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Prediction of the basic density of tropical woods by near-infrared spectroscopy

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TECHNOLOGY OF FOREST PRODUCTS

ABSTRACT

Background: Determining the basic density of the wood is usually defined as a simple process, but it requires caution and the operator's skill to avoid errors in the analysis. In addition, it involves sample preparation and time to saturate the wood until obtaining the dry sample mass. The development of alternative measurement techniques could reduce the time to obtain the results and provide reliable values. Therefore, this study aimed to develop multivariate models to estimate the basic density of native woods using near-infrared spectra (NIR). Basic densities were determined by the water immersion method, and the values were associated with NIR signatures. Spectra were directly collected on the wood transversal and radial faces with an integrating sphere. Partial least squares regression (PLS-R) was calibrated and validated to estimate basic density based on spectral signatures.

Results: In the cross-validation and prediction of the models, the results were promising. The coefficients of determination varied from 0.87 to 0.93 with a standard error of 0.01 %. The partial least squares discriminant analysis (PLS-DA) efficiently classified the wood species. The ratio of performance to deviation obtained satisfactory values, a minimum of 2.81 and a maximum of 4.20.

Conclusion: The statistical parameters of the models based on NIR spectra showed potential for density measurements in floors, furniture, and solid wood products.

Keywords: Multivariate statistics, Non-destructive analysis, Amazonian species, wood identification, classification.

HIGHLIGHTS

The NIRS technique was effective in predicting the basic density of tropical woods.
The determination coefficients ranged from 0.87 to 0.93 with a standard error of 0.01%.
For all models, PC1 explained more than 90% of the data variation, and the PLS-DA classified the species with 100% success.
Bowdichia nitida showed higher NIR absorbance due to the high density of the wood (0.81 g.cm^{-3}).

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INTRODUCTION

Density is the main physical property in the technical evaluation of wood and can be determined by the relationship between mass and volume (Dias et al. 2018). Studies address different destructive methods in tree sampling (Zaque et al. 2018). However, there are fast, reliable, and non-destructive methods that can be used in predicting wood density. These methods do not damage trees and allow field evaluations, such as near-infrared spectroscopy (NIR) (Costa et al. 2018; Arriel et al. 2019). The NIR is a method that allows the estimation of characteristics: physical (Amaral et al. 2021), chemical (Mancini et al. 2021), mechanical (Ma et al. 2021), and anatomical (Sánchez et al. 2013) of wood, and can be applied in real-time monitoring, including on-site analysis.

NIR spectroscopy has several advantages when compared to traditional methods, such as little or no sample preparation, quick obtainment of information, and low operating cost, in addition to the non-production of chemical residues (Pasquini 2018). This method is based on vibrational spectroscopy, consisting of exposing the samples to electromagnetic radiation in the wavelength region ranging from 4,000 cm^{-1} to 12,500 cm^{-1}, to obtain the absorbance spectra of the bond vibrations from organic molecules (Muñiz et al. 2012; Arriel et al. 2019). Spectral information analysis requires the application of multivariate statistical tools (Soares *et al*. 2017). Spectra obtained from NIR contain information on the material properties and/or chemical constituents and can generate qualitative and/or quantitative information when associated with multivariate analyses (Amaral et al. 2020). In qualitative studies, the main multivariate technique applied in the wood sector is Principal Component Analysis (PCA), which is used to discriminate and group unknown samples. On the other hand, Partial Least Squares Regression (PLS-R) is the most used for quantitative analyses to estimate material properties (Sandak et al. 2016).

Multivariate regression models developed from NIR spectra have been successfully applied to estimate the technological wood properties in different forest species (Ramalho et al. 2018; Arriel et al. 2019; Shukla et al. 2021). Among the fundamental properties of the industrial wood sector, density has been adopted as a quality indicator, mainly for particleboards production, pulp and paper, charcoal, and the mechanical performance of wood-based products (Hein et al. 2017; Mascarenhas et al. 2021).

Several studies have successfully reported the prediction of basic density by NIR technology (Diesel et al. 2014; Hans et al. 2015; Arriel et al. 2019; Ayanleye and Avramidis 2021). However, most studies have focused on wood from planted forests, especially *Eucalyptus* sp. Costa et al. (2018) investigated the settings of the NIR parameter to identify the most suitable anatomical plane of *Eucalyptus urophylla* × *Eucalyptus grandis* wood pieces for spectra acquisition and generation of density models. Similarly, Amaral et al. (2021) studied different models to predict the wood density of *Eucalyptus urophylla* × *Eucalyptus grandis* using NIR spectroscopy, founding values for the coefficient of determination of prediction = 0.53.

Therefore, research involving wood from natural forests must define a more appropriate way to apply the technique and generate more accurate predictions for native woods. According to Medeiros et al. (2021), tropical woods are traded in different countries and used in producing furniture, plywood panels, musical instruments, and flooring. Among the central woods are *Manilkara huberi, Bowdichia* sp., *Caryocar villosum*, and *Piptadenia suaveolens* (Teixeira et al. 2019). Studies have shown the potential of NIR to predict the basic density of the wood from native species (Pace et al. 2019; Nascimento et al. 2021). This indicates that obtaining wood density through non-destructive monitoring is a crucial step to simplify and optimize the management routine of forest-based companies, allowing greater predictability of the technological behavior of their products.

In this context, the objective of the study was to evaluate the potential of NIR spectroscopy associated with multivariate statistics to develop predictive models for the basic density of Brazilian native woods.

MATERIAL AND METHODS

Obtainment and preparation of the samples

Wood samples of the tropical species: *Manilkara huberi* Ducke, *Caryocar villosum* Pers., *Bowdichia nítida* Spruce, and *Piptadenia suaveolens* Miq. were selected in this study considering the commercial demand for wood flooring. The woods were obtained from commercial establishments that operate under the terms of environmental legislation, located in Curitiba, State of Paraná, Brazil (latitude 25°42'84" S, longitude 49°27'33" W, and altitude 930 m). From each species, 20 heartwood samples with dimensions 4.7 × 2.5 × 1.8 cm were obtained. The samples were kept in acclimatized room adjusted to a humidity of 65 % and temperature of 25 °C until the wood reached moisture ~12 %. Subsequently, all samples had their faces polished with sandpaper.

Determination of basic density

The basic density of the wood was obtained by the ratio between the anhydrous wood mass and its saturated volume (moisture > fiber point saturation). Ten samples were evaluated for each species through the procedures based on the standard ASTM D2395-17 (ASTM 2022). The density of the woods were categorized employing the classification suggested by INDEA (2011).

Near-infrared spectroscopy

The spectral acquisition was performed in a Tensor 37 Fourier Transform Near-infrared spectrometer (Bruker, USA) via integrating sphere, in diffuse reflectance mode, recording the log (1/R); where R = reflectance every 4 nm (Figure 1). The range acquisition varied from 4,000 cm^{-1} to 10,000 cm^{-1} with a resolution of 4 cm^{-1} and 64 scans. A standardized gold was used as a measurement reference before the NIR spectral readings.

Ten different samples from those evaluated for density were used to collect the spectra directly on the wood faces. Five spectra acquisitions were performed on the transversal plane and five on the radial plane of each sample. Posteriorly, 100 wood spectra of each species were collected and saved in the software OPUS 7.5 storage. The spectra were obtained in an acclimatized room, with a temperature of 25 ºC and relative humidity close to 65 % until reaching the equilibrium moisture for the region (~12%).

Multivariate statistical analyzes

After acquiring the spectral matrix, multivariate statistics were applied using the statistical package Chemoface 1.61 (Nunes *et al*. 2012). Principal component analysis (PCA) was applied to species discrimination to assess the spectral similarity of the different species. The partial least squares discriminant analysis (PLS-DA) was performed to classify the four wood classes, with values evaluated by the number and percentage of correct responses. For calibration and validation of the predictive basic density models, partial least squares regression (PLS-R) was applied to correlate the values predicted by the technique and measured in the laboratory.

The models were generated from the spectra measured on the radial and transversal wood faces, being validated by cross-validation (leave-one-out) and independent (60 % of samples for calibration and 40 % for validation). The original data were treated by the first derivative (13-point filter and second-order polynomial) and second derivative (25-point filter and second-order polynomial) for noise suppression (Lazzarotto *et al*. 2016; Ramalho *et al*. 2018).

The number of latent variables (LV) was determined based on the reduction of standard error and maximization of validation of R^2. The performance ratio of standard deviation (RPD) was obtained by the ratio between the standard deviation of the measured values and the standard error of cross-validation and independent. The statistical parameters used to select the most robust models were the coefficient of determination of cross-validation and independent (R^2cv and R^2p), the root of the average standard error of the validations (RMSEV and RMSEp), number of latent variables (LV), and performance ratio of standard deviation (RPD).

RESULTS AND DISCUSSION

Basic density of the tropical species

The highest values of basic density were obtained for the woods of the species *Bowdichia nitida* and *Manilkara huberi*, which did not differ statistically. The wood of *Piptadenia suaveolens* presented the lowest density value, while *Caryocar villosum* obtained intermediate results (Table 1).

Considering the classification proposed by INDEA (2011), *Piptadenia suaveolens* was classified as moderately heavy, and the species *Bowdichia nitida*, *Manilkara huberi*, and *Caryocar villosum* categorized themselves as heavy woods. The results obtained in the present study are similar to those verified by Texeira et al. (2019) for the same species. The basic density of wood directly influences the strength of parts, as in floors made of wood, in which you can provide more or less susceptibility to scratches due to the intense movement of people (Medeiros et al. 2021).

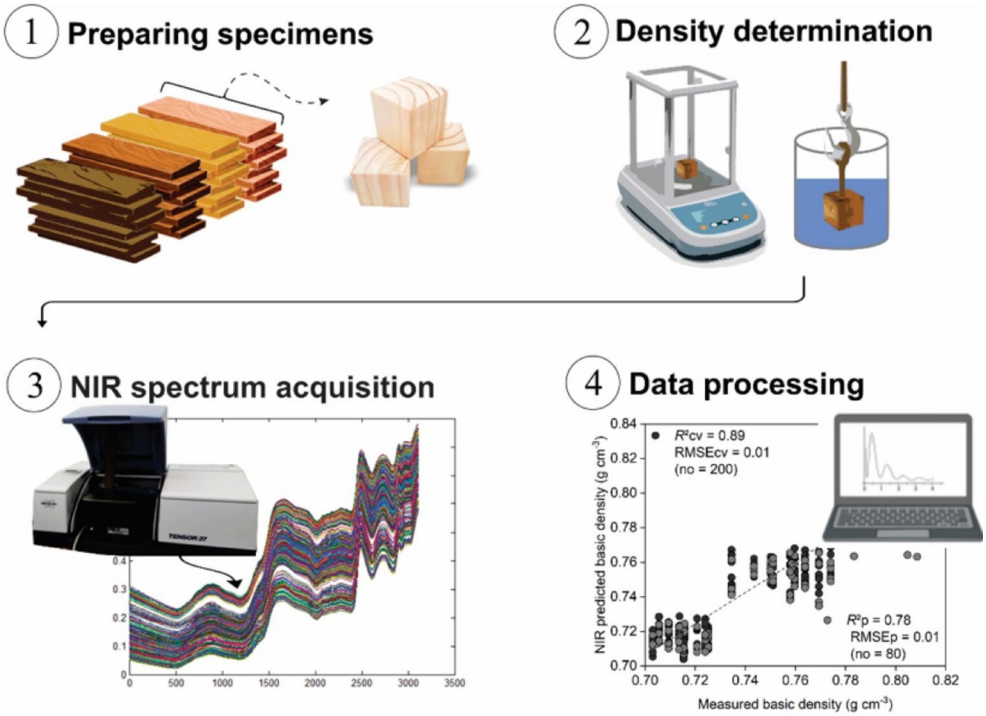

Figure 1: Steps for the obtainment of the samples and NIR spectra.

Table 1: Basic density of the Brazilian tropical woods.

Scientific name	Family	Basic density (g.cm^{-3})
Bowdichia nitida	Fabaceae	0.81 ± 0.01 a
Manilkara huberi	Sapotaceae	0.80 ± 0.02* a
Caryocar villosum	Caryocaraceae	0.76 ± 0.01 b
Piptadenia suaveolens	Fabaceae	0.71 ± 0.01 c

*Standard deviation. Values followed by distinct letters in the same column differ statistically by the Tukey test ($p < 0,05$).

Spectral signatures of the woods

The most prominent absorption bands were seen between 5,200 cm^{-1} and 4,500 cm^{-1} for both the original (Figure 2a) and the treated spectra (Figure 2b). Throughout the entire range (10,000 cm^{-1} to 4,000 cm^{-1}), the highest NIR absorbance intensities in decreasing order were observed for *Bowdichia nitida*, *Manilkara huberi*, *Caryocar villosum* and *Piptadenia suaveolens* (Figure 2a). This intensity behavior can be associated with wood density since the property showed the same decreasing trend. This variation in absorbance according to wavelength is according to results found by Pace *et al*. (2019) for native woods from the Atlantic Forest. The first derivative modified the behavior of the NIR spectra, improving the spectral signal by smoothing the noise and highlighting the main absorption bands, similar results were seen by Costa *et al*. (2018), Arriel *et al*. (2019) and Amaral *et al*. (2021).

According to Tsuchikawa and Korobi (2015), spectral variation allows us to identify species from correlation with the wood chemical components (cellulose, lignin, hemicelluloses, and extractives). Basic density is highly related to the chemical and anatomical aspects of the wood. Greater entanglement and fiber aggregation imply greater basic density. On the other hand, a more significant number of pores on the transversal face may suggest lower density, as well as a greater amount of parenchymal rays, which are generally formed by reserve and nutrition tissues (Zobel and Van Buijtenen 2012; Fromm 2013; Tyree and Zimmermann 2013).

Nisgoski *et al*. (2016) observed that wavenumbers between 7,000 cm^{-1} and 6,287 cm^{-1} comprise the amorphous and crystalline cellulose regions. Bands from 6,000 cm^{-1} to 5,970 cm^{-1} are associated with hemicelluloses and extractives (Vieira *et al*. 2021). The range between 5,974 cm^{-1} and 5,850 cm^{-1} makes it possible to identify the vibration of lignin aromatic rings. Wavelengths between 4,808 cm^{-1} to 4,739 cm^{-1} are attributed to cellulose (Schwanninger *et al*. 2011). Because it is denser (0.81 g cm^{-3}) and has more significant amounts of fiber, it is natural that *Bowdichia nítida* wood shows higher NIR absorbance intensities in the entire spectral range. The opposite trend can be attributed to the *Piptadenia suaveolens* wood, which is lighter (0.71 g cm^{-3}). This spectral behavior for all wavelengths according to density was also reported by Soares *et al*. (2017) when discriminating six different Amazon kinds of wood by NIR spectroscopy.

Principal component analysis

The contribution of principal components (PC) was performed through untreated NIR spectra and with mathematical pre-treatment (Table 2). Untreated PCA showed the highest cumulative results (PC1 + PC2). However, for all models, with pre-treatment or original spectrum, PC1 explained more than 90 % of the data variation, in agreement with studies conducted by Nisgoski et al. (2017) and Pace et al. (2019).

Table 2: Contribution (%) of the principal components (PC) to the original and pre-treatment spectra as a function of the analyzed woods.

Treatment	PC1	PC2	Total
Untreated	99.49	0.45	99.94
1d	98.76	0.71	99.47
2d	90.62	2.78	93.40

1d = first derivative; 2d = second derivative.

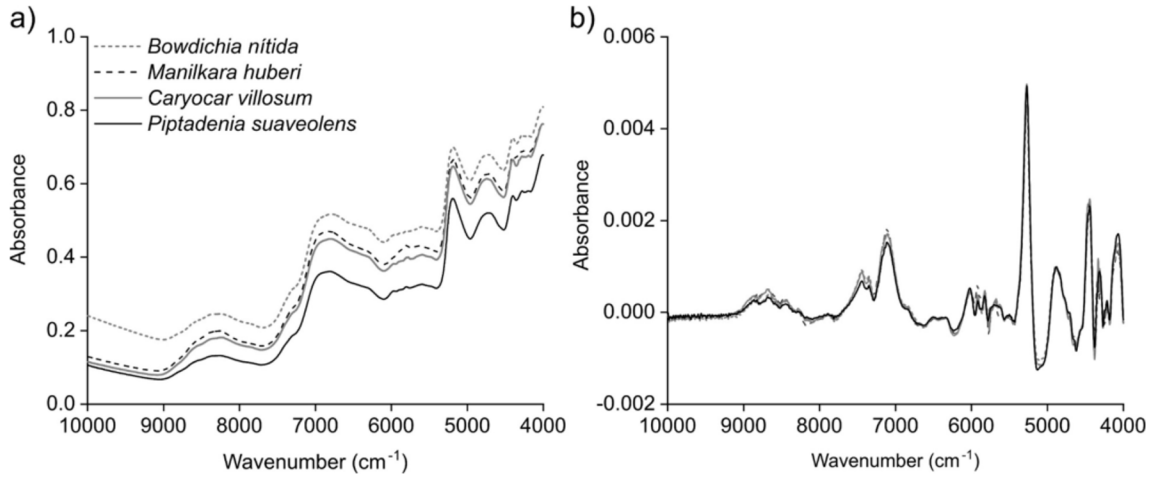

Figure 2: Average profile of the original NIR spectra (a) and treated with first derivative (b) of tropical Brazilian woods.

After treatment with the second derivative, the explanatory power of data variances by PC1 (90.6%) and PC2 (2.8%) for the species allowed to observe clusters close to *Bowdichia nítida* and *Piptadenia suaveolens* woods because both species belong to the same botanical family (Figure 3). The proximity of *Caryocar villosum* scores to the *Piptadenia suaveolens* group may be related to the densities similar range (0.71 to 0.76 g cm^{-3}).

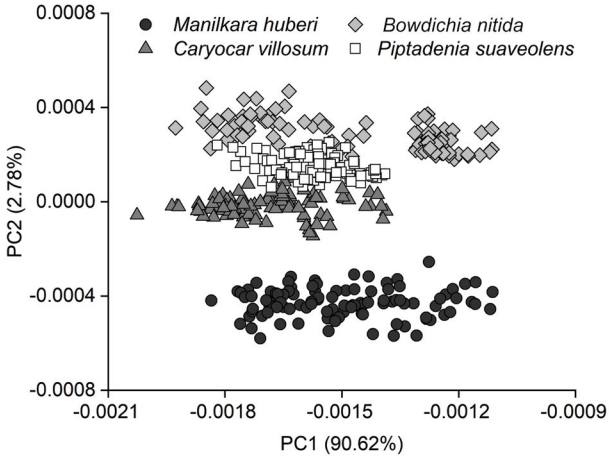

Figure 3: Scores of PCA with the second derivative treatment (2d) from spectra collected on the radial and transverse surface of tropical woods.

The data grouping by species in the score perceptual map showed satisfactory discrimination. The species group may be associated with the density variation between woods since this property is directly influenced by the chemical constitution and anatomical elements arrangement (Fahey et al. 2019; Liang et al. 2020). The application of mathematical pre-treatments in the spectra did not increase the total percentage of explained variance, although it visually clarified the species discrimination. This trend was also reported by Ramalho et al. (2018) when identifying groups of native woods. Yang et al. (2015) related that identifying wood species using non-destructive means can reduce processes and optimize production, enabling quickness and data reliability. This effect was observed in the present work because, from the PCA, the four species analyzed were organized clearly and precisely. In addition, PCA simplified the size of the spectral matrix database, and the information was grouped into a few components.

Partial least squares discriminant analysis

The classification of species using PLS-DA through cross-validation and independent was performed with 100% of success (Table 3). Therefore, it is possible to affirm that spectral signals showed a high correlation with the intrinsic characteristics of the woods, which resulted in high discrimination accuracy.

The results found in this study were encouraging and allowed the achievement of the objectives, considering woods from natural and heterogeneous forests. These aspects highlight the importance of the present work in demonstrating the possibility of using the NIR technique to identify wood and its by-products in various sectors of the forestry market. The observed trends are in harmony with other research. Pace et al. (2019) identified wood from 20 native species from Brazilian Atlantic Forest by NIR spectroscopy and multivariate analysis, founding PLS-DA models adjusted with reliability and efficiency of 93.3% of correct classifications.

Ramalho et al. (2018) differentiated wood from planted forests of *Eucalyptus* sp. with 90% of success and wood from natural forests (*Cedrela* sp., *Apuleia* sp., *Aspidosperma* sp., and *Jacaranda mimosifolia*) with percentages of success ranging between 45 and 90%. Toscano et al. (2017) used NIR spectroscopy and PCA to discriminate bark and wood of the species *Fagus sylvatica*, *Pinus* sp., and *Abies* sp. aiming to optimize quality control for pellets production.

Models to estimate the basic density of the woods

Parameters of PLS-R models showed significant modelling for predicting the wood's basic density (Table 4). The coefficients of determination were high, both for cross-validation (R^2cv) which varied between 0.88 and 0.93 and for independent validation (R^2p), which ranged from 0.87 to 0.93. RPD exposed values from 2.90 to 3.80 for cross-validation and from 2.81 to 4.20 for prediction.

Table 3: Wood classification by cross-validation and independent prediction in PLS-DA models without mathematical pre-treatment of the spectral matrix.

Validation type	NIR (spectra classification by species)					Correct classification	
	Species	*B. nitida*	*M. huberi*	*C. villosum*	*P. suaveolens*	N°	%
Cross-validation	*B. nitida*	100				100	100
	M. huberi		100			100	100
	C. villosum			100		100	100
	P. suaveolens				100	100	100
Test set	*B. nitida*	40				40	100
	M. huberi		40			40	100
	C. villosum			40		40	100
	P. suaveolens				40	40	100

Table 4: PLS-R parameters by cross-validation and independent to estimate the basic density of the woods.

Face	Model	Treatments	LV	Cross-validation (n = 200)			Test set validation (n = 80)		
				R^2cv	RMSEcv	RPD	R^2p	RMSEp	RPD
Transversal	1	Untreated	10	0.93	0.01	3.80	0.93	0.01	4.20
	2	1d	8	0.93	0.01	3.76	0.93	0.01	3.93
	3	2d	6	0.89	0.01	3.00	0.87	0.01	2.82
Radial	4	Untreated	8	0.91	0.01	3.42	0.89	0.01	3.02
	5	1d	6	0.92	0.01	3.62	0.92	0.01	3.56
	6	2d	6	0.88	0.01	2.90	0.87	0.01	2.81

1d= first derivative; 2d = second derivative; LV = number of latent variables; R^2cv = coefficient of determination of cross-validation; RMSEcv = standard error of cross-validation; R^2p = coefficient of determination of prediction; RMSEp = standard error of prediction; RPD = performance ratio of standard deviation.

When considering the face of spectral acquisition, for the transversal face of the wood, models 1 and 2 presented the same results, even though model 1 did not pass by pre-treatment in the database. However, model 2 has fewer latent variables, being more robust.

Latent variables are not directly observed but are strongly inferred through a mathematical model from other variables observed and measured directly. On the radial face, model 5 exhibited the main coefficients of determination in cross-validation and prediction. Further, it can be seen that both models were satisfactory, and the best model to predict the basic density was developed when treated with the first derivative and measured on the transversal face.

Variations in the model's performance developed on the different faces (transversal and radial) can be explained by morphological differences and arrangement of the xylem elements, which change according to the anatomical planes (Costa *et al*. 2018). Amaral *et al*. (2021), evaluating NIR spectra acquisition on wood faces to predict basic density, found that transversal face was the most appropriate to develop the models, corroborating the results of this study. However, Santos *et al*. (2012) obtained more robust models when the spectra were collected on the radial face.

Liang *et al*. (2019) cited that model treated with the first derivative also showed suitable correlation fits, obtaining values: R^2cv = 0.98 and R^2p = 0.95. The same authors applied the second derivative to the spectral data and obtained R^2cv = 0.98 and R^2p = 0.94, a result superior compared to this study. According to Honorato *et al*. (2007), the first derivative aims to mitigate the systematic displacement of the baseline, while the second derivative minimizes linear variations.

The RPD results were in agreement with the values reported in the literature. According to Williams and Sobering (1993), RPD between 2 and 3 are considered adequate for estimates and RPD between 3 and 5 are classified as satisfactory in forest sciences. Santos *et al*. (2012), adjusted models to estimate the wood basic density of *Acacia melanoxylon* with RPD greater than 2.5, which is consistent with this study. When analyzing the global model dispersion, with basic density values measured in the laboratory and predicted by NIR (Figure 4), suitable correlation fits were obtained. In cross-validation, the coefficient of determination was 0.89, whereas in prediction the value found was 0.78.

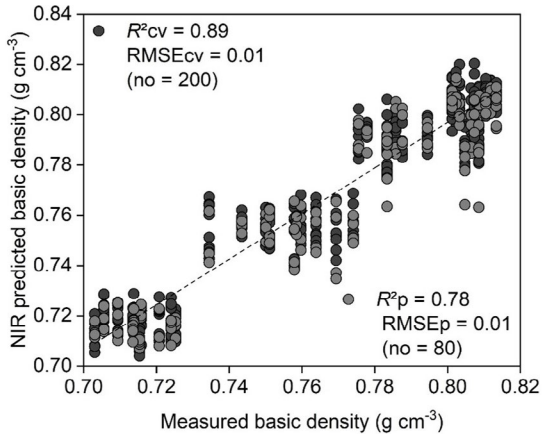

Figure 4: Global model: measured values versus predicted values by NIR for basic density of tropical woods by cross-validation and independent.

The estimated accuracy of verified NIR in the cross-validation and independent is the best way to evaluate the performance of quantitative models (Hein *et al*. 2009). Honorato *et al*. (2007) reported that the global model is indispensable, as it covers all possible variations contained in the spectra, making the model more complex, as it includes the entire spectral matrix. Furthermore, the non-destructive method used in this study showed promising results, justifying the use of NIR spectroscopy to evaluate wood properties. Another great advantage is the possibility of carrying out the analyzes in loco, after calibration in the laboratory, which could allow cost savings and no need to transport heavy materials. This highlights the potential for application in the wood sector, mainly for floor and furniture applications, as it is a rapid, practical, and reliable method that simplifies laboratory analyses. New studies could be carried out to estimate wood moisture for different purposes, such as particleboard production, pulp production, sawn wood, and carbonization.

CONCLUSION

NIR technology, associated with multivariate statistics, could predict the basic density of tropical woods and can be applied to estimate this property in

floor industries and possible applications in furniture and other wood-based products. The first derivative was the mathematical pre-treatment that most improved the model's performance. PC1 explained more than 90 % of the data variation for all models with pre-treatment or original spectrum. The PLS-DA classified the woods by species with 100% accuracy of the data. *Bowdichia nítida* wood presented higher NIR absorbance intensities for this spectral range because of its high density (0.81 g cm^{-3}). The models for predicting wood basic density were more appropriate when the spectra acquisition was measured on the transversal face. Model 2 was considered the most robust, with R^2 of 0.93 in the cross-validation and the independent validation.

AUTHORSHIP CONTRIBUTION

Project Idea: DTM, RRM, PHGC, PGR

Database: DTM, FGB

Processing: DTM, RRM, FGB

Analysis: DTM, FGB, ARPM

Writing: DTM, FGB, ARPM

Review: ARPM, RRM, PGR

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