CALIBRATIONS BASED ON NEAR INFRARED SPECTROSCOPIC DATA TO ESTIMATE WOOD-CEMENT PANEL PROPERTIES

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Some scientific contributions have used near infrared (NIR) spectroscopy as a rapid and reliable tool for characterizing engineered wood products. However, to our knowledge, there are no published papers that used this technique in order to evaluate wood-cement panels. The main objective of this paper was to evaluate the ability of NIR spectroscopy to estimate physical and mechanical properties in wood-cement panels. The wood-cement panels were produced using *Eucalyptus grandis x E. urophylla*, *Pinus taeda*, and *Toona ciliata* woods with Portland cement under different manufacturing conditions. Wood-cement panels were characterized by traditional methods, and Partial Least Squares regressions were used to build calibrations. Our cross-validated models for MOR, IB, and TS24h of the panels yielded good coefficients of determination (0.80, 0.82, and 0.91, respectively). Based on the significant absorption bands and regression coefficients of the PLS models, our results indicate that cellulose and aromatic groups in lignin are components that play an important role in the calibrations.

Keywords: Partial least squares regression; Wood products; MOE; MOR; Internal bond; Water absorption; Thickness swelling

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INTRODUCTION

Wood-cement panels are made from particles or fibers of vegetal biomass (aggregate), cement (mineral binder), and water, which are consolidated under pressure and room temperature. Normally, most ligno-cellulosic materials may be used to produce these boards, including forestry and agro-industrial residues. However, chemical substances, especially extractives, present in the wood may delay or sometimes even impede the cement cure, making necessary the use of some kind of process that provides chemical compatibilization between the vegetal biomass and the cement by the minimization of these components' effects on mineral binder hydration (Savastano Junior et al. 2000). According to Latorraca and Iwakiri (2000), softwood species are more used to cement-wood panels production than hardwoods due to their properties, especially the chemical ones, which do not affect the cure and hardening of the cement. Iwaki and Prata (2008) evaluated the potential of *Eucalyptus grandis* and *Eucalyptus dunnis* for cement-

wood panels production. They showed that these woods were suitable for panel manufacture and no treatment was required.

According to Moslemi (1974), the wood-cement panel is a material of structural use that can provide excellent thermal and acoustic isolation, besides having high resistance to fire, biodegradation agents, and water. Moreover, the mineral board is harder and more resistant than its components alone, achieving lower cost and density than concrete. In addition, they also present lower energy consumption compared to conventional particleboards, since wood-cement panels are pressed at room temperature, and they have higher dimensional stability and due not involve the use of formaldehyde.

The product engineering industry requires new solutions for controlling the quality of their products in order to achieve property specifications (Campos et al. 2009). In general, the performance of a wood-cement panel is related to its physical and mechanical properties. In spite of these tests being relatively quick, the sample preparation is laborious and time-consuming. Furthermore, to obtain data on stiffness (modulus of elasticity), the use of high capacity machine testing and also deformation measuring is required (Hein et al. 2009). Moreover, after testing, the samples become unusable. One alternative to overcome these inconveniences is the use of near infrared (NIR) spectroscopy, which is an indirect, fast, and accurate method for the prediction of many wood properties (Pasquini, 2003). The use of this technique in the evaluation of physical and mechanical properties may reduce the sample preparation time, cost, and material waste.

In this study, wood-cement panels were produced using *Eucalyptus grandis* x *Eucalyptus urophylla* hybrid clone, *Pinus taeda* and *Toona ciliata* woods, and Portland cement, under different manufacturing conditions. NIR spectra were measured on the panel samples under two conditions: (a) on the rough panels, generating the information labeled as "rough NIR spectra" and (b) on the surfaced panels (sanded with 300-grit sandpaper), producing the "surfaced NIR spectra". We used the two types of NIR information to develop predictive models. To our knowledge, there are no published papers that used near infrared (NIR) spectroscopy technology associated to multivariate data analysis in order to investigate wood-cement panels. For this reason, the objectives of this paper were: (i) to evaluate the ability of NIR spectroscopy to estimate physical and mechanical properties in wood-cement panels; (ii) to build general NIR calibrations for estimating these properties in these wood-cement panels, and (iii) to generate a better understanding of why NIR spectroscopy is able to estimate mechanical and physical properties in wood-cement panels.

EXPERIMENTAL

Wood-Cement Panels Manufacture

The raw materials used to manufacture the wood-cement panels investigated in this study were: (a) 18-year-old *Pinus taeda* wood (basic density of 0.36 g/cm^3) obtained from experimental plantation located at Lavras, Minas Gerais State, Brazil; (b) 7-year-old *Eucalyptus grandis* x *Eucalyptus urophylla* hybrid clone wood (basic density of 0.56 g/cm^3), obtained from Companhia Mineira de Metais located at Vazantes, Minas Gerais

State, Brazil, and (c) veneering residues of 18-year-old *Toona ciliata* M. Roem. wood (basic density of 0.32 g/cm^3) obtained from commercial plantation located at Marechal Floriano, Espírito Santo State, Brazil.

The *Toona ciliata* M. Roem wood was obtained from veneering process residues of the first log. The *Pinus taeda* and *Eucalyptus* clones logs were sawed above 1.3 m, transformed into boards, reduced to small samples of 90 mm x 200 mm x 25 mm dimension, and finally transformed into strand particles. The woods were milled to produce slivers, which were mechanically sifted in order to remove the finest particles. The slivers were then submerged in water and, after immersion for 24 hours, the water was drained and the particles were dried in an acclimatized room (20 ± 2°C and 60 ± 3%). Under these conditions, wood samples reached an equilibrium moisture content of 12%.

For the manufacture of the wood-cement panels, Portland cement (often referred to as OPC, from Ordinary Portland Cement) of high initial strength was used as binder material, which was mixed with water (water/cement ratio: 1/2.5 and hydration water/cement ratio: 1/4), a chemical additive ($CaCl_2$ at 4%), and the particles, in a cement mixer. The wood-cement panels were manufactured using a wood/cement ratio of 1/2.75 and were made by using a 10-minute press closing time, pressure of 40 kgf/cm², and room temperature. The pressing procedure was adapted to a system of clamps, and pressure was maintained during 24 hours until the panels had hardened. The target density and dimensions for all panels were 1.10 g/cm^3 and 480 mm x 480 mm x 15 mm, respectively.

Wood-Cement Panels Composition

Different proportions of *Toona ciliata*, *Pinus taeda*, and *Eucalyptus grandis* x *Eucalyptus urophylla* hybrid clone wood were combined into three classes of composition of wood-cement panels. The panels of each combination were cured using two different procedures: (i) 28 days in acclimatized room (20 ± 2°C and 60 ± 3%) and (ii) 10 days in a vapor chamber (60°C), after which the panels were kept in the acclimatized room (same condition as before) for 18 more days in order to stabilize the moisture content at 12%. Six treatments were investigated with three repetitions of each combination (3 compositions x 2 curing procedures), totaling 18 wood-cement panels (Table 1).

Table 1. Combinations of the Wood-Cement Panels

Treatments	Composition class	Content (%)			Cure
		T	P	E	
T1	T	100	-	-	AR
T2	T+P	50	50	-	
T3	T+E	50	-	50	
T4	T	100	-	-	VC
T5	T+P	50	50	-	
T6	T+E	50	-	50	

T - *Toona ciliata* M. Roem.; P - *Pinus taeda*; E - *Eucalyptus grandis* x *Eucalyptus urophylla* hybrid clone; AR - acclimatized room; VC - vapor chamber.

Specimens Preparation and Physical and Mechanical Tests

After the 28th day, the specimens were cut from the panels and stabilized in an acclimatized room (20 ± 2°C and 60 ± 3%) for the determination of physical and mechanical properties and also for NIR spectral scanning. Under these conditions, samples reached an equilibrium moisture content of 10%. 14 specimens were cut from each panel: 4 specimens of 250 mm x 50 mm x 10 mm of dimension were used for modulus of elasticity (MOE) and modulus of rupture (MOR) measurements; 2 specimens with 150 mm x 150 mm x 10 mm of dimension for water absorption after immersion for 24 hours (WA24H) and thickness swelling after immersion for 24 hours (TS24H); 4 specimens with 50 mm x 50 mm x 50 mm of dimension for compression (Comp); 4 specimens with 50 mm x 50 mm x 10 mm of dimension for internal bond (IB) measurements. WA24H and TS24H were performed according to the ASTM Standard 1982: D1037-100. The IB was determined according to ASTM standard 1982: D1037-28 while MOE and MOR, both determined in bending tests, and Comp determined according to DIN 52362 (1982) standard. The mechanical tests were performed on a universal testing machine (DL - 30 kN model, EMIC, Paraná, Brazil).

NIR Spectroscopy Measurement

The NIR spectra were measured in the diffuse reflectance mode with a NIR 900 PLS spectrophotometer (Femto instrument Ind & Com, São Paulo, Brazil) in an acclimatized room (20 ± 2°C and 60 ± 3%). This NIR spectrometer is based on a Czerny-Turner monochromator and uses a continuous diffraction to perform the reflectance analysis of solids using a tungsten-halogen 55W light source. The NIR spectra were obtained directly from the sample surface at 5 nm intervals over the wavelength range of 1100 nm to 2500 nm.

NIR scanning results were recorded before treatment in the same four specimens used for the IB tests. Eight (8) scans (four scans per side) were collected and averaged into a single NIR spectrum for each specimen. The specimens were turned in the equipment measuring cell. Thus, the light beam can capture different spots of the heterogeneous surface, providing a more representative averaged spectrum of the sample and reducing the data noise.

First, NIR spectra were measured on the rough panel samples, generating the information labeled as "rough NIR spectra". After that, the panels were sanded using 300-grit sandpaper for approximately 30 seconds, and new spectra were recorded, labeled as "surfaced NIR spectra". We used the two types of NIR information to develop predictive models.

Calibration Statistics

Partial Least Squares (PLS) regressions were developed in order to describe the relationship between the NIR spectra and wood-cement panel properties using The Unscrambler (CAMO AS, Norway) software version 9.7. PLS models were performed in full cross-validation mode with a maximum of twelve latent variables (LV). The LV number adopted for each model presented minimal residual variance (sometimes this value was similar to the suggested by the software), and the NIR spectra considered as outliers were indentified from the student residuals and leverage value plot analyses. In

order to enhance the quality of the calibration adjustment, the significant wavelengths were selected by the Martens uncertainty test (Westad and Martens, 2000). To enhance the calibration quality, first (13-point filter and a second order polynomial) and second derivatives (25-point filter and a third order polynomial) were applied on the NIR spectral data using the Savitsky and Golay (1964) algorithm. R²cv - coefficient of determination of the cross-validation, RMSECV - root mean square error of cross-validation, and RPD value (ratio of performance to deviation) were used as criteria to select the predictive model to estimate the properties of the wood-cement panel. Formulas used to estimate the RMSEC and RMSECV are given in Mora and Schimleck (2008) and should be as low as possible, while the coefficient of determinations should be high. The RPD value is the ratio of the standard error of cross-validation (or prediction) to the standard deviation (SD) of the original data. This statistic provides a basis for standardizing the standard error of prediction (Williams and Sobering, 1993) and makes it possible to compare calibrations for different parameters.

RESULTS AND DISCUSSION

Laboratory Data

The descriptive statistics of the wood-cement panel properties for each treatment including internal bond, modulus of rupture, modulus of elasticity, compression, thickness swelling, and water absorption after immersion for 24 hours measured by conventional methods are presented in Table 2. The mean value presented for each treatment in Table 2 represents the average value of 3 panels. For each panel, 4 specimens were used for MOE, MOR, Comp, and IB tests and 2 for WA24H and TS24H measurements. The coefficient of variation for each treatment and for each property is showed in parenthesis.

Table 2. Descriptive Statistics of the Wood-cement Panel Properties for Each Treatment Including Average and Coefficient of Variation (%, in parenthesis)

	IB	MOR	MOE	Comp	TS24h	WA24h
T1	8.29 (1.5)	93.67 (2.8)	47892 (8.4)	115 (17)	0.73 (5.5)	14.33 (16.4)
T2	10.32 (11.3)	84.17 (4.4)	45336 (7.9)	123 (7.9)	0.68 (22)	13.62 (6.3)
T3	10.4 (9.6)	62.25 (8.4)	42945 (9.9)	106 (6.8)	2.59 (23)	12.71 (8.3)
T4	11.48 (6.2)	75.34 (5.5)	44539 (6.7)	99 (12.3)	0.47 (40)	11.6 (1.8)
T5	8.53 (5.7)	74.42 (6.1)	42835 (14)	89 (8.1)	0.69 (2.5)	11.1 (1.9)
T6	8.29 (8.4)	51.58 (14)	35280 (9.5)	84 (16)	0.57 (17)	12.09 (6.4)
No. of samples per test	72	72	72	72	36	36

IB - internal bond (Kgf/cm²), MOR - modulus of rupture (Kgf/cm²), MOE - modulus of elasticity, (Kgf/cm²), Comp - compression (Kgf/cm²), TS24h - thickness swelling after 24 hours (%), and WA24h - water absorption after 24 hours (%)

The wood-cement panels investigated in this study yielded a wide range of values for each property (Table 2). The property which gave a higher range of variation was

thickness swelling after immersion for 24 hours, because treatment 3 resulted in a higher mean value (2.59%). In each treatment, this property was measured with 6 repetitions (2 specimens from 3 panels) and yielded a coefficient of variation of 22% for treatment 2; 23% for treatment 3; and 40% for treatment 4), and the water absorption ranged from 10.9 to 17%. The effects of the treatments on the properties of the wood-cement panels are not within the scope of this paper, and a deeper discussion about these issues are provided in Sá et al. (2009).

The calibration and cross-validation set used in this study is summarized in Table 3. The variability of these data is very important to develop predictive models: the higher the range of property variation, more chances to get a better coefficient of determination for the calibration model. As our main objective is developing general NIR calibration for estimating such properties, we are interested primarily in the range of variation (minimum and maximum value) for each property. According to Mora and Schimleck (2008), the calibration models must include all possible sources of variation that can be encountered later in real applications, because the goal is to estimate the properties in new samples.

Table 3. Summary of the Wood-Cement Panel Properties of Calibration and Cross-Validation Set

	IB	MOR	MOE	Comp	TS24h	WA24h
Average per panel	9.6	73.6	43138	102.7	0.96	12.6
SD per panel	1.43	14.7	5367	17.66	0.79	1.51
Min value	7.6	43.8	31815	69	0.32	10.9
Max value	12	96.3	51893	133.8	3.17	17
CV (%)	15	19.98	12.44	17.2	82.61	12.01
No. of samples per panel	18	18	18	18	18	18

IB - internal bond (Kgf/cm²), MOR - modulus of rupture (Kgf/cm²), MOE - modulus of elasticity, (Kgf/cm²), Comp - compression (Kgf/cm²), TS24h - thickness swelling after 24 hours (%), and WA24h - water absorption after 24 hours (%)

NIR Spectral Information

Wood is a complex material composed basically of cellulose, hemicellulose, lignin, and extractives. According to European Standard EN197.1, "Portland cement clinker is a hydraulic material which shall consist of at least two-thirds, by mass, of calcium silicates ($3CaO.SiO_2$ and $2CaO.SiO_2$), the remainder consisting of aluminium- and iron-containing clinker phases and other compounds. The ratio of CaO to SiO_2 shall not be less than 2.0. The magnesium content (MgO) shall not exceed 5.0% by mass." Wood-cement panel combines different kinds and contents of chemical substances and solid materials, generating a more complex material.

Figure 1 shows the first derivative NIR spectra measured on the specimens with the NIR spectrometer. Each spectrum represents the averaged NIR spectra of each treatment used for manufacturing the cement-wood panels. Variation in NIR absorbance values between the six types of treatments could be observed in specific points of the full spectrum. For instance, at the 1550 nm, treatments 1 and 2 showed clear differences in comparison to the others. Similarly, these treatments showed differences in their

absorbance values at 2200 nm. These differences reflect the variations in chemical composition of these panels, the interaction of wood species, and its influence on the curing rate and compatibilization with the cement.

In the original NIR spectra, such differences showed the same patterns. The higher variation in absorbance values were found at the same points (1550 nm and 2200 nm); however, we observed that rough NIR spectra presented higher values of NIR absorbance along the spectra. We supposed that such relative variation was caused by differences in surface roughness. The reason for this trend can be attributed to the physical effects due to the sanding operation, but it is still matter of debate. Perhaps, after sanding, the wood fibers become more exposed to NIR radiation, as part of the cement is removed from the panel surface. Another possible explanation for this trend is the NIR absorption of the calcium silicate molecules present within the panels. This complex information demands sophisticated statistical tools to allow proper interpretations and enhancement of NIR spectral data applications.

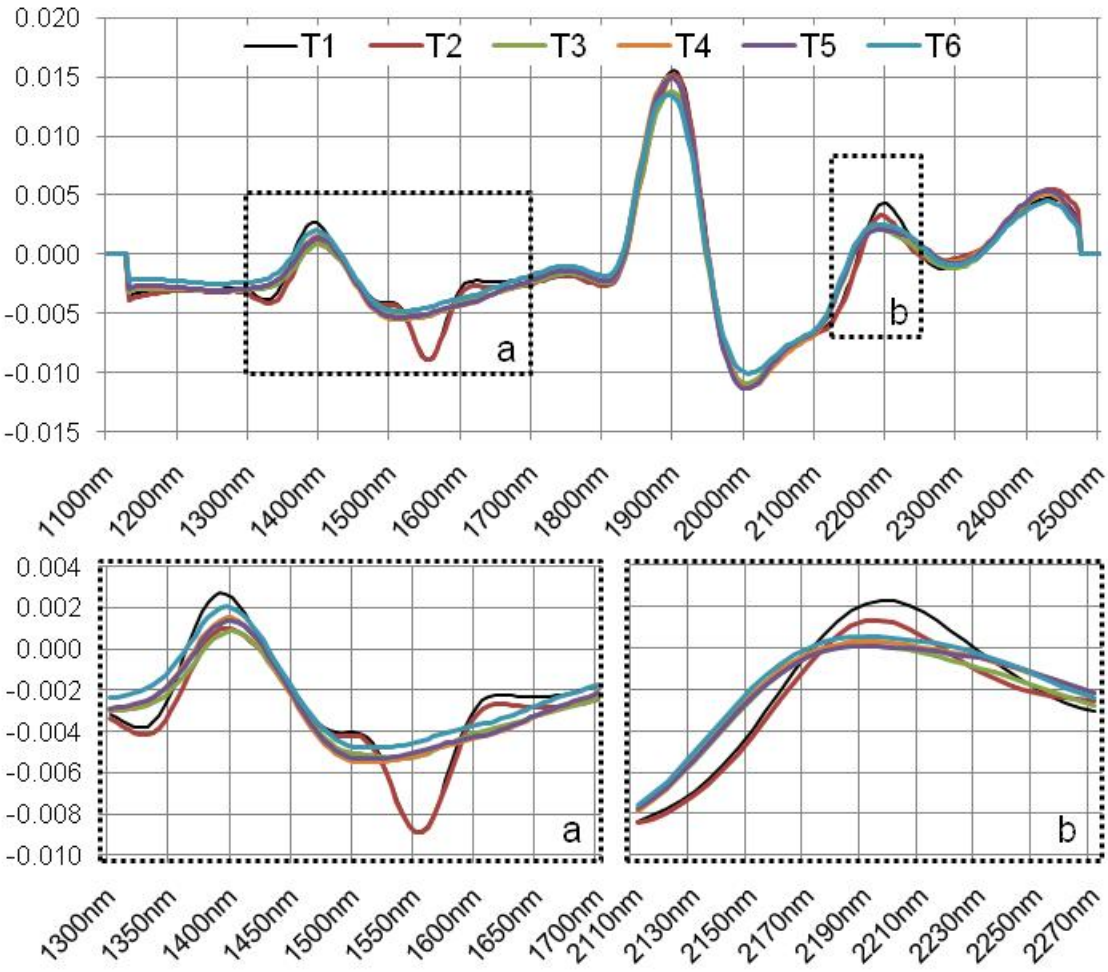

Fig. 1. Averaged first derivative NIR spectra recorded from surfaced wood-cement panels manufactured with different treatments

The NIR spectra "finger print" reflects the energy interaction with chemical bonds from different wood species, the nature of each component, and the content of the woods and cement, as well as their interaction, as recently affirmed by Campos et al. (2009). Thus, the NIR absorbance values (Fig. 1) represent a great variety of NIR radiation interactions along the wavelength range involving the wood cement panels' chemical and physical properties. NIR spectra from a complex surface such as wood-cement panels are consequently not merely driven by the underlying chemistry, but also by the solid structure (Gierlinger et al. 2004). NIR spectra also represent the light deviation and scattering due to surface roughness, particles size and shape, among other factors. In the present study, we analyzed the NIR spectra using the student residuals and leverage value plots. This procedure was used recently by Hein et al. (2009) in order to identify anomalous NIR readings. In the present investigation, only four NIR spectra (from a total of 576 measures) were detected as outliers, and they were removed from the data set.

PLS Models Based on Rough NIR Spectra

Table 4 presents the calibration and cross-validation models based on NIR spectra measured from the rough surface of the wood-cement panels in order to estimate the investigated properties.

Table 4. Summary of PLS Models based on Rough NIR Spectra

Model	Properties	Treat.	R²c	RMSEC	R²cv	RMSECV	LV´s	RPD
1	IB	1d	0.74	0.71	0.59	0.95	4	1.5
2	MOR	1d	0.84	5.63	0.8	6.71	2	2.2
3	MOE	1d	0.57	3402	0.54	3747	1	1.4
4	Comp	1d	0.53	11.71	0.47	13.24	1	1.3
5	TS24h	none	0.97	0.13	0.91	0.24	7	3.3
6	WA24h	1d	0.58	0.95	0.41	1.19	2	1.3

Treat. - mathematical treatment; 1d - first derivative; R^2c - coefficient of determination of the calibration model (%); RMSEC - root mean square error of calibration; R^2cv - coefficient of determination of the cross-validation (%); RMSECV - root mean square of cross-validation (%); LV - number of latent variables, and RPD - ratio of performance to deviation.

The cross-validated PLS models for MOR and TS24h of the wood-cement panels provided good statistics, showing coefficients of determination for cross-validation of 0.80 and 0.91 and RPD values of 2.2 and 3.3, respectively. These properties could be evaluated in unknown samples with satisfactory accuracy. The calibration for IB gave moderate statistics, being able to discriminate panels' performance. On the other hand, MOE, Comp, and WA24h provided low coefficients of determination, and RPD values and did not provide acceptable predictive models.

Figure 2 shows the plots of laboratory-measured versus NIR-fitted values for modulus of rupture by the PLS Model 2 developed from the NIR spectra measured on rough wood-cement panels.

Williams and Sobering (1993) claim that a RPD greater than 2.5 is considered satisfactory for screening, although it has been shown that a RPD value of approximately 1.5 indicates that NIR spectroscopy can be used as an initial screening tool (Schimleck et

al. 2003). In this study, our predictive models for internal bond (Model 1) gave acceptable statistics for initial screening (>1.5), while the models for MOR (Model 2) and TS24h (Model 5) presented quite satisfactory RPD values for screening (≈2.5).

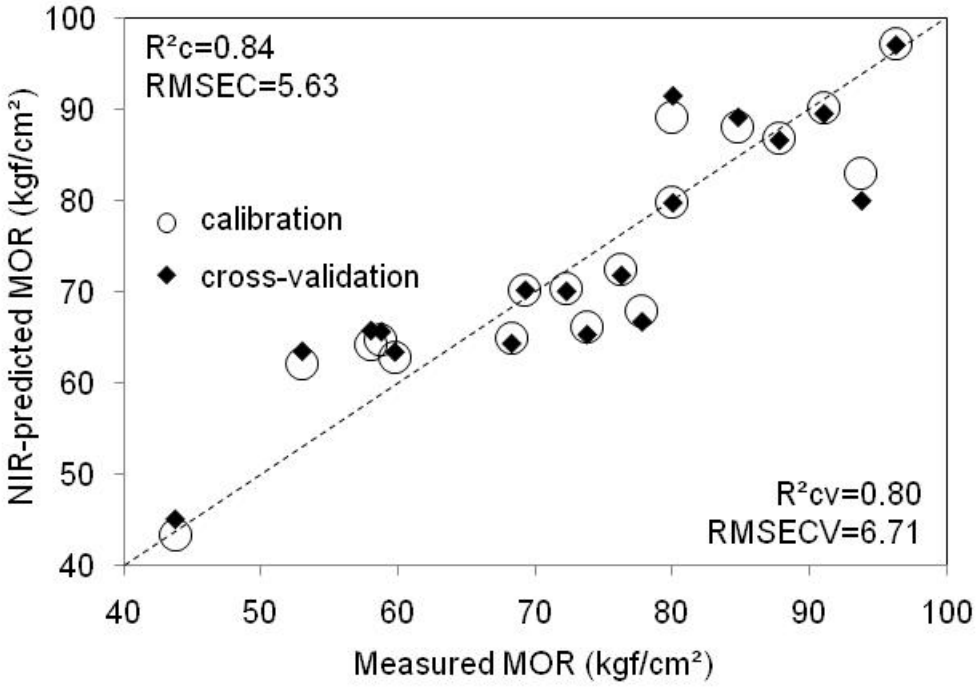

Fig. 2. Relationship between laboratory-measured versus NIR-predicted values for modulus of rupture using rough NIR spectra. The dotted line represents the 1:1 equivalence.

PLS Models based on Surfaced NIR Spectra

Table 5 shows the statistics associated with the PLS models based on surfaced NIR spectra to predict the properties of the wood-cement panels.

Table 5. Summary of PLS Models based on Surfaced NIR Spectra

Model	Properties	Treat.	R²c	RMSEC	R²cv	RMSECV	LV´s	RPD
7	IB	none	0.98	0.21	0.82	0.62	9	2.3
8	MOR	none	0.88	4.97	0.78	7.04	5	2.1
9	MOE	none	0.43	3916	0.35	4462	1	1.2
10	Comp	1d	0.75	8.52	0.31	15.05	6	1.2
11	TS24h	1d	0.9	0.24	0.53	0.55	7	1.4
12	WA24h	1d	0.41	1.12	0.31	1.29	2	1.2

Treat. – mathematical treatment; 1d - first derivative; R^2c - coefficient of determination of the calibration model (%); RMSEC - root mean square error of calibration; R^2cv - coefficient of determination of the cross-validation (%); RMSECV - root mean square of cross-validation (%); LV - number of latent variables, and RPD - ratio of performance to deviation.

Similar to the PLS models presented in Table 4, the calibrations developed from surfaced NIR spectra for internal bond (Model 7) and modulus of rupture (Model 8)

showed good statistics; however, TS24h calibration did not provide reasonable predicted values. The surfaced NIR spectra generated PLS models with higher R²cv (0.82) in comparison with the IB calibration (0.59) from rough NIR spectra. For Internal bond, the sanding operation seemed to improve the ability of light to capture information from the panels. The RMSECV, Number of LV of these models for IB were very different, indicating that the model statistics were highly influenced by the sanding operation. However, this trend was not observed for the MOR model. The statistics associated with models generated by rough NIR spectra were slightly similar (R²cv=80 and RMSECV= 6.71 for rough and R²=0.78 and RMSECV=7.04 for surfaced panels samples). After the sanding operation, the samples lose important information for thickness swelling after immersion for 24h, and the NIR spectra did not generate good calibrations. Similarly to Table 4, modulus of elasticity, compression, and water absorption after 24h could not be evaluated by NIR spectroscopic models: these properties had unsuitable R²cv and RPD values.

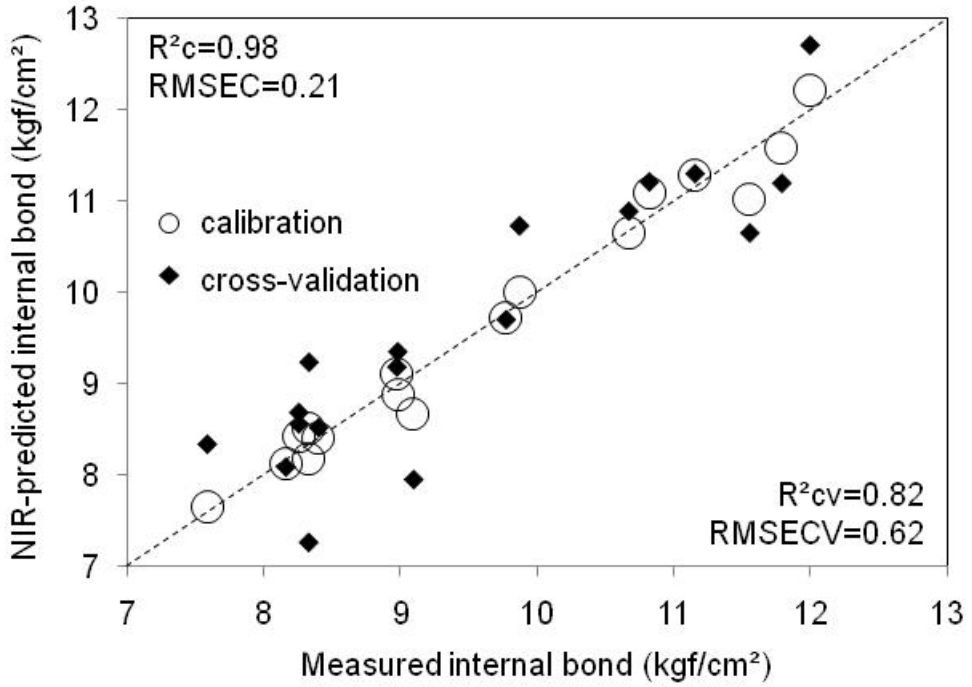

Fig. 3. Relationship between laboratory-determined values and NIR-predicted values for the internal bond using the surfaced NIR spectra. The dotted line represents the 1:1 equivalence.

In regard to the mathematical treatments of the surfaced NIR spectra (Table 5), the statistics of the models presented different results in comparison to Table 4. By using the rough NIR spectra, only TS24h provided the best statistics for calibration models from untreated NIR spectra. We applied first and second derivatives on NIR spectra, but only first derivative improved the models. Most of the predictive models were developed using the first derivative of the spectra information. After sanding, the model calibrations for IB and MOR did not require mathematical treatment to supply good results. The other properties (MOE, Comp, TS24H and WA24H) yielded low coefficients of determination,

and the transformation of NIR spectra using the derivative algorithm did not result in any advantages.

Figure 3 shows the plots of laboratory-measured versus NIR-fitted values for internal bond by the PLS Model 7 developed from the NIR spectra measured on surfaced wood-cement panels.

In this study, the cross-validated models for internal bond (Model 7), modulus of rupture (Models 2 and 8), and thickness swelling after immersion for 24h (Models 5) gave good statistics and had potential to predict these properties with reasonable accuracy in unknown wood samples.

NIR Spectroscopy in Panels

Many investigations have demonstrated the potential of the combination of NIR spectroscopic and multivariate data analysis as a promising technique for characterizing wood products (Niemz et al. 1994; Muller et al. 2009). In regard to the prediction of mechanical properties by NIRS, some studies have tested the ability of this technique to estimate some properties in engineered panels. For instance, Rials et al. (2002) used NIR spectroscopy for predicting the mechanical properties of wood composites, while Meder et al. (2002) estimated veneer stiffness in mini-LVL panels. Our results confirm the above-mentioned studies, which demonstrated that it is possible to evaluate a range of properties of wood-based panels using near infrared spectroscopy and multivariate analysis techniques. However, how this technique is able to evaluate mechanical and physical properties in engineered products on the basis of vibrational spectroscopic analysis is still matter of debate. To generate a better understanding of why NIR spectroscopy is able to estimate mechanical properties in wood-cement panels by the PLS calibration, we used two types of analysis simultaneously: (i) we investigated the regression coefficients of the calibrations for MOR (Model 2) and IB (Model 7), and (ii) we analyzed the important absorption bands for wood, based on the table presented previously by Fujimoto et al. (2008). These authors evaluated NIR spectroscopy recorded in wood of *Larix* hybrids, and we used their information merely for comparative purposes.

The general plot of weighted regression coefficients against the wavelengths for MOR and IB, and the assignment of peaks in the NIR spectrum are presented in Fig. 4. The gray rectangles indicate the uncertainty limits from cross-validations using Martens' uncertainty tests (Westad and Martens 2000).

The $CaO.SiO_2$ and $2CaO.SiO_2$ molecules present in the Portland cement may affect the NIR spectra; however, their influence on the calibration and on the panel´s properties is outside the scope of this paper, since the cement content of each panel was the same (wood/cement ratio = 1/2.75).

The assignments of peaks in the NIR spectrum to specific wood components are indicated by numbered arrows in Fig. 4. Arrows 1, 2, 5, 6, and 7 indicate absorption bands for cellulose, while arrow 3 indicates the absorption band for aromatic groups in lignin. In regard to the cellulose, the peaks at 1366 nm (arrow 1) and at 2270 nm (arrow 7) represent the pure cellulose, the peaks at 1550 nm (arrow 2) and 2080 nm (arrow 5) represent the crystalline regions in cellulose, and the peak at 2140 nm (arrow 6)

represents the amorphous regions in cellulose. The arrow 4 indicates an absorption band for water.

Figure 1 shows a remarkable difference in absorbance values for manufacturing treatments at 1550 nm. This result suggests that variations in crystalline regions in cellulose are linked to the variations in the curing rate and compatibilization of wood with the cement, which reflects its mechanical performance (especially for internal bond).

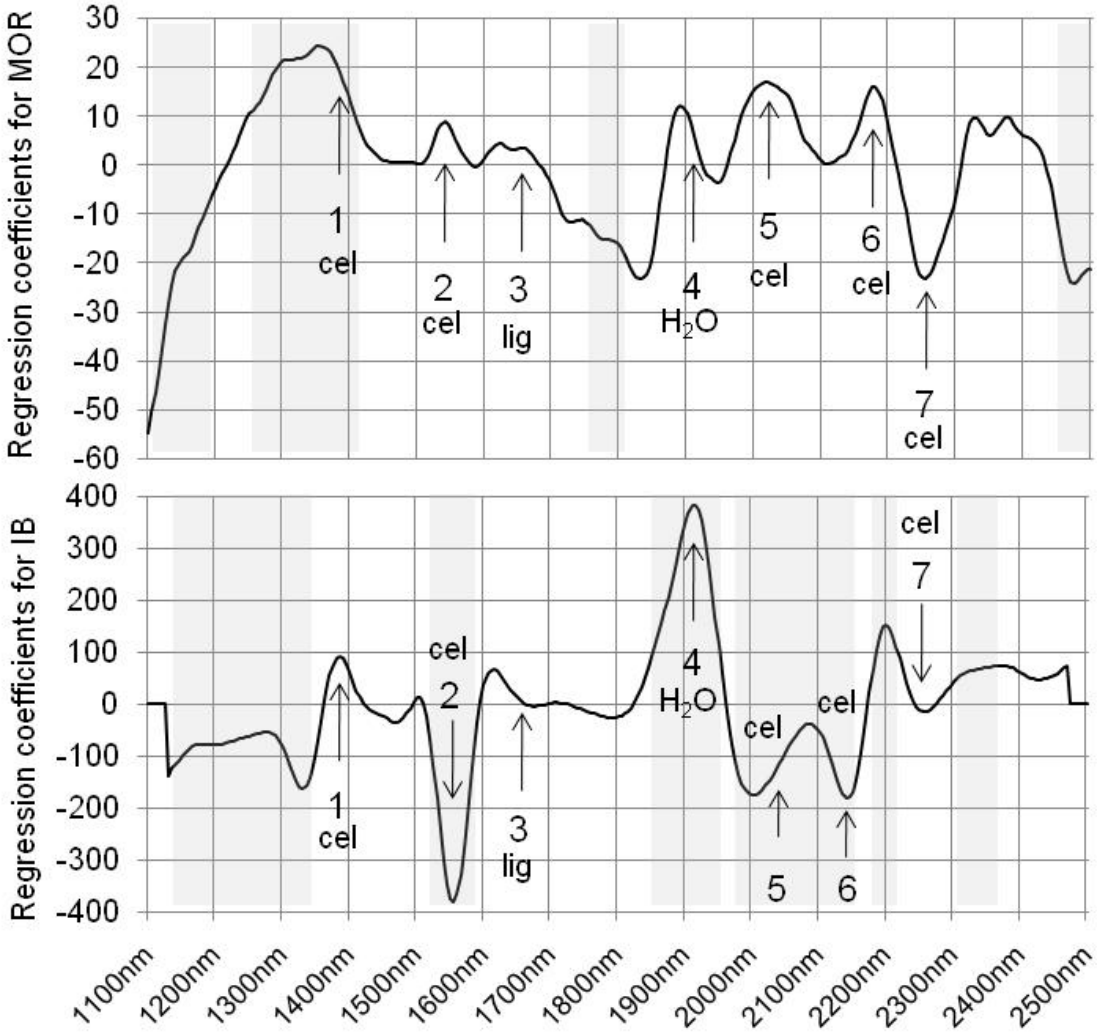

Fig.4. Significant absorption bands according to Martens' uncertainty test and regression coefficients of the models for modulus of rupture (using first derivative of NIR spectra) and internal bond (using untreated NIR spectra)

In addition, according the important absorption bands for wood along the wavelength axis (Fig. 4), cellulose seems to be a component of wood-cement panels that play a major role in the calibrations. There is evidence that the mechanical properties of the cement wood panels will change depending of the cellulose content of the wood used to manufacture it.

Particularly for the internal bond, the moisture content of the panels played a considerable role in the models (See Fig. 4 - 1940 nm). The panels were kept in an acclimatized room until stabilization, and the internal bond of the samples was measured under the same conditions; however, there were difference in moisture content between samples. We suppose that this difference in moisture content is due the interaction between wood and cement, which results in more or less water molecules within its complex chemical structure. The high coefficient regression in the water region of the NIR spectra (1940nm) indicated that the chemical interaction of wood and cement, which affects moisture content, is an important parameter for the internal bond of the panels.

The major limitation of the present work was the number of samples available for analysis. We had only six treatments, and only 3 panels were manufactured per treatment, totaling 18 wood-cement panels.

CONCLUDING REMARKS

These results demonstrated that NIR spectroscopy coupled with multivariate statistical analysis has the potential to evaluate some physical and mechanical properties in wood-cement panels. Using rough NIR spectra, our cross-validated PLS models for modulus of rupture and thickness swelling after immersion for 24h of the wood-cement panels yielded good coefficients of determination (0.80 and 0.91, respectively), while for internal bond, the calibrations developed using surfaced NIR spectra showed the best statistics ($R^2cv=0.82$). These results confirm that the near infrared spectroscopy technique can be a useful tool in order to evaluate MOR, TS24h, and IB quickly and suitably by NIR calibrations. On the other hand, the NIR spectra measured on wood-cement panels did not provide acceptable predictive models for modulus of elasticity, compression, and water absorption after immersion for 24h. Based on the significant absorption bands and regression coefficients of the models, our results indicate that cellulose is a component that plays an important role in the calibrations for modulus of rupture and internal bond. However, further studies are indispensable to generate a better understanding of how this technique can evaluate mechanical and physical properties on the basis of vibrational spectroscopic analysis.

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