Near Infrared (NIR) Spectroscopy to Predict Physical Properties of *Acacia mangium* at Three Different Age Classes

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Abstract

Near Infrared (NIR) spectroscopy has been used to predict several properties of wood. This is one of the nondestructive testing (NDT) methods providing fast and reliable wood characterization analysis which can be applied in various manufacture industry, included forest sector, in control and process monitoring task. Moisture content and wood density are important properties related to strength properties. The aim of this study was to evaluate NIR technique in obtaining calibration models for determining moisture content and wood density of *Acacia mangium* in the age of 5, 6, 7 years-old. Spectra were measured in both solid and ground wood samples. Laboratory testing of physical properties were determined by volumetric and gravimetric methods. The laboratory values were correlated with the NIR spectra using multivariate analysis statistic of Partial Least Square (PLS). The calibration-validation model of this relationship was evaluated by using the coefficient of determination (R^2), root means square error of calibration (RMSEC) and cross-validation (RMSECV) values. Generally, a better accuracy was obtained by using calibration model of ground wood compared to that of solid wood samples. At age of 7 years-old, the R^2 allowed the use of NIR spectra of solid samples to develop calibration and validation model, especially for wood density. Based on ratio of performance to deviation (RPD) and RMSE, ground samples demonstrated a higher value of RPD, RMSEC, and RMSECV compared to solid wood for all properties.

Keywords: near infrared (NIR), wood density, *Acacia mangium*, nondestructive testing (NDT), Partial Least Square (PLS).

Introduction

Near Infrared (NIR) is one of the emerging technologies which already being utilized to evaluate the properties of wood and wood products. There are some advantages of NIR spectroscopy technology including more rapid analysis and the ability to operate in a number in-field or on-line/at-line environments. Some material property analyses need destructive analyses, use specific equipment analysis and require long time testing. NIR spectroscopy technique of non destructive testing is used as an alternative method to assess material properties through fast and reliable analysis. NIR spectroscopy uses electromagnetic spectrum region extended from wave length of 780 nm to 2500 nm. In this region, spectra may be characterized by band assignments of chemical component vibration as the response of the absorption bands in NIR region. In wood, overtone and combination bands (stretching and deformation) of fundamental vibrations due to large force constants and low masses involved through hydroxyl group bonds (So *et al.* 2004; *et al.* 2011). NIR spectroscopy has been widely applied in agricultural products such as cereal, beverages, dairy products, textiles, and pharmaceutical applications (Burns and Ciurzak 2008). In forest product industry, NIR spectroscopy has been used at processing stage to control the product quality. It is a rapid assessment tool to predict wood quality, chemical properties of wood, pulp and paper properties, density, moisture content, grain angle and surface roughness, anatomical properties and mechanical properties (Schimleck *et al.* 2001a,b; So *et al.* 2004; Tsuchikawa 2007).

In analyzing of NIR spectroscopy to predict wood properties, chemometric approach is involved through multivariate data analysis. Partial Least Squares (PLS) and Principal Component Analysis (PCA) are two multivariate characterization data analyses commonly used (Antti 1999; Burns and Ciurzak 2008). In Indonesia, research of NIR spectroscopy to predict wood and wood products is still limited. The aim of this study was to predict wood density and moisture content of tropical hardwood of acacia species (*Acacia mangium*) using NIR spectroscopy technology at three age classes.

Materials and Methods

The study used samples of *Acacia mangium* aged 5, 6, and 7 years-old collected from plantation forest of Perum Perhutani at RPH Maribaya, BKPH Parung Panjang, West Java. Four trees were felled from each sample age. The samples were then cut into planks. The samples were processed using band saw for thickness, width and length of 3 x 10 x 25 cm.

The NIR spectra were taken for both solid and ground wood samples using NIRFlex N-500 fiber optic from Buchi® (Switzerland products) (Figure 1). Solid wood samples were tested by mounting a sensor into smooth and clean surface. The measurements were conducted using a contact, diffuse reflectance, fiber optic sensor on three sample surface. Each scanning gave a single spectrum. The ground samples were solid samples which were then milled using a standard Wiley® knife mill until those passed the 40 mesh screen. Samples were held in a sample holder. All

measurements were conducted using a noncontact, diffuse reflectance, fiber optic sensor. The spectra were collected at 0.4 nm interval over the wave length of 1000 ~ 2500 nm. Three scan readings were collected for each sample. NIR diffuse reflectance spectra (R) were obtained from NIR scanning. The absorbance spectra were calculated as $\log(1/R)$.

Chemometric analysis with multivariate analysis (MVA) was conducted using the Unscrambler®X10.2 trial version, CAMO software. A multivariate statistical tool was used to group the data and to develop prediction models (Antti 1999). Partial least square (PLS) is a regression method that is used to near infrared spectra with referenced response variables of moisture content and wood density. The spectral data from each test were handled as untreated data (original), first derivatives and second derivatives. For the 1st and 2nd derivatives, the data were differentiated using Savitsky-Golay (S-G) algorithm method (20-point filter, a second order polynomial and 20 maximum components). The cross validation models were used to identify following calibration parameters: the best pre-treatments, the number of latent variables, outlier samples. The accuracy and stability of models were evaluated by coefficient of determination (R^2) and root mean square error (RMSE), respectively. Four criteria for model selection as following: 1) higher correlation coefficient of cross validation, 2) higher ratio of deviation performance (RDP), 3) lower number of latent variables used in calibration, and 4) standard error of

cross validation (Hein 2010). The root mean square error of calibration (RMSEC) is used to assess calibration performance. RMSEC is determined from residuals of final calibration as a predictor of the model performance. The root mean square error of cross-validation (RMSECV) is calculated from the residuals of each cross validation phase to optimize the calibration model. RMSEC is part of internal validation. Ratio of performance to deviation (RPD) is usually used to present predictive ability of calibration. RPD is calculated by standard deviation of the reference value divided by error of prediction or the RMSECV. This value explained a basis for standardizing the standard error of prediction. Previous study (Schimleck *et al.* 2005; Jones *et al.* 2005; Hein *et al.* 2009; Hein 2010) claimed that a RPD in a range of 1.5 to 2.5 is good enough in considering the model for screening and preliminary prediction of wood properties. The higher RPD points out the better performance of model prediction.

Results and Discussion

Near infrared spectra were collected for at least forty five samples with three scan readings over the range of 1000 to 2500 nm. Each scanning gave a single spectrum. The original spectra for each of the individual sample age were averaged and are shown in Figure 2 for both solid and ground wood samples.

Figure 1. NIR spectroscopy testing using NIRFlex N-500 fiber optic; (a) solid wood sample, (b) ground wood sample.

Figure 2. Average value of raw absorbance NIR-spectra from solid and ground wood samples of *A. mangium* (Karlinsari *et al.* 2012)

There are some obvious visual differences between the absorbance spectra of solid and ground wood samples from one species, as it noticeably shifted upward for solid wood samples. However, little difference of absorbance values

were quantitatively found between the individual spectra from each age. Surface roughness and fiber orientation response to radiation direction seemed to influence NIR absorbance spectra.

Table 1. Results of moisture content and density of *Acacia mangium* wood.

Age	Moisture content (%)	Air-dry Wood density (g/cm ³)	Oven-dry Wood density (g/cm ³)
5 years (n=50)	18.93 (2.27)	0.54 (0.11)	0.46 (0.088)
6 years (n=50)	15.46 (3.78)	0.54 (0.06)	0.47 (0.047)
7 years (n=45)	21.51 (8.89)	0.60 (0.06)	0.50 (0.050)
All years (145)	18.63	0.56	0.48

n: number of samples, number in parentheses denotes standard deviation.

Table 2. Summary of NIR calibration for each age of *Acacia mangium* in ground and solid wood samples.

Physical properties	Age	Wood sample type	Treatment	LV	R ² C (%)	R ² CV (%)	RMSEC	RMSECV	RPD _{CV}
Moisture content	5 years	Ground	1d	6	81.6	59.0	1.060	1.595	1.42
		Solid	1d	8	63.2	54.0	1.376	1.545	1.47
	6 years	Ground	0d	11	83.8	77.2	1.417	1.694	2.23
		Solid	2d	12	78.2	61.5	1.752	2.343	1.61
	7 years	Ground	0d	12	73.4	57.4	4.533	5.767	0.90
		Solid	1d	14	84.8	49.5	3.435	6.307	0.82
	All year	Ground	0d	16	55.2	42.0	4.090	4.070	1.51
		Solid	2d	18	77.4	51.0	2.653	3.920	1.43
	5 years	Ground	1d	5	61.0	37.2	0.063	0.080	1.35
		Solid	1d	10	42.1	13.9	0.082	0.099	1.09
Wood density (air-dried)	6 years	Ground	0d	12	80.3	70.1	0.026	0.032	1.73
		Solid	2d	7	48.7	7.7	0.045	0.053	1.05
	7 years	Ground	0d	14	77.6	58.5	0.029	0.041	1.55
		Solid	1d	15	85.6	51.3	0.024	0.044	1.44
	All year	Ground	0d	16	56.7	43.8	0.059	0.060	1.35
		Solid	0d	17	41.7	24.2	0.065	0.074	1.15
Wood density (oven-dried)	5 years	Ground	1d	5	58.7	31.2	0.052	0.068	1.30
		Solid	1d	8	33.8	11.5	0.071	0.083	1.06
	6 years	Ground	0d	11	79.4	70.5	0.023	0.028	1.69
		Solid	0d	17	61.6	14.9	0.029	0.044	1.08
	7 years	Ground	0d	13	75.6	57.1	0.023	0.030	1.53
		Solid	1d	13	78.7	42.9	0.021	0.035	1.31
	All year	Ground	0d	16	53.6	37.0	0.043	0.050	1.26
		Solid	0d	12	41.2	20.2	0.052	0.061	1.11

0d: original data, 1d: first derivatives, 2d: second derivatives, RPD: ratio of performance to deviation, LV: latent variables, R²C: coefficient of determination of calibration model (%), R²CV: coefficient of determination of cross-validation model (%), RMSEC: root mean square error of calibration, RMSECV: root mean square error of cross validation.

The descriptive statistic for the laboratory testing of moisture content and wood density of the samples are given in Table 1. The average values of moisture content were 21.51, 15.46 and 18.93 for 7, 6 and 5 year-old samples, respectively. The air-dried wood density presented the highest value of 0.60 g/cm³ obtained by 7 year-old wood samples while lower values were resulted from 6 and 5 year-old wood samples with the same average values of 0.54 g/cm³. The oven-dried wood density values were ranging from 0.46 to 0.50 g/cm³.

Original absorbance spectra data were analyzed as well as differentiated data to determine the smoothed value for each point. A valuable spectral pre-treatment method is the use of 1st or 2nd derivatives. Table 2 provides a summary of the partial least squares (PLS) analysis from the ground and solid wood samples.

Results in Table 2 indicated that pre-treatment of first derivate of absorbance spectra was used to determine all physical properties for both ground and solid samples at age of 5 years-old. Meanwhile, original absorbance spectra were applied on ground samples to determine moisture content and wood density at age of 6, 7 and all years. The number component of latent variables (LV) used were mostly a slightly higher in solid samples compared to ground samples. Twenty components in maximum were set on derivation of multivariate calibration model in this study.

The LV in 6 and 7 years-old presented more factors number as well as all years than at age 5 years-old. It denoted that more unwanted variation in spectra was removed in those ages as filtering NIR spectra. The age, correlated with complexities of characteristics of material, was supposed to be influenced on combination of pre-treatment of NIR spectra and latent variables.

Coefficient of determination of calibration (R^2C) was ranged from 0.412 to 0.838, while coefficient of determination of cross-validation (R^2CV) was from 0.202 to 0.848. Poor relationships were presented by a lower value of R^2C and R^2CV as found in solid samples compared to ground samples, except for R^2C of solid wood in age 7 years-old in calibration model (Figure 2). Figure 2b and 2c allowed the use of solid samples to predict wood density at older age of 7 years-old as shown by increasing R^2C and R^2CV significantly. This information pointed out relation of anatomical structure with the age of tree. Further research can be done to know the trend by age on wood properties using multivariate models based on NIR spectra. Previous study by Thygesen (1994), Schimleck *et al.* (2005), Hein *et al.* (2009) and Hein (2010) on wood density using solid samples of temperate region species, *Picea abies*, *Pinus taeda*, and Eucalyptus, revealed that R^2C values ranging from 0.384 to 0.87 using 3 to 11 latent variables.

Figure 2. The value of coefficient determination calibration sample in solid and ground wood sample for moisture content and wood density properties.

RMSE is total error indicated the stability of calibration or validation model. The RMSEC values were lower than RMSECV values of predictive models for all wood properties. This was in line as noted by Burns and Ciurczak (2008) which RMSEC was lower than predictive model parameter of RMSEC or RMSE prediction (RMSEP). RMSEC was known as extremely optimistic estimation of the model performance when more factors were included in calibration model so that the RMSEC always lower. Almost all values for both RMSEC and RMSECV of ground values were lower than those of solid wood samples as shown in Table 2. Those were related to stability of the models. The optimal model will be defined from the significantly lower RMSE. Study on prediction model of pulp yield of *Acacia mangium* (Zhang 2011), prediction of air-dry density of *Pinus taeda* (Schimleck *et al.* 2005), and estimation of wood stiffness of *E. delegatensis*, *P. radiata* (Schimleck *et al.* 2002) concluded the same results.

According to Table 2 the PLS model provided RPD values ranging from 0.82 to 2.23. The RPD values less than 1.0 were found in moisture content properties at age 7 years-old. Most of RPD values were higher in ground wood samples compared to those of solid samples. The unsatisfactory values of RPD revealed could be avoided by adding more samples and minimizing technical aspects such as sample distribution and variation, complexity of the calibration problem and noise level.

Using nondestructive testing of NIR spectroscopy to estimate physical properties of moisture content and wood density of tropical hardwood species of acacia species have shown positive values. The ground wood samples spectra represented that calibration and validation model were better than those of solid wood samples at age 5 to 6 years-old. At the age of 7 years old, the model allowed the use of solid sample based on parameters obtained from the model. However, the statistical parameters can be improved by a large number of samples included considering some technical aspects such as considering a noise level and smoothing data technique.

Conclusions

NIR spectroscopy is promising to predict both moisture content and wood density of *Acacia mangium*'s wood. The Partial Least Square (PLS) calibration for estimating moisture content of wood presented satisfactory coefficient of determination (R^2 more than 0.50).

The predictive calibration and cross validation models showed better correlation and stability model of ground wood samples compared to solid wood. However, solid samples at the age of 7 years-old seemed to be allowed to use in determining wood density.

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