

## NEAR INFRARED SPECTROSCOPY TO EVALUATE COMPOSITION OF AGRO-BASED PARTICLEBOARDS

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Particleboards can be manufactured from particles of any lignin-cellulosic material that can be combined with an adhesive and consolidated under the action of temperature and pressure. Because the raw materials in the industrial process are continually changing, the particleboard industry requires methods for monitoring the quality of their products. Hence, the aim of this paper was to evaluate the composition of the agro-based particleboards by near infrared spectroscopy. In this study, agro-based particleboards produced with different compositions of *Eucalyptus* and *Pinus* wood particles and sugar cane bagasse were evaluated by NIR spectroscopy and partial least square (PLS) regression. The PLS models to estimate the *Eucalyptus* and *Pinus* particles and sugar cane bagasse contents presented a strong coefficient of determination (0.90, 0.88 and 0.84, respectively), but also high magnitudes of standard errors of cross-validation were observed (ranging from 8.84 to 11.27%). Development work would be required in order to reduce the standard errors and improve predictive model performance to build robust models that could be applied as quality control tool.

*Keywords:* Near infrared spectroscopy; *Eucalyptus*; *Pinus*; Cane bagasse; Particleboard; Calibration; Composition

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### INTRODUCTION

Wood is frequently modified by an engineering process to give stiffness or homogeneous mechanical properties (Tsuchikawa 2007). Agro-based particleboards are manufactured from various lignin-cellulosic materials, usually wooden and mainly in the particle form, which are combined with an adhesive that consolidates under the action of temperature and pressure (Rowell et al. 2000). The advantages of particleboards are mainly attributed to their homogeneous structure and use of raw materials without restrictions as to shape and size. The main factors that influence the properties and quality of the panels are the density of the panel, geometry, and moisture content of the particles, the pressing cycle, and the quantity and type of adhesive (Kelly 1977; Maloney 1993). The raw material in the process is always changing due to variations in the relative amount of wood species, differences in moisture content, storage effects, etc. Therefore,

the product engineering industry, such as that of particleboards, requires new solutions for controlling the quality of their products in order to maintain the same quality standard.

A possibly suitable technique for characterization of variability in biomaterials is near infrared (NIR) spectroscopy (Burns and Ciurczak 2001). NIRS is a non-destructive technique used for rapid assessment of some properties in biological materials. The technique requires minimal or no sample preparation and measures the interaction of electromagnetic radiation with matter (Pasquini 2003). The tendency of the material to absorb different wavelengths of light depends on various vibrational modes of the component molecules. The method involves collecting the NIR spectra of a set of characterized samples, developing a regression equation [commonly using partial least squares (PLS) regression] and using the equation to predict the properties of further samples (the validation set) based on their NIR spectra (Jones et al. 2008). NIR radiation interacts with polar molecules and especially with O-H structural groups such as in water, C-H as in biomass, but also with C-O and C-O-H bonds and C=C double bonds frequently found in biomass. Biomass contains mostly C, O, and H atoms. Thus, reflectance in the NIR wavelength region senses most of the covalent bonds in biomass feedstocks, except for the C-C bonds in carbon chains (Lestander et al. 2008).

The combined approaches of Near Infrared Spectroscopy and Multivariate Analysis can make it possible to monitor raw material changes and correlate them to variations in the quality of the final product such as plywood (Sjoblom et al. 2004). For this reason, Tsuchikawa et al. (2003) suggested the application of NIRS for a reasonable classification of wood used at the factory. Thus, engineered wood products (for example, plywood, particleboard, etc.) are being manufactured for which NIR spectroscopy could be utilized as an online process control (Tsuchikawa 2007). Possibly the first study involving NIRS and engineered wood products was published by Kniest (1992), who used NIR spectroscopy to characterize wood particles treated with urea resin. Two years later Niemz et al. (1994) evaluated resin content in chip mixtures by NIR-spectroscopy. Currently some scientific contributions have shown the power of combining spectroscopic data with multivariate data analysis as a rapid and reliable tool for characterizing agro-based particleboards products (Meder et al. 2002; Rials et al. 2002; Dolezel-Horwath et al. 2005; Kelley et al. 2005).

In the particleboards industry panels are being produced using cotton stalks, jute, grain plant stalks (corn wheat, etc), and other plant resources (Rowell et al. 2000). In the present work, agro-based particleboards were produced from *Eucalyptus* and *Pinus* wood particles and sugar cane bagasse under different manufacturing conditions. The work was done in order to evaluate the ability of NIR spectroscopy techniques to classify and predict the composition of the agro-based particleboards.

## **MATERIAL AND METHODS**

### **Preparation of Agro-Based Particleboards**

Table 1 shows the composition classes used to produce the agro-based particleboards with different combinations of three raw materials. Softwood, hardwood,

and cane bagasse were obtained from experimental plantations located at the southeast of Minas Gerais State, Brazil. Wood particles were sampled from *Pinus oocarpa* (31 years old and average basic density of 0.50 g/cm<sup>3</sup>) and *Eucalyptus urophylla* (8 years old and average basic density of 0.56 g/cm<sup>3</sup>). The raw material underwent milling, and the slivers produced were classified at the mill by a shaker (0.8 x 0.8 mm) to remove most of the fines. The slivers were dried to a mean moisture content of 3% in a convection oven.

Different proportions of cane bagasse, *Pinus*, and *Eucalyptus* slivers were combined into seven composition classes of particleboards, according to Table 1. For each class, three levels of phenol-formaldehyde and urea-formaldehyde resin (6, 9, and 12% of the oven-dry weight of the agro-based slivers) were applied to the slivers using a rotary blender. Three replicates per resin level were produced. The total press time was 8 minutes at 180 °C (for phenol-formaldehyde) and 160 °C (for urea-formaldehyde), and pressure of 40 kgf/cm<sup>2</sup>. The target board density and dimensions for all panels were 0.70 g/cm<sup>3</sup> and 480 x 480 x 15 mm, respectively.

From each panel, two samples were removed for NIR scanning. The specimens were sawn from the center area of each panel and stabilized in an acclimatizing room, at 20 ± 2 °C and 60 ± 3% relative humidity, where they were also submitted to spectral scanning. Under these conditions the moisture content of the samples stabilized at 12%. A total of 252 samples were analyzed: 7 composition combinations x 2 resins x 3 resin levels x 3 replicates x 2 samples.

**Table 1.** Composition of Agro-Based Particleboards for Each Treatment

Composition Class	Resin	Content (%)		
		C	P	E
1-C75P25	Phenol-Formaldehyde /Urea-Formaldehyde (6, 9, and 12%)	75	25	0
2-C50P50		50	50	0
3-C25P75		25	75	0
4-C75E25		75	0	25
5-C50E50		50	0	50
6-C25E75		25	0	75
7-C100		100	0	0

\*C (cane bagasse); P (*Pinus oocarpa*) and E (*Eucalyptus urophylla*).

By applying different adhesives and different concentrations into a same composition class (our reference values), we tried to simulate what would be a realistic approach to NIR spectroscopy coupled with multivariate statistical analysis applied as an online, real time quality control tool. These heterogeneous classes are more likely to build robust predictive models that are needed for such applications.

### NIR Spectra Acquisition

The NIR spectra were measured in the diffuse reflectance mode in a FEMTO spectrophotometer (NIR 900 PLS model) in a climatized room with temperature around 20°C and relative humidity around 65%. This Fourier transform spectrometer is designed for reflectance analysis of solids with an integrating sphere. The NIR spectra were

obtained at 5 nm intervals over the wavelength range of 1100 nm to 2500 nm. NIR spectral information was taken directly from the sample surface. Four scans were measured and averaged for each sample. This procedure involved turning the sample in the equipment measuring cell so that the light beam would capture different spots of the heterogeneous surface, providing more representative averaged spectrum of the sample and reducing the data noise.

### **Calibration Statistics**

Principal Component Analysis (PCA) and Partial Least Squares (PLS) regression analyses were performed using the Unscrambler (CAMO AS, Norway) software, version 9.2. The PLS calibrations were developed in order to describe the relationship between the NIR spectra and particleboards properties. PCA and 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, and the outlier samples were identified from the student residuals and leverage value plot analyses. The outlier detection and the selection of the number of PLS factors follow the suggestion described in Hein et al. (2009). In order to enhance the quality of the calibration adjustment, the significant wavelengths were selected by the Martens uncertainty test (Westad and Martens 2000), and mathematical pretreatments including first and second derivatives (Savitzky and Golay 1964) were applied to the NIR spectra data.

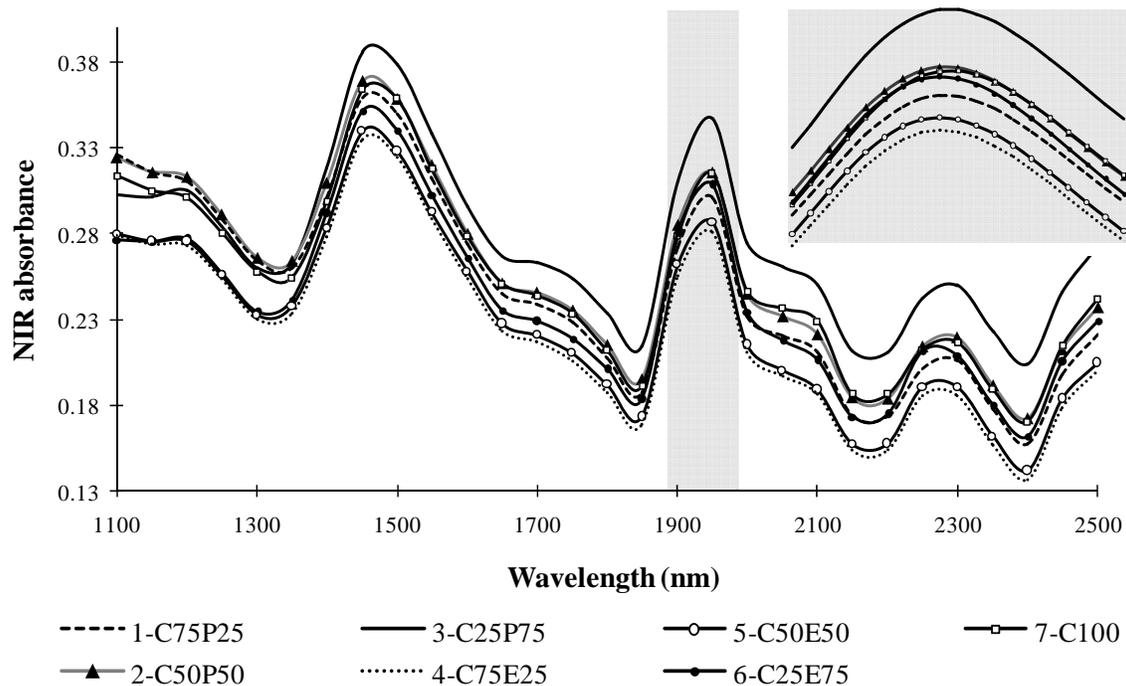
### **Selection of the Predictive Model**

The parameters  $R^2_{cv}$  - coefficient of determination of the cross-validation, SECV - standard error of cross-validation, and RPD value (ratio of performance to deviation) were used as selection criteria to select the predictive model to estimate the particleboards composition, as suggest by Jones et al. (2008). Formulas used to estimate the SEC and SECV are given in Schimleck et al. (2001) and should be as low as possible, while the coefficient of determinations should be high. The RPD value is the ratio of the SEP to the standard deviation (SD) of the original data. This statistic provides a basis for standardizing the SEP (Williams and Sobering 1993) and makes it possible to compare different calibration parameters such as spectra information obtained from different agro-based particleboards. Williams and Sobering (1993) state that a RPD greater than 2.5 is considered satisfactory for screening, although it has been shown that with RPD of approximately 1.5 indicates that NIR spectroscopy can be used as an initial screening tool (Schimleck et al. 2003).

## **RESULTS AND DISCUSSION**

### **NIR spectral Information**

The NIR spectral information consists basically of overtones and combination bands of fundamental molecular vibrations, especially stretching and bending, of O-H, N-H and C-H functional groups (Workman and Weywer 2007). Figure 1 shows original spectra data obtained from agro-based particleboards with the NIR spectrometer. Each spectrum represents the averaged spectra of each treatment.



**Fig. 1.** Original NIR spectral data from agro-based particleboards

The absorbance spectra measured on the particleboard surfaces showed interesting results (Fig. 1). The panels manufactured with different contents of cane bagasse and *Pinus* wood (1-C75P25; 2-C50P50; 3-C25P75) presented higher absorbance values when compared to *Eucalyptus* based panels (4-C75E25; 5-C50E50; 6-C25E75). On both *Pinus*- and *Eucalyptus*-based particleboards, the increased content of wood (or decreased content of cane bagasse) present in panels composition seemed to augment the absorbance value throughout the NIR spectral range. Panels produced only with cane bagasse (treatment 7-C100) showed an intermediate absorbance value.

As a complex material, particleboards can be described as a combination of different kinds and contents of chemical substances, anatomical tissues, and solid materials. NIR spectra from such a complex surface are consequently not merely driven by the underlying chemistry, but also by the solid structure (Gierlinger et al. 2004). The absorbance values (Fig. 1) represent a great variety of interactions of the NIR radiation along the wavelength range and the particleboard's chemical and physical properties. Therefore, the spectral "finger print" reflects the energy captured (or reflected) by chemical bonds from different resins (phenol-formaldehyde and urea-formaldehyde) and resin levels, wood components nature and contents (cellulose, lignin, hemicelluloses, extractives), as well as their interactions. Spectra also represent the refraction and scattering of light due to surface roughness, particles size and shape, among other factors. This complex information demands sophisticated statistical tools, such as PCA and PLS, to allow proper interpretations and enhance NIR spectral data applications.

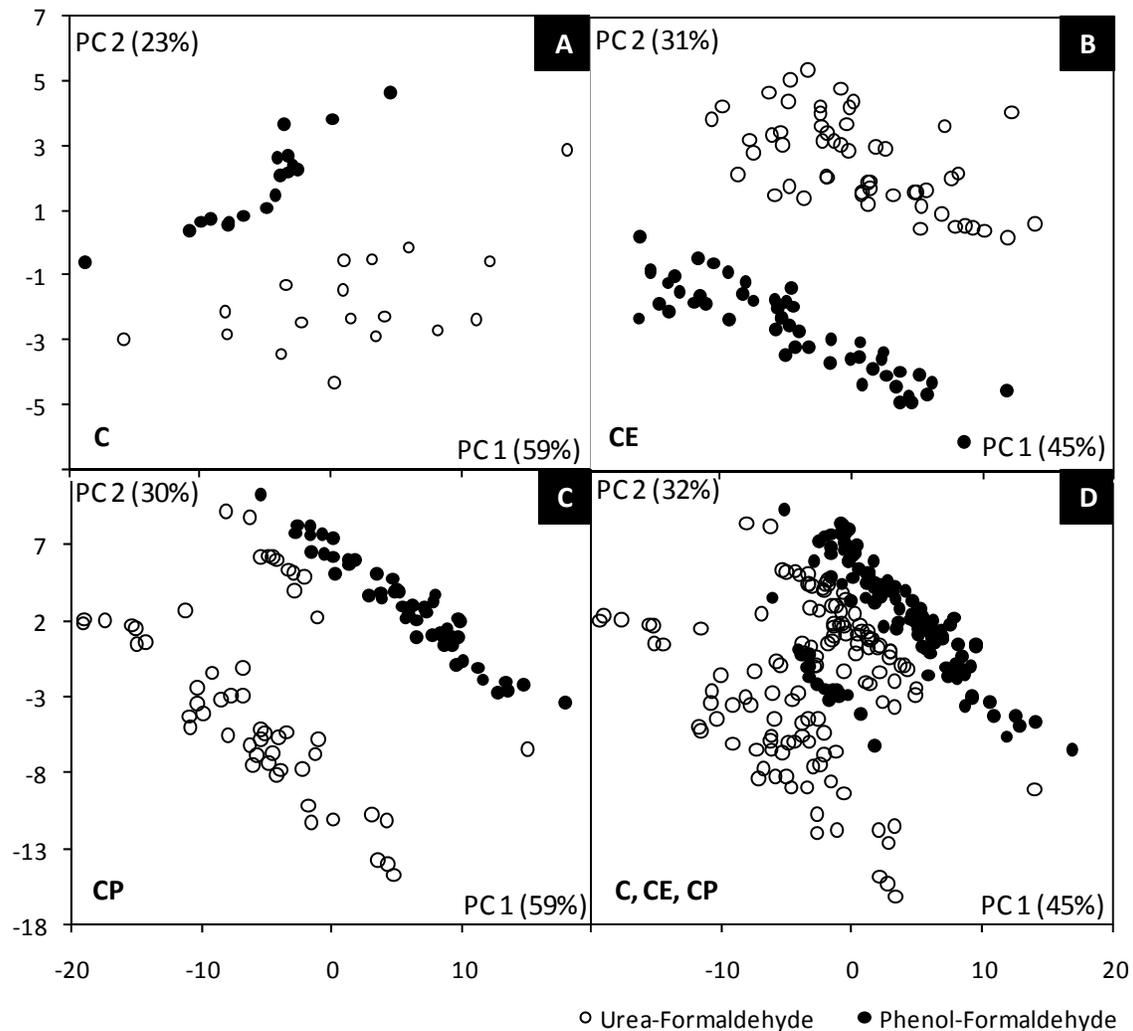
## Principal Component Analysis

Principal Component Analysis is the most common and versatile method to analyze NIR spectral information (Pasquini 2003). NIR spectral differences between the particleboards composition can be simply recognized by the application of principal component analysis (Fujimoto et al. 2008). These chemometric tools showed promising results in a range of studies involving engineering products (Dolezel-Horwath et al. 2005; Kelley et al. 2005). For instance, recently Muller et al. (2009) evaluated particleboards and medium density fibreboard (MDF) of *Abies grandis* [Douglas ex D. Don] lindl. and *Fagus sylvatica* L., employing fourier transform infrared (FTIR) attenuated total reflectance spectroscopy in combination with multivariate statistical methods in order to investigate the homogeneity of the wood-based panels. They showed that principal component analysis of FTIR spectra was able to discriminate wood, fibers, particles, MDF and particleboards of both species during the production process for panels.

This qualitative analysis can be used to explore information from NIR spectra measurements, and it is useful to remove redundant data. They also allow the visualization of the majority of significant data in two or three-dimensional spaces (Ciosek et al. 2005). In this study, the particleboards were divided into three classes to perform PCA: C with panels composed by only bagasse cane; CE representing panels composed by different contents of bagasse cane and *Eucalyptus* wood chips; and CP composed by panels manufactured by different contents of bagasse cane and *Pinus* wood chips. Figure 2 shows the two-dimensional scatter plots of scores for PC 1 and PC 2 from PCA of the first derivative spectra data for each of the three classes (A to C) and for all the 252 particleboards samples (D). The principal component (PC) 1 and 2 accounted for 76 to 89% of the NIR spectra variability.

The plots of PC scores (Fig. 2) presented clusters among the particleboard samples due to their resin (phenol-formaldehyde and urea- formaldehyde). However, the discrimination of the type of resin or particleboard composition (CE, CP and C) is not clear in Fig. 2-D. These graphics allow one to infer that the major amount of variability of the NIR spectra data is mainly influenced by the resin type, which is the response of NIR absorbance by the chemical bonds present in both adhesives.

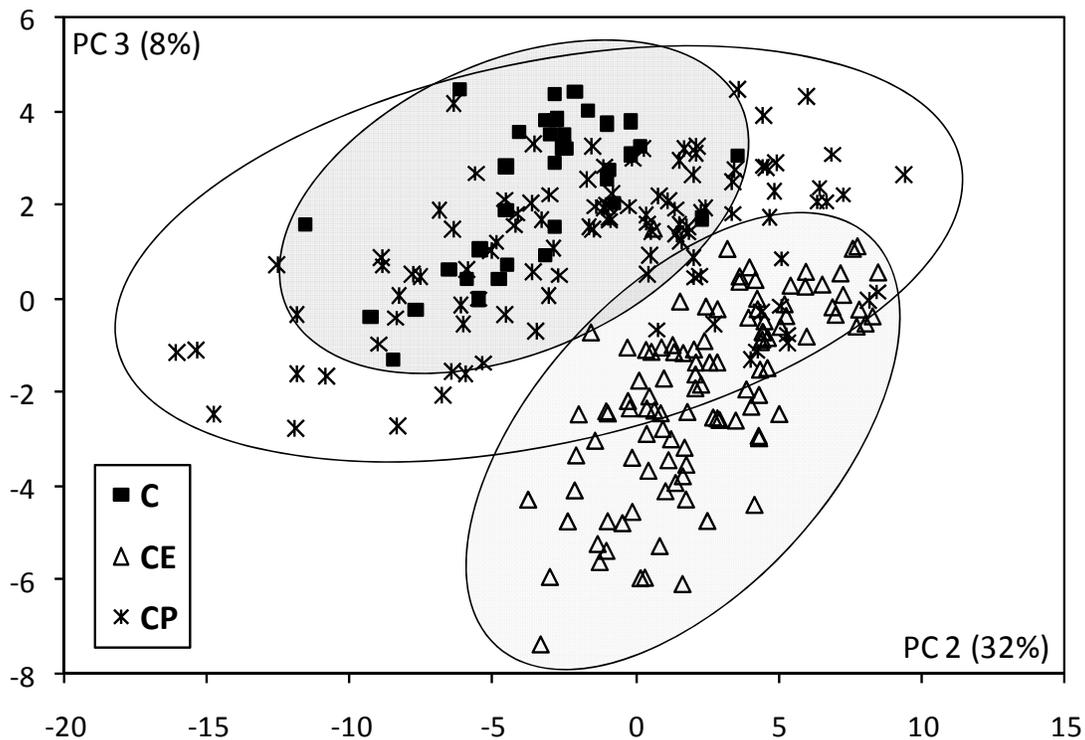
In this study, three principal components accounted for 85% of the total NIR spectra variability (PC 1 with 45%, PC 2 with 32%, and PC 3 with 8%). Figure 3 shows the scores plot for PC 2 and PC 3, in which two classes of particleboards (C and CE) were easily distinguishable from each other, with clearly different patterns of response. However, the CP particleboards manufactured were not discriminated from the first well-distinguished clusters. Figure 3 brings some insight that the composition nature of the particleboards may be partially responsible for the third major amount of variability within NIR spectra data. However, it was not sufficiently strong to allow PCA analysis to discriminate the composition classes accurately. Spectral information along the NIR range can be further analyzed with more powerful tool, such as PLS-R model, in order to find spectral variability related to agro-based particleboard composition.



**Fig. 2.** Two dimensional scatter plots of scores for PC 1 and PC 2 from principal component analyses of the first derivative spectra data of the particleboards made of pure cane bagasse (A), cane bagasse and *Eucalyptus* wood (B), cane bagasse and *Pinus* wood (C), and all the agro-based particleboards produced (D)

### Partial Least Squares Regression

The PLS-R models were calibrated from the original NIR spectra data and after mathematical treatment of the spectra scanned from the panels. The statistics associated with calibration and cross-validation models to estimate the composition of the agro-based particleboards are reported in Table 2.



**Fig. 3.** – Two-dimensional scatter plots of scores for PC 2 and PC 3 from principal component analysis of the first derivative spectra data of the particleboards

**Table 2.** Summary of NIRS Calibration to Predict Agro-Based Particleboards Composition

Biomaterial	Treat	$R^2_c$	SEC	$R^2_{cv}$	SECV	LV	Outliers	RPD
<i>Eucalyptus</i>	none	0.89	9.416	0.87	9.93	8	5 (1.98%)	2.8
	1der	0.91	8.619	0.90	9.06	6	4 (1.58%)	3.1
	2der	0.92	7.854	0.90	8.84	7	4 (1.58%)	3.2
<i>Pinus</i>	none	0.89	9.126	0.87	9.93	10	6 (2.38%)	2.8
	1der	0.89	9.140	0.88	9.85	7	1 (0.39%)	2.9
	2der	0.89	9.444	0.87	10.14	8	1 (0.39%)	2.8
<i>Cane bagasse</i>	none	0.83	10.507	0.81	11.12	7	4 (1.58%)	2.3
	1der	0.85	9.837	0.84	10.46	6	3 (1.19%)	2.5
	2der	0.82	10.841	0.81	11.27	5	1 (0.39%)	2.3

Treat - pretreatment applied; 1d - first derivative; 2d - second derivative;  $R^2_c$  - coefficient of determination of the calibration model (%); SEC - standard error of calibration (%);  $R^2_{cv}$  - coefficient of determination of the cross-validation (%); SECV - standard error of cross-validation (%); LV - number of latent variables and RPD - Ratio of Performance to Deviation.

The *Eucalyptus* particle content and the spectral data presented strong coefficients of determination, ranging from 0.87 (raw NIR spectra data) to 0.90 (first and second derivatives). The RPD of these models ranged from 2.8 to 3.2, and the standard errors of cross-validation were lower when compared to those of the PLS-R models to estimate *Pinus* wood and sugar cane content. The calibrations to predict *Pinus* particle content were similar to those obtained using *Eucalyptus* particles. The  $R^2$  of the *Pinus* particle content cross-validated PLS-R model ranged from 0.87 to 0.88 with a RPD value ranging from 2.8 to 2.9. Although the associated statistics seems to be quite close, it is important to note that raw NIR spectra data required 10 latent variables and the removal of 6 outliers against 7 latent variables and 1 outlier from the first derivative PLS-R model. The standard errors of cross-validation to estimate *Pinus* content were, comparatively, higher (9.85 to 10.13) than *Eucalyptus* chip content models (8.84 to 9.92). The cross-validation to predict sugar cane bagasse content gave, comparatively, weaker statistics ( $R^2=0.81$  to 0.84; SECV=10.45 to 11.27 and RPD=2.3 to 2.5).

The RPD value relates the SECV to the standard deviation of the original reference data. According to Williams and Sobering (1993) the RPD value should be as high as possible, and values of 5 to 10 are adequate for quality control. However, RPDs of this magnitude are rarely encountered in NIR analysis from lignin-cellulosic materials. The RPD value of 1.0 means that the SECV and the SD are the same, and the instrument is not capable of predicting the parameter accurately, using that calibration (Williams and Sobering 1993). In breeding programs, an RPD value of 2.5 or higher is satisfactory for screening, but to control the panel production quality we did not find any reference value. In this initial work, the calibration models presented RPD values ranging from 2.3 to 3.2.

The statistics associated with calibration and cross-validation PLS-R models were affected after mathematical pretreatments (first and second derivatives) of the NIR spectral data. This can be explained because, although diffuse reflectance measurement of solid samples is a distinguishing measurement mode employed in NIR spectroscopy (Pasquini 2003), in diffuse reflectance, scattering and absorbance by solid granules contribute to change the signal intensity. Hence, to solve these problems, mathematical pretreatments on the NIR spectra data were performed.

According to Delwiche and Reeves (2004) the performance of PLS regression equations that utilize NIR spectra data benefits from a mathematical transformation of the spectra before the PLS processing. In this study, the mathematical treatment in the spectra improved the *Eucalyptus* particle content prediction, raising the coefficient of determination from 0.87 (raw NIR spectra data) to 0.90 (first and second derivatives), decreasing the SECV from 9.93 to 8.84 and improving the RPD from 2.8 to 3.2. Schimleck et al. (1999), Jones et al. (2005), and Hein et al. (2009) reported the same effect when applied to mathematical treatments in the NIR spectral information.

At first sight, the high values presented by the coefficients of determination (0.81 to 0.90) seem to indicate powerful predictive models, but the standard errors of cross-validation also presented high magnitudes (ranging from 8.84 to 11.27%). For example: the *Eucalyptus* particle content and the NIR spectral data presented the strongest coefficients of determination (0.90) and standard error of cross-validation of 9.06. Although this PLS-R model presents a high  $R^2_{cv}$ , it would allow a prediction uncertainty of ~30% to ~70% of *Eucalyptus* wood content for a panel with 50%. To enhance the

performance of such predictive PLS-R models, more samples would be required with gradual content rising (from 0 to 100% for each component) for the calibration step, in order to reduce standard error.

## CONCLUSIONS

These results suggest that NIR spectroscopy coupled with multivariate statistical analysis has the potential to classify and quantify agro-based particleboards composition. The high values of standard error of cross-validation, encountered for the calibrated PLS-R models within this work, limit their application as predictive models for the composition of panels. In order to reduce the standard errors and improve predictive model ability, we recommend the following: i. the enlargement of the reference samples within the range of composition and in smaller intervals (i.e. 0, 5, 10...100%); ii. ground and screen particleboard samples to NIRS scanning in order to reduce the spectral noise. Development work would be required to build robust models that could be applied as quality control tools.

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