

ESTIMATION OF DENSITY, MOISTURE CONTENT AND STRENGTH PROPERTIES OF *Tectona grandis* WOOD USING NEAR INFRARED SPECTROSCOPY

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ABSTRACT

Near infrared spectroscopy is non-invasive and may be applied as a rapid and cost effective technique for assessment of quality parameters of timber. Near infrared spectra of *Tectona grandis* (teak) wood samples were collected before measuring physical (density, equilibrium moisture content) and strength (flexural and compressive) properties using conventional methods. Partial least squares regression was used to develop calibration models between measured wood properties and near infrared data. The best near infrared spectra pre-processing methods differed by property. Linear calibration models with high R², low error and high ratio of performance to deviation values were observed from partial least squares analysis for different wood properties. These linear models may be applied for rapid and precise estimation of the properties examined in testing and evaluation procedures for commercially valuable teak wood.

Keywords: Calibration models, compression properties, density, equilibrium moisture content, flexural properties, NIRS, timber

INTRODUCTION

The original impetus for research in non-destructive testing and evaluation (NDT&E) of wood is to provide methodologies for assessing quality parameters so that more accurate decisions can be made about rational utilization of this resource. The marketplace has become increasingly global and shipment of timber and wood products between countries is commonplace. In recent years, near infrared spectroscopy (NIRS) has become more common in many sectors as it meets the criteria of being rapid, accurate, reliable, non-destructive and inexpensive (Burns and Ciurezak 2007). It has also gained wide acceptance within many industries such as pulp and paper, pharmaceutical, food etc. for raw material testing, product quality control and process monitoring (Prieto *et al.* 2017, Roggo *et al.* 2007, Schwanninger *et al.* 2011, Tsuchikawa and Kobori 2015, Tsuchikawa and Schwanninger 2013, Williams 2001).

Research on using NIRS technique in the area of wood, wood composites and their products has been carried out only by a few research groups. Schimleck *et al.* (2000) reviewed applications of NIR spectroscopy as a rapid and inexpensive method for assessing the pulp quality from eucalypts. So *et al.* (2004) discussed about rapid assessment of solid wood properties using NIR spectra. NIRS in association with multivariate analysis techniques were also used to predict various wood quality parameters (Kelley *et al.* 2004, Mora and Schimleck 2009, Schimleck and Evans 2002, Schimleck *et al.* 2001, Tsuchikawa *et al.* 2005). Bailleurs and Durand (2000) evaluated the wood quality of plantation-grown teak (*Tectona grandis*) using several non-destructive techniques. Naimeke *et al.* (2014) reported rapid prediction of phenolic compounds as chemical markers for the natural durability of teak heartwood by near infrared spectroscopy. Kokutse *et al.* (2010) estimated the

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dimensional stability of teak wood using NIRS and described that inclusion of additional samples may lead to stronger prediction models to be used on a wide range of woods of different plantation origins. They presented that the best prediction models may be applied for shrinkage screening and prediction of fibre saturation point of teak wood. Via *et al.* (2005) investigated the ability of NIR spectroscopy to monitor air-dry density distribution and its variation in wood. Tsuchikawa and Siesler (2003) also reported that NIRS is quite useful for estimating wood quality parameters with acceptable accuracy.

Economical utilization of available timber resources calls for a vast range of basic studies including techno-mechanical properties of wood. Teak is one of the most sought-after timbers known worldwide (FAO 2015). In tropical regions, teak is a very important and highly-valued hardwood due to its excellent wood quality, the attractiveness due to its grain and texture, high strength to weight ratio, and high durability of its heartwood against biological attack (Anda 2019, Tewari 1999). Teak is also easy to work and process without appearance of defects. Multiple ends uses of teak include building and construction, furniture and cabinets, parquet flooring, joinery, ship and vehicle body building, decorative veneer, reconstituted products, among others (Pandey and Brown 2000, Tewari 1999). Despite being a high value timber, its demand is increasing in domestic markets. As a result, teak is being imported from different countries (FAO 2015). Authenticity of species and wood quality parameters of such imported timbers is of concern. Hence, quick and non-destructive procedures may be developed for estimating various wood properties and species confirmation. Not many studies have been reported on using NIRS for rapid estimation of wood quality parameters of teak (Bailleres and Durand 2000, Kokutse *et al.* 2010, Naimeke *et al.* 2014). The aim of the present study was to develop the calibration models based on NIRS data for rapid and precise estimation of various important wood quality parameters of teak such as density, moisture content and strength properties.

MATERIALS AND METHODS

Wood material

Fifteen boards from different trees of teak (*Tectona grandis* L.f.) of the size 5 cm - 6 cm (thickness), 30 cm - 45 cm (width) and 180 cm - 240 cm (length) were procured from a saw miller and imported teak supplier. The teak trees were originated from Myanmar (locally called Burma teak). A total of about 1200 wood specimens of different sizes were prepared for testing and evaluation of various physical and strength properties (BIS 1986).

Physical properties

Density

The specimen size was 20 mm × 20 mm in cross-section and 60 mm in length. Total number of specimens was about 220. The specimens were weighed correctly to 0,001 g and dimensions were measured correctly to 0,01 mm. Volume of wood specimens was determined after immersing in water. Density of wood specimen was calculated using following Equation 1:

$$Density = \frac{W_1}{V_1} \quad (1)$$

Where W_1 is weight (kg) and V_1 is the volume (m^3) of specimen at test condition.

Equilibrium moisture content (EMC)

All the specimens 20 mm × 20 mm × 25 mm were weighed initially with an accuracy of 0,001 g. A set of about 48 small blocks were exposed to the eight saturated solutions of different salts in desiccators (Arabhosseini 2005). Weights of 5 samples were also recorded at regular intervals to monitor the changes in moisture uptake/release. Samples were kept in different desiccators for a period of about 4 months and equilibrated until the difference between two weights of individual wood block was less than 0,2 % over two

successive days. Once equilibrium was reached, wood blocks were taken out, kept in a small desiccator and NIR spectra were collected. Final weights of these samples were recorded after drying in an oven at $103 \text{ }^\circ\text{C} \pm 2^\circ\text{C}$ for 48 h. The EMC of test samples was computed as Equation 2:

$$EMC (\%) = \frac{W_1 - W_0}{W_0} \times 100 \quad (2)$$

Where W_1 = weight of specimen at test (g) and W_0 = oven-dry weight of specimen (g).

Mechanical properties

Flexural strength (modulus of rupture-MOR), stiffness (modulus of elasticity-MOE) and compressive strength parallel to grain (maximum crushing stress-MCS) as described in Indian standard (BIS 1986) were evaluated using 50 kN universal testing machine (Model: AG-50, Shimadzu). Clear wood samples having perfect radial and tangential surfaces were selected for testing as described below:

Flexural strength and stiffness

Wood specimens for static bending were kept in a room for conditioning to $12 \% \pm 2 \%$ moisture content. The size of specimens was $20 \text{ mm} \times 20 \text{ mm} \times 300 \text{ mm}$ with a span length of 280 mm. Rate of loading was kept constant at 1,0 mm/min and load was applied on the tangential surface. MOR and MOE were computed using following Equation 3a and Equation 3b:

$$MOR = \frac{(3 * P_{\max} * l)}{(2 * b * h^2)} \quad (3a)$$

$$MOE = \frac{(P * l^3)}{(4 * D * b * h^3)} \quad (3b)$$

Where P = load (kN), and D = deflection (mm) at limit of proportionality; P_{\max} = maximum load (kN); l = span (mm), b = breadth (mm) and h = depth (mm) of test specimen.

Compressive strength parallel to grain

Defect free test specimens $20 \text{ mm} \times 20 \text{ mm}$ in cross-section and 80 mm in length were selected. The ends were sanded perfectly at right angles to length. Specimen was placed such that center of movable head was vertically above the center of cross-section of specimen. During the testing, load was continuously applied at a constant rate of 0,6 mm per min. MCS was calculated by the Equation 4:

$$MCS = \frac{P'}{A} \quad (4)$$

Where P' = maximum load at break point (kN), A = area of cross-section of specimen (mm^2).

Measurement of NIR spectra

A NIR spectrophotometer (MPA, Bruker Optics,) was used to collect diffuse reflectance NIR spectra of wood samples over the wavelength range 780 nm to 2500 nm (4000 cm^{-1} to 12820 cm^{-1}) with a resolution of 8 cm^{-1} using integrating sphere. Integrating sphere fitted with detector permits a direct measurement of large sample area (15 mm spot size). A total of 32 scans were collected for each position and total collected scans were averaged into a single spectrum to compensate for the natural variation present. For each surface, two

spectra each from tangential longitudinal (TL) and radial longitudinal (RL) were collected, providing four positions per sample.

Preprocessing of spectra and development of calibration models

For spectral noise reduction and smoothing, raw spectra were subjected to pre-processing algorithms to convert into useful spectral information. For comparisons, different pre-processing techniques such as first derivative with multiplicative scattering correction (MSC), straight line subtraction (SLS) and standard normal variate (SNV) were the most appropriate treatment procedures for the models through an optimization procedure. When first and second derivatives were applied, spectra were smoothed and differentiated by applying the algorithm proposed by Savitzky and Golay (1964) using 17 smoothing points. Bruker OPUS 5.5 Quant package was used for optimization and model development. For other analysis and data plotting, SigmaStat (Version 3.5, Systat Software Inc.) and SigmaPlot (Version 10.0, Systat Software Inc.) software were used.

NIR spectral data with multivariate statistical modeling was used for analysis and estimation of wood properties. For spectral analysis and partial least squares (PLS) modeling, models developed for density, EMC and mechanical properties were validated and their robustness evaluated. Full cross-validation was used for validating the results and selecting the number of factors giving minimum residual variance using inbuilt algorithm of OPUS Quant package. Based on whole spectral range, a region of higher value of coefficient of estimation (R^2), cross-validation (R_p^2) and ratio of performance to deviation (RPD) between spectral data and wood property as well as lower values of root mean square errors of estimation (RMSEE) and cross-validation (RMSECV) were used for evaluation of PLS regression models. More than one pre-processing method gave good models; however, only the best calibrations are presented for each wood property.

RESULTS AND DISCUSSION

Density

The average wood density of teak was $622,20 \text{ kg/m}^3 \pm 48,36 \text{ kg/m}^3$ at $12 \% \pm 2 \%$ moisture content. The coefficient of variation was $7,77 \%$ and density values ranged from $525,89 \text{ kg/m}^3$ to $805,20 \text{ kg/m}^3$. Unprocessed teak NIR spectra subjected to first derivative plus multiplicative scattering correction (MSC) algorithm (smoothing points 17) are shown respectively in Figure 1a and Figure 1b.

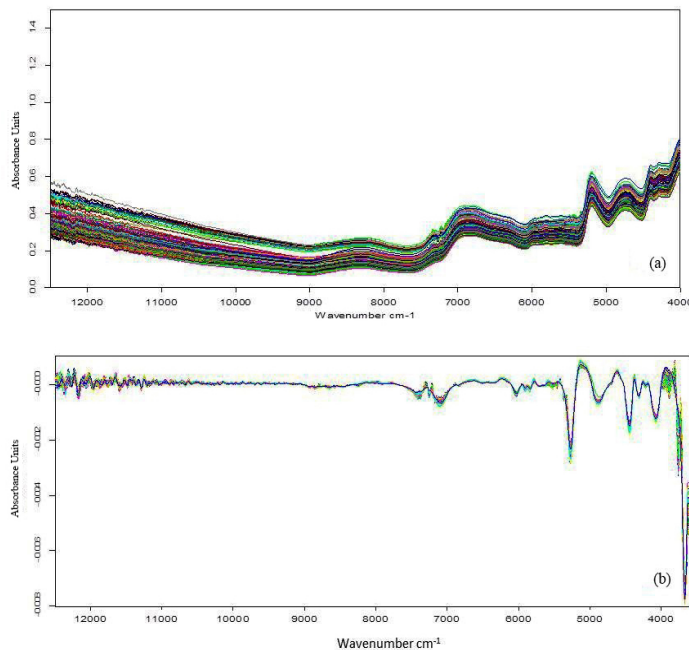


Figure 1: NIR spectra of density samples of teak wood (a), preprocessed spectra by First Derivative plus MSC (b).

Table 1 summarizes PLS-R calibration model between measured wood density and NIR spectral data. Several pre-treatments were carried out using spectral ranges between 9000 cm⁻¹ and 4000 cm⁻¹ with optimization of model parameters. Figure 2 shows correlation between measured and NIR predicted density values. The calibration and validation sets have shown linear correlation with high values of R² between measured and NIR predicted density values and lower values of RMSECV. The optimum values of rank and RPD were 5,0 and 3,58 respectively. It may be noted that application of NIRS predictive models for forest science considers models having RPD values above 1,5 to be satisfactory for preliminary prediction (Schimleck *et al.* 2003).

Table 1: Summary of NIRS calibration of density, EMC, MOR, MOE and MCS of teak.

Wood property	Preprocessing (Treatment)	R ² (R _p ²)	Rank (Latent variables)	RMSEE (RMSECV)	RPD	Frequency band (cm ⁻¹)
Density (kg/m ³)	First derivative + MSC (17 point)	0,94 (0,92)	5	9,29 (10,1)	3,58	7502,5-5450,3, 4601,4-4247,8
EMC (%)	MSC	0,97 (0,96)	9	0,48 (0,51)	5,19	7100,8-6398,8, 5303,4-4948,6
MOR (MPa)	Min-Max normalization	0,93 (0,92)	6	2,95 (3,11)	3,54	7502-4598
MOE (MPa)	MSC	0,95 (0,94)	5	251,0 (309,0)	3,94	8192-6098, 5454 -4246,6
MCS (MPa)	SNV	0,96 (0,95)	8	1,38 (1,43)	4,49	5453,8-4597,6

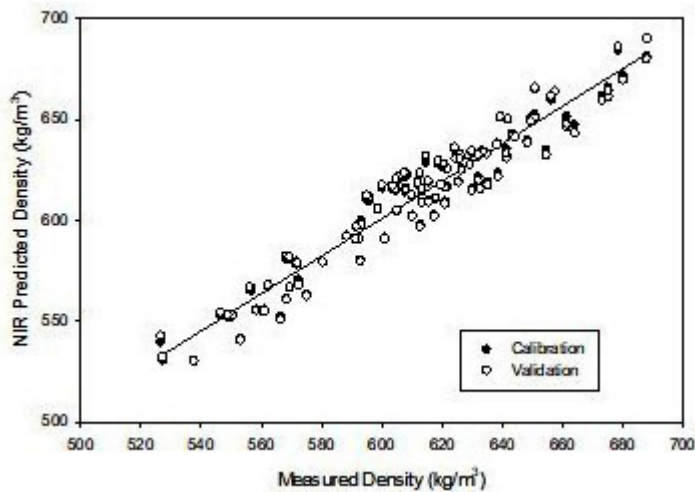


Figure 2: NIR predicted vs. measured (reference) density of teak wood.

As listed in Table 1, two optimum frequency ranges of 1333 nm - 1835 nm and 2173 nm - 2354 nm in combination gave the best models for air-dry density of teak. Many researchers have also used 1000 nm – 2000 nm and 1111 nm – 2500 nm regions for developing the models (Carneiro *et al.* 2010). Fujimoto *et al.* (2012) used band positions of 7320 cm⁻¹, 7160 cm⁻¹ and 7000 cm⁻¹ for developing wood density models independent of moisture conditions of samples. Only a few researchers have used entire range of 12800 cm⁻¹ to 4000 cm⁻¹ for development of models for different wood properties (Hein *et al.* 2009a, Hein *et al.* 2009b). Robustness

of models based on NIR spectra to predict basic density and other properties of *E. urophylla* was reported by Hein *et al.* (2010). Schimleck and Evans (2003) investigated air-dry density calibration of radiata pine using 5 factors. Mora *et al.* (2008) reported NIR calibration models for estimation of wood density in softwood using repeated measurements. Raturi *et al.* (2012) also applied this technique for prediction of specific gravity of dry *E. tereticornis* solid wood samples.

EMC

Figure 3 shows measured and NIR predicted EMC of teak for the calibration and predictions sets. The correlation coefficients and root mean square errors for the calibration and prediction sets were (0,97 %; 0,48 %) and (0,96 %; 0,51 %) respectively.

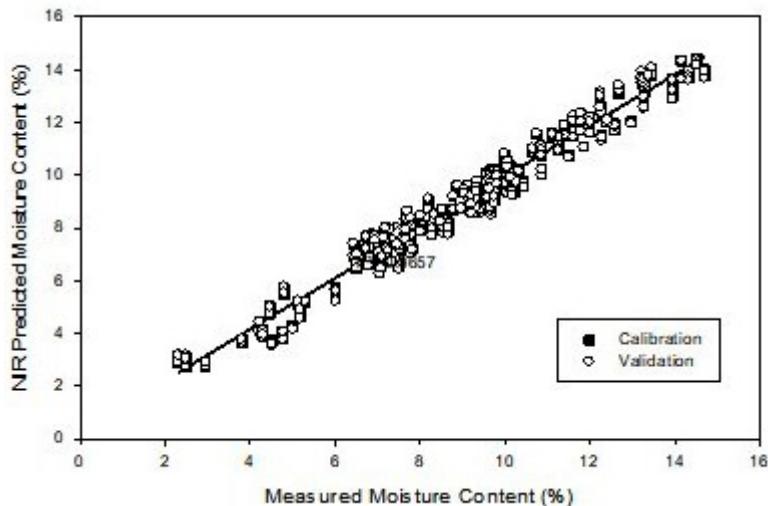


Figure 3: Measured vs. NIR predicted EMC of teak wood.

Development of model for EMC was carried out using PLS-R analysis between measured wood EMC values and NIR spectra. Different pre-processing methods were applied using different NIR spectral ranges between 9000 cm^{-1} and 4000 cm^{-1} with optimization of model parameters. Summary of NIRS calibration of EMC is shown in Table 1. Two frequency regions for optimum performance were $7100,8\text{ cm}^{-1} - 6398,8\text{ cm}^{-1}$ and $5303,4\text{ cm}^{-1} - 4948,6\text{ cm}^{-1}$. The wavenumbers above 9000 cm^{-1} and below 4000 cm^{-1} were avoided due to the low and high intensity of absorbance bands in these regions respectively.

Moisture content of wood plays an important role in the NIR spectrum and represented by several bands (Schwanninger *et al.* 2011). Absorption spectra of water have been resolved into component bands centered at 5200 cm^{-1} associated with water molecules. Hydroxyl groups are often involved in hydrogen bonding and 1st OT of O-H stretching depends on the strength of the hydrogen bonds, which is important for bands of cellulose. The band at 6944 cm^{-1} is assigned to free O-H groups and bands at 6710 cm^{-1} and 6494 cm^{-1} to H-bonded O-H groups. Similarly, the absorption bands at 6770 cm^{-1} , 6945 cm^{-1} and 7073 cm^{-1} have been assigned to the 1st overtone of cellulosic O-H, adsorption of water and water-water H-bonds respectively. The band found at 5200 cm^{-1} is attributable to hydroxyl (O-H) and carbonyl (-C=O) groups, the band at 7000 cm^{-1} to hydroxyl groups (Schwanninger *et al.* 2011). Both are strongly influenced by the moisture present in the solid teak wood samples. The teak wood spectra indicated that the absorption bands with peak maxima around 4761 and 5924 cm^{-1} were associated with C-H and O-H combination bands and 5880 and 4545 cm^{-1} overtones. Haddadi *et al.* (2016) also applied visible-NIRS technique to determine the moisture content of wood. A comprehensive review on using NIRS for monitoring the moisture content and density of solid wood is presented by Leblon *et al.* (2013). Defo *et al.* (2007) studied applicability and usefulness of NIRS in estimating the moisture content and density of fresh-sawn red oak wood. Cooper *et al.* (2011) reported that average moisture content of air-dried wood can be estimated accurately within $\pm 2\%$ to 5% .

Mechanical properties

Flexural MOR

Average values of MOR of teak along with standard deviation, COV and range (maximum, minimum) are listed in Table 2. Maximum-minimum algorithm was used for preprocessing treatment of the original spectra. Figure 4 shows measured vs. NIR predicted MOR for calibration and prediction sets. The optimum rank and RPD values were 7 and 3,54 respectively.

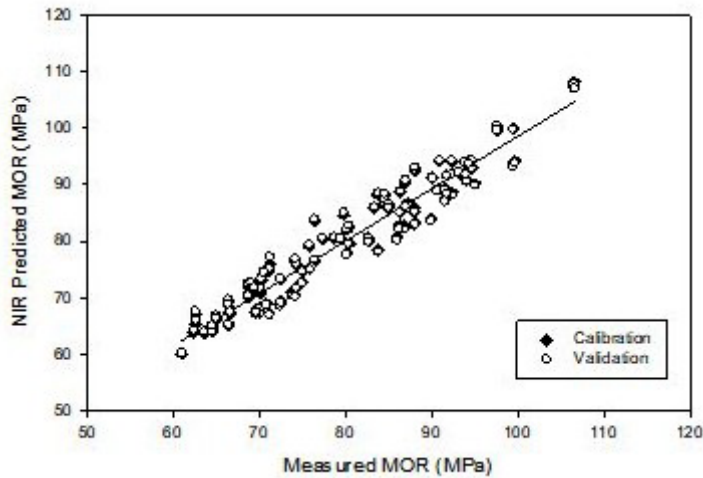


Figure 4: Measured vs. NIR predicted MOR of teak wood.

Table 2: Average values of MOR, MOE and MCS of teak wood.

Mechanical property	Number of samples (N)	Average value (MPa)	Standard deviation (SD, MPa)	Coefficient of variation (COV, %)	Maximum MOR (MPa)	Minimum MOR (MPa)
MOR	200	79,19	17,09	21,58	116,54	41,35
MOE	200	10695,69	1935,44	18,10	25763,48	6560,64
MCS	197	47,94	7,87	16,42	65,92	31,63

Treatments with various pre-processing methods were carried out using different spectral ranges between 9000 cm⁻¹ and 4000 cm⁻¹ for optimization of model parameters. Table 1 also provides the summary of NIRS calibration analysis of MOR. Min-max pre-processing and wavenumbers in the range of 7502 cm⁻¹ - 4598 cm⁻¹ were responsible for development of the best models for MOR. R_p², rank, RMSECV and RPD were 0,92 MPa, 6 MPa, 3 MPa, 11 MPa and 3,54 MPa respectively for the best MOR calibration.

Flexural MOE

Summary of development of PLS regression models between measured MOE and NIR spectral data (pre-processed using MSC) is shown Table 1. Linear plots between measured and NIR predicted MOE for calibration and prediction sets are shown in Figure 5.

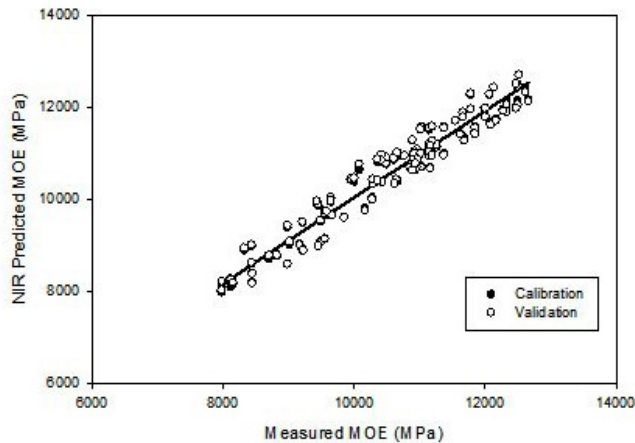


Figure 5: Measured vs. NIR predicted MOE of teak wood.

The pre-processing methods were applied using different spectral ranges between 9000 cm^{-1} and 4000 cm^{-1} to optimize the model parameters. Two frequency regions, $8192\text{ cm}^{-1} - 6098\text{ cm}^{-1}$ and $5454\text{ cm}^{-1} - 4246,6\text{ cm}^{-1}$ gave the best model for MOE. The optimum number of latent variables (rank) and RMSECV values were 5 MPa and 309,0 MPa respectively. Similarly, the best R_p^2 and RPD values for MOE were found to be 0,94 and 3,94 respectively. NIRS and multivariate calibration technique was applied to predict the stiffness of *Liriodendron tulipifera* (Adedipe *et al.* 2008). Kothiyal and Raturi (2011) studied certain mechanical properties (MOR, MOE) of *E. tereticornis* wood samples using NIRS coupled with multivariate analysis. PLS-R calibrations models were developed showing good relationships between measured and predicted values. It is also reported that coefficient of determination for calibration and prediction ranged from 0,76-0,83 and 0,58-0,77 SEPs were slightly higher compared to SECs. Kelley *et al.* (2004) used NIRS to measure the mechanical properties (MOR and MOE in three-point bending) of solid loblolly pine wood. Gindl *et al.* (2001) reported the relationship between NIR spectra of radial surfaces and mechanical properties of wood. Andrade *et al.* (2010) applied NIRS technique to obtain calibrations for mechanical properties of *E. urophylla* hybrid. They reported that NIRS technique is suitable to estimate MOR in solid wood with high values of r and RPD. MOR and compressive strength presented R values below 0,9.

MCS

Average values along with standard deviation, COV and range of MCS of teak wood are listed in Table 2. After comparison of different pre-processing techniques, SNV was found to be the most appropriate treatment procedure for the MCS models. Figure 6 shows the plots between the measured and predicted MCS values for the calibration and prediction sets. Table 1 demonstrates the summary statistics for the best calibration model developed using PLS-R method between measured MCS and teak NIR spectra. Wavenumbers in the range of $5453,8\text{ cm}^{-1} - 4597,6\text{ cm}^{-1}$ were found to be responsible for development of best models for MCS with R_p^2 and RPD values of 95,05% and 4,49 respectively. Similarly, the number of latent variables (rank) and RMSECV were 8 and 1,43 MPa for best MCS calibration model. Hoffmeyer and Pedersen (1995) also estimated the compressive strength of *Picea abies* wood by using NIRS method. NIR calibrations for compression strength proved that the NIR technique is an excellent non-destructive method ($R^2 \geq 0,9\%$). The authors concluded that this method is very versatile in non-destructive evaluation of wood properties. Andrade *et al.* (2010) also reported the suitability of NIRS to estimate the compressive strength of *E. urophylla* hybrid with $R < 0,9\%$.

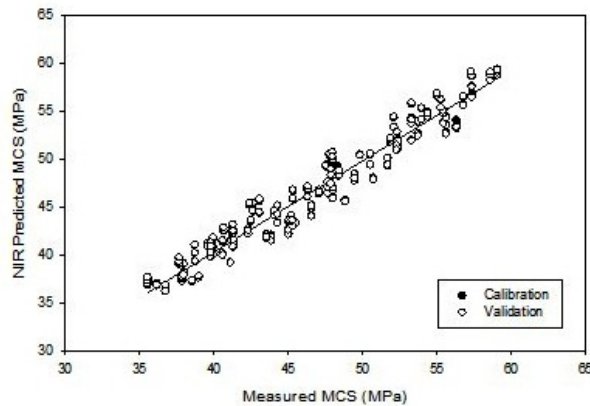


Figure 6: Measured vs. NIR predicted MCS of teak wood.

The strength properties of solid wood are generally related with the specific gravity, microfibril angle (MFA) and slope of grain (Butterfield 1997). Tsehaye *et al.* (1997) discussed a strong relationship between MOE and cellulose content. As NIR spectra of wood contain information on its different chemical constituents, its various properties such as specific gravity, MFA and cellulose content may be correlated (Kelley *et al.* 2004). Their results also showed that the strength properties of wood samples could be accurately predicted from the NIR spectra. Figure 1b shows the 2nd derivative of NIR spectra of teak wood where peaks are attributed to second overtones of hydroxyls and third overtones of C-H stretching vibrations. The first overtone of cellulose and hemicellulose hydroxyls are observed between 7142,9 cm⁻¹ and 6024,1 cm⁻¹ (Kelley *et al.* 2004). Strong vibration at 4950,5 cm⁻¹ to 4444,4 cm⁻¹ has also been allocated to cellulose hydroxyl vibrations. The first overtone of the lignin and carbon/hydrogen vibrations have been visualized between 5479 cm⁻¹ and 6116 cm⁻¹ (Schwanninger *et al.* 2011). Some of the lignin hydroxyl vibrations were found to overlap with cellulose hydroxyl vibrations, e.g., first overtone of lignin hydroxyl vibrations occurs between 7142,9 cm⁻¹ and 6578,9 cm⁻¹. Michell and Schimleck (1996) assigned the bands at 5995 cm⁻¹, 5974 cm⁻¹ and 5938 cm⁻¹ in 2nd derivative wood spectra to extractives and lignin. The bands near 4690 cm⁻¹ and 5974 cm⁻¹ were designated to 1st overtone of C-H from lignin. Similarly, Schwanninger *et al.* (2011) assigned the bands at 6003 cm⁻¹, 5950 cm⁻¹, 5816 cm⁻¹ and 5656 cm⁻¹ to 1st overtone of C-H stretching vibrations from methyl groups derived from hemicelluloses in wood.

CONCLUSIONS

NIRS coupled with multivariate regression analysis (PLS-R) has been applied to develop calibration models for different physical and mechanical properties of teak. NIR spectra of teak wood samples were collected before measuring different properties using conventional methods. Linear models were developed between measured and NIRS estimated values of density, EMC, MOR, MOE and MCS. These predictive models may be applied for non-destructive and rapid evaluation of teak avoiding conventional destructive, time consuming and expensive test methods. However, such models and libraries are required to be strengthened continuously for more accurate and precise estimations of various wood properties of commercially valuable timber species.

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