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DETERMINATION OF *E. benthamii* PROPERTIES BY INFRARED SPECTROSCOPY AND PLS

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Resumo

Determinação de propriedades de E.benthamii por espectroscopia infravermelho e PLS. Considerando o interesse na espécie *Eucalyptus benthamii* e a busca de alternativas para determinação de algumas propriedades físicas de forma rápida e confiável, o objetivo deste estudo foi construir modelos de calibração multivariada por meio da técnica de espectroscopia de infravermelho próximo (NIR) e regressão dos mínimos quadrados parciais (PLS) para densidade básica da madeira (DB) e umidade (U) do cavaco. Árvores foram amostradas em cinco classes diamétricas e utilizadas para avaliar DB, em função de 3 idades e 3 regiões de produção de *E. benthamii.* Considerou-se apenas plantios na idade de 7 anos em fase de colheita e amostras de cavacos considerando intervalos pré-definidos de dez dias, desde a data de colheita das árvores até 90 dias, em duas estações do ano (inverno e primavera) para análise de umidade. Para DB e U foram calibrados e validados modelos NIR utilizando a regressão pelos mínimos quadrados parciais (PLS). Os modelos de calibração foram avaliados pelos coeficientes de correlação (R^2), raiz quadrada do erro médio (RMSE) e relação de desempenho do desvio (RPD). Os modelos para espectroscopia de infravermelho próximo (NIR) apresentaram valores de R^2, que variaram entre 0,60 e 0,68 para densidade básica e 0,72 para umidade. O melhor modelo para DB observado foi o que considerou apenas as amostras do DAP. Concluiu-se que a técnica NIR foi adequada para estimar as propriedades da umidade e densidade de madeira, nesta espécie avaliada, *E. benthamii*.

Palavras-chave: madeira, análise não destrutiva, calibração

Abstract

Considering the interest in the *Eucalyptus benthamii* species and the search for alternatives to determine some physical properties in a fast and reliable way, the objective of this study was to build multivariate calibration models through the near infrared spectroscopy (NIR) technique and partial least squares regression (PLS) for wood basic density (BD) and chip moisture (U). Trees were sampled in five diametric classes and used to evaluate DB, as a function of 3 ages and 3 production regions of *E. benthamii*. We considered only plantations at 7 years of age in the harvest phase and chip samples considering pre-defined intervals of ten days, from the date of tree harvest to 90 days, in two seasons of the year (winter and spring) for moisture analysis. For DB and U, NIR models were calibrated and validated using partial least squares (PLS) regression. Calibration models were evaluated by correlation coefficients (R^2), root mean square error (RMSE), and variance performance ratio (RPD). The models for near infrared (NIR) spectroscopy showed R^2 values ranging from 0.60 to 0.68 for basic density and 0.72 for moisture. The best model for DB observed was the one that considered only the DAP samples. It was concluded that the NIR technique was suitable for estimating the properties of moisture and wood density in this evaluated species, *E. benthamii.*

Keywords: wood, non-destructive analysis, calibration

INTRODUCTION

The preference for fast-growing species and the diversity of uses for wood highlight the *Eucalyptus* genus in Brazil, as well as the prominence of silvicultural practices, evolution of technologies, and forest management have been fundamental for the growth of the forestry sector and consolidation of eucalyptus cultivation. The *Eucalyptus benthamii* Maiden et Cambage is a species of interest for cold climate regions and has been used for energy purposes. Even with great knowledge about silvicultural and management practices applied to planted forests, the variations in growth and properties of this species are still not sufficiently known.

To know the suitability of a species for a particular use, the technological properties of this material are evaluated. The basic density is one of the most studied properties in the technological characterization of wood, given its importance in correlation with the physical and mechanical properties (NASCIMENTO *et al*., 2017), given its importance in correlation with the physical and mechanical properties, in order to qualify it for the most varied uses of wood. This property can bring information for the decisions of the manager and for the planning and destination of his forest and or for the adequacy of its use, before its harvest.

Especially for the use of forest biomass for energy generation, humidity is one of the main variables to be controlled before wood processing, because high humidity influences the increase of fuel consumption in the water evaporation phase, contributes to heat and gas losses, and makes it difficult to control the combustion process. Moreover, the presence of water has an inversely proportional relationship with the calorific value of the fuel (BRAND, 2010).

The control of moisture content in wood or biomass destined for energy use is a concern for industries, both for the remuneration of suppliers as a function of product quality, segregation of loads for consumption and for the negative impacts that excess moisture can bring on production and energy yield. Among these impacts, one can highlight the lower performance of the boiler operation, higher energy consumption, in addition to increased costs with energy inputs.

In both cases, whether for the determination of wood density or moisture, the use of conventional or destructive methods become costly and expensive, because they require more resources and time to obtain the results. The use of non-destructive methodologies for the determination of several wood attributes is gaining space in the forest sector, due to the advantages that they offer over traditional methods, in order to contribute with fast, precise and reliable information. One of the tools that has been successfully employed in the forestry area, used as an alternative to traditional destructive methods, is near infrared spectroscopy and multivariate regression modeling. This methodology is based on the adjustment of predictive models, relating dependent variables such as wood properties and independent variables: reflectance or absorbance of electromagnetic radiation (HEIN *et al.*, 2010).

For the basic density of wood, the most recent studies adopt sampling at DBH level, without the need to fell the tree, to estimate the wood properties and by non-destructive analysis, from the calibration of models compared to a reference method (LAZZAROTTO *et al.*, 2016, ESTOPA *et al.*, 2017). For moisture, there are few studies with non-destructive techniques for its determination, but the calibration model by near infrared spectroscopy (NIR) for moisture can be an option to the oven method and assist in the rapid determination in samples of wood chips, before their use in boilers, providing immediate information that contributes to better utilization of energy from this input.

Given the above, the objective of this work was to build multivariate calibration models by near infrared spectroscopy and PLS for the determination of wood basic density and wood chips moisture for *E. benthamii.*

MATERIAL AND METHODS

Material Sampling and Selection Basic Density

The trees used to determine the basic density of the wood were selected from commercial *Eucalyptus benthamii* plantations in the municipality of Guarapuava-PR. The experimental sketch included three regions (R1, R2 and R3) and three ages (5, 6 and 7 years). Three trees were randomly chosen within the interval of each class, totaling 45 trees at each age. Table 1 shows the average diameters of each tree and their respective diametric class in the sampled regions.

Table 1. Diameter of E. benthamii trees sampled at each age and region, in the municipality of Guarapuava-PR
Tabela 1. Diâmetro das árvores de E. benthamii amostradas em cada idade e região, no Município de Guarapuava-PR

Center of Class	**5 years**			**6 years**			**7 years**		
(cm)	**R1**	**R2**	**R3**	**R1**	**R 2**	**R 3**	**R 1**	**R2**	**R3**
7	5.4	5.6	5.6	5.9	5.1	6.0	5.6	6.0	5.6
	7.0	8.0	6.7	7.3	6.4	7.6	7.0	7.6	6.4
	8.0	8.8	8	8.3	7.0	8.9	8.3	8.6	6.4
11	10.5	9.9	10.2	10.5	8.9	9.9	9.7	9.5	9.3
	11.1	11.3	11.5	11.3	10.2	11.1	10.0	11.0	9.3
	12.1	12.1	12.4	12.4	11.8	12.4	11.4	11.5	10.3
15	14.3	13.8	13.4	13.1	13.4	13.7	13.6	14.3	13.1
	14.8	15.0	15.1	14.7	14.3	15.6	14.8	16.0	13.9
	16.5	16.2	16.7	15.6	15.3	16.9	16.0	17.0	15.3
19	17.5	17.5	17.8	17.3	17.2	18.1	17.5	17.6	17.8
	18.5	17.5	18.8	18.1	18.3	19.4	18.9	19.5	18.6
	19.7	20.1	20.4	19.4	20.1	20.7	20.7	20.7	20.4
23	21.6	21.3	21.3	21.0	21.0	22.0	22.3	22.2	22.3
	23.6	22.3	22.9	22.6	22.3	22.4	23.8	22.8	21.5
	23.9	24.2	25.0	23.0	24.7	23.9	27.8	24.2	22.9

Legend: R1 = Region 1, R2 = Region 2 and R3 = Region 3. Center of class = Center of the diametric class in which the trees were sampled..

After felling the trees, five disks were removed from each tree at heights corresponding to 0; 1.30 m; 25%; 50% and 75% of the total height of the tree. Opposite wedges were marked on each disc and used to determine the basic density, which followed the method adapted from ASTM D2395-14 (AMERICAN SOCIETY FOR TESTING AND MATERIALS, 2009). The results were presented in volume-weighted form.

Sampling and selection of material for determining the moisture content of *E. benthamii* wood chips

A commercial *Eucalyptus benthamii* plantation, at 7 years of age (rotation age), was selected to determine the moisture content in the caving process. Whole trees were harvested with a Feller buncher and bundles were formed in the stand. A representative area of the stand was selected, in which the harvest date of the trees was identified. Immediately following this step, sequential sampling of minimum, average, and maximum diameter trees from that population was conducted within ten days of the start of harvest. The whole trees were dragged to the whole tree chipper for further chipping. From the chopped volume, in each evaluation period three composite samples (leaves, branches, wood, and fines) were collected for the determination of whole-tree chip moisture content. Sampling was performed in the winter and spring periods, up to 90 days after the beginning of harvesting. This period was chosen to evaluate the behavior of whole-tree moisture loss, evaluated from the chips produced. Chip moisture was determined by the kiln method, based on ASTM D1762-84.

Sample Preparation and Spectra Collection

The same samples (Figure 1A) used for basic density, for each sampling position, were read in a spectrophotometer (Figure 1C). For chip moisture, it was not necessary to transform the sample. A representative portion of the original sample (Figure 1B) collected in each evaluation period (wood, leaves, twigs, bark, fines) was selected to obtain the spectra.

Figure 1. Wood and chip samples and Spectrophotometer used to collect spectra
Figura 1. Amostras de madeira e cavaco e Espectrofotômetro utilizado para coleta de espectros

The spectra were obtained from a Bruker FTNIR spectrometer with MPA accessory (Figure 1C). The wood samples were read in triplicate in the radiation range of 4000 to 12000 cm^{-1} and spectral resolution of 4 cm^{-1}. Between readings, the sample was mixed to homogenize it. Every one hour of reading, the blank was read for calibration, reference and reliability of the spectra results.

Calibration, validation and model selection

The models were generated by Opus 7.2 software. The spectra received 1st derivative or SNV (normal signal standardization) pre-treatment to reduce spectral noise and to improve signal quality. The data were centered to the mean. For each model the best pre-treatment that optimized the calibration quality was selected. A preliminary analysis of the data was performed to identify anomalous samples, using the frequency histogram. Samples that did not contribute to the model were excluded to improve the peformance of the models.

The models were fitted by the partial least squares (PLS) regression method, assuming a maximum of 10 latent variables. The number of latent variables for each model, however, was chosen according to the value that maximized the residual variance of calibration and validation, increased the correlation coefficients, and reduced the cross-validation error.

Cross validation was used because of the number of samples available for some models. Cross-validation consisted of separating 90% of the samples for model building and 10% of the remaining samples for internal validation. The procedure is repeated for each sample until the entire initial data set is explored or has participated in the validation (FERREIRA, 2015).

The choice of the prediction models considered the correlation coefficient of the model in cross-validation (R^2), square root of the mean error (RMSECV) that measures the efficiency of the calibration model in predicting the property of interest in cross-validation, number of latent variables (VL) used in calibration and deviation performance ratio (RPD).

RESULTS

Figure 2 (A, B, C, D, E) shows the spectra collected for samples of *E. benthamii* used for calibration of the models of basic density, in the range between 4000 and 12000 cm $^{-1}$. It was possible to observe for all models, the same pattern of peaks in the spectral region, however with different intensities.

The most pronounced peaks were between the regions of 6900 -7000 cm^{-1}, 5000 to 5500 cm^{-1} and in the range of 4000 cm^{-1}, which are observed as a function of the chemical constituents present in the wood samples, representing mainly CH and CH_2 groups.

The spectra of the *E. benthamii* samples used for the moisture model of chips obtained from whole trees (Figure 2F), show peaks between the regions of 5000 and 7000 cm^{-1}, indicating water-related bands. The presence of water in the sample reduces the quality of the spectrum and the higher the water content in the sample, the higher the intensity of the band in the spectrum.

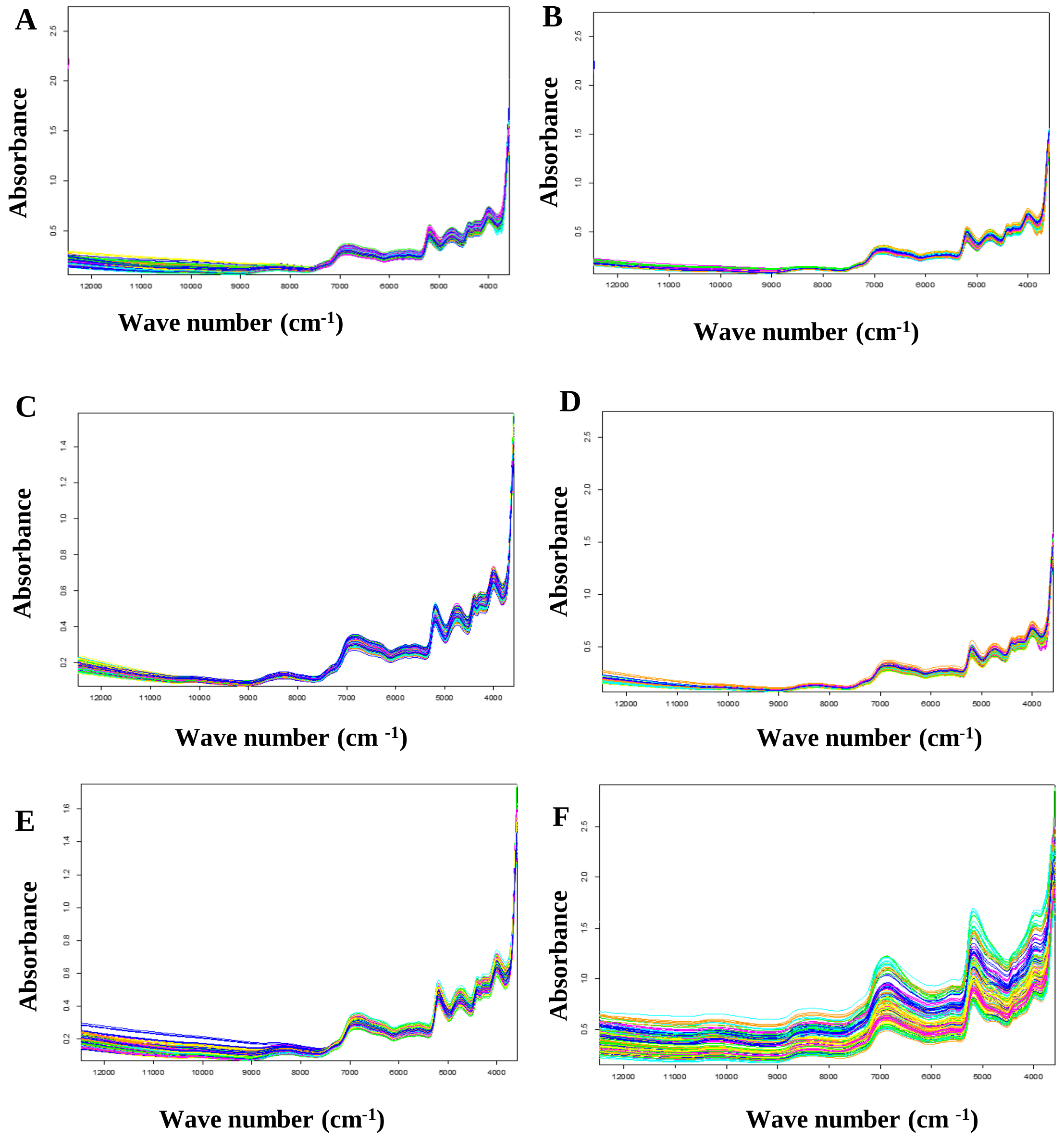


Figura 2. Espectros obtidos para amostras de *E. benthamii* para modelos de densidade básica (DB) e umidade (U). **A** (DB com todas as posições de amostragem). **B** (DB posição DAP). **C** (DB 5 anos). **D** (DB 6 anos). **E** (DB 7 anos). **F** (Espectros das amostras para o modelo de umidade).

Figure 2. Spectra obtained for *E. benthamii* samples for basic density (BD) and moisture content (U) models. **A** (DB with all sampling positions). **B** (DB DAP position). **C** (DB 5 years). **D** (DB 6 years). **E** (DB 7 years). **F** (Spectra of the samples for the moisture model).

Table 2 shows the results of the calibration and cross-validation models for the basic density models, with their respective spectral treatments. For the model with all samples the correlation coefficient was $R^2c = 0.63$, and the cross-validation error was 0.0154 g cm^{-3} and RPD = 1.58.

Tabela 2. Resultados da calibração e validação cruzada de modelos NIR para a densidade básica da madeira de *E. benthamii*

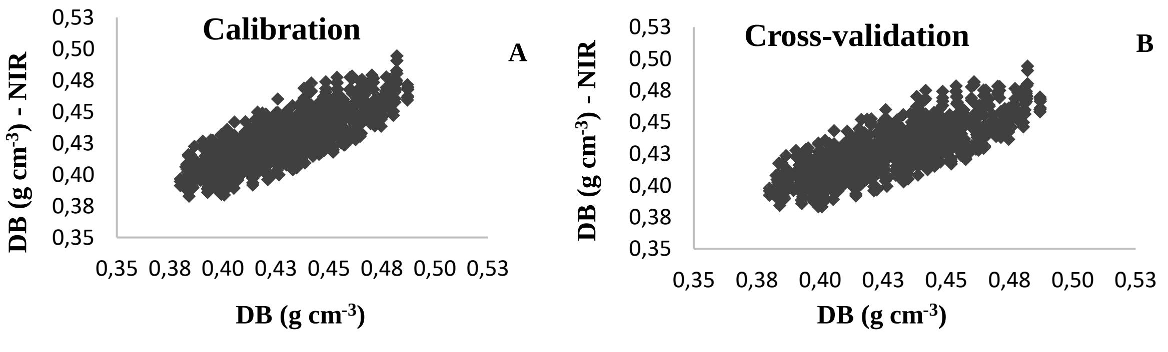
Table 2. Results of calibration and cross-validation of NIR models for the basic density of *E. benthamii* wood

Property	**Pre-treatment**	**Model**	**VL**	**R^2**	**RMSE**	**RPD**
Basic Density	1st derivative	Calibration	10	0.637	0.0148	1.66
		Cross-validation	10	0.601	0.0154	1.58
Basic Density	1st derivative	Calibration	5	0.681	0.0131	1.77
DAP		Cross-validation	5	0.626	0.0139	1.64
Basic Density	1st derivative	Calibration	6	0.649	0.0126	1.69
(5 years)		Cross-validation	6	0.603	0.0133	1.59
Basic Density	1st derivative	Calibration	7	0.486	0.0168	1.40
(6 years)		Cross-validation	7	0.419	0.0177	1.31
Basic Density	1st derivative	Calibration	10	0.664	0.0154	1.73
(7 years)		Cross-validation	10	0.595	0.0167	1.57

For the model built only with samples collected at the DBH position (1.30 m), slightly more than 25% of the total samples available, 5 latent variables were used. The correlation coefficient for this model was higher than the model with all sampling positions ($R^2 = 0.68$). In cross-validation, this model showed lower cross-calibration error, RMSECV = 0.0139 g cm^{-3} and RPD = 1.64 (Table 14). This shows that the representativeness of the samples may be a more important factor than a high number of samples, that is, at the DBH position, there is a better representativeness of the samples in relation to the basic density of the tree.

For models built at different ages, the best results were obtained for calibration and cross-validation at the age of 7 years, followed by the model for 5 years, with $R^2c = 0.66$ and 0.64, respectively. In cross-validation, the smallest errors were observed for the 5-year age (0.013 g cm^{-3}) and the 7-year model (0.016 g cm^{-3}). On the other hand, the model built for basic density at the age of 6 years showed $R^2c = 0.48$ for calibration and $R^2 = 0.41$ for cross-validation and presented RMSECV value = 0.0177 g cm^{-3}, considered the highest error among all models evaluated. The RPD values for models at the age of 6 years were less than 1.5, which together with other statistics show an inaccurate model for estimating the basic density of *E. benthamii*.

The observed and predicted values for the NIR models of basic density are presented in Figure 3. Figure 3 (C and D) shows the values of basic density for calibration and validation models, only for DBH positions. It can be observed for the cross-validation plot (Figure 3 D), that absence of some samples in the range between 0.40-0.43 g cm^{-3} may have contributed to a median value in the value of the correlation coefficient for this model.



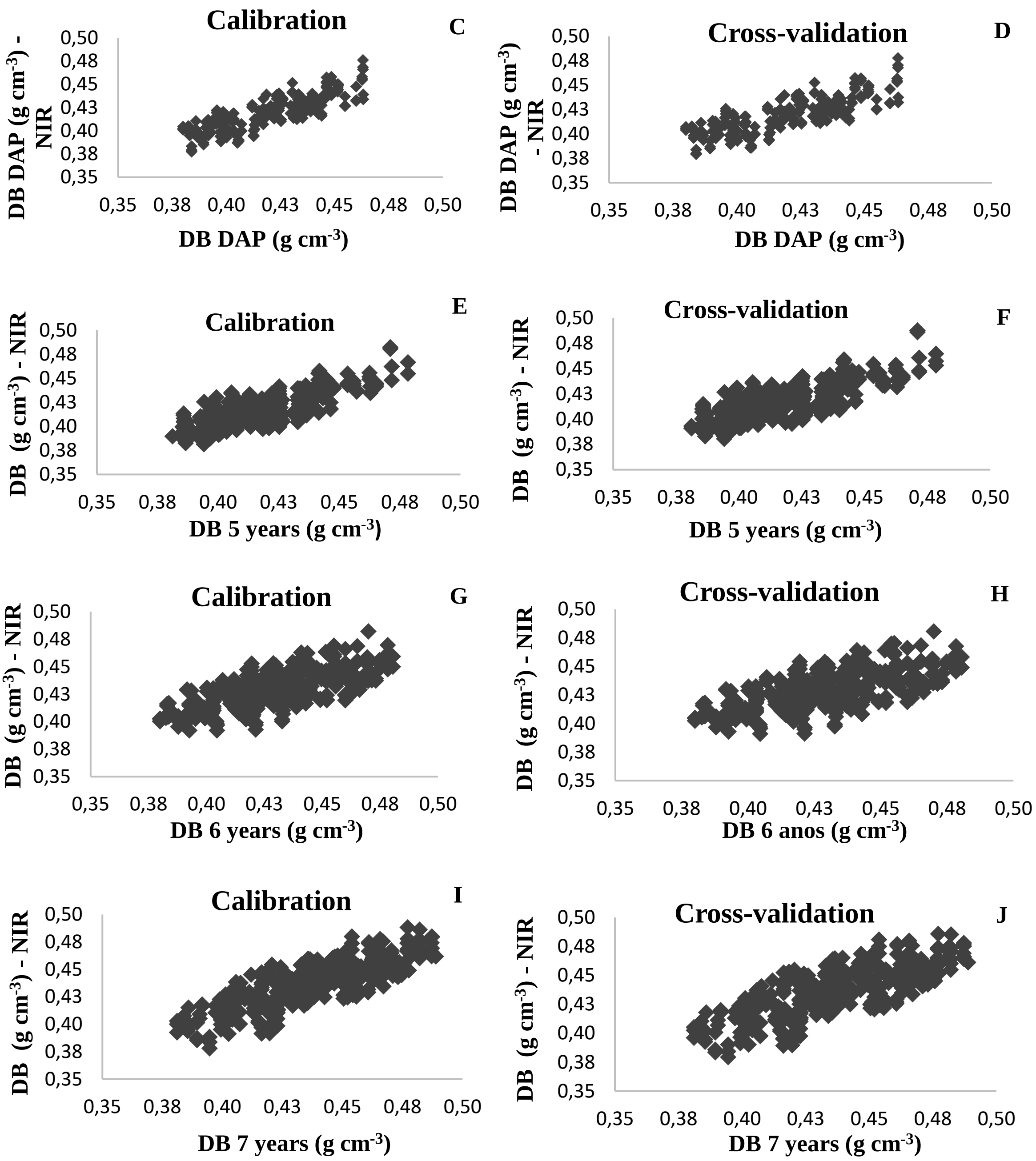


Figure 3. Values of basic density of *E. benthamii* determined by the conventional method and values predicted by near infrared spectroscopy for calibration and cross-validation models. A and B (DB all samples); C and D (DB DAP samples); E and F (DB 5 years); H and G (DB 6 years); I and J (DB 7 years)

Figura 3. Valores de densidade básica de *E. benthamii* determinados pelo método convencional e valores preditos por espectroscopia no infravermelho próximo para modelos de calibração e validação cruzada. A e B (DB todas as amostras); C e D (DB amostras DAP); E e F (DB 5 anos); H e G (DB 6 anos); I e J (DB 7 anos)

Humidity model calibration

Table 3 shows the results of the calibration and validation models for moisture (%) and its respective pretreatment. The calibration showed R²c = 0.72, square root of the mean cross-validation error of 2.92 % and RPD of 1.79. The number of latent variables that optimized the correlation coefficient and reduced the cross-validation error was 1.

Table 3. Results and statistics of calibration and validation of NIR moisture models of *E. benthamii*
Tabela 3. Resultados e estatísticas de calibração e validação de modelos NIR umidade de *E. benthamii*

Property	Pre-treatment	Model	VL	R²	RMSE	RPD
Humidity (%)	SNV	Calibration	1	0.72	2.79	1.89
		Cross-validation	1	0.68	2.92	1.79

Where: VL = Number of latent variables used for the model. R² = correlation coefficient of the model. RMSE = Square root of the mean error (%), being considered RMSEC = for calibration models, RMSECV = for cross-calibration models. RPD = Variance performance ratio.

Figure 4 shows the values determined and predicted by the NIR models for moisture. By evaluating the distribution of points on the calibration graphs, it is possible to verify that the absence of samples between some moisture ranges may have influenced the median values of the coefficient of determination. This is due to the sampling method adopted (10 in ten days after felling the tree), also indicating the effect of the loss of free water from the wood at the beginning of the process and also the relative humidity of the air during the sampling period. Although the models did not present high values of determination coefficients, they were sufficient to identify the moisture points of interest for the production and use of *E. benthamii* chips.

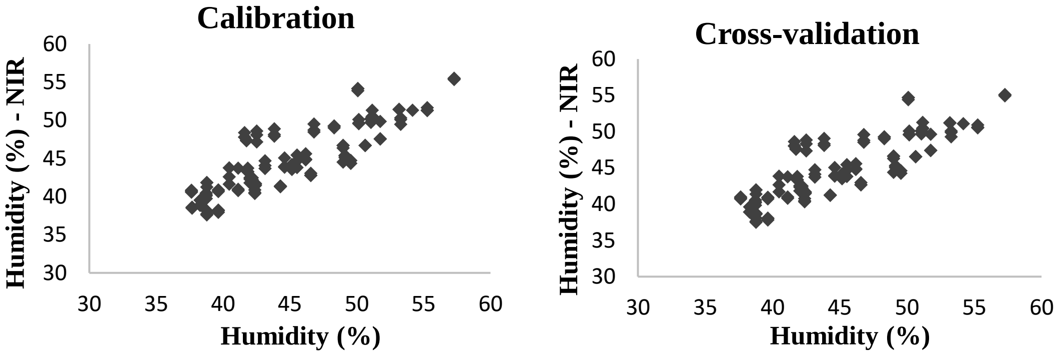


Figure 4. Moisture values (%) of whole-tree chips of *E. benthamii* determined by the oven method and values predicted by near-infrared spectroscopy
Figura 4. Valores de umidade (%) de cavaco de árvores inteiras de *E. benthamii* determinadas pelo método de estufa e valores preditos por espectroscopia no infravermelho próximo

DISCUSSION

The results found for the model with DBH samples corroborate the values found by Lazarotto and Magalhães (2014). These researchers developed models for 40 trees of *E. benthamii* and 44 trees of *Eucalyptus pelitta* and found results for these models developed by the near infrared spectroscopy (NIR) technique with R² = 0.78 and error of 21 kg m^{-3}. In the external validation, performed with 28 samples, the error for the model was 27 kg m^{-3} and the R² = 0.62.

The PLS models, in general, had a RPD of less than 2. The higher the RPD the better the accuracy, however, Prades *et al.* (2014) proposes that models with RPD greater than 1.5 are considered sufficient screening and preliminary predictions.

Milagres *et al.* (2013) evaluated the effect of age on the construction of NIR models for the prediction of wood properties of *Eucalyptus sp*, and concluded that the choice of model for sample prediction should be according to the characteristics of the samples to be predicted, i.e., in case of samples with different ages from those used in the development of the model will not provide good predictions. It was observed that only the models for basic density with different ages of samples were able to accurately predict the group property.

In general, the average errors of calibration and cross-validation found for the models of basic density in this study, are lower than those reported in the literature, with a maximum observed value of 0.0154 g cm^{-3} or 15.4 kg m^{-3}, except for the model of basic density at five years, which is not recommended for use in predicting this property for *E. benthamii*. Baldin *et al.* (2020) used samples of 87 trees of *E. benthamii*, obtained in the region of DBH, to calibrate models for the basic density and obtained fits with R² = 0.73 and RMSEC = 16 kg m^{-3}. In this case the author worked with three latent variables and the 1st derivative as spectral pre-treatment.

Estopa *et al.* (2017) developed NIR models for basic density, using samples of *E. benthamii* in the Santa Catarina region, in order to evaluate the species in improvement programs. The results for the correlation coefficients were R² = 0.37, R² = 0.33 and R² = 0.13, for the calibration model, cross-validation and external validation, respectively. In all three cases, 5 latent variables were selected and the RPD was 1.4. The correlation was considered low to use the model in predicting this property. For *Eucalyptus sp*, the statistics of the models developed by the near infrared spectroscopy technique were R2 = 0.83 and the cross-validation error was 45 kg m^{-3} (LAZZAROTTO e MAGALHÃES, 2014).

Studies with other species also found satisfactory results in determining the basic density. Nascimento *et al.* (2017) tested different types of pre-treatments and obtaining spectra in radial and tangential directions, for the wood of *Eschweilera odora*, obtaining models with correlation coefficients greater than 0.90 and consequently low calibration and validation errors. The authors concluded that the NIR technique is suitable for estimating the basic density of the species. For *Pinus maximoii* and *Pinus tecunumanii*, the authors developed models with 54 samples and obtained correlation coefficient of 0.94 and validation error of 30 kg m^{-3}. In the external validation, the error value was 47 kg m^{-3} and the RPD = 1.9, indicating that the model is satisfactory for predicting new samples of the species (LAZAROTTO *et al.*, 2016).

Other authors have also concluded from their results, about the efficiency of the NIR technique associated with PLS regression for estimating wood technological properties, such as basic density (MORA *et al.*, 2011, MILAGRES *et al.*,2013, BALDIN *et al*., 2020).

Humidity

There are few studies reporting the use of near infrared spectroscopy to determine the moisture content of wood, some models developed under laboratory conditions and other more recent ones demonstrate the interest in applying this methodology using spectroscopy and PLS modeling.

Mora *et al.* (2011) evaluated the moisture content of green *Pinus taeda* logs using near infrared spectroscopy, by obtaining spectra on the cross-section of logs aged between 13 and 19 years. The results for the correlation coefficient for the calibration model was $R^2 = 0.85$ and mean square error of 2.1%, as 9 latent variables.

For Korean Pine, the near infrared spectroscopy technique was employed for wood moisture prediction. Seven wavenumbers were selected and partial least squares (PLS) regression was used. The best result was obtained between the 1000 to 2100 cm^{-1} wavenumber, where the model showed validation correlation coefficient of 0.98 and RMSEP = 0.0465 % (ZHANG *et al.* 2011). The study by Santos *et al.*, (2017) demonstrated that NIR spectroscopy is an efficient technique to estimate wood moisture and monitor water desorption of *Eucalyptus urophylla* x *Eucalyptus grandis*.

To evaluate the water desorption of *E. urophylla* x *E. grandis* chips during the process of chip drying, Amaral *et al.* (2020), conducted an experiment where they collected spectra and moisture of the chips in predetermined periods, under laboratory conditions, according to the loss of mass of the samples. For the moisture models, they obtained the best calibration results with application of the first derivative treatment, presenting R^2cv greater than 0.90. They concluded that the technique is efficient for moisture determination, as well as samples in chip form are more suitable for obtaining spectra.

Amaral (2020) classified NIR models for moisture by moisture ranges, ranging from 0 to 40%, 40 to 80%, and > 80%. The best calibration and cross-validation results were obtained for the 40 % range, with values of $R^2 = 0.96$, SECV = 2.50 %, RDP = 5.33. When submitting the data for external validation, all models were appropriate for moisture estimation, however, the best performing range was 0 to 40 % with external validation coefficient of determination $R^2p = 0.96$.

The moisture models showed potential for estimating the property for whole tree chips of *E. benthamii*. The introduction of new samples to the model tends to improve its predictive ability, and can be used for rapid determination of the moisture content for chip samples.

CONCLUSIONS

- The NIR models were appropriate for predicting wood properties, considering the figures of merit of the models for density and moisture;
- The forest manager can use average basic density information from his stands, using sampling at DBH level and NIR models developed for basic density, to accurately determine the value of samples of his interest.

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