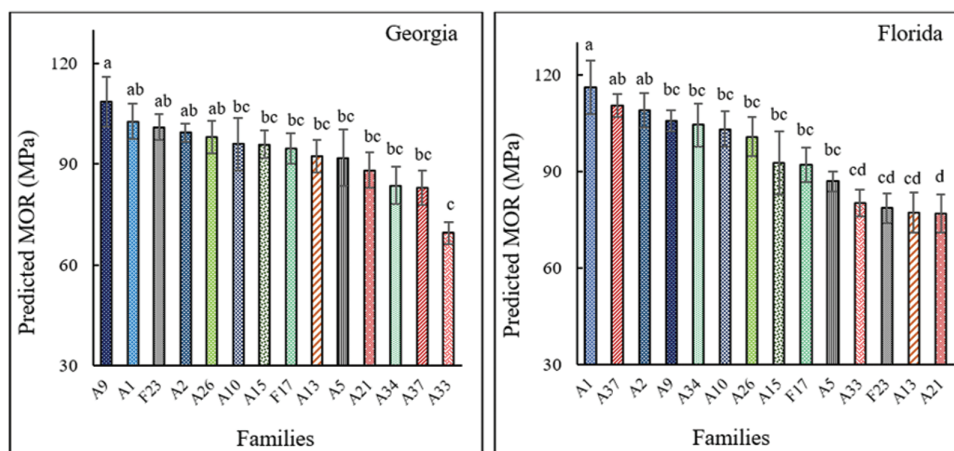


Figure 3. Rank of loblolly pine families for MOR on the two sites. \*Bars with different letters are significantly different at 95% confidence level (Tukey's HSD test).

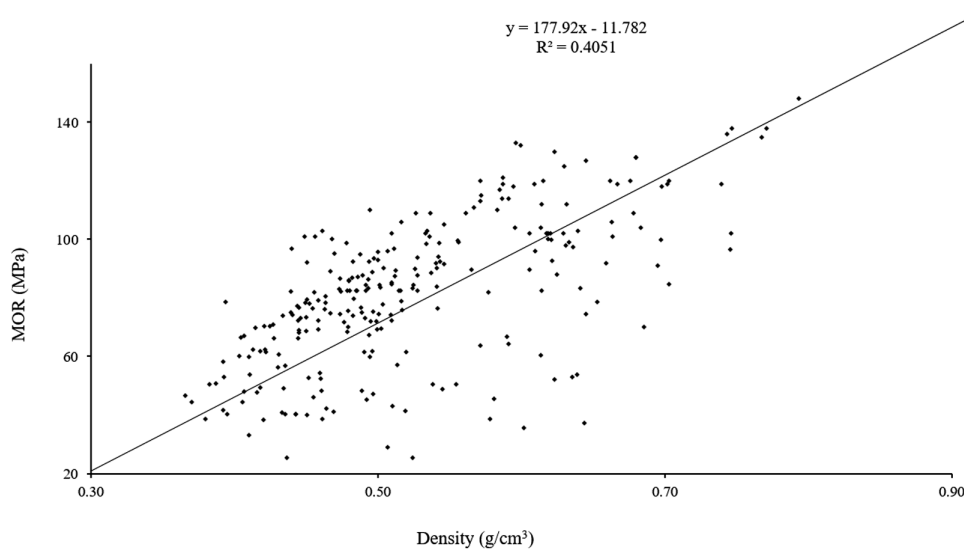


Figure 4. Correlation between the density and MOR of wood.

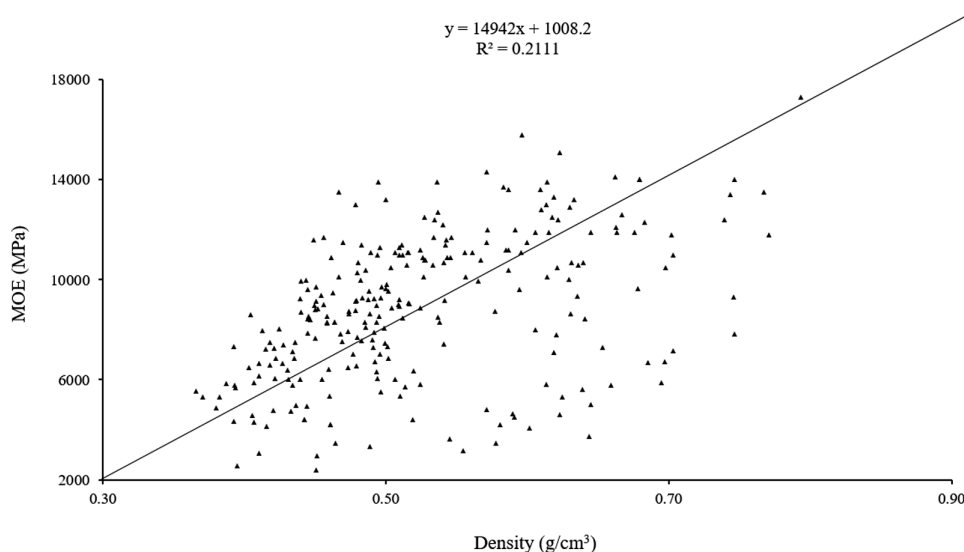


Figure 5. Correlation between the density and MOE of wood.



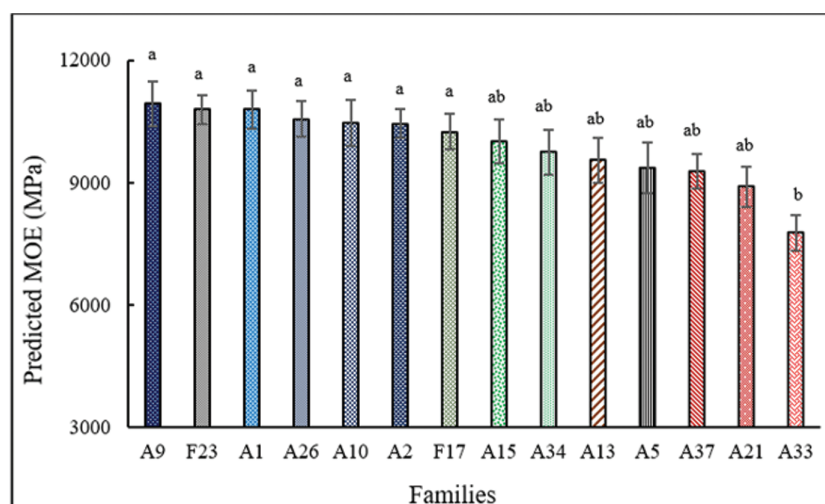


Figure 6. Rank of loblolly pine families for MOE. \*Bars with different letters are significantly different at 95% confidence level (Tukey's HSD test).

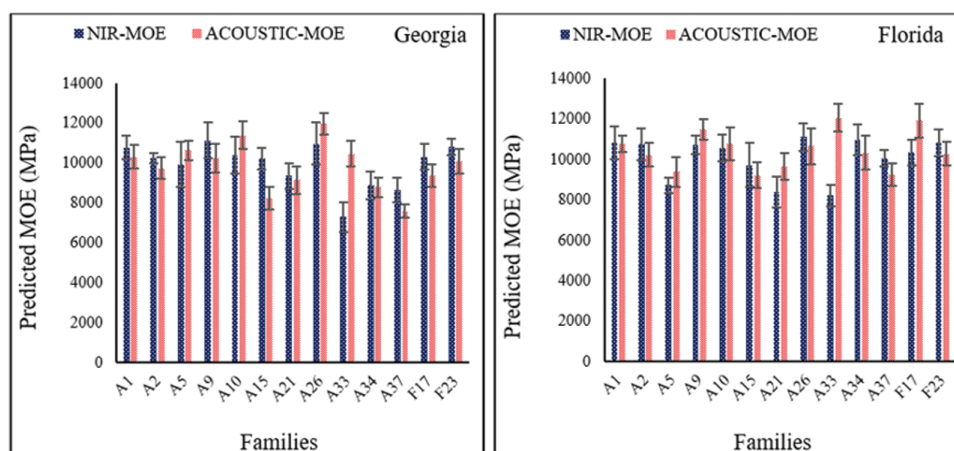


Figure 7. MOE of the loblolly pine families as predicted by NIR and acoustics on the two sites.

### Comparison of NIR-Predicted MOE Using Acoustics-Predicted MOE

The stiffness of the elite pine families as predicted by NIR and acoustics was compared as a further measure of ascertaining the robustness of developed NIR models. One-way ANOVA of the two tools was not significant, with an F-value of 0.58 and a  $P$ -value of 0.45 ( $\alpha = 0.05$ ). For better insight, the NIR predicted versus acoustics predicted was plotted for each of the families separately for the two sites. As can be seen in Figure 7, the MOE of the families was estimated more or less equally by the two nondestructive techniques. However, NIR consistently underestimated the stiffness of A33. It had been determined earlier that this family was among those with low densities. As such, the MFA might be a bigger contributor to the MOE (Tsehaye et al. 1998, Evans and Ilic 2001, Ivkovic et al. 2009) of this particular family than density. However, as already stated, further studies of the anatomical features of the elite families will be required to test this hypothesis. The underestimation of the MOE of this family on both study sites by NIR might also be an indication of the limitation of the tool in being less sensitive to the MFA.

### Conclusions

This study demonstrated that NIR spectroscopy can be used as a high throughput tool to non-destructively screen standing trees for important wood traits. NIR-based PLS-models were developed and used to predict the density, bending strength (MOR), and stiffness (MOE) of 351 living trees representing 14 elite loblolly pine families planted on two forest sites. MOR values of the loblolly pine families were noted to be comparable to what have been reported in the literature for older loblolly pine trees, suggesting that the elite families have improved strength. Analysis of Variance showed an effect of family on the three traits that dictate wood quality for structural applications. The significant interaction term is an indication that the density and MOR of a family could vary depending on the environment. Further studies with more sites would, however, be helpful in estimating the extent of the family by site interaction. Such knowledge would be valuable for tree breeders and landowners in decisions to plant families with desired traits in certain environments. The developed methodology can be directly applied to the other hundreds of loblolly pine families in tree improvement programs, and also leveraged



toward other wood species. This will facilitate endeavors to make the right feedstock available to support the conventional forest products industry.

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