https://doi.org/10.26525/jtfs2018.30.1.117125

DETERMINATION OF HEAT-TREATED *EUCALYPTUS* AND *PINUS* WOOD PROPERTIES USING NIR SPECTROSCOPY

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Submitted April 2017; accepted August 2017

Near-infrared (NIR) spectroscopy associated with multivariate statistics has been successfully applied to a range of studies involving wood properties and its products. However, the use of NIR spectroscopy for technological classification of heat-treated wood is less common. Thus, the aim of this study was to estimate the equilibrium moisture content (EMC), dimensional stability, stiffness and strength of heat-treated wood samples of *Eucalyptus urophylla* and *Pinus oocarpa* by NIR spectroscopy. Specimens taken from central boards were heat treated at 125, 150, 175, 200 and 225 °C. After heat treatment, the woods were kept in a climatic chamber until EMC was reached. NIR spectra were recorded and correlated to the wood properties determined in laboratory using multivariate statistics. It was possible to develop satisfactory models for estimating EMC (cross-validation coefficient (R^2cv) = 0.94, ratio of performance to deviation (RPD) = 4) and modulus of rupture (R^2cv = 0.86, RPD = 2.7) when considering both *Eucalyptus* and *Pinus* wood samples together. The model for volumetric swelling presented better statistics (R^2cv = 0.93 and RPD = 3.8) when only *Eucalyptus* samples was considered.

Keywords: Timber quality, nondestructive evaluation, multivariate analysis, vibrational spectroscopy

INTRODUCTION

Heat treatment of wood uses heat up to 260 °C. This temperature range degrades hemicelluloses in the wood, the main component responsible for its hygroscopic character (Brito et al. 2008). This will cause reduction in moisture adsorption capacity, resulting in lower equilibrium moisture content (EMC), and radial and tangential swellings (Zanuncio et al. 2014, Severo et al. 2012). However, the process can also decrease the mechanical properties of wood including its stiffness and strength (Candelier et al. 2015). A major attraction of this process is the low environmental impact since there is no use of chemicals (Korkut 2012).

In order to improve the quality control of heat treatment processes, it is necessary to use tools that provide rapid, accurate and reliable results. In this context, the use of near infrared (NIR) spectroscopy has shown promising results in characterisation of wood (Tsuchikawa & Schwanninger 2013). This technology has been applied for estimating a range of wood traits such as basic density (Rosso et al. 2013), wood anatomic features (Viana et al. 2009), wood moisture content (Yang et al. 2017), microfibril angle of cellulose deposition in wood cell wall (Hein et al. 2010), wood chemical composition (Leblon et al. 2013), physical and mechanical properties (McLean et al. 2014, Todorovic et al. 2015) and wood genetic parameters (Hein & Chaix 2014). There are studies reporting the use of NIR spectroscopy associated with multivariate analyses for predicting heat-treated wood properties (Tsuchikawa & Kobori 2015). The aim of this study was to develop multivariate models in order to estimate the EMC, linear and volumetric swellings, stiffness and strength of heat-treated wood samples of *Eucalyptus urophylla* and *Pinus oocarpa* by NIR-spectroscopy.

MATERIALS AND METHODS

Materials and preparation of specimens

Heat-treated wood of three 15-year-old *E. urophylla* and three 18-year-old *P. ocarpa* trees were investigated. Central boards were removed from the first logs (taken below 1.3 m height) and air dried until EMC was reached. Samples with nominal dimensions of 20 mm × 20 mm × 30 mm (radial × transverse × longitudinal) were cut for physical tests and specimens of 20 mm × 20 mm × 300 mm were produced for mechanical essays. The specimens were taken along the radial direction and samples coming from the same position within the boards were proportionally distributed in each heat treatment in order to avoid bias. The samples were dried at 103 °C until constant weight to achieve the anhydrous condition.

Heat treatment

Specimens were heat treated at 125, 150, 175, 200 and 225 °C. The heating rate was 5 °C per min, residence time was 4 hours at atmospheric pressure with presence of air. The specimens were conditioned in a climatic chamber at 23 °C and 50% relative humidity until mass stabilisation.

Wood characterisation

Equilibrium moisture content (EMC) of the specimens was calculated according to equation 1:

$$EMC(\%) = \left(\frac{Wm - Dm}{DM}\right) \times 100 \qquad (1)$$

where Wm = wet mass and Dm = dry mass. The specimens were saturated in water to determine the volumetric swelling according to equation 2:

$$VS(\%) = \left(\frac{Vsw - Vdw}{Vdw}\right) \times 100 \qquad (2)$$

where VS (%) = volumetric swelling, Vsw = volume of saturated wood and Vdw = volume of dried wood. The radial swelling was determined using equation 3:

$$RS(\%) = \left(\frac{RLs - RLd}{RLd}\right) \times 100 \qquad (3)$$

where RS = radial swelling, RLs = radial length of saturated wood and RLd = radial length of dry wood. The tangential swelling was calculated according to equation 4:

$$TS(\%) = \left(\frac{TLs - TLd}{TLd}\right) \times 100 \qquad (4)$$

where TS = tangential swelling, TLs = tangential length of saturated wood and TLd = tangential length of dry wood. Finally, the anisotropy factor was determined by the ratio between tangential and radial swellings.

Modulus of elasticity (MOE), which indicates wood stiffness, and modulus of rupture (MOR) were calculated from 20 mm × 20 mm × 300 mm specimens according to the D143 procedure described in ASTM (1997), using a universal testing machine. Thirty samples were used per species.

NIR spectroscopy

NIR spectra were obtained using transform spectrometer in diffuse reflectance mode. The NIR spectral scanning was performed between 12,500 and 4000 cm^{-1} with spectral resolution of 8 cm^{-1}. Spectra were obtained from the radial–longitudinal surface of the specimens. Each NIR spectrum represented the average of 32 scannings and two NIR spectra were obtained per sample. A sample NIR spectra was calculated from 64 scans and used for carrying out the multivariate models.

Calibration and validation

Partial least squares regressions were developed to compare all physical and mechanical values determined in the laboratory with the NIR spectra. The principal component analysis (PCA) was calculated to classify the NIR spectra using the statistical software (Unscrambler, version 9.7, 2007), as described by Hein et al. (2009). Partial least squares (R) calibration and cross-validations and PCA were calculated with a maximum of 12 latent variables (LV) or principal components (PC) respectively. The number of LVs or PCs adopted for each model was identified during the cross-validation process and corresponded to the first minimal residual variance obtained from the residual Y variance by PC or LV plot, as suggested by Viana et al. (2009).

The models were validated by cross-validation method in which the total sampling was divided into six batches at random. The selection of the wavelengths was performed according to the incertitude Martens test. The anomalous samples (outliers) were detected by Student residuals and leverage values and were not included in the calibration and validation models.

To evaluate the robustness of the developed models, the following statistics were considered: coefficient of determination of the model in cross-validation (R^2cv), the root mean standard error of cross-validation, number of LV used in the calibration and the ratio of performance to deviation (RPD). The RPD is the ratio between standard deviation of the reference value and root mean standard error of cross-validation.

RESULTS AND DISCUSSION

Variability of heat-treated wood properties

Table 1 presents the descriptive statistics of the physical properties of heat-treated woods of *E. urophylla* and *P. oocarpa*. The range of these wood traits is essential to develop satisfactory calibrations and robust validations.

Wood samples had EMC values ranging from 6.06 to 12.13%, showing a variation close to 100%, whereas tangential swelling showed 440% variation (2.25% minimum and 12.3% maximum). Variations between minimum and maximum were higher when considering *E. urophylla* and *P. oocarpa* samples together. Considering the two species together is interesting to obtain discrepant values between the samples, which facilitates the creation of NIR models. In addition, in Brazil, it is common to find sawmills working with softwood and hardwood. A higher coefficient of determination for models will be obtained when there are greater variety in values of parameters (Hein et al. 2012). Heat treatment reduced EMC and radial and tangential swellings as much as 49, 57 and 39% in *Eucalyptus grandis* heat-treated wood at 220 °C respectively (Calonego et al. 2012). Heat treatment at 220 °C reduced EMC and radial and tangential swellings to 23, 15.1 and 15.6% respectively in *Pinus elliottii* (Severo et al. 2012).

Variation in mechanical properties was similar to that found in physical properties (Table 2). Considering samples of both species, mean MOE was 2917.5 MPa (range between 1187.5 and 4897.6 MPa), while mean MOR was

Table 1 Descriptive statistics of equilibrium moisture content (EMC), linear swelling in tangential (Tan) and radial direction (Rad), volumetric swelling (VS) and anisotropy factor (AF) of the samples used for calibration

Property	N	Mean	SD	Min	Max
			Eucalyptus urophylla + Pinus oocarpa		
EMC (%)	234	9.34	1.815	6.06	12.13
Tan (%)	175	5.71	2.185	2.25	12.30
Rad (%)	175	3.46	1.486	1.23	6.20
VS (%)	234	12.88	3.445	7.41	19.67
AF	175	1.73	0.409	0.90	3.17
			Eucalyptus urophylla		
EMC (%)	119	8.83	1.700	6.16	11.01
Tan (%)	88	6.82	2.308	2.83	12.30
Rad (%)	88	4.60	1.223	2.27	6.20
VS (%)	119	14.44	3.790	7.73	19.67
AF	88	1.47	0.234	0.90	2.09
			Pinus oocarpa		
EMC (%)	115	9.87	1.784	6.06	12.13
Tan (%)	87	4.59	1.315	2.25	6.76
Rad (%)	87	2.32	0.548	1.23	3.33
VS (%)	115	11.27	2.062	7.41	16.02
AF	87	1.99	0.383	1.22	3.17

SD = standard variation, N = number of samples

Table 2 Descriptive statistics of modulus of elasticity (MOE) values and modulus of rupture (MOR) of the samples used for calibration

Property	Mean	SD	Min	Max	N
		Eucalyptus urophylla + *Pinus oocarpa*			
MOE (MPa)	2917.5	949.6	1187.5	4897.6	224
MOR (MPa)	65.21	29.15	18.02	139.40	224
		Eucalyptus urophylla			
MOE (MPa)	3737.4	595.0	2414.1	4897.6	111
MOR (MPa)	83.49	27.28	38.17	139.40	111
		Pinus oocarpa			
MOE (MPa)	2112.1	356.4	1187.5	3211.6	113
MOR (MPa)	47.26	17.45	18.02	90.61	113

SD = standard variation, N = number of samples

65.21 MPa (18.02 and 139.40 MPa). With wide variation between samples in their physical and mechanical parameters, NIR will be able to detect the differences and create a more precise model to predict these properties accurately.

NIR-spectral variation due to heat treatment

Figure 1 shows the NIR-spectral signatures recorded in heat-treated wood samples. From wavelength 12,500 to 7000 cm^{-1} it was possible to visually detect differences in NIR spectra in function of heat treatment. This region of the spectra is more sensitive to the colours of the wood. Specimens heat treated at 225 °C were darker and had higher absorbance in this spectra zone. There was no visual difference in NIR spectra from 7000 to 3500 cm^{-1}.

Using PCA, it was possible to detect differences between wood samples according to heat treatment temperatures. Figure 2 shows the dispersion of wood samples for each heat treatment in principal components 1 and 2 plots for *Eucalyptus* and *Pinus*. The first and second principal components explained 73 and 25% respectively of the spectral variability in samples of *Eucalyptus* while those of *Pinus*, 60 and 38% respectively.

The spectral difference can be easier distinguished in the wood samples treated at 200 and 225 °C (Figure 2). At lower temperatures, heat treatment results in volatilisation of the polar components of wood extractives (Mészáros et al. 2007). Due to their low proportion in wood, any alteration in these substances does not change the absorbance values of the wood. At higher temperatures, wood changes are more intense, resulting in high degradation of hemicellulose (Brito et al. 2008, Cademartori et al. 2013), forming formaldehyde, furfural and other aldehydes (Tjeerdsma et al. 1998). These compounds react differently to NIR spectra, allowing the differentiation of heat-treated samples at 200 and 225 °C.

Calibration for estimating physical properties

Table 3 shows the statistics associated with predictive NIR-based models for physical properties of heat-treated wood of *Eucalyptus* and *Pinus*. Except for the anisotropy factor, the models developed from NIR spectra were able to predict physical properties of the heat-treated wood satisfactorily.

The calibration for estimating physical properties of heat-treated wood were developed according to species (*P. oocarpa* and *E. urophylla*), separately and together. Accuracy of the models was assessed by RPD. Higher RPD value results in better model fit (Fujimoto et al. 2008).

The model to estimate EMC from NIR spectra showed better statistics, with $R^2cv = 0.94$ when using both or only *Eucalyptus* samples. NIR spectroscopy has shown promising results for predicting wood moisture (Leblon et al. 2013). NIR-based models for moisture of *Fagus sylvatica* and *Pinus sylvestris* wood without heat treatment had $R^2cv = 0.98$ and 0.99 respectively (Kobori et al. 2013), while models for estimating moisture of heat-treated wood of *Pinus pinaster* and *Eucalyptus*

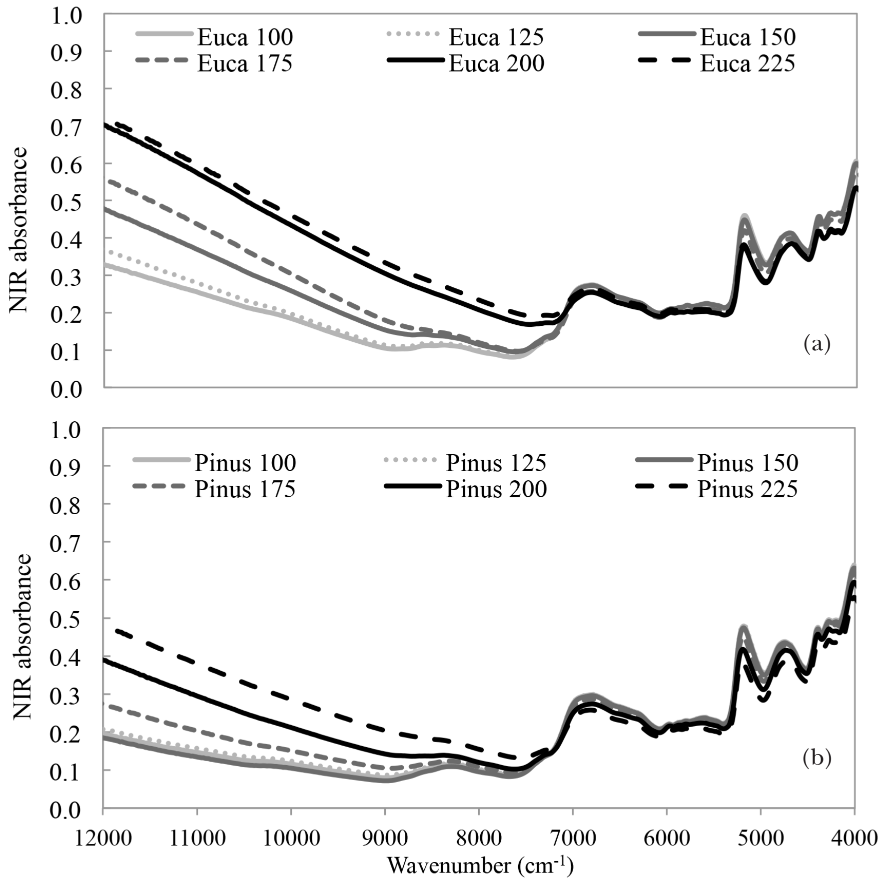

Figure 1 Absorbance of NIR spectra according to heat treatments for (a) *Eucalyptus urophylla* (Euca) and (b) *Pinus oocarpa* (Pinus) wood; for each treatment (species and temperature employed), there is a curve for the relation of the graph

globulus had coefficient of determination between 0.73 and 0.95 (Esteves & Pereira 2008).

The best models to predict the linear and volumetric swellings were generated when considering both samples together or only the specimens of *E. urophylla* (Table 3 and Figure 3). The major difference was the radial swelling whereby the model considering all samples showed $R^2cv = 0.92$ and 0.90 while the model considering only the wood of *P. oocarpa* had $R^2cv = 0.58$ and 0.50 respectively. All models generated for linear and volumetric variation showed similar R^2cv values.

It was not possible to accurately estimate the anisotropy factor through NIR spectroscopy. The model including all samples presented $R^2cv = 0.44$ while models considering only *Pinus* or *Eucalyptus* samples presented R^2cv lower than 0.20. The anisotropy factor was derivated from two other parameters, i.e. the radial and tangential swellings, making it more difficult to calibrate using NIR spectra.

Models for EMC presented RPD values ranging from 3.9 to 4.1 (above 3 is good) probably because the wood samples showed low coefficients of variation by treatment, which facilitated the correlation between spectra and the respective EMC values. On the other hand, the anisotropy factor showed unsatisfactory models, with RPD values between 1.0 and 1.3 due to high variation between wood samples in the same treatment (Table 3).

Calibration for estimating mechanical properties

Table 4 shows the statistics of the models based on NIR spectra for estimating MOE and MOR of heat-treated wood samples. Calibration presented greater accuracy for estimating the mechanical properties of the two species when grouped in the same model. The models for predicting MOE showed good statistics when both species were considered together ($R^2cv > 0.8$, RPD =

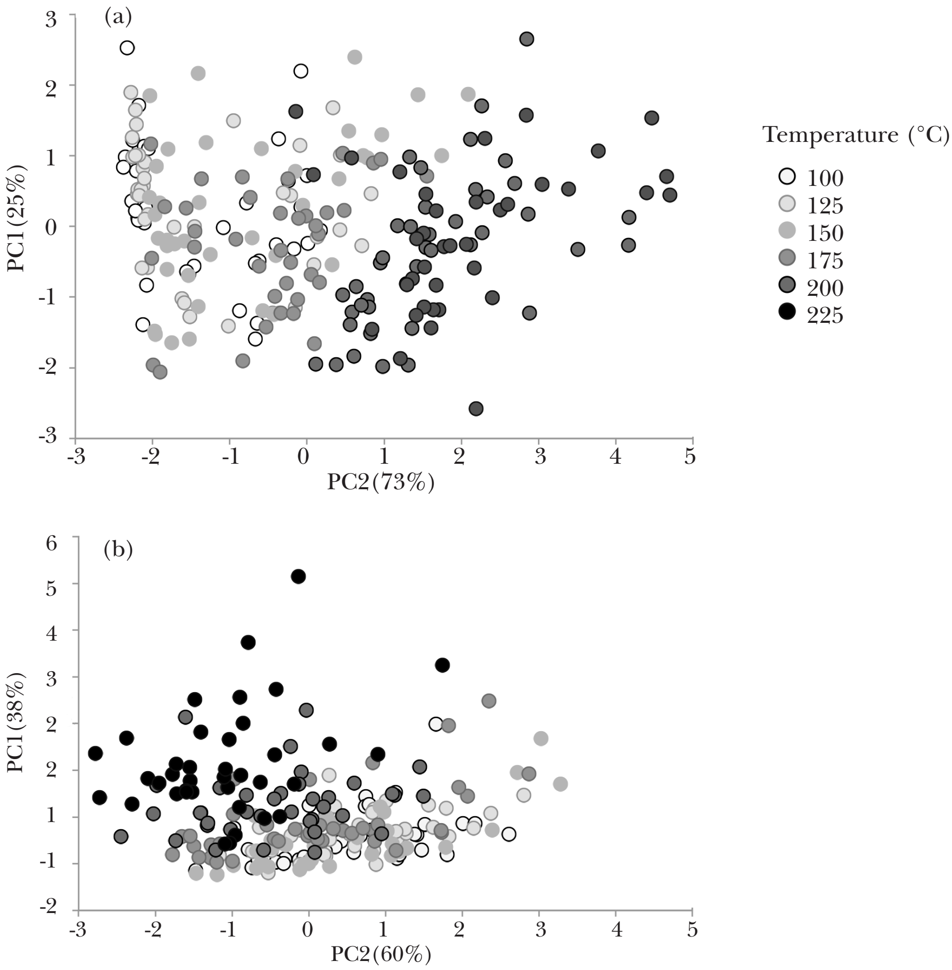

Figure 2 Principal component analysis (PCA) for (a) *Eucalyptus urophylla* and (b) *Pinus oocarpa* under different thermal treatments

Table 3 Statistics associated with calibrations to estimate physical properties by NIR spectroscopy

Property	Database	R^2c	RMSEC	R^2cv	RMSECV	LV	RPD
	E+P	0.94	0.438	0.94	0.453	5	4.0
EMC	Euca	0.95	0.385	0.94	0.412	4	4.1
	Pinus	0.94	0.429	0.93	0.461	5	3.9
	E+P	0.84	0.840	0.82	0.876	5	2.5
Tan	Euca	0.87	0.836	0.84	0.918	4	2.5
	Pinus	0.82	0.560	0.78	0.632	6	2.1
	E+P	0.92	0.431	0.90	0.471	8	3.2
Rad	Euca	0.88	0.416	0.86	0.454	4	2.7
	Pinus	0.58	0.354	0.50	0.388	4	1.4
	E+P	0.89	1.15	0.88	1.19	5	2.9
VS	Euca	0.93	0.97	0.93	1.00	4	3.8
	Pinus	0.75	1.02	0.72	1.08	5	1.9
	E+P	0.50	0.287	0.44	0.306	6	1.3
AF	Euca	0.19	0.209	0.13	0.221	2	1.1
	Pinus	0.15	0.351	0.10	0.366	3	1.0

E+P = *Eucalyptus urophylla* and *Pinus oocarpa* heat-treated wood, Euca and Pinus = *E. urophylla* and *P. oocarpa* heat-treated wood respectively, EMC = equilibrium moisture content, Tan = linear swelling in tangential direction, Rad = linear swelling in radial direction, VS = volumetric swelling and AF = anisotropy factor, R^2c = coefficient of determination of the prediction model, RMSEC = root mean square error, R^2cv = cross-validation coefficient, RMSECV = root mean standard error of cross-validation, LV = latent variables, RPD = ratio of performance to deviation

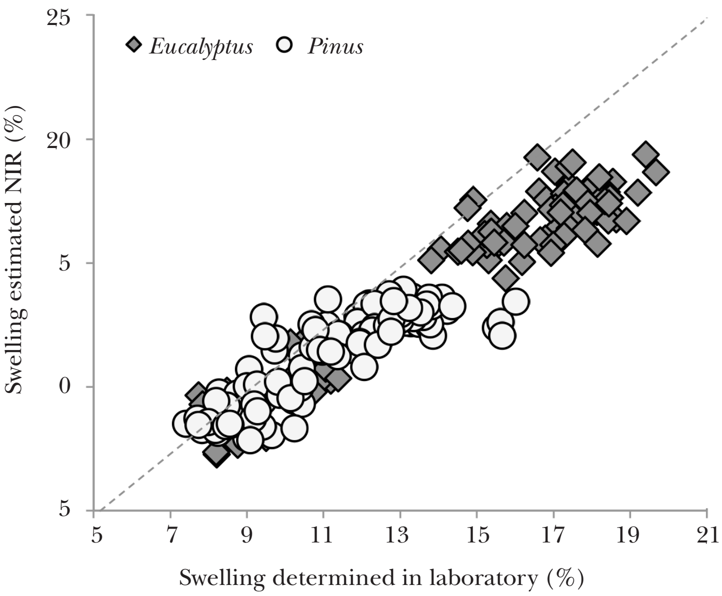

Figure 3 Bi-dimensional plot for volumetric swelling values estimated by NIR versus the values determined in the laboratory for *Eucalyptus urophylla* and *Pinus oocarpa*

Table 4 Statistics associated with calibrations to estimate the mechanical properties by NIR spectroscopy

Property	Database	R^2c	RMSEC	R^2cv	RMSECV	LV	RPD
MOE	E+P	0.83	393.1	0.81	416.4	8	2.3
	Euca	0.44	441.7	0.42	457.4	1	1.3
	Pinus	0.29	298.2	0.29	301.6	1	1.2
MOR	E+P	0.88	9.97	0.86	10.81	8	2.7
	Euca	0.85	10.63	0.83	11.71	5	2.3
	Pinus	0.79	7.98	0.79	8.27	3	2.1

R^2c = coefficient of determination of the prediction model, RMSEC = root mean square error, R^2cv = cross-validation coefficient, RMSECV = root mean standard error of cross-validation, LV = latent variables, RPD = ratio of performance to deviation; E+P = *Eucalyptus urophylla* and *Pinus oocarpa* heat-treated wood, Euca and Pinus = *E. urophylla* and *P. oocarpa* respectively; MOE = modulus of elasticity, MOR = modulus of rupture

2.3). However, when the models were calibrated using *Eucalyptus* or *Pinus* samples separately, the statistics of the models were not good. Similarly, for MOR, the models showed good results when both species were grouped together in the same model (R^2cv = 0.86, RPD = 2.7). The specific models for MOR also presented satisfactory statistics, with R^2cv = 0.83 and RPD = 2.3 for *E. urophylla* wood and R^2cv = 0.79 and RPD = 2.1 for *P. oocarpa* wood.

The robustness of the models to predict mechanical properties using NIR spectroscopy varied greatly according to the wood characteristics. Gindl et al. (2001) have obtained NIR-based models and MOR with coefficient of determination of the prediction model (R^2) = 0.98 (for MOE) and 0.96 (for MOR) in *Larix decidua* wood samples. Bachle et al. (2010) reported models presenting coefficient of determination of the prediction model (R^2c) of 0.36 (for MOE) and 0.16 (for MOR) for heat-treated wood of *Picea abies* and *F. sylvatica.*

Figure 4 presents the relationship between MOR values obtained from laboratory tests and those estimated by NIR. From estimated values by NIR spectroscopy and those determined by the universal testing machine, it was possible to ascertain the quality of the models considering both samples (Figure 4). It was also possible to observe that the NIR-based models were able to

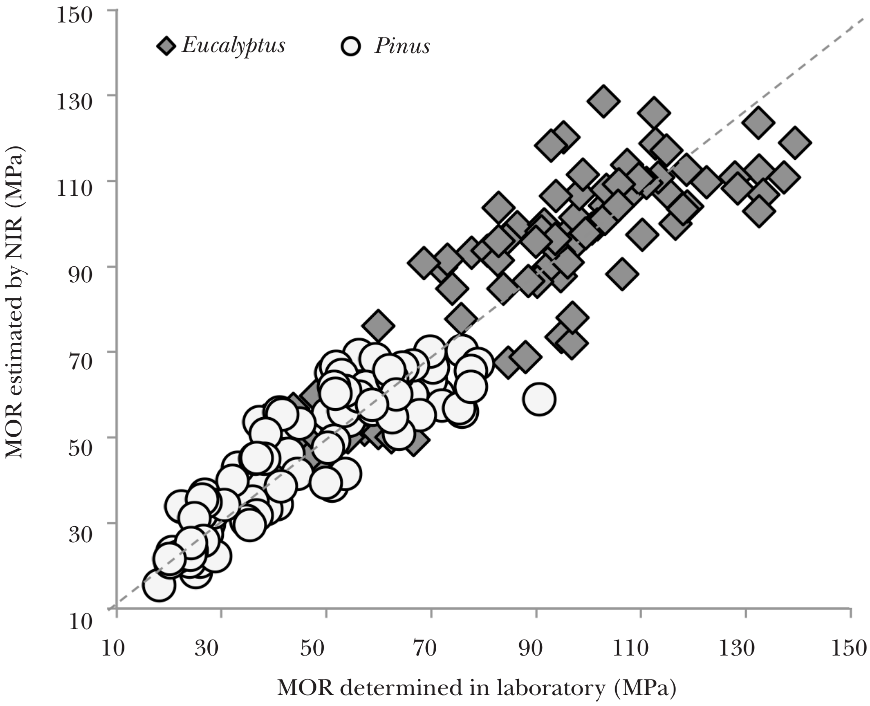

Figure 4 Two-dimensional plot for modulus of rupture (MOR) estimated by NIR versus the values obtained in the laboratory for *Eucalyptus urophylla* and *Pinus oocarpa*

predict MOR without trends or homoscedasticity for *P. oocarpa* and *E. urophylla* wood.

In short, the findings showed that NIR spectroscopy associated with multivariate analysis were able to generate efficient models to predict physical and mechanical properties of heat-treated wood of *E. urophylla* and *P. oocarpa*. Predictive models based on NIR spectra for estimating physical and mechanical wood properties rarely reach present RPD values above 2.5 (Todorovic et al. 2015). Therefore, this technology can be used by industries for monitoring quality of heat-treated hardwood and softwood to increase their quality process control.

CONCLUSIONS

It was possible to develop calibrations for estimating the physical and mechanical properties of *P. oocarpa* and *E. urophylla* heat-treated wood from NIR spectroscopy. The best statistics were presented by models for estimating EMC (R^2cv = 0.94, RPD = 4) and MOR (R^2cv = 0.86, RPD = 2.7) when both species were considered together, and by the models for volumetric swelling when considering only *Eucalyptus* samples (R^2cv = 0.93, RPD = 3.8).

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