ESTIMATE OF THE DENSITY OF *Eucalyptus grandis* W. Hill ex Maiden USING NEAR INFRARED SPECTROSCOPY

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ABSTRACT: This study aimed to analyze use of near infrared spectroscopy (NIRS) to estimate wood density of *Eucalyptus grandis*. For that, 66 27-year-old trees were logged and central planks were removed from each log. Test pieces 2.5 x 2.5 x 5.0 cm in size were removed from the base of each plank, in the pith-bark direction, and subjected to determination of bulk and basic density at 12% moisture (dry basis), followed by spectral readings in the radial, tangential and transverse directions using a Bruker Tensor 37 infrared spectrophotometer. The calibration to estimate wood density was developed based on the matrix of spectra obtained from the radial face, containing 216 samples. The partial least squares regression to estimate bulk wood density of *Eucalyptus grandis* provided a coefficient of determination of validation of 0.74 and a ratio performance deviation of 2.29. Statistics relating to the predictive models had adequate magnitudes for estimating wood density from unknown samples, indicating that the above technique has potential for use in replacement of conventional testing.

Key words: Bulk density, *Eucalyptus*, hardwood, NIRS.

PREDIÇÃO DA MASSA ESPECÍFICA DE *Eucalyptus grandis* W. Hill ex Maiden POR ESPECTROSCOPIA NO INFRAVERMELHO PRÓXIMO

RESUMO: *Conduziu-se este estudo, com o objetivo de analisar a espectroscopia no infravermelho próximo (NIR) aplicada à predição da massa especifica da madeira de Eucalyptus grandis. Foram coletadas 66 árvores, com 27 anos de idade, transformadas em pranchões centrais e, da base de cada pranchão foram retirados os corpos de prova, medindo 2,5 x 2,5 x 5,0 cm, no sentido medula-casca, destinados à determinação da massa especifica (aparente e básica ambas a 12% de umidade base seca) e, posterior obtenção dos espectros em espectrofotômetro de infravermelho, da marca Bruker, Tensor 37, conforme os sentidos radial, tangencial e transversal. A calibração para predizer a massa específica da madeira foi ajustada a partir da matriz de espectros obtidos a partir da face radial contendo 216 amostras. A regressão dos mínimos quadrados parciais para a estimativa da massa especifica aparente da madeira de Eucalyptus grandis apresentou coeficiente de determinação em validação de 0,74 e relação de desempenho do desvio de 2,29. As estatísticas associadas aos modelos preditivos apresentaram magnitudes adequadas para estimar a densidade da madeira de amostras desconhecidas, indicando que a técnica tem potencial para substituir os ensaios convencionais.*

Palavras-chave: Densidade aparente, Eucalyptus, folhosas, NIR.

1 INTRODUCTION

With the growing shortage of native forests due to agricultural expansion and the increasing need for superior quality wood, new sources of raw material are required to supply the manufacturing industry. Use of fast-growing, exotic species such as those of the genus *Eucalyptus* is being regarded as a potential alternative to meet the market demands for strong, resilient wood (ROSSO, 2010).

An important parameter for technical characterization of timber is density. It is a physical property that, according to Barrichelo et al. (1983), varies between species, between individuals and proveniences of the same species and between the longitudinal and radial directions within a single tree.

Wood density, despite easily measurable (ZOBEL; JETT, 1995) by traditional destructive methods, becomes a real challenge when it comes to real-time mensuration in manufacturing plants that need to sort wood according to grade based on that characteristic (ACUNA, 2006).

Overall, destructive evaluations are the most commonly used methods for determining physical and mechanical properties of wood. Attaining results with that methodology, however, can be costly on account of the time consumed in preparing test pieces and the cost of material. Nondestructive evaluations, on the other hand, enable access to more accurate and comprehensive information, allowing more samples to be measured as a result of test agility (STANGERLIN et al., 2008). A fast and accurate

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alternative method is to use near infrared spectroscopy (NIRS). According to So et al. (2002), analysis of wood properties using NIRS spectra is an expanding field with great potential for improving wood quality and, ultimately, enabling genetic improvement of trees.

Hein et al. (2009a) argued that use of near infrared spectroscopy to evaluate wood properties is feasible, for the reason that the expression of every property is one way or another related to its chemical composition. Therefore, the NIRS technique can be used to simultaneously evaluate the chemical, physical, mechanical and anatomical properties of wood.

Nondestructive methods are for the most part based on analysis of correlations and calibration models between the properties of interest and other properties more easily measurable, which characterizes them also as indirect methods (ABRAHÃO, 2005).

Several studies have been conducted in the forestry sector using the near infrared technique (NIRS) and multivariate analysis for estimation of wood basic density (HOFFMEYER; PEDERSEN, 1995 apud NISGOSKI, 2005; SCHIMLECK et al., 1999), with a report of successful development of a calibration model for sawdust wood to estimate basic density of *Eucalyptus globulus*.

With the above in mind, the objective of this study was to evaluate the use of near infrared spectroscopy to estimate wood density for *Eucalyptus grandis* W. Hill ex Maiden.

2 MATERIAL AND METHODS

The wood material used for the density analysis was obtained from 66 27-year-old trees of *Eucalyptus grandis* Hill ex Maiden from a commercial stand established in the northern coastline of Rio Grande do Sul, with trees spaced 3 x 2 meters apart. From each felled tree, two 4-meter logs were removed from the trunk section between DBH and 25% of the tree length. From these logs, central planks were removed containing pith, around 8-cm thick and having variable width depending on the tree diameter. After being left to air dry, the central planks were transported to the joinery of *UFSM* university for removal of test pieces from the base of each plank.

The test pieces (41 x 2.5 x 2.5 cm) were used for determination of bulk density at 0% and 12% moisture content (reference values). Measurements were taken of the weight at 12% and volume at 12% of samples, then samples were dried to 0% moisture content and again subjected to measurements of weight and volume for calculation of bulk density at 0%.

For measurement of near infrared spectra, samples around 2.5 x 2.5 x 5.0 cm (thickness, width and length) in size were used, with anatomical planes of section oriented in the RD (radial), TG (tangential) and TR (transverse) directions, all samples being sanded before the readings (Figure 1).

Figure 1 – Illustration of a test piece indicating each plane of section for the NIRS readings.

Figura 1 – *Esquema que demonstra como será avaliado o corpo de prova no NIR, utilizando a amostra.*

The test pieces were acclimated in a temperature-controlled environment (20°C±3°C temperature and 65%±1% relative humidity) until they reached 12% moisture content. After the acclimation period in the *LANAQM* laboratory, near infrared spectra were recorded using a Bruker spectrophotometer (Tensor 37, Bruker Optik GmbH, Ettlingen, Germany) based on Fourier transform, using a halogen lamp as source of light radiation and an integrating sphere for capturing the reflected radiation. Information was recorded by software application OPUS version 4.2 which was coupled to the equipment. Spectral acquisition was done in the region 9000-4000 cm^{-1} with spectral resolution of 8 cm^{-1} in diffuse reflection mode. A gold-plated surface base was used as reference in an instrumentation test run prior to the series of readings. The spectrum of each sample was determined from the arithmetic average of 64 scans of the three planes of section (RD, TG, TR), with the sample fixed.

A partial least squares regression (PLS regression) was applied to describe the correlation of bulk density values ($MEA_{0\%}$) with the near infrared spectra, using the software application The Unscrambler® version 9.7. Calibrations were developed by the PLS-1 method with a maximum of 12 latent variables (VL). The leave-one-out cross-validation method was used to validate the models. For the purpose of comparing calibrations, five latent variables were adopted in each model. An analysis of the plot of studentized residuals and leverage value was done for detection of anomalous samples (outliers), as described in Hein et al. (2009b).

For selection of spectral variables, the Martens' uncertainty test (WESTAD; MARTENS, 2000) was used, available for PLS modeling in the statistical

application used. Samples perceived to be outliers were excluded from the calibration and validation stage. Mathematical treatments such as first and second derivatives (SAVITZKY; GOLAY, 1964) were applied to spectral information to try and improve signal quality.

For the first derivative, polynomials of second order with a spectral window of six variables were adopted (six points before and six points after), while for the second derivative polynomials of third order with a spectral window of 12 variables were adopted (12 points before and 12 points after).

Calibrations were developed using four routines, namely routine 1: calibration with all samples and spectral variables; routine 2: calibration without anomalous samples (outliers) and with all spectral variables; routine 3: calibration without anomalous samples (outliers) and with spectral variables selected by the Martens' uncertainty test, and routine 4: 1st and 2nd derivative of spectral information. For validation of the calibration, cross-validation and independent-validation methods were used for all routines. For the cross-validation test, samples were divided into 16 subsets, and in each subset eight samples were selected at random for model validation. For the independent-validation test, 80 samples were used for calibration and 40 were used for validation, with selection done at random. The determination of calibration and validation parameters was based on preliminary analyses.

Criteria used for selecting the prediction model followed the recommendations of Fujimoto et al. (2008) and Hein et al. (2009b) and included: (a) coefficient of determination of cross-validation (R^2cv); (b) Standard error of cross-validation (SECV); (c) ratio performance deviation (RPD); (d) Number of latent variables (VL) used in calibration.

The tests were run in the wood anatomy and quality laboratory *(LANAQM)* of the Federal University of Paraná *(UFPR)*, in conjunction with the forest products laboratory *(LPF)* of the Federal University of Santa Maria *(UFSM)*.

3 RESULTS AND DISCUSSION

Table 1 provides results of mean, minimum and maximum bulk density at 0% and 12% moisture for 27-year-old *Eucalyptus grandis*, used for calibrating the models based on partial least squares regression. The mean values in Table 1 proved lower than results found by Santini Junior et al. (2009) for 21-year-old *Eucalyptus grandis* Hill ex-Maiden in a study on variation of basic density along the radial plane under the influence of fertilization. These authors found a mean basic density of 560 $kg.m^{-3}$, with 450 $kg.m^{-3}$ in the radial position 0% relative to the pith, against 690 $kg.m^{-3}$ in the radial position 100% relative to the pith (bark).

Table 1 – Descriptive statistics of bulk density for 27-year-old *Eucalyptus grandis*.

Tabela 1 – *Estatística descritiva para massa específica aparente para Eucalyptus grandis aos 27 anos de idade.*

	$MEA_{0\%}$ (g/cm³)	$MEA_{12\%}$ (g/cm³)
No. of test pieces	216	216
Mean	0.464	0.527
Standard Deviation	0.074	0.084
CV (%)	15.9	15.9
Minimum	0.322	0.366
Maximum	0.738	0.837

Please note: CV = coefficient of variation; MEA = bulk density

In a study exploring density of 26-year-old *Eucalyptus saligna* and 21-year-old *Eucalyptus grandis*, Santos and Ballarin (2002) found mean values of 0.845 g/m³ and 0.615 g/cm³ respectively.

The analysis of near infrared spectra as measured in the radial, tangential and transverse directions revealed different behaviors depending on the plane of section of the piece, as is illustrated in Figure 2.

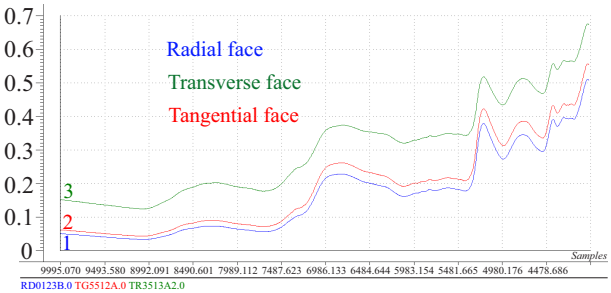

Figure 2 – Spectra of *Eucalyptus grandis* read on radial, tangential and transverse surfaces.

Figura 2 – *Espectros da madeira de Eucalyptus grandis medidos na superfície radial, tangencial e transversal.*

A higher absorbance was found in the transverse direction for the wood of *Eucalyptus grandis*. This finding was similar to results found by Hein et al. (2009b) and Ribeiro (2009) for the same species, using NIRS technique.

The principal component analysis (PCA) applied to the matrix of data containing spectra from 216 samples,

according to different planes of section (RD, TG and TR), revealed an overlap of the radial and tangential directions, whereas the transverse direction formed a distinct cluster, as is illustrated in the scores plot (Figure 3). A similar result was found by Ribeiro (2009) in a study evaluating *Eucalyptus spp*.

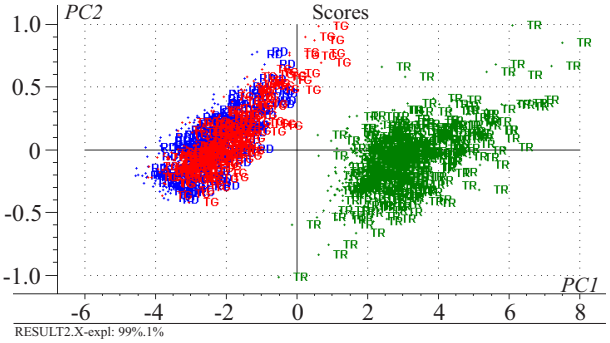

Figure 3 – PCA score plot of spectral information for RD, TG and TR surfaces.

Figura 3 – *Diagrama dos scores obtidos pela pca aplicada às informações espectrais medidas nas superfícies RD, TG e TR.*

Two principal components explain 100% of variability in data, out of which 99% are explained by principal component 1 (PC1) and 1% is explained by principal component 2 (PC2). The difference between radial and tangential directions in relation to transverse direction can be explained by the different disposition of anatomical elements, with the radial and tangential planes having most elements arranged axially, while the transverse plane has these anatomical elements arranged along the growth rings. It can also be justified by microfibril angle and by density which, according to the spectra, revealed high correlations, associating these variables to the chemical composition of the wood. The NIRS method provides outstanding correlations between technological properties of wood and its chemical composition. The method offers qualitative and quantitative information on the chemical constituents of biomass as a result of the interaction between electromagnetic waves and the sample (PASQUINI, 2003).

Partial least square regressions were run to estimate bulk density at 0% moisture of *Eucalyptus grandis*, as is illustrated in Table 2. Calibrations were based on solid wood and tested by independent validation. Pasquini (2003) recommends use of independent validation because the results provided by this method are closer to real situations. In independent validation, estimation of new samples is not dependent on data used for constructing and optimizing the model. Spectra were acquired on three planes of section, namely radial, tangential and transverse faces, noting that the radial face provided the best performance model.

The radial face provided a calibration with coefficient of determination of 0.736 and ratio performance deviation of 2.29 with six latent variables. Hein (2008) found higher values for the radial plane in wood of *Eucalyptus urophylla*, 0.896 for coefficient of determination and 3.1 for ratio performance deviation. Ribeiro (2009) studied *Eucalyptus* spp, investigating all three planes of section, and reported that it was the tangential plane that best estimated the values of density, 0.64 for coefficient of determination of calibration, and 0.50 for cross-validation.

Schimleck et al. (1999) investigated wood of *Eucalyptus globulus* at age eight years having basic density

Table 2 – Calibrations of bulk density at 0% moisture ($MEA_{0\%}$) in the radial direction.

Tabela 2 – *Calibrações para massa específica aparente a 0% de umidade ($MEA_{0\%}$) no sentido radial das amostras.*

Routine	Treatment	Outliers	R^2c	RMSEC	R^2v	RMSEV	LV	RPD
1	-		0.695	0.0407	0.684	0.0416	3	1.77
2	-	3	0.753	0.0350	0.727	0.0370	7	1.99
3	-	3	0.726	0.0370	0.714	0.0380	4	1.94
1	SNV + 1D		0.768	0.0354	0.703	0.0400	9	1.84
2	SNV + 1D	3	0.795	0.0313	0.739	0.0356	8	2.07
3	SNV + 1D	3	0.778	0.0326	0.735	0.0359	6	2.05
4	SNV+ 1D	3	0.784	0.0340	0.736	0.0322	6	2.29

Please note: R^2c = coefficient of determination of calibration; R^2v = coefficient of determination of validation; RMSEC = standard error of calibration; RMSEV = standard error of validation; LV = number of latent variables; Outlier = number of discarded samples; RPD = ratio performance deviation; 1D = first derivative; SNV= standard normal variate.

between 0.378 and 0.656 g/cm³. These authors calibrated models by using mean spectra, with correlation coefficient (R) ranging between 0.62 and 0.77 (r^2=0.384 to r^2=0.593), using 4 to 10 principal components (latent variables). In the same study, they demonstrated that using a second derivative spectral pretreatment did improve the models. Filtering spectral information minimized error (SEC and SEP) and increased R from 0.63 to 0.80 (r^2=0.397 to r^2=0.64).

Jones et al. (2005) studied basic density on 120 samples of *Pinus taeda*. From pure spectra, they found 0.82 for R^2, 0.551 for SEP and 1.89 for RPD. In the same study, these authors demonstrated that applying a first derivative spectral pretreatment did not affect the coefficient of determination, which remained the same (R^2=0,82), though it reduced SEP from 0.551 to 0.458 and increased RPD from 1.89 to 2.28. The higher the RPD value, the more robust the upcoming model will be (FUJIMOTO et al., 2008).

The effect of spectral pretreatment on reducing the SEP and increasing the RPD of calibrations is also reported by Hein et al. (2008), who evaluated 14 species of wood. These authors found, in calibrations for basic density, that the coefficients of determination in cross-validation ranged between 0.922 and 0.951 and the ratio performance deviation ranged between 3.55 and 4.47.

Figure 4 illustrates estimated density versus calculated density. The values found, 0.78 for R^2cal and 0.74 for R^2val, were very close, indicating that estimated values were similar to calculated values.

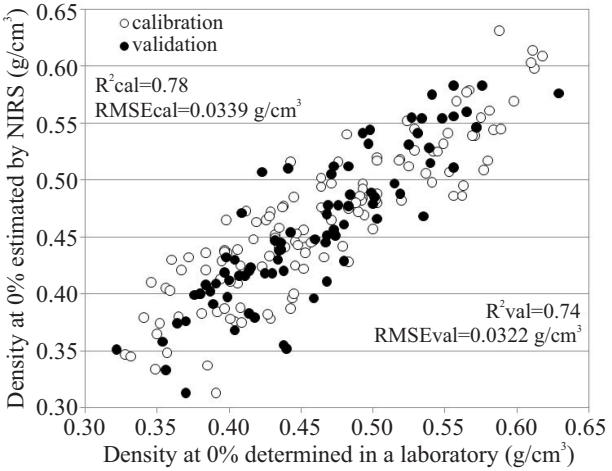

Figure 4 – Density values as measured by traditional methods and estimated by near infrared spectroscopy.

Figura 4 – *Valores de massa específica medidos por métodos tradicionais e preditos por espectroscopia no infravermelho próximo.*

4 CONCLUSIONS

Results found reveal that procedures such as mathematical treatment of spectra, detection and discarding of anomalous samples, and selection of spectral variables (wavelengths) significantly optimized the statistics of the models to estimate wood density. The radial face provided the best performance model, corroborating results already existing in literature.

Models developed by partial least squares regression (PLS), based on original spectra, provided a coefficient of determination of 0.736 and ratio performance deviation of 2.29 with six latent variables for estimation of bulk density at 0% of *Eucalyptus grandis*.

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