SCREENING *PINUS TAEDA* (LOBLOLLY PINE) FAMILIES FOR PHYSICAL AND MECHANICAL PROPERTIES USING VIBRATIONAL SPECTROSCOPY

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Abstract—In a bid to control the loblolly pine decline complex, stakeholders are using the selection and deployment of genetically superior families that are disease tolerant. It is vital that we do not compromise other important properties while breeding for disease tolerance. In this preliminary study, near infrared spectroscopy was utilized in conjunction with data collected via conventional methods to develop partial least squares regression models that were used to rapidly predict the basic density, stiffness and ultimate strength of 14 genetically superior loblolly pine families that have been selectively bred to be disease tolerant. Calibration and independent validation data were from southern pines acquired from a commercial sawmill. Seven or eight latent variables were used in model development. The coefficients of determination of the predictive models were more promising for MOE (0.58) and MOR (0.42).

INTRODUCTION

Pinus taeda (loblolly pine) is the most economically important tree species in the USA. With 30 million acres in plantations in the southern US alone, it provides 110,000 jobs and contributes approximately $30 billion to the economy of this region. However, over the past 50 years, reduced growth, decline and eventual mortality have been associated with loblolly pine trees (Eckhardt and others 2010). In a bid to control this decline complex, stakeholders are using the selection and deployment of genetically superior families that are disease tolerant. It is vital that we do not compromise other important properties while breeding for disease tolerance, and the only way to determine this is to measure them. However, with the large number of trees involved in tree breeding programs, it will not be feasible to determine these properties with the conventional methods that require considerable sample preparation thus time consuming, expensive and mostly destructive. There is therefore the need for alternative analytical tools that have high throughput and are cost effective.

In this preliminary study, near infrared spectroscopy (NIRS) coupled with multivariate data analysis was used to characterize and then rapidly predict the basic density, modulus of elasticity (MOE) and modulus of rupture (MOR) of loblolly pine families that have been selectively bred to be disease tolerant. These properties are important because any changes in them will impact the yield and/or quality of final products – whether it be pulp, paper, lumber or engineered wood product. Furthermore, it is essential that strength is not compromised else mortality due to reasons other than forest disease such as wind failure could occur.

MATERIALS

Fourteen year old loblolly pine trees were harvested from two forest sites. Seven families were harvested from Yulee FL and seven from Waycross GA in February and March of 2014 respectively. The diameter at breast height (DBH) of these trees ranged from 12.4 cm to 21.6 cm. Trees were crosscut into 1.5 m bolts along the bole, then 50 cm 'disks' were taken from the mid portions for further processing into the final test specimen. Nominal 2 x 4-in southern pine boards were also acquired from West Fraser Inc., a commercial sawmill located in Opelika AL.

METHODS

Conventional Testing

For mechanical testing, the three-point bending test as specified in ASTM D143 was employed. Test specimens cut to final dimensions of 2.5 x 2.5 x 41 cm were conditioned to an average moisture content (MC) of about 9 percent in a control chamber (temp: 22 °C; relative humidity: 55 percent) before testing. Samples were 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 MOE (i.e. Stiffness) was computed as the slope of the linear portion of the

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load-deflection curve. MOR (i.e. Ultimate strength) was calculated as

$$\frac{a \cdot Pmax}{(b \cdot h^3/12)}$$

where

a: Distance between loading point and nearest support
Pmax: Maximum load
b: Width
h: Height

Basic density was determined as the ratio of the mass of a test specimen to its volume.

Near Infrared Spectroscopy (NIRS)

The infrared region is the wavelength range of 780 nm – 1 mm (wave number range 12820 – 10 cm^{-1}) that lies between the visible and microwaves regions of the electromagnetic spectrum. NIRS measures the amount of near infrared light a sample absorbs, transmits or reflects based on its chemical composition. Spectra was collected from samples ground to pass an 80-mesh screen (Jiang and others 2014) with a PerkinElmer Spectrum Model 400 NIR spectrometer. A sample was scanned thirty-two times at a resolution of 4 cm^{-1} and averaged into one spectrum for analysis.

Multivariate Data Analysis – Model Calibration and Validation

PerkinElmer Spectrum Quant+ software was used to develop Partial Least Squares regression (PLS) models. The PLS1 algorithm, utilized the raw untreated NIR spectra (i.e. absorption of near infrared light at different wavelengths) as the independent (X) variables and regressed them on the results acquired from the conventional testing methods (i.e. dependent – Y variables). Thirty-one samples from the southern pines stock was used for calibration. An additional thirteen samples from this material that were not part of the calibration set were used in independent validation. Several statistics including standard error of calibration (SEC), standard error of prediction (SEP), coefficient of determination (R^2) and residual predictive deviation / ratio of performance to deviation (RPD) were used to evaluate the performance of our models. Models that had the least error values were selected and used to predict the basic density, MOE and MOR of the fourteen genetically superior loblolly pine families.

RESULTS AND DISCUSSION

NIR Spectra

Raw NIR spectra characteristic of loblolly pine used for model development is shown in figure 1. Spectra highlights some important peaks that have been assigned to specific chemical components of wood. For instance, the peak occurring at 4080 cm^{-1} is known to result from cellulose, while that at 6945 cm^{-1} has been attributed to the presence of lignin and extractives (Schwanninger and others 2011). These chemical constituents are the basis for the application of NIRS in the prediction of density and strength of wood – wood chemistry have been shown to have strong correlations with these non-chemical properties. Cellulose has a strong relationship with density, which in turn has an influence on the stiffness and ultimate strength of wood (Via and others 2003).

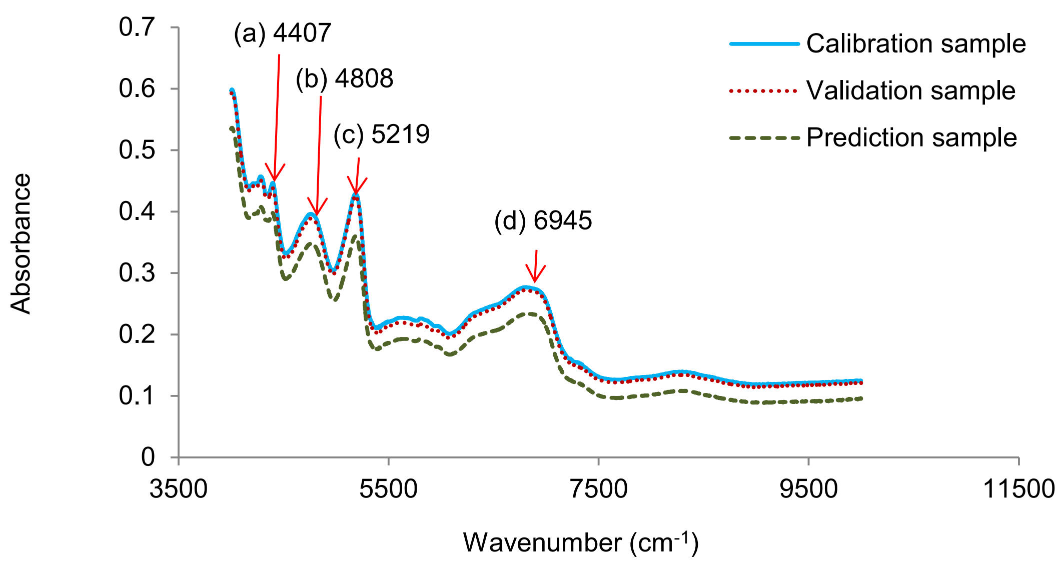

Figure 1—Characteristic NIR spectra of loblolly pine wood showing some important wavenumbers. (a) - cellulose and hemicelluloses; (b) - cellulose; (c) - water; (d) - lignin and extractives.

Table 1—Conventional lab results used for model development and independent prediction

	Basic Density (g/cm^3)	*MOE (MPa)*	*MOR (MPa)*
Calibration set (n = 31)			
Min	0.43	5800	46.2
Max	0.69	15100	125.0
Average	0.56	10100	83.4
SD	0.07	2500	17.3
Validation set (n = 13)			
Min	0.47	6200	41.2
Max	0.62	13900	110.0
Average	0.44	9400	77.6
SD	0.03	2100	17.8
Prediction set (n = 14)			
Min	0.51	6010	40.3
Max	0.65	15100	130.0
Average	0.56	10091	97.4
SD	0.04	2601	40.3

Conventional Lab Results

The density, MOE, and MOR of loblolly pine measured using the conventional methods are presented in table 1. Average density was 0.56 cm^{-3} for the calibration set and 0.51 cm^{-3} for the independent test set. Stiffness for all samples used in this study ranged from a low of 5800 MPa to a high of 15100 MPa. The calibration set was slightly stiffer than the validation set. For ultimate strength, the range of the calibration set was wider than the range of the validation set -- i.e. 78.8 MPa and 68.8 MPa respectively.

Although means of the calibration and validation sets had good overlap which help with model performance, the range of especially the mechanical properties were not as wide as seen in the literature. MOE have been reported to be as low as 2200 MPa, and as high as 26000 MPa (Kelley and others 2004, So and others 2002).

PLS Model Performance

Fit statistics showing how models performed are presented in table 2. Seven or eight latent variables (LVs) were used in developing PLS models. LVs are linear combinations of the x and y variables extracted in such a way that variation in both response and regressors are optimally explained. The proportion of variance accounted for by the first LV is the maximum, and this progressively decreases as successive LVs are extracted. SEC was used to evaluate how precisely the regression line fitted the data, while SEP measured the precision of a model's predicting ability during validation. The closer these errors are to zero, the better the model. A small difference between calibration and validation errors is an indication that the test sets were well predicted by calibration models. Ideally, the SEP of a model should not be more than 1.3 times its SEC (Acquah and others 2015). Only the models for predicting stiffness and ultimate strength passed this criteria. The SEC and SEP were respectively 1800 MPa and 1900 MPa for MOE, and 13.5 MPa and 17.4 MPa for MOR.

R^2 is a measure the total variance between measured and predicted that can be modeled linearly. Usually, R^2 keeps increasing as more LVs are added in the development of a model. To ensure models were not overfitted, we computed the adjusted R^2, a measure that penalizes if insignificant LVs are added to a model just so the R^2 get inflated. R2adj values ranged from a low of 0.51 for MOR, to a high of 0.64 for basic density. Furthermore, another statistic, the RPD was used to evaluate the SEP in terms of SD of the reference data (i.e. y-variables). In order for a model to predict a parameter with precision, the SEP should be considerably lower than SD. RPD values for all three models were less than 1.5. For a model to be

Table 2—Fit statistics of PLS regression models using raw NIR spectra

Property	LVs	SEC	SEP	SD	RPD	R^2	R^2_{adj}	R^2_{pred}
Basic Density (g/cm^3)	8	0.05	0.08	0.07	0.9	0.65	0.64	0.19
Modulus of Elasticity (MPa)	8	1800	1900	2500	1.3	0.63	0.62	0.58
Modulus of Rupture (MPa)	7	13.5	17.4	17.3	1.0	0.53	0.51	0.42

applicable for preliminary predictions in tree selection for improvement programs, this threshold should be exceeded (Hein and others 2009).

Although the model developed for basic density had the highest adjusted R^2, its RPD was less than 1. Thus as expected, it performed worst when it was applied in parameter prediction for the fourteen genetically superior loblolly pine families. Even though the models for stiffness and ultimate strength were not the best, their predictive ability was promising when applied to the loblolly pine families that were not included in model calibration, table 2.

CONCLUSIONS

In this preliminary study, PLS regression models were developed with NIR spectra of southern pines and reference data acquired through standard methods. These models were used to rapidly predict the basic density, Modulus of Elasticity (Stiffness) and Modulus of Rupture (Ultimate strength) of fourteen *Pinus taeda* (loblolly pine) families that have been selectively bred to be disease tolerant.

Although the number of LVs used in model development was relatively high, the similar R^2 and adjusted R^2 values was an indication that models were not overfitted. RPD values for models developed in this study were however low. Inspite of this, the linear relationship between measured and NIR predicted parameter were promising for the loblolly pine families that were not included in model calibration. (i.e. MOE = 0.58; MOR = 0.42).

Work is ongoing to increase the precision of the predictive ablity of these models. At the completion of this project, models should be able to rapidly predict not only the studied properties of these 14 families, but also other genetically superior loblolly pine families that are currently being deployed by stakeholders. The long term goal of this study is to make the right feedstocks available for the conventional forest industry, as well as to support the emerging bioeconomy.

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