ESTIMATION OF THE MECHANICAL PROPERTIES OF WOOD FROM
Eucalyptus urophylla USING NEAR INFRARED SPECTROSCOPY

Carlos Rogério Andrade[1], Paulo Fernando Trugilho[2], Alfredo Napoli[3],
Renato da Silva Vieira[4], José Tarcísio Lima[2], Leonardo Chagas de Sousa[5]

ABSTRACT: Mechanical properties studies of wood usually involve destructive wood assessments, with time-consuming tests that use large amounts of resource (wood). Although this is not a limiting factor, it could be attenuated by the use of a nondestructive technique known as near infrared spectroscopy (NIRS). This technique has been applied to evaluate compounds containing C-H, N-H, S-H or O-H bonds, and involves quick analyses and can be applied to process control tasks. The objective of this work is to use the NIRS technique to obtain calibrations for mechanical properties of *Eucalyptus* sp. wood. A natural *E. urophylla* hybrid at age 7 was used as obtained from V&M Florestal crops. Spectra were measured directly in solid wood (radial, tangential and transverse faces) and in ground wood, in diffuse reflectance mode, using a Bruker spectrometer in the 800 to 1,500 nm range. The NIRS technique proved suitable to estimate modulus of elasticity in solid wood, with values r=0.91 and RPD=2.6, and in ground wood, with values r=0.87 and RPD=2.0. Modulus of rupture and compressive strength presented r values below 0.9. First and second derivative pretreatments provided a slight increase in correlation values for the properties in question. Calibrations for different plank faces did not present a defined variation pattern. Solid wood and ground wood presented similar correlation values for all properties.

Key words: NIRS, Eucalyptus, characteristics.

ESTIMATIVA DE PROPRIEDADES MECÂNICAS DA MADEIRA DE *Eucalyptus*
urophylla USANDO A ESPECTROSCOPIA NO INFRAVERMELHO PRÓXIMO

RESUMO: O estudo das propriedades mecânicas da madeira comumente é realizado por meio de uma avaliação destrutiva na madeira demandando um extenso tempo para realização dos testes e com grande uso de recursos (madeira). Esse fator, embora não seja limitante para a realização dos ensaios poderia ser amenizado com o uso de uma técnica não-destrutiva como a espectroscopia no infravermelho próximo (NIRS). Essa técnica vem sendo aplicada na avaliação de compostos que contenham ligações C-H, N-H, S-H ou O-H, apresenta rapidez nas análises e pode ser aplicada no controle de processos. O objetivo, neste trabalho, foi utilizar a técnica NIRS para obter calibrações para as propriedades mecânicas da madeira de Eucalyptus sp. Foi utilizado um híbrido natural de E. urophylla com 7 anos, da empresa V&M Florestal. Os espectros foram medidos diretamente sobre madeira sólida (faces radial, tangencial e transversal) e moída, em modo de reflexão difusa, utilizando-se um espectrômetro Bruker na gama de 800 a 1500 nm. A técnica NIRS se mostrou adequada para estimar o módulo de elasticidade obtido na madeira sólida com r = 0,91 e RPD = 2,6 e moída com r = 0,87 e RPD=2,0. O módulo de ruptura e a resistência a compressão apresentaram r abaixo de 0,9. Os pré-tratamentos de primeira e segunda derivada proporcionaram um ligeiro aumento nos valores das correlações para as propriedades analisadas. Calibrações para as diferentes faces da madeira não apresentaram padrão de variação definido. Madeira sólida e moída apresentaram valores de correlação semelhantes para todas as propriedades.

Palavras-chave: NIRS, eucalipto, características.

1 INTRODUCTION

Properties relating to wood resistance include bend strength, also known as modulus of rupture, compressive strength, tensile strength, shear strength, stiffness, among others. And properties relating to wood elasticity include modulus of elasticity, Poisson coefficient, plasticity and resilience (KOLLMANN & CÔTÉ JUNIOR 1968).

Strength in compression parallel to grain is the maximum load in the direction of the length a sample is

capable of sustaining (KOLLMANN & COTÊ JÚNIOR 1968).

Modulus of rupture reflects a sample's ability to resist deformation when it is subjected to bending and is equivalent to the maximum moment shown by the sample (GREEN et al. 1999). Klock (2000) reports that high MOE values indicate a material's high strength and low deformation characteristics, which means it will qualify for structural works.

Typically, mechanical characterization of wood is done by destructive analysis of samples, using specific equipment that involves extensive, standard-based testing routines and capable of providing reliable results. However, this type of assessment requires a long lead time between preparing samples and arriving at final values for the relevant property.

Many researches have been conducted into alternative methods for fast and reliable wood characterization that, along with traditional methods, can result both in analysis quality and time gains. Among recently developed nondestructive methods for wood characterization is the near infrared spectroscopy technique (NIRS). According to Baillères et al. (2002), near infrared spectroscopy is a noninvasive analysis technique for organic materials, largely used by various manufacturing companies, forest sector included, in quality control and process monitoring tasks.

The above method can offer advantages in relation to traditional methods, as it is considered nondestructive and fast (one minute or less per sample) (PASQUINI 2003).

The basic principle is measuring absorbance spectra in the near infrared region of relevant samples, which then have their properties determined by traditional destructive analysis in a laboratory. NIR spectra are then subjected to regression analysis by the partial least square method (PLS), which is the most common method in calibration for model construction, using spectral signals (GELADI & KOWALSKI 1986). Once the model has been calibrated for a given property, calibration should be tested for accuracy by a process known as validation. Model validation can be accomplished in two ways, namely external validation and cross validation (MARTENS & NAES 1996).

External validation is considered simpler, yet it has the disadvantage of requiring another sample set, implying use of spectral measurement and conventional analysis and thus increasing time on task and procedure costs (SOTELO 2006). In cross validation, samples awaiting validation are the same samples used in the calibration test. Some samples are set aside, a model is constructed with the remaining samples and prediction is done in relation to samples initially set aside. This type of validation is recommended where a small number of samples is involved.

Several works have demonstrated the use potential of near infrared spectroscopy to determine wood properties, as, for example, studies developed by Fujimoto et al. (2008) and Schimleck et al. (1999) into density and also into strength and stiffness properties. Viana et al. (2009), by using NIRS technique for predicting the morfological characteristics and basic density, found interesting results. Hein (2010) found similar results for basic density in *Eucalyptus* hybrids. Studies were also conducted into mechanical strength (THUMM & MEDER 2001). While studying *E. grandis* and *E. urophylla*, Hein et al. (2009) evaluated estimation of strength and elasticity in compression parallel to grain and found coefficients of determination of cross validation of 0.78 and 0.75 respectively. Ribeiro (2009) used the same technique to predict physical and mechanical properties of softwoods and hardwoods and found it to be a very effective technique, economically and practically speaking. Kelley et al. (2004) demonstrated the use potential of the NIRS technique to reliably predict mechanical properties of wood.

Being a noninvasive technique, the use of NIRS to investigate mechanical properties of wood is extremely promising, potentially translating as considerable gains in time and resources (wood).

With that said, Pasquini (2003) reports that where measurements are taken in diffuse reflectance mode, the NIRS technique can pose some problems, e.g. light dispersion and inaccuracies, due to differences in sample thickness. Therefore, using techniques that will help reduce, eliminate or standardize the effect of unwanted factors on spectra and yet maintain the spectroscopic information unchanged (SIESLER et al. 2002) is extremely important. Examples of such techniques include first and second derivative and multiplicative signal correction methods.

This research used the NIRS technique to estimate mechanical properties of wood from a *Eucalyptus urophylla* hybrid, as determined by static bending and compression parallel to grain testing.

2 MATERIAL AND METHODS

2.1 Biological material and sampling

An *Eucalyptus urophylla* hybrid aged 7 years was obtained from a commercial crop grown by V&M Florestal,

a company located in the municipality of Paraopeba, Minas Gerais state, Brazil.

Sampling was done using a single tree divided into lengthwise and widthwise sections. The stem was divided into 5 logs, each log being 5 meters long. Each log section was divided into planks, each plank being 150 cm. This material was processed using a band saw and defect-free specimens were taken for static bending tests (25 x 25 x 410 mm in size) and compression parallel to grain tests (25 x 25 x 100 mm in size) from the outer, central and inner portions of the plank, with well-oriented radial and tangential faces. Mechanical tests were performed in a universal testing machine EMIC DL 30000. Tests were run according to the ASTM D 143 - 94 standard (1997).

2.2 NIR spectral acquisition

For spectral acquisition, a Bruker MPA-R spectrometer was used, equipped with software Opus version 5.5. This equipment uses the Fourier transform and has an integrating sphere. Spectral acquisition was conducted in the 800 to 1,500 nm range, in diffuse reflectance mode, with spectral resolution of 8 cm^{-1} and 64 scans per reading. Readings were taken of solid wood spectra (on radial, tangential and transverse faces), then samples were ground and spectral readings were taken in ground wood retained in a 60-mesh sieve. The procedure of spectral acquisition is illustrated in Figure 1.

2.3 Parameters of calibration, validation and model selection

Software The Unscrambler® version 9.1. was used as a calibration tool. Models were fitted using the partial least square regression method (PLS-1), with a maximum of 10 latent variables, and the value adopted for each model was suggested by the statistical program. The validation method used in this work was cross validation. To eliminate some of the noise and improve signal quality, first and second derivative pretreatments were applied to spectral information, using the algorithm proposed by Savitzky & Golay (1964). Second order polynomials were used with a spectral window of seven variables. A third pretreatment–multiplicative signal correction (MSC)–was also adopted to minimize the chance of any interference in solid wood spectral data. Outliers were detected by using the plot of Student's residuals as a parameter and were not included in model calibration. Model selection was based on the following criteria: (1st) higher correlation coefficient of cross validation (r); (2nd) higher ratio of deviation performance, (3rd) lower number of latent variables used in calibration and (4th) standard error of cross validation SECV.

3 RESULTS AND DISCUSSION

3.1 Mechanical properties of wood

Table 1 summarizes results of compression parallel to grain and static bending tests.
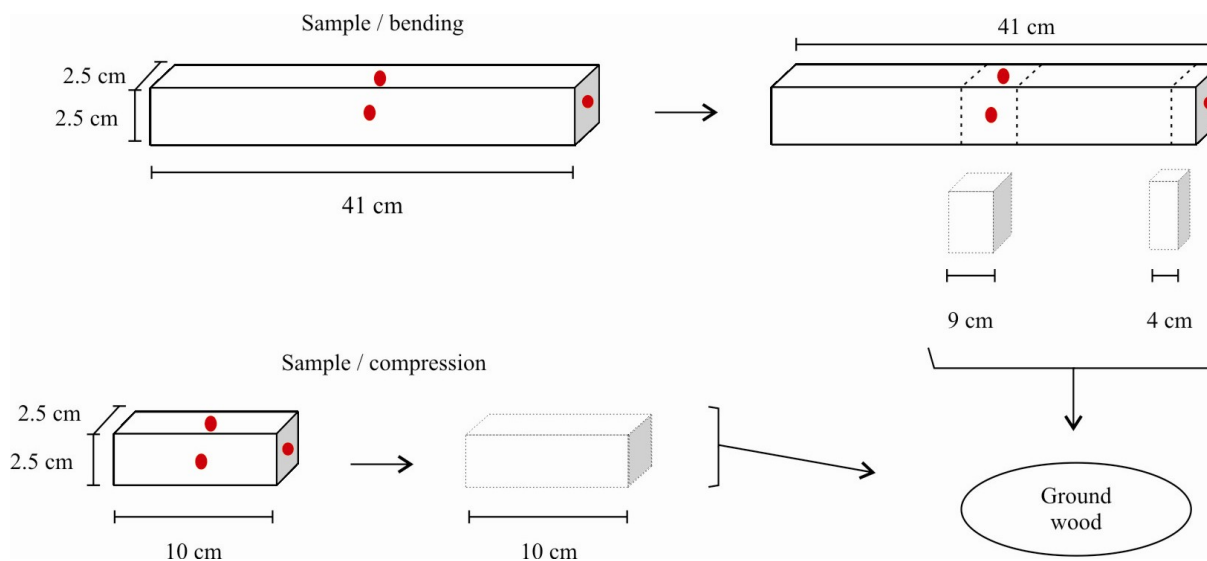

Figure 1 – Points of spectral acquisition in solid wood and portions used for ground wood readings.

Figura 1 – Pontos de aquisição espectral na madeira sólida e regiões utilizadas para a obtenção da madeira moída.

Table 1 – Results of compression parallel to grain and static bending tests.

Tabela 1 – Ensaio de compressão paralela às fibras e flexão estática.

Test type	Prop.	Mean (MPa)	Sd	Min (MPa)	Max (MPa)	n
Compression parallel to grain	MOE	7934.0	1466.7	5388.3	10794.3	25
	CS	52.1	6.9	40.1	63.6	25
Static bending	MOE	15972.2	2751.1	11054.4	20178.1	19
	MOR	94.4	15.4	69.9	115.8	19

MOE – Modulus of elasticity; CS – Strength in compression parallel to grain; MOR – Modulus of rupture; Prop.- Property; Sd – Standard deviation; Min – Minimum; Max – Maximum; n – Number of samples.

Values shown in Table 1 for modulus of elasticity, strength in compression parallel to grain and modulus of rupture are similar to results found by Hein et al. (2009), who obtained 7848 MPa for modulus of elasticity in compression parallel to grain testing while investigating *Eucalyptus grandis* and *E. urophylla* at age 6.5 years. Silva et al. (2005) worked with *Eucalyptus grandis* at different ages and obtained 129230 kgf/cm^2 (equivalent to 12673 MPa) as means of modulus of elasticity in static bending testing. Cruz et al. (2003) assessed *Eucalyptus* clones at age 5.5 to 10.5 years and obtained 15044 MPa as mean of modulus of elasticity in static bending testing, and 7657 MPa as mean of modulus of elasticity in compression parallel to grain testing.

3.2 Calibration of mechanical properties of wood

Cell exposure in wood will differ depending on the face being assessed–radial (Rd), tangential (Tg) or transverse (Tr). It is thus expected, for instance, that the greater exposure of parenchyma cells on the radial face will produce different spectral information, and this information in turn can correlate positively or negatively with mechanical properties of wood (HEIN et al. 2009). Based on this information, solid wood calibrations included all 3 faces–radial (Rd), tangential (Tg) and transverse (Tr)–, then an average spectral value of all three faces was worked out (mean), followed by new calibrations.

Table 2 summarizes best calibration results for mechanical properties of wood based on spectral information read on the radial, tangential and transverse faces of solid wood, in addition to spectral means.

Results of cross validation correlation are similar to those found by Hein et al. (2009), who worked with *Eucalyptus grandis* and *E. urophylla* clones at age 6.5

years and reported a coefficient of determination (R^2) of 0.75 in cross validation for modulus of elasticity (MOE) in compression parallel testing.

For strength in compression parallel (CS), the model presented a R^2_p of 0.73 in cross validation. In static bending testing, the best observed values for modulus of elasticity were r=0.91 and RPD=2.5, using tangential face spectra. The best observed values for modulus of rupture was the spectral mean of all three faces, with values r=0.75 and RPD=1.5. So et al. (2002) obtained good calibrations to estimate modulus of elasticity (R^2=0.87) while performing bending tests in wood from *Pinus taeda* L. at ages 15 to 55 years.

As regards use of pretreatments, best correlations were obtained when original spectra were used, except for compressive strength which had its best observed value when multiplicative signal correction was applied. Overall, best correlation values for compression parallel testing were obtained when using transverse face spectra, followed by tangential face and then radial face spectra. As for bending testing, best responses were observed when using tangential face spectra, followed by transverse face and then radial face.

Differences in spectral information depending on the face measured–whether the radial, tangential or transverse section–are yet to be better established (HEIN et al. 2009). Fujimoto et al. (2008) used spectral information from tangential (R^2 between 0.61 and 0.84) and radial (R^2 between 0.73 and 0.89) faces to estimate mechanical properties of two species of genus *Larix* and concluded that both spectra present the same overall tendency as to calibration. It should be noted that in this particular work calibrations for mechanical properties were based on a small number of samples (n), and also that new studies should be undertaken to investigate the influence of

Table 2 – Summary of NIRS calibrations for mechanical properties in solid wood.

Tabela 2 – Resumo das calibrações NIRS para as propriedades mecânicas da madeira sólida.

Wood	Test type	Face	Prop	Treat	r	LV	*Outlier*	SECV	RPD	n
Solid	Comp.	Rd	MOE	2d	0.72	3	0	996.4	1.5	25
			CS	1d	0.74	5	0	4.5	1.5	25
		Tg	MOE	1d	0.74	6	0	962.9	1.5	25
			CS	1d	0.77	6	0	4.3	1.6	25
		Tr	MOE	osd	0.91	3	0	566.2	2.6	25
			CS	osd	0.89	5	0	3.1	2.2	25
		Mean	MOE	osd	0.89	3	0	648.3	2.3	25
			CS	MSC	0.90	10	0	2.8	2.5	25
	Bending	Rd	MOE	1d	0.71	1	0	1879.7	1.5	19
			MOR	osd	0.44	1	0	13.6	1.1	19
		Tg	MOE	osd	0.91	10	0	1099.9	2.5	19
			MOR	MSC	0.74	6	0	10.6	1.5	19
		Tr	MOE	1d	0.77	6	0	1764.1	1.6	19
			MOR	MSC	0.65	10	0	13.0	1.2	19
		Mean	MOE	1d	0.78	6	0	1680.2	1.6	19
			MOR	osd	0.75	6	0	10.4	1.5	19

Prop – Properties, Treat – Treatment, r – Cross validation correlation, LV – Number of latent variables, *Outlier* – Number of discarded samples, SECV – Standard error of cross validation, RPD – Ratio of performance deviation, n – Total number of samples used, Comp. – Compression parallel to grain, Rd –Radial, Tg – Tangential, Tr – Transverse, Mean – Spectral mean of 3 faces, MOE – Modulus of elasticity, CS – Strength in compression parallel to grain, MOR – Modulus of rupture, osd – Original spectral data, 1d – First derivative, 2d – Second derivative, MSC – Multiplicative signal correction.

spectra depending on the face measured, preferably with a larger number of samples and ensuring grain orientation is well defined throughout.

Figure 2 provides a correlation graph between estimated and measured modulus of elasticity in compression parallel to grain testing on the transverse face. There is a strong association between observed and predicted values, indicating the use potential of NIRS to estimate modulus of elasticity from solid wood.

Table 3 summarizes best calibration results for mechanical properties using ground wood spectra.

It was noted that modulus of elasticity in compression parallel testing presents the best value of cross validation correlation, with r=0.87 and RPD=2. Modulus of elasticity also presents the same number of latent variables and *Outlier* in both tests, compression and bending, LV=3 and *Outlier*=1.

First and second derivative pretreatments provided the best correlation values overall. According to Schimleck et al. (2003), a RPD value higher than 1.5 is considered satisfactory for preliminary readings and predictions (*screenings*), and for tree selection in forest improvement programs.

According to Tables 2 and 3, it can be said that calibrations obtained from solid wood spectra were slightly higher than calibrations obtained from ground wood spectra, considering both tests and each of the three properties being tested–MOE, CS and MOR.

Cross validation correlations for mechanical properties using solid wood spectra and ground wood spectra overall presented positive values. However, these values can be improved if in future works a larger number of samples is included in the calibration lot.
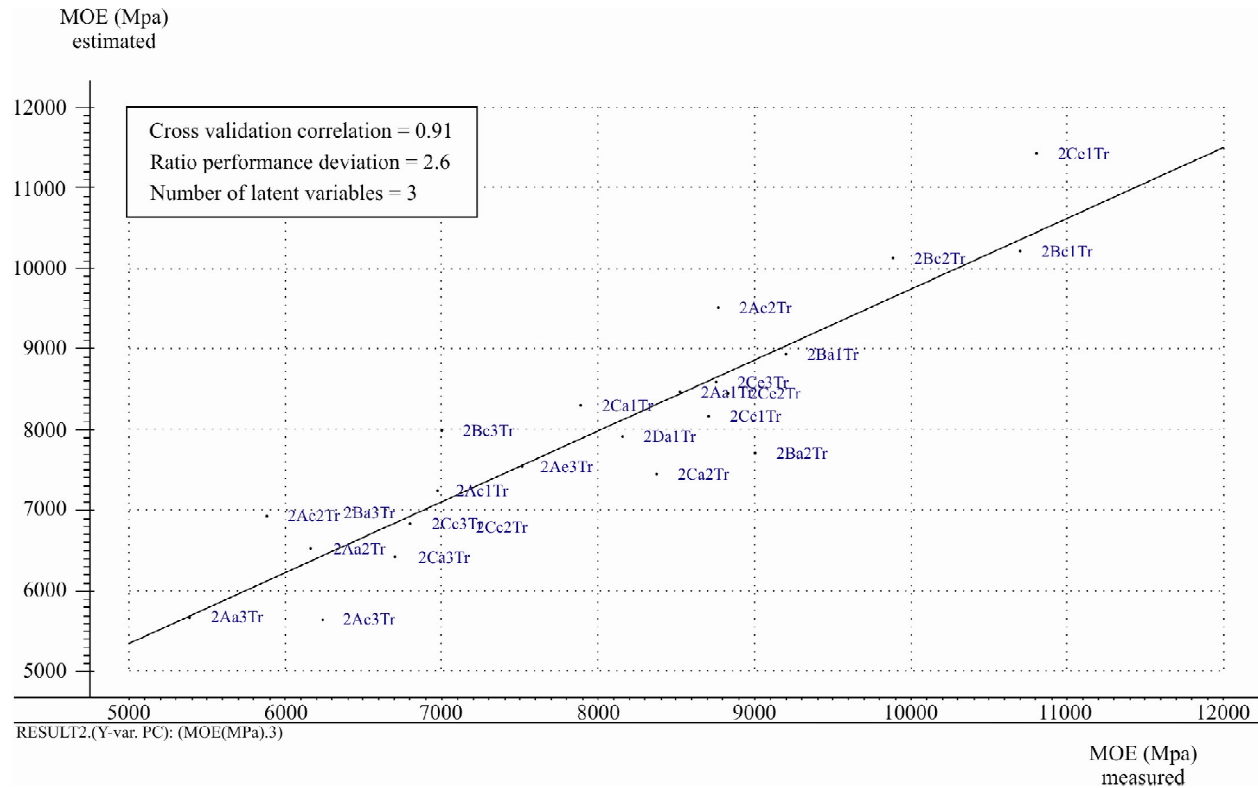
Figure 2 – Correlation graph between estimated and measured modulus of elasticity in compression parallel to grain testing on transverse face.

Figura 2 – Gráfico da correlação entre o módulo de elasticidade estimado e o medido no ensaio de compressão paralela às fibras, face transversal.

Table 3 – Summary of NIRS calibrations for mechanical properties in ground wood.

Tabela 3 – Resumo das calibrações NIRS para as propriedades mecânicas da madeira moída.

Wood	Test type	Prop	Treat	r	LV	Outlier	SECV	RPD	n
Ground	Comp.	MOE	2d	0.82	3	1	866.3	1.7	25
		CS	1d	0.89	4	0	3.0	2.3	25
	Bending	MOE	1d	0.87	3	1	1346.6	2.0	19
		MOR	1d	0.72	5	0	10.7	1.4	19

Prop – Property, Treat – Treatment, r – Cross validation correlation, LV – Number of latent variables, *Outlier* – Number of discarded samples, SECV – Standard error of cross validation, RPD – Ratio of performance deviation, n – Total number of samples used, Comp. – Compression parallel to grain, MOE – Modulus of elasticity, CS – Strength in compression parallel to grain, MOR – Modulus of rupture, 1d – First derivative, 2d – Second derivative.

4 CONCLUSIONS

Best correlation coefficients to estimate modulus of elasticity and compressive strength, in compression parallel to grain testing, were obtained when using original spectra from solid wood.

Modulus of elasticity in static bending testing was best estimated when solid wood spectra were subjected to first derivative treatment, while modulus of rupture presented best calibrations when original spectra were used.

As for ground wood spectra, higher values of cross validation correlation were obtained when using first and second derivative pretreatments than when using original spectra.

5 ACKNOWLEDGMENTS

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