PREDICTING THE MORPHOLOGICAL CHARACTERISTICS AND BASIC DENSITY OF *Eucalyptus* WOOD USING THE NIRS TECHNIQUE

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ABSTRACT: This research aimed to apply the near infrared spectroscopy technique (NIRS) for fast prediction of basic density and morphological characteristics of wood fibers in *Eucalyptus* clones. Six *Eucalyptus* clones aged three years were used, obtained from plantations in Cocais, Guanhães, Rio Doce and Santa Bárbara, in Minas Gerais state. The morphological characteristics of the fibers and basic density of the wood were determined by conventional methods and correlated with near infrared spectra using partial least square regression (PLS regression). Best calibration correlations were obtained in basic density prediction, with values 0.95 for correlation coefficient of cross validation (Rcv) and 3.4 for ratio performance deviation (RPD), in clone 57. Fiber length can be predicted by models with Rcv ranging from 0.61 to 0.89 and standard error (SECV) ranging from 0.037 to 0.079 mm. The prediction model for wood fiber width presented higher Rcv (0.82) and RPD (1.9) values in clone 1046. Best fits to estimate lumen diameter and fiber wall thickness were obtained with information from clone 1046. In some clones, the NIRS technique proved efficient to predict the anatomical properties and basic density of wood in *Eucalyptus* clones.

Key words: Near infrared spectroscopy, timber, specific gravity, anatomical elements, hardwood.

PREDIÇÃO DAS CARACTERÍSTICAS MORFOLÓGICAS E DA DENSIDADE BÁSICA DA MADEIRA DE *Eucalyptus* PELA TÉCNICA NIRS

RESUMO: *Neste trabalho, objetivou-se aplicar a técnica da espectroscopia no infravermelho próximo (NIRS) para rápida predição da densidade básica e das características morfológicas das fibras da madeira em clones de Eucalyptus. Foram utilizados seis clones de Eucalyptus, com idade de três anos, provenientes de plantios localizados nas regionais de Cocais, Guanhães, Rio Doce e Santa Bárbara, no Estado de Minas Gerais. As características morfológicas das fibras e a densidade básica da madeira foram determinadas por método convencional e correlacionadas com os espectros no infravermelho próximo por meio da regressão dos mínimos quadrados parciais (PLS regression). As melhores correlações nas calibrações foram obtidas para prever a densidade básica da madeira, com valores de coeficientes de correlação na validação cruzada (Rcv) de 0,95 e relação de desempenho do desvio (RPD) de 3,4 para o clone 57. O comprimento das fibras pode ser predito por modelos com Rcv, entre 0,61 e 0,89 e erro padrão (SECV), variando de 0,037 a 0,079 mm. O modelo para predição da largura das fibras da madeira apresentou os maiores valores de Rcv (0,82) e RPD (1,9) para o clone 1046. Os melhores ajustes para estimar o diâmetro do lume e a espessura da parede das fibras foram obtidos com as informações das amostras do clone 1046. Para alguns clones, a técnica NIRS mostrou-se eficiente para predição das propriedades anatômicas e da densidade básica da madeira dos clones de Eucalyptus.*

Palavras-chave: Espectroscopia no infravermelho próximo, lenho, massa específica, elementos anatômicos, folhosas.

1 INTRODUCTION

The ever-increasing market demand for wood to attend to the needs of various forestry-related sectors of the Brazilian industry, along with the declining availability of native forests, have sparked an interest in studying alternative, fast-growing forest species.

In Brazil, genus *Eucalyptus* has been drawing the attention of businesses and research institutions due to the steady expansion of new plantations, as a function of significant gains from forest productivity, to meet the increasing demand for wood both as an energy source and for the pulp and paper industry (SOCIEDADE BRASILEIRA DE SILVICULTURA 2005).

Wood is known to be a heterogeneous material, with varying properties within a single species (SHIMOYAMA 1990) and within a single clone (EVANS et al. 2000, TOMAZELLO FILHO 1985). Knowledge of these

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properties is of crucial importance to recognize and define potential uses for this material.

Traditionally, experiments attempting to evaluate technological characteristics of wood have been accurate but also painstaking, time-consuming, costly and relying on a limited number of samples, thus obstructing suitable characterization of this raw material. It is thus necessary to investigate the possibility of developing new tools to enable simpler measurement methods.

With the above considerations in mind, the use of nondestructive techniques for suitable characterization of wood properties should be investigated. These measurement techniques should be easy and quick to determine, allowing a large number of samples to be analyzed so as to provide representative results.

Near infrared spectroscopy (NIRS) has been used as a nondestructive method to characterize wood. According to Pasquini (2003), NIRS is a vibrational type of spectroscopy that uses the near infrared region of the electromagnetic spectrum (from about 750 nm to 2,500 nm). It enables qualitative and quantitative information on biomass constituents by allowing near infrared electromagnetic waves to interact with the sample.

The NIRS method involves acquisition of an absorbance/reflectance spectrum which is generated when near infrared radiation penetrates the sample, using a NIR spectrometer. With multivariate analysis, the spectrum is correlated with obtained values using conventional analysis techniques (SEFARA et al. 2000).

The NIRS technique offers advantages in relation to traditional methods in that it is fast (one minute or less per sample) and non-invasive, suitable for use in the production line, can be applied to any molecule containing C-H, O-H, N-H, S-H and C=O bonds and requires minimal sample preparation (PASQUINI 2003).

In the forestry sector, early works using near infrared spectroscopy and multivariate analysis techniques focused on predicting wood chemical composition and paper properties. Studies have yet demonstrated the success of this method for determining the anatomical, physical, and mechanical properties of wood.

Schultz & Burns (1990) used near infrared spectroscopy to determine concentrations of lignin, cellulose and extractives in wood. Studies conducted by Easty et al. (1990) and Garbutt et al. (1992) found that wood anatomical properties can be estimated using near infrared spectroscopy. Hoffmeyer & Pedersen (1995), Schimleck et al. (1999) and Thygesen (1994) found that NIR can be used to predict wood density. Schimleck et al. (2002) and Thumm & Meder (2001), working with *Pinus radiata*, concluded that NIRS can be used to predict the mechanical properties of wood in this species. In a recent study with *Eucalyptus urophylla*, Hein (2008) found that the chemical properties of its wood can be estimated by the NIRS technique, based on measured spectra on solid wood and on disintegrated wood.

Near infrared spectroscopy has been found to be an accurate analytical tool for application in several areas. However, to ensure successful application of this technique, it is necessary that the relevant sample should represent the entire range of variability existing in a population, and that the reference measurements should be taken correctly and accurately.

Therefore, the objective of this work is to apply the NIRS technique to predict the basic density and morphological characteristics of wood fibers in *Eucalyptus* clones.

2 MATERIAL AND METHODS

2.1 Vegetal material

Six *Eucalyptus* clones (Table 1) aged three years were used in this experiment, as obtained from commercial plantations, with spacings of 3.0 m x 3.3 m, in the municipalities of Cocais, Guanhães, Rio Doce and Santa Bárbara, in Minas Gerais state.

In each experimental location and clone, a plot was demarcated containing 10 x 10 plants. In the plot, all tree diameters were measured (DBH), then the mean diameter and standard deviation of the mean were determined, so that trees falling within the mean DBH value plus or minus the standard deviation were selected, to a total of five trees per clone per location. With the experiment comprising four locations and six clones, 24 plots were measured in total.

2.2 Basic density

To determine basic density, the tree trunks were sectioned into logs of 1m in length at 0%, 25%, 50%, 75% and 100% of the commercial height of the trunk – minimum of 7 cm in diameter. The logs were then cut into chips, and the chips formed a mixture so as to include all longitudinal sampling points along the trunks. The compound sample was then used for determination of average basic density (ABD).

2.3 Determination of fiber and vessel measurements

The method used for analysis and determination of fiber measurements was the hydrogen peroxide method

Table 1 – *Eucalyptus* clones.

***Tabela 1 –** Clones de Eucalyptus.*

Genetic material	Species
Clone 57	natural hybrid of *Eucalyptus grandis*
Clone 1046	hybrid of *E. grandis X E.urophilla*
Clone 1213	hybrid of *E. grandis X E.urophilla*
Clone 1215	hybrid of *E. grandis X E.urophilla*
Clone 1274	hybrid of *E. grandis X E.urophilla*
Clone 7074	*Eucalyptus grandis*

(H_2O_2), as described by Ramalho (1987), using a 1:1 ratio maceration solution of hydrogen peroxide 30% and glacial acetic acid. Sample slides were stained with astra blue. Measurements were taken of 45 fibers per tree, using an Olympus BX 41 optical microscope coupled to a WinCELL image analysis system.

2.4 Spectral acquisition

Samples for the near infrared analysis were collected from tree discs in the trunk portion 1.30 m above the ground (Figure 1) and stored in an environmental chamber. Spectral acquisition was conducted in the 750 to 2,500 nm range, with a spectral resolution of 2nm in diffuse reflectance mode, using a Bruker spectrometer and OPUS version 4.2 software. Diffuse reflectance readings were taken on sample specimens measuring 0.8 cm x 2.5 cm x 6.0 cm, directly onto the crosswise surface pre-polished with 120 sandpaper. An average 32 scans were acquired per spectrum.

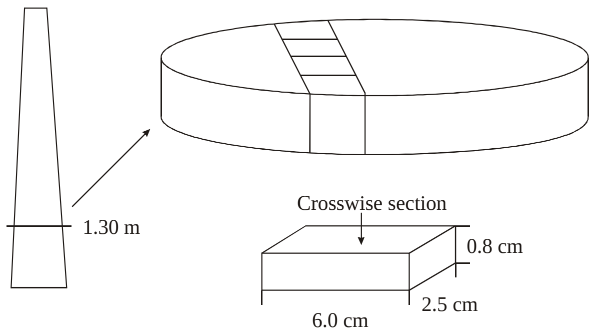

Figure 1 – Sample position in relation to tree trunk.

***Figura 1 –** Esquema da posição das amostras no fuste da árvore.*

2.5 Calibration, validation and selection of models

A partial least square regression (PLS regression) was used to establish a relationship between spectral information and properties being investigated, using The Unscrambler® 9.1 version software. Models were fitted with a maximum of twelve factors. The number of factors adopted for each model was the value that minimized the residual variance of the calibration and cross validation. Calibrations were performed based on original spectra and mathematically treated spectra using the Savitzky-Golay first derivative method (SAVITZKY & GOLAY 1964). Samples considered to be *outliers* in the *Student* residual plot were not included in the models. A full cross validation was adopted to validate the calibration models. Criteria to select the prediction models included: correlation coefficient of cross validation (Rcv), standard error of cross validation (SECV), number of PLS factors used in the calibration, and ratio performance deviation (RPD).

3 RESULTS AND DISCUSSION

Mean values of average basic density and anatomical characteristics are illustrated in Table 2. The results agree with data in existing literature relating to eucalyptus wood.

According to Schimleck et al. (2003), calibrations with ratio performance deviation (RPD) values of 1.5 or above suffice for initial readings. Near infrared spectroscopy can thus be used for initial predictions intended for selection of young trees with potential for selection.

NIRS calibrations for estimation of wood basic density in six *Eucalyptus* clones are illustrated in Table 3.

The values of correlation coefficient of cross validation (Rcv) for basic density ranged from 0.22 (clone

Table 2 – Average values of basic density and anatomical characteristics of wood fibers.

Tabela 2 *– Valores médios da densidade básica e dos caracteres anatômicos das fibras.*

Clones		BD (g/cm^3)	FL (mm)	FW (µm)	LD (µm)	WT (µm)
57	Mean	0.43	1.00	15.58	8.49	3.56
	CV (%)	2.84	9.36	7.95	12.49	7.93
	N	20	20	20	20	20
1046	Mean	0.43	1.04	16.18	8.51	3.86
	CV (%)	2.02	7.92	10.35	15.32	8.53
	N	20	20	20	20	20
1213	Mean	0.46	1.04	16.29	8.91	3.7
	CV (%)	3.05	8.29	9.2	12.16	8.89
	N	20	20	20	20	20
1215	Mean	0.45	1.01	15.86	8.82	3.54
	CV (%)	2.78	6.28	11.07	15.86	8.65
	N	20	20	20	20	20
1274	Mean	0.45	1.02	15.77	8.48	3.66
	CV (%)	4.07	9.05	9.98	16.21	11.81
	N	20	20	20	20	20
7074	Mean	0.37	0.96	16.05	9.5	3.29
	CV (%)	5.53	10.31	11.18	16.94	11.45
	N	20	20	20	20	20

BD: basic density, FL: fiber length, FW: fiber width, LD: lumen diameter, WT: wall thickness, CV: coefficient of variation, N: number of samples.

BD: densidade básica, FL: comprimento da fibra, FW: largura da fibra, LD: diâmetro do lume, WT: espessura da parede, CV: coeficiente de variação, N: número de amostras.

Table 3 – NIRS calibrations of wood basic density.

Tabela 3 *– Calibrações NIRS para a densidade básica da madeira.*

Clone	Filter	Rc	Rcv	Factors	SEC	SECV	Outlier	N	RPD
57	original	0.99	0.95	8	0.001	0.004	2	20	3.4
1046	original	0.72	0.58	2	0.006	0.008	2	20	1.2
1213	original	0.99	0.78	9	0.000	0.007	2	20	2.1
1215	2nd derivative	0.99	0.93	5	0.002	0.004	0	20	2.9
1274	original	0.46	0.22	1	0.015	0.017	1	20	1.0
7074	original	0.67	0.60	1	0.015	0.017	1	20	1.2

Rc: correlation coefficient of calibration, Rcv: cross validation correlation coefficient, SEC: standard error of calibration (g/cm³), SECV: standard error of cross validation (g/cm³), N: number of samples used in the calibration, RPD: ratio performance deviation.

Rc: coeficiente de correlação da calibração, Rcv: coeficiente de correlação da validação cruzada, SEC: erro padrão da calibração (g/cm³), SECV: erro padrão da validação cruzada (g/cm³), N: número de amostras utilizadas na calibração, RPD: relação de desempenho do desvio.

1274) to 0.95 (clone 57). Clones 57, 1213 and 1215 had the highest Rcv and RPD values, with special mention to clone 57 (Figure 2) due to its low SEC (0.001 g/cm³) and SECV (0.004 g/cm³) values and high RPD (3.4) value. To calibrate this model eight PLS factors were used and two outlier samples were eliminated.

Meder et al. (1999), using four PLS factors to estimate wood basic density in *Pinus radiata*, obtained models with coefficients of correlation and calibration (R) whose values were 0.93 and 0.77 respectively, RMSEC value of 21.6 kg/m³ and RMSEP value of 36.9 kg/m³.

Schimleck et al. (1999) used PLS regression to estimate wood basic density in trees of *Eucalyptus globulus* aged eight years, and obtained models with correlation and calibration coefficients (R) from 0.62 to 0.80. The standard error of calibration coefficients (SEC) and standard error of validation (SEV) values ranged from 27 to 33 Kg/m³.

Gindl et al. (2001) studied the physical properties of *Larix decidua* Mill. trees and obtained a calibration model for basic density with a correlation coefficient (R) value of 0.986 and an RMSEC value of 0.018 g/cm³. Also, in the cross validation the authors obtained a correlation coefficient of 0.975 and an RMSECV value of 0.023 g/cm³.

Tables 4 and 5 provide a summary of PLS calibrations to predict fiber length and width in *Eucalyptus* wood.

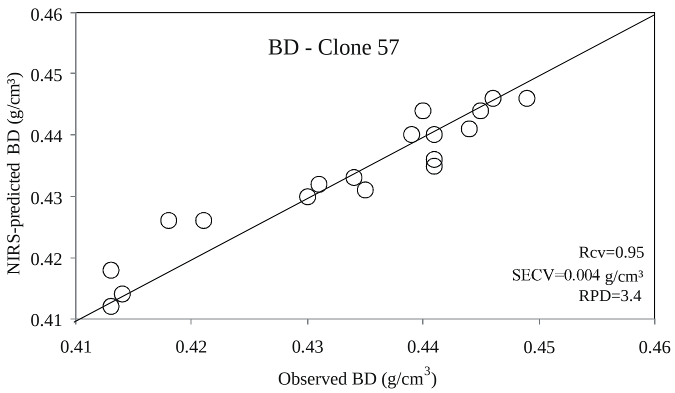

Figure 2 – Correlation between predicted and observed values for average basic density of wood in *Eucalyptus* clones.

Figura 2 – *Correlação entre os valores preditos e medidos para a densidade básica média da madeira de clones de Eucalyptus.*

Table 4 – NIRS calibration of fiber length.

Tabela 4 – *Calibração NIRS para comprimento da fibra.*

Clone	Filter	Rc	Rcv	Factors	SEC	SECV	Outlier	N	RPD
57	original	0.98	0.67	8	0.015	0.079	1	20	1.2
1046	original	0.79	0.66	3	0.049	0.062	0	20	1.3
1213	original	0.83	0.61	5	0.044	0.070	1	20	1.2
1215	2nd derivative	0.84	0.72	2	0.028	0.037	2	20	1.8
1274	1st derivative	0.96	0.83	5	0.024	0.051	0	20	1.8
7074	1st derivative	0.94	0.89	3	0.032	0.044	1	20	2.3

Rc: correlation coefficient of calibration, Rcv: cross validation correlation coefficient, SEC: standard error of calibration (mm), SECV: standard error of cross validation (mm), N: number of samples used in calibration, RPD: ratio performance deviation.

Rc: coeficiente de correlação da calibração, Rcv: coeficiente de correlação da validação cruzada, SEC: erro padrão da calibração (mm), SECV: erro padrão da validação cruzada (mm), N: número de amostras utilizadas na calibração, RPD: relação de desempenho do desvio.

The calibration of fiber length generated models with cross validation correlation coefficient (Rcv) ranging from 0.61 to 0.89, and standard error (SECV) ranging from 0.037 to 0.079 mm. The generated model for clone 7074, using three PLS factors and first derivative treatment, provided the values: 0.94 and 0.89 for correlation coefficients of calibration and cross validation (Rc and Rcv) respectively, 0.044 mm for standard error of cross validation (SECV) and 2.3 for ratio performance deviation (RPD).

As for fiber width, clone 1046 provided the best prediction model, using five PLS factors, with the highest correlation coefficients of calibration (0.93) and cross validation (0.82), and also the highest RPD value (1.9).

Rcv values in this work were higher, and SECV values, smaller, than those found by Magalhães et al. (2005) for calibration of fiber characteristics as described in Tables 4 and 5. These authors fitted models to determine wood fiber measurements in species *Pinus*. To estimate fiber width, calibrations were performed on 20 samples and provided a correlation coefficient (R) of 0.65 and a standard error of cross validation (SECV) of 3.870 µm. To estimate fiber length, the calibration models provided a correlation coefficient (R) of 0.63 and a SECV of 0.290 mm.

Table 6 illustrates calibration data to predict fiber lumen diameter in six *Eucalyptus* clones.

Table 5 – NIRS calibration of fiber width.

Tabela 5 – *Calibração NIRS para largura da fibra.*

Clone	Filter	Rc	Rcv	Factors	SEC	SECV	Outlier	N	RPD
57	original	0.92	0.63	7	0.399	0.895	2	20	1.4
1046	original	0.93	0.82	5	0.547	0.868	2	20	1.9
1213	original	0.79	0.47	5	0.904	1.562	0	20	1.0
1215	1st derivative	0.76	0.61	2	1.079	1.372	1	20	1.3
1274	1st derivative	0.88	0.78	3	0.675	0.895	3	20	1.8
7074	original	0.84	0.72	4	0.970	1.243	0	20	1.4

Rc: correlation coefficient of calibration, Rcv: cross validation correlation coefficient, SEC: standard error of calibration (µm), SECV: standard error of cross validation (µm), N: number of samples used in calibration, RPD: ratio performance deviation.

Rc: coeficiente de correlação da calibração, Rcv: coeficiente de correlação da validação cruzada, SEC: erro padrão da calibração (µm), SECV: erro padrão da validação cruzada (µm), N: número de amostras utilizadas na calibração, RPD: relação de desempenho do desvio.

Table 6 – NIRS calibration of lumen diameter.

Tabela 6 – *Calibração NIRS para diâmetro do lume.*

Clone	Filter	Rc	Rcv	Factors	SEC	SECV	Outlier	N	RPD
57	original	0.58	0.12	5	0.856	1.392	0	20	0.8
1046	original	0.90	0.84	3	0.569	0.726	2	20	1.8
1213	original	0.76	0.35	5	0.698	1.320	0	20	0.8
1215	1st derivative	0.84	0.56	2	0.699	1.092	1	20	1.3
1274	original	0.90	0.69	6	0.432	0.928	3	20	1.5
7074	original	0.71	0.46	4	1.120	1.518	0	20	1.1

Rc: correlation coefficient of calibration, Rcv: cross validation correlation coefficient, SEC: standard error of calibration (µm), SECV: standard error of cross validation (µm), N: number of samples used in calibration, RPD: ratio performance deviation.

Rc: coeficiente de correlação da calibração, Rcv: coeficiente de correlação da validação cruzada, SEC: erro padrão da calibração (µm), SECV: erro padrão da validação cruzada (µm), N: número de amostras utilizadas na calibração, RPD: relação de desempenho do desvio.

As with the calibration to predict fiber width, clone 1046 provided the best results to estimate fiber lumen diameter. To obtain such values, a model with three PLS factors was used and two outlier samples were discarded. Clone 1046 provided Rc and Rcv values of 0.90 and 0.84 respectively, an RPD of 1.8, and lowest standard error of cross validation (SECV), 0.726 µm.

Schimleck & Evans (2004), fitting models to predict radial diameter of tracheids in *Pinus radiata*, found correlation coefficients (R) values of 0.55 and 0.53, standard error of prediction (SEP) of 2.7 and 1.9 µm, and ratio performance deviation (RPD) of 1.0 and 1.1. In the same work, they developed models to predict tangential diameter of tracheids, with R values of 0.83 and 0.89, SEP value of 1.2 and 1.7 µm, and RPD value of 1.5 and 1.1.

Table 7 provides a summary of PLS calibrations to predict fiber wall thickness.

Table 7 provides highly ranging Rcv values – the lowest being 0.10 for clone 57, the highest being 0.86, for clone 1046 –, when using PLS regression to predict fiber wall thickness in the *Eucalyptus* clones. Again clone 1046 provided the best results, with the lowest SEC and SECV values (0.072 and 0.175 µm) and highest RPD (1.9).

These values are below those found by Schimleck & Evans (2004), who found correlation coefficients of 0.94 and 0.95 for tracheid wall thickness, SEP value of 0.2 and 0.5 µm, and RPD value of 1.4 and 2.7, when evaluating the anatomical characteristics of *Pinus radiata.*

According to Magalhães et al. (2005), fiber wall thickness in *Pinus* can be estimated by a calibration with R equal to 0.91 and standard deviation of cross validation equal to 0.34 µm.

Figure 3 illustrates correlations between the observed values and values predicted by the most suitable models to predict the morphological characteristics of woods.

Table 7 – NIRS calibration of fiber wall thickness.

Tabela 7 *– Calibração NIRS para espessura da parede da fibra.*

Clone	Filter	Rc	Rcv	Factors	SEC	SECV	Outlier	N	RPD
57	original	0.43	0.10	1	0.255	0.297	0	20	1.0
1046	original	0.97	0.86	7	0.072	0.175	2	20	1.9
1213	original	0.73	0.32	5	0.225	0.350	0	20	0.9
1215	original	0.79	0.67	3	0.158	0.193	2	20	1.6
1274	original	0.85	0.78	3	0.189	0.225	1	20	1.9
7074	original	0.84	0.63	5	0.190	0.287	2	20	1.3

Rc: correlation coefficient of calibration, Rcv: cross validation correlation coefficient, SEC: standard error of calibration (µm), SECV: standard error of cross validation, N: number of samples used in calibration (µm), RPD: ratio performance deviation.

Rc: coeficiente de correlação da calibração, Rcv: coeficiente de correlação da validação cruzada, SEC: erro padrão da calibração (µm), SECV: erro padrão da validação cruzada (µm), N: número de amostras utilizadas na calibração, RPD: relação de desempenho do desvio.

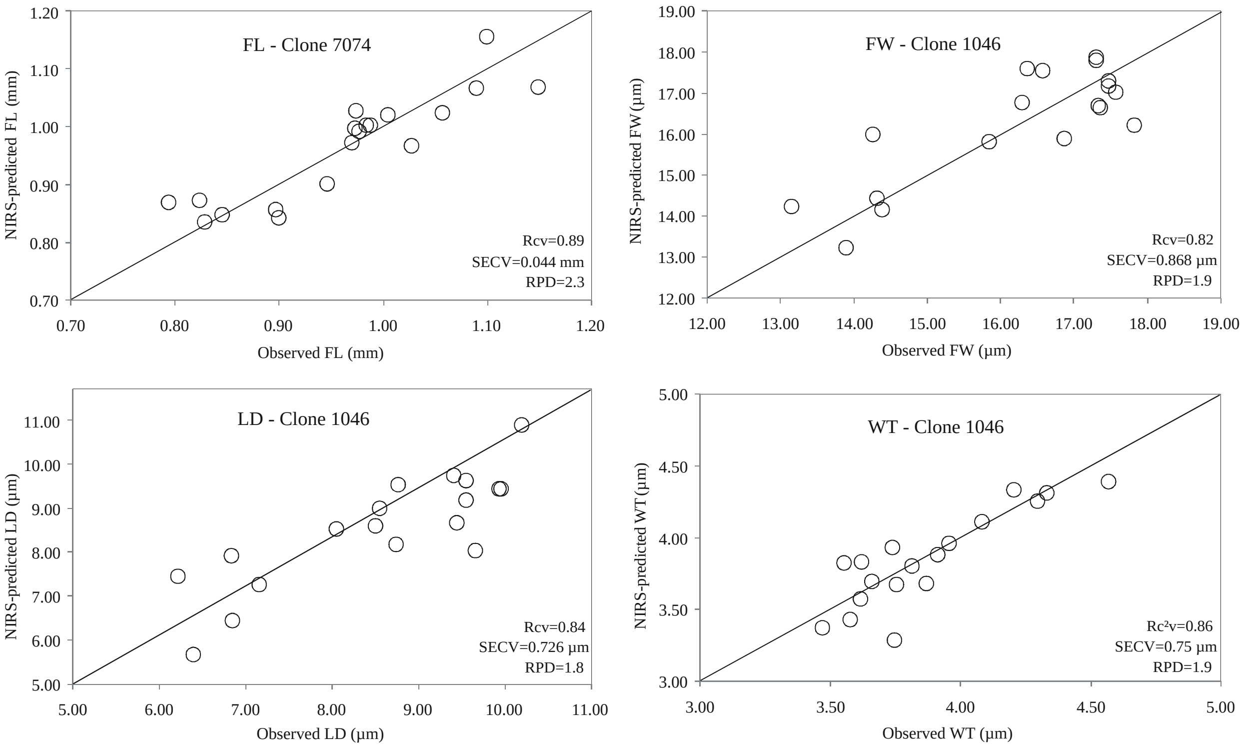

Figure 3 – Correlation between predicted and observed values for morphological characteristics of wood in *Eucalyptus* clones.

Figura 3 – *Correlação entre os valores preditos e medidos para as características morfológicas da madeira de clones de Eucalyptus.*

4 CONCLUSIONS

Based on the above results, it can be said that:

- Overall, the NIRS technique proved efficient for prediction of wood basic density in *Eucalyptus* clones, with best cross validation correlations being found for this property.

- The morphological properties of wood fibers in some *Eucalyptus* clones can be estimated by using NIR spectroscopy.

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