NEAR INFRARED SPECTROSCOPY FOR ESTIMATING WOOD BASIC DENSITY IN *Eucalyptus urophylla* AND *Eucalyptus grandis*

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ABSTRACT: Wood basic density is indicative of several other wood properties and is considered as a key feature for many industrial applications. Near infrared spectroscopy (NIRS) is a fast, efficient technique that is capable of estimating that property. However, it should be improved in order to complement the often time-consuming and costly conventional method. Research on wood technological properties using near infrared spectroscopy has shown promising results. Thus the aim of this study was to evaluate the efficiency of near infrared spectroscopy for estimating wood basic density in both *Eucalyptus urophylla* and *Eucalyptus grandis.* The coefficients of determination of the predictive models for cross validation ranged between 0.74 and 0.86 and the ratio performance of deviation (RPD) ranged between 1.9 and 2.7. The application of spectral filter, detection and removal of outlier samples, and selection of variables (wavelength) improved the adjustment of calibrations, thereby reducing the standard error of calibration (SEC) and cross validation (SECV) as well as increasing the coefficient of determination (R^2) and the RPD value. The technique of near infrared spectroscopy can therefore, be used for predicting wood basic density in *Eucalyptus urophylla* and *Eucalyptus grandis.*

Key words: NIRS, partial least square regression, hardwood.

ESPECTROSCOPIA NO INFRAVERMELHO PRÓXIMO PARA ESTIMATIVA DA DENSIDADE BÁSICA DA MADEIRA DE *Eucalyptus urophylla* E *Eucalyptus grandis*

RESUMO: *A densidade básica da madeira é indicadora de várias outras propriedades, sendo considerada como característica-chave para muitas aplicações industriais. A espectroscopia no infravermelho próximo (NIRS) é uma técnica rápida, eficiente e capaz de predizer essa propriedade, no entanto, deve ser aprimorada a fim de auxiliar o método convencional, que muitas vezes é demorado e oneroso. Objetivou-se, neste trabalho avaliar a eficiência da espectroscopia no infravermelho próximo para estimativa da densidade básica da madeira de Eucalyptus urophylla e Eucalyptus grandis. Os coeficientes de determinação dos modelos de predição variaram entre 0,74 e 0,86 e a relação de desempenho do desvio (RPD) variou entre 1,9 e 2,7. A aplicação de filtro espectral, a detecção e descarte de amostras anômalas (outliers) e a seleção de variáveis (comprimento de ondas) melhoraram o ajuste das calibrações, reduzindo o erro padrão da calibração e validação cruzada (SEC e SECV), e aumentando o coeficiente de determinação (R^2) e a RPD. Assim, a técnica NIRS pode ser usada para estimativas da densidade básica da madeira de Eucalyptus urophylla e Eucalyptus grandis.*

Palavras-chave: NIRS, regressão dos mínimos quadrados parciais, folhosas.

1 INTRODUCTION

Wood basic density is defined as the ratio of dry matter content to green volume of a sample. Basic density is a critical property as it has a strong correlation with other properties (PANSHIN & ZEEUW, 1980), affecting, for instance, cellulose pulp yield (ZOBEL & BUIJTENEN, 1989). In order to use wood in a wide range of industrial applications it is desirable that its basic density be high and uniform. However, basic density or specific gravity as some authors prefer to call it, can present considerable variation among and within trees of the same species. Genetic and environmental factors and the interaction between these two factors are both responsible for such variation (ZOBEL & JETT, 1995). According to Zobel & Buijtenen (1989), growth rate and consequently fiber and wood properties are affected by site quality and local silvicultural methods.

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Many methods have been studied for determination of wood basic density in recent years, with gravimetric methods being the most common. Scaramuzzi (1966) compared various methods for determination of basic density and concluded that the maximum moisture content method was fast and easier to perform. In comparison to indirect methods, however, traditional methods can be slow and cost can be a limiting factor where large samples are involved in genetic improvement programs. By the traditional method, samples often become unusable, thus demonstrating another disadvantage of methods consisting of water immersion and drying.

With the increasing demand for the development of fast methods for determining wood technological properties, new techniques have been devised for indirect measurement of basic density. Alternative methods have been researched to replace traditional methods. Diniz (2003), Oliveira (2001), Raymond & MacDonald (1998), Rosado (1982), Rosado et al. (1983) and Thiersch et al. (2006) correlated the penetration depth of Pilodyn® striking pin to wood basic density. Chantre & Rozenberg (1997), Gantz (2002), Isik & Li (2003), Lima et al. (2006, 2007) and Rinn et al. (1996) reported promising results for estimation of average wood basic density with a Resistograph® device. Though these methods provide satisfactory results, their application to forestry is yet limited and requires further investigation.

The development of rapid, accurate and industrially feasible methods becomes therefore necessary for characterization and classification of raw material in the forestry-related industry, including paper and cellulose or steel production (charcoal), since these companies require methods that will cover a large number of samples.

Near infrared spectrometry (NIRS) is a fast, non-destructive technique (one minute or less) applicable to any biological material, including on-line processes, demanding little or no sample preparation (PASQUINI, 2003). It is based on vibrational spectroscopy and measures the interaction between light and the relevant material (NÆS et al. 2002). It is determined by vibration of chemical bonds specific to sample constituents. According to Baillères et al. (2002), the method is based on selective absorption of light by chemical compounds.

The extraction of quantitative information present in near infrared spectra consists in associating the absorbance spectra to wood properties determined by a conventional method. Using chemometric tools (multivariate statistics, computer science, mathematics) a calibration is adjusted (MAGALHÃES et al. 2005). This method has been widely used in forestry. Studies have demonstrated, based on information contained in spectra measured in the near infrared region alone, that it is possible to investigate several technological properties in wood, as, for instance, cellulose paste yield (RAYMOND et al. 2001), cellulose content (BIRKETT & GAMBINO, 1988), lignin content (BRINKMAN et al. 2002), basic density (HOFFMEYER & PEDERSEN, 1995), strength (THUMM & MEDER, 2001), natural durability (JACQUES et al. 2002), microfibril angle (YANG & EVANS, 2003), fiber morphology (SCHIMLECK & EVANS, 2004), and also to classify wood (SCHIMLECK et al. 1996) and wood products (CAMPOS et al. 2009). The NIRS technique can simultaneously evaluate chemical, physical, mechanical and anatomical properties in wood.

Different criteria exist for selecting NIRS-based predictive models. The most common are coefficient of determination (R^2), standard error of prediction (SEP) or cross validation (SECV), number of latent variables (LV) and ratio performance deviation (RPD).

The RPD value represents the relationship between the standard deviation of basic density values as measured by the conventional method and the standard error of cross validation (SECV) or prediction (SEP) which, despite not being used very often, is the most informative statistic for model rating. The RPD value provides a basis for SEP standardization (WILLIAMS & SOBERING 1993) and allows comparison between calibrations of different treatments and properties.

For practical application of NIRS calibrations, Williams & Soberings (1993) classify calibrations with RPD values between 5 and 10 as suitable for quality control, and above 2.5 as satisfactory for approximate predictions in improvement programs. Likewise, a 1.0 RPD indicates that the standard error of prediction and the standard deviation of reference values are equal and the tool is incapable of predicting the parameter with precision based on that calibration.

Regarding the application of NIRS predictive models to forest sciences, Schimleck et al. (2003) consider models with RPD values above 1.5 to be satisfactory for preliminary predictions and readings, while for tree selection in improvement programs Schimleck & Evans (2004) consider models with RPD values of 2.5 to be sufficient.

This work aimed to evaluate the performance of near infrared spectroscopy for estimating wood basic density in *Eucalyptus grandis* and *Eucalyptus urophylla*.

2 MATERIAL AND METHODS

2.1 Sampling

In this study we used 70 samples of *Eucalyptus grandis* and 67 samples of *Eucalyptus uroplhylla* aged 6.5 years from commercial crops of company Plantar S.A., located in Curvelo, central Minas Gerais state. Samples measuring 20 x 20 x 20 mm with well-defined tangential, radial and transverse faces were cut from the central, middle and external portions of one meter long planks which in turn had been removed from the trunk base.

2.2 NIR spectra measurement

Bruker spectrophotometer (model Vector, Bruker Optik GmbH, Ettlingen, Germany) was used in the diffuse reflectance mode. This FT-IR spectrometer is designed for reflectance analysis of solids with an integrating sphere. NIR spectral acquisition was performed in the range of 4,000 to 12,500 cm^{-1} at 8 cm^{-1} spectral resolution. A sintered gold standard was used as background. NIR spectra were measured directly on the two transverse faces of each sample, with one spectrum representing the average reading of 32 scans. The spectrum used in the calibration was derived from the average between the two readings (upper and lower transverse faces). Spectral measurements were done in a acclimatized room under controlled temperature of $\cong$ 20°C, and $\cong$ 60% relative air humidity.

2.3 Basic density

The basic density was obtained through the method described by the Associação Brasileira de Normas Técnicas - ABNT (2003). The volume of each sample was determined through the immersion method after complete saturation in water. Samples were subsequently oven-dried at a temperature of 103 ± 2ºC until constant mass. The dry matter content was determined using a precision scale with 0.01 g accuracy.

2.4 Calibration parameters

Partial Least Square (PLS) regressions were fitted to describe the relationship between wood basic density values (dependent variable) and near infrared spectra (independent variable) using multivariate statistics program The Unscrambler® (9.7 v.). PLS calibrations were fitted through the PLS-1 method, with a maximum of 12 latent variables (LV). Model validation was performed using full cross validation and external validation. The number of latent variables adopted for each model was that suggested by the statistical program. To detect outliers, Student's residual and leverage values plot were analyzed as suggested by Hein et al. (2009). Samples classified as outliers were not included in the model calibration and validation phase. In order to improve signal quality, standard normal variation - snv, first derivatives - 1d (13-point filter and a second order polynomial), second derivatives - 2d (25-point filter and a third order polynomial) and combinations were applied in the NIR spectra data. The derivatives were performed using Savitsky and Golay (1964) algorithm.

2.5 Selection of calibrations

Criteria adopted for selecting the prediction model included: (a) coefficient of determination of cross validation (R^2cv); (b) standard error of cross validation (SECV); (c) ratio performance deviation (RPD), and (d) number of latent variables (LV) used in the calibration.

SECV measures the efficiency of the calibration model in predicting the property of interest in a set of unknown samples differing from the samples that form the calibration set (SCHIMLECK et al. 2001), and is given by:

$$SECV = \sqrt{\frac{\sum_{i=1}^{NP}(\hat{y}_i - y_i)^2}{NP - 1}}$$

where $\hat{y}_i$ is the estimated value of average basic density of sample i, y_i is the known value for average basic density of sample i and NP is the number of samples.

3 RESULTS AND DISCUSSION

3.1 Basic density

Table 1 presents average wood basic density as obtained by conventional method (g/cm³), standard deviation (g/cm³), minimum (g/cm³) and maximum values (g/cm³), coefficient of variation (%) and number of samples of the calibration set.

3.2 Spectral information

Measurement of near infrared spectra was done in diffuse reflectance mode directly on the material. Figure 1 presents near infrared absorption spectra for *Eucalyptus* wood and the overtones and combination bands regions of absorption bands. Spectral acquisition was performed in the range of 4,000 cm^{-1} to 12,500 cm^{-1} at 8 cm^{-1} spectral resolution.

Table 1 – Wood basic density of the calibration set.

Tabela 1 – *Densidade básica da madeira das amostras do lote de calibração.*

	Mean	St. Dev.	Min.	Max.	CV (%)	N
BD (g/cm³)	0.546	0.064	0.407	0.708	11.730	137

BD – basic density; St. Dev. – standard deviation; Min. – minimum value; Max. – maximum value; CV – coefficient of variation and N – number of samples

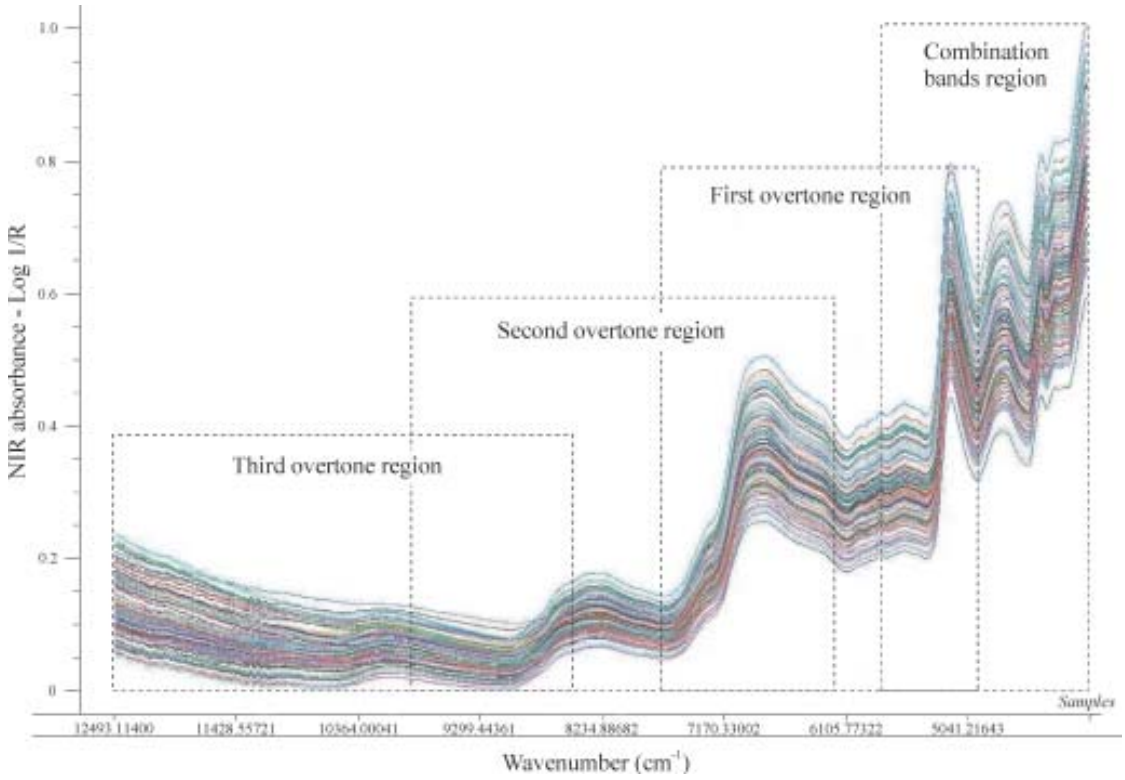

Figure 1 – Near infrared spectra of *Eucalyptus urophylla* and *Eucalyptus grandis* wood, and overtones and regions of absorption bands combination.

Figura 1 – *Espectros no infravermelho próximo da madeira de Eucalyptus urophylla e Eucalyptus grandis e regiões de sobreposição e combinação das bandas de absorção.*

According to Workman & Weywer (2007), the range between 4,000 and 5,000 cm^{-1} is the combination band region. This region has highly overlapping absorbance peaks, which is a result of the interaction between different chemical bonds. It is thus informative and has low noise level but is not clear enough to be chemically analyzed. The range between 5,000 and 9,000 cm^{-1} represents the first and second harmonic region. It is an informative region, having low noise level. The spectral range extending from 9,000 to 12,500 cm^{-1} has high noise level and corresponds to the third harmonic region. In this range absorption is low, and that results in poor information quality.

NIR spectral ranges selection results in better fit of data to the calibration model. Several studies have investigated spectral ranges that explain variables of interest, and in most cases wavelengths were incident on the first and second harmonic regions, which are the most informative (GHASEMI et al. 2003; LEARDI et al. 2002).

Some wavelength ranges are informative for certain chemical components in wood.

Table 2 illustrates the absorption bands of some specific organic chemical bonds and relevant wavelengths promoting the absorption of energy incident on the material. It also presents types of deformation caused by wavelength incidence as well as harmonic regions.

Chemical bonds constitute a vibration system whose frequency is contingent on the type of molecular bond. Therefore, the energy absorption from a near infrared electromagnetic wave is regulated by the frequency of vibration and by the nature of the chemical bond (PASQUINI, 2003).

What makes the use of near infrared spectroscopy feasible in assessing wood properties is based in the fact that the expression of all its properties is somehow influenced by the chemical constitution of wood.

3.3 NIRS Calibrations

The selection of wavelengths (LEARDI et al. 2002) and spectral filters (DELWICHE & REEVES, 2004) during modeling are important procedures for optimizing PLS regression models. The precision of calibration models can be checked by two methods, namely cross validation and external validation (GEMPERLINE, 2006). Here, models were calibrated using different mathematical treatments of spectra, with selection by the variable uncertainty test, for both validation methods. Table 3 presents statistics associated with calibration and cross validation models for wood basic density based on original spectra and spectra after mathematical treatment.

Statistics associated with calibration and validation models by external test set for estimation of wood basic density are illustrated in Table 4. Models were calibrated based on original spectra and after mathematical treatment of the spectra of wood from *Eucalyptus uroplhylla* and *Eucalyptus grandis*.

Results in Tables 3 and 4 indicate that wood basic density in *Eucalyptus uroplhylla* and *Eucalyptus grandis* wood can be assessed using near infrared spectrometry, agreeing with results found in other studies. The application of spectral filter, exclusion of outliers and selection of variables (wavelengths) improved statistics associated with models.

A similar situation was reported by Jones et al. (2005), who studied the basic density of 120 samples of *Pinus taeda* and, based on pure spectra, obtained 0.82 for R^2, 0.551 for SEP and 1.89 for RPD. These researchers demonstrated that application of spectral data pretreatment of the first derivative reduced the standard error of prediction (SEP) from 0.551 to 0.458 and increased RPD from 1.89 to 2.28.

The role of spectral pretreatment in reducing SEP and increasing RPD in calibrations was also reported by

Table 2 – Near infrared specific absorption bands.

Tabela 2 – *Bandas de absorção específicas no infravermelho próximo.*

Wavenumber (cm^{-1})	Chemical structure and type of deformation	Region
11760-11630	Aromatic	3rd harmonic
11490-11300 and 11110-10990	$-CH_3$: stretch	3rd harmonic
10640-10470	-OH: bend	2nd harmonic
9350-9220	2*CH stretch + 2*C-C stretch	combination
8850-8770	Aromatic	2nd harmonic
8700-8580; 8840-8370 and 8400-8330	$-CH_3$: stretch	2nd harmonic
7170-7020	-OH	1st harmonic
7090-7040 and 6940-6900	2*CH stretch + CH bend	combination
5950-5920	Aromatic	1st harmonic
5850-5870; 5700-5630 and 5650-5600	$-CH_3$: stretch	1st harmonic
4850-4780	OH stretch + OH bend	combination
4400-4380	CH stretch + CH bend	combination
4220-4180	-OH: stretch	2nd harmonic

Table 3 – Comparison between mathematical treatments on calibration models by cross validation.

Tabela 3 – *Comparação dos tratamentos matemáticos nos modelos de calibração e validação pelo método de validação cruzada.*

Treat	N	LV	R^2c	SEC	*Outlier*	R^2cv	SECV	*Outlier*	RPD
osd	137	4	0.76	0.031	2	0.74	0.032	1	2.0
d1	137	7	0.85	0.024	4	0.73	0.028	1	2.3
d1+snv	137	7	0.87	0.023	5	0.82	0.027	1	2.4
d2	137	5	0.82	0.027	4	0.79	0.029	1	2.2
d2+snv	137	6	0.85	0.024	3	0.80	0.028	1	2.3
snv	137	11	0.83	0.026	3	0.74	0.032	1	2.0

treat – treatment; osd – original spectral data; d1 – first derivative; d2 – second derivative; snv - standard normal variate; N – number of samples; LV – number of latent variables; R^2c - coefficient of determination of calibration; SEC – standard error of calibration (g/cm^3); outlier – number of discarded samples; R^2cv - coefficient of determination of cross validation; SECV – standard error of cross validation (g/cm^3) and RPD – ratio performance deviation.

Table 4 – Comparison between mathematical treatments on calibration models by external validation.

Tabela 4 – *Comparação dos tratamentos matemáticos nos modelos de calibração e validação pelo método de validação externa.*

Treat	Ncal / Nval	LV	R^2c	SEC	*Outlier*	R^2p	SEP	*Outlier*	RPD
osd	97 / 40	11	0.86	0.023	2	0.86	0.026	0	2.5
d1	97 / 40	5	0.81	0.028	4	0.85	0.024	0	2.7
d1+snv	97 / 40	6	0.83	0.024	5	0.87	0.026	0	2.5
d2	97 / 40	4	0.80	0.027	4	0.79	0.030	1	2.1
d2+snv	97 / 40	6	0.88	0.021	3	0.77	0.034	0	1.9
snv	97 / 40	10	0.80	0.028	3	0.83	0.025	0	2.6

treat – treatment; osd – original spectral data; d1 – first derivative; d2 – second derivative; snv - standard normal variate; Ncal – number of calibration samples; Nval – number of validation samples; LV – number of latent variables; R^2c - coefficient of determination of calibration; SEC – standard error of calibration (g/cm^3); outlier – number of discarded samples; R^2cv - coefficient of determination of cross validation; SECV – standard error of cross validation (g/cm^3) and RPD – ratio performance deviation.

Hein et al. (2008), who evaluated 14 species of wood. These authors obtained values between 0.922 and 0.951 for coefficient of determination of cross validation and values between 3.55 and 4.47 for ratio performance deviation, in calibrations for basic density.

In a study of basic density of 169 samples of *Picea abies*, Thygesen (1994) obtained values between 0.504 and 0.792 for coefficient of determination, using six to eight latent variables in a calibration based on near infrared spectra.

Schimleck et al. (1999) investigated *Eucalyptus globulus* aged eight years with basic density between 0.378 and 0.656 g/cm³. These authors calibrated models with coefficients of determination ranging between 0.384 and 0.593, using 4 to 10 latent variables.

Via et al. (2003) evaluated 10 *Pinus palustris* aged 41 years and obtained calibrations with coefficients of determination ranging between 0.71 and 0.86, using multiple linear regression and principal component regression. The standard error (RMSEC) obtained by these authors ranged between 0.0432 and 0.0510 g/cm^3.

4 CONCLUSIONS

Wood basic density in *Eucalyptus urophylla* and *Eucalyptus grandis* wood can be estimated through near infrared spectrometry.

The calibrations for estimating wood basic density presented satisfactory coefficients of determination for both cross validation and for external test set validation.

The application of spectral filter, detection and

discarding of anomalous samples, and selection of wavelengths improved adjustment of calibrations, reducing the standard error of calibration and cross validation (SEC and SECV), and increasing the coefficient of determination (R^2) and the ratio performance deviation (RPD).

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