NEAR INFRARED SPECTROSCOPY FOR ESTIMATING SUGARCANE BAGASSE CONTENT IN MEDIUM DENSITY FIBERBOARD

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Medium density fiberboard (MDF) is an engineered wood product formed by breaking down selected lignin-cellulosic material residuals into fibers, combining it with wax and a resin binder, and then forming panels by applying high temperature and pressure. Because the raw material in the industrial process is ever-changing, the panel industry requires methods for monitoring the composition of their products. The aim of this study was to estimate the ratio of sugarcane (SC) bagasse to *Eucalyptus* wood in MDF panels using near infrared (NIR) spectroscopy. Principal component analysis (PCA) and partial least square (PLS) regressions were performed. MDF panels having different bagasse contents were easily distinguished from each other by the PCA of their NIR spectra with clearly different patterns of response. The PLS-R models for SC content of these MDF samples presented a strong coefficient of determination (0.96) between the NIR-predicted and Lab-determined values and a low standard error of prediction (~1.5%) in the cross-validations. A key role of resins (adhesives), cellulose, and lignin for such PLS-R calibrations was shown. PLS-DA model correctly classified ninety-four percent of MDF samples by cross-validations and ninety-eight percent of the panels by independent test set. These NIR-based models can be useful to quickly estimate sugarcane bagasse vs. *Eucalyptus* wood content ratio in unknown MDF samples and to verify the quality of these engineered wood products in an online process.

Keywords: MDF panel; Fiberboard, Sugarcane bagasse; Eucalyptus; NIR spectroscopy; Principal component analysis; Partial least square regression; Discriminant analysis

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INTRODUCTION

The use of natural fibres as a complement for wood composites has generated much interest due to their low cost, concerns about environmental protection, and the possibility to use locally available renewable resources. Dry sugarcane bagasse is usually used as a fuel for the sugar factory, but the heating value is low (Han et al. 2010). In this context it makes sense that engineering lignocellulosic panels are being manufactured using this residue as a renewable alternative.

Several applications involving NIR spectroscopy have been proposed, making this technique a promising tool for evaluating lignocellulosic products (Tsuchikawa 2007). Recent studies have confirmed the potential of NIR spectroscopy to evaluate a range of wood traits, including mechanical (Fujimoto et al. 2008), physical (Mora et al. 2008), chemical properties (Zahri et al. 2008; Kelley et al. 2005), and morphological features (Viana et al. 2009). Currently, the pulp and paper industry uses such a tool for screenings and for selecting candidate genotypes or clones in breeding programs.

For industrial products based on lignocellulosic materials, a range of applications for NIR spectroscopy have been proposed. For instance, Rials and Kelley (2002) applied NIR spectroscopy for predicting the mechanical properties of wood composites. Horwath et al. (2005) tested this tool to control the manufacturing process of wet-processed hardboards. Hein et al. (2009) estimated physical and mechanical properties of wood-cement panels from NIR spectroscopic data. Taylor and Via (2009) applied NIR models for quantifying resin content in oriented strand boards (OSB). Campos et al. (2009) used the technique to determine the percentage of raw material in particleboards manufactured with different percentages of fibers of *Eucalyptus*, pine and sugarcane bagasse. Continuing this work, Hein et al. (2011) used these particleboards samples to develop NIR calibrations for mechanical and physical properties. Muller et al. (2009) successfully employed infrared attenuated total reflectance (IR-ATR) to investigate the homogeneity of particleboards and medium density fiberboard (MDF). These studies presented encouraging results, indicating the potential of NIR spectroscopy as a tool for quality control in manufacturing processes.

While countless studies have shown the potential of combining spectroscopic data with multivariate data analysis as a rapid and reliable tool for characterizing engineered wood products (Hein et al. 2011), there is a gap between academic studies and industrial applications (Hubbe 2010). However, further research is still required at the laboratory scale to discover the fundamental relationships and processes of using NIR to monitor SC bagasse composites. Successful methodology development in the laboratory will help to define the fundamental process necessary for calibration as well as justify the capability of utilizing NIR for quality control. Thus the aim of the present study was to evaluate the suitability of NIR spectroscopy to predict SC bagasse particle content in *Eucalyptus* medium density fibreboards.

Here, MDF panels were manufactured with different compositions of sugar cane and *Eucalyptus* fibers, and NIR spectra were measured. Then, Principal Component Analysis (PCA), Partial Least Square (PLS) regressions and PLS-Discriminant Analysis (PLS-DA) were performed, presented, and discussed.

EXPERIMENTAL

Collect and Prepare of Sugarcane Bagasse and *Eucalyptus* Fibers

Samples of sugarcane (SC) bagasse were collected at the sugarcane plant Usina Açucareira São Manuel S.A. (São Manoel, São Paulo, Brazil), stored in plastic containers, and oven-dried at 105 °C, resulting in a 5% moisture content. This prevented the development of microorganisms. The oven-dried samples were screened in a

Produtest, a vibratory sieve shaker, and the fraction bellow 2.0 mm representing 60% of the bagasse, were used for panel manufacture.

Samples of *Eucalyptus* fibers (thermo-mechanical pulp) were collected in an industrial line process at the Duratex S.A, in Botucatu (São Paulo, Brazil) after refining and previous to the addition of resin and wax. The refining conditions of *Eucalyptus* wood chips were: (i) heating time = 4 min, (ii) refiner pressure = 8.0 bar, and (iii) specific energy consumption = 100 kWh/t.

Medium Density Fiberboard Manufacture

The *Eucalyptus* fibers and bagasse particles were dried (70 °C; 2-3% moisture content) and weighed in different fractions according to the treatments (Table 1), including the urea formaldehyde resin and wax emulsion. In sequence, these materials were transferred to the blender, and urea formaldehyde resin and wax emulsion were pulverized over the sugarcane particles-eucalyptus fibers through spray nozzles and processed with the fiber mass mixture for 3 minutes, aiming to reach 7±0.5% final moisture.

The fiber mat was formed in a square wooden deckle frame that was resting on an aluminum plate, and these materials were transferred to a hydraulic system for removing air. The fiber mats were disposed and pressed in an experimental Siempelkamp press with pressure control for panel thickness. The pressure levels and timing were: 0 to 100 bar (10 s); 100 bar (5 s); 100 to 20 bar (20 s); 20 to 10 bar (20 s); 10 bar (70 s); 10 to 30 bar (10 s); 30 bar (50 s); and 30 to 0 bar (5 s), at constant temperature (190 °C) to manufacture the panels. The wax content was 0.8% for all treatments. The pressed fiberpanels were transferred at room temperature (tabs were placed between them to allow cooling) and cut at pre-fixed dimensions (15 x 370 x 370 mm) with variable densities, as indicated in Table 1.

Table 1. Composition of the MDF Panels Indicating the Density (kg m^{-3}), Resin Content and Fiber (SC, sugarcane and E, Eucalyptus) Composition for each Treatment

Treat.	Panel density	Resin content	Fiber Content		Treat.	Panel density	Resin content	Fiber Content	
			SC	*E*				SC	*E*
1	751	13%	-	100%	7	763	13%	15%	85%
2	763	16%	-	100%	8	793	16%	15%	85%
3	722	13%	5%	95%	9	755	13%	20%	80%
4	767	16%	5%	95%	10	760	16%	20%	80%
5	750	13%	10%	90%	11	764	13%	25%	75%
6	795	16%	10%	90%	12	775	16%	25%	75%

Two MDF panels per treatment were manufactured for 12 treatment combinations (Table 1). For each panel, three specimens measuring 50 mm x 50 mm x 15 mm were sawn from the center of each panel. The samples were conditioned in an acclimatized room (20±2 °C and 60±3% relative humidity), where they were also submitted to NIR spectroscopic analysis. Under this condition, the equilibrium moisture content of the panels was 7%.

NIR Spectra Measurements

NIR spectra were measured in the diffuse reflectance mode with a Bruker spectrometer (model Vector 22/N, Bruker Optik GmbH, Ettlingen, Germany). This Fourier transform spectrometer is designed for reflection analysis of solids with an integrating sphere (diameter of measured area = 10 mm). Spectral analysis was performed within the 12,500 to 3,500 cm^{-1} (800 to 2,850 nm) range at 8 cm^{-1} resolution.

A sintered gold standard was used as reference or as background. Sixty-four (64) scans were performed and averaged for each measure. These were compared to the standard in order to obtain the reflectance spectrum of the sample. Averaging 64 spectra into one single spectrum improves the signal to noise ratio.

Seventy-two (72) specimens (six samples per treatment) were subjected to NIR analysis, scanning in four conditions, which were labeled: (i) press surface; (ii) rough lateral surface; (iii) lateral surface after sanding, and (iv) ground panels, according Fig. 1. Three NIR spectra were recorded on the press line face; two NIR spectra on the rough and on sanded lateral surface and six NIR spectra were recorded from the ground panel. A spinning cup was used for the NIRS measurements on ground samples. The lateral surface of the panel samples were manually and softly sanded with 300-grit sandpaper for approximately 45 seconds in order to produce a polished and homogeneous surface. The MDF samples were ground in a Retsch rotating-knife grinder (SM 100).

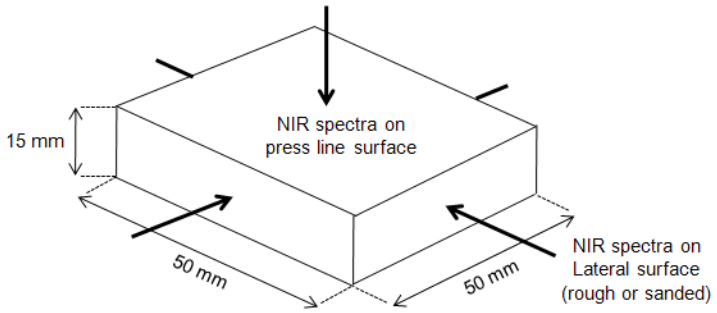

Fig.1. MDF sample dimensions and NIR spectra measurements

Chemometric Analysis

Principal Component Analysis (PCA), Partial Least Squares (PLS) regression, and PLS-DA (discriminant analyses) were performed using the Unscrambler (CAMO AS, Norway) software version 9.8. The PLS-R calibrations were developed in order to describe the relationship between the NIR spectra and sugarcane bagasse content of the MDF panels. PCA and PLS-R models were performed in full cross-validation mode with a maximum of 12 latent variables (LV). The selection of the model was based on the residual variance to number of latent variables (LV´s) plot. The model presenting the minimum residual variance and the optimal LV number was selected. In order to enhance the quality of the calibration adjustment and reduce noise, mathematical pre-treatments including Standard Normal Variate (SNV) transformation followed by second derivative (Savitzky and Golay 1964), de-trend, and combinations were applied to the NIR spectra. Moreover, the significant wavelengths for the PLS regressions were selected by the Martens uncertainty test (Westad and Martens 2000).

To evaluate the cross-validated PLS-R calibrations for SC bagasse by *Eucalyptus* wood content ratio, the following statistics were used: (1) coefficient of determination between measured and cross-validated values (R^2_{cv}); (2) root mean of standard error of cross-validation (RMSECV); (3) the bias, and (4) the number of latent variables (LV). The bias is useful to verify if there are systematic deviations between reference and NIR-based values.

The samples were discriminated by PLS-DA, supervised analysis (PCA is an unsupervised method), which were used to separate classes of objects according to their X-variables (NIR spectra). The six classes of SC were recorded as dummy variable. For each class, the samples belonging to the target class were coded as 1 and the samples belonging to the non-target class were coded as 0. Thus, PLS-2 were performed on the six variables (0, 5, 10, 15, 20, and 25%) coded by 0 and 1 simultaneously and to the pre-treated NIR spectra (SNV and second derivative) measured on the natural and sanded surfaces of the panels. The PLS-DA models were validated by cross-validation and by independent validation sets. Cross-validations were carried out on six groups (selected at random) to optimize the model, i.e., to find the number of latent variables that promotes the best classification rate. A sample was assigned to the target group when its predicted value fell around 1 for target class. The established PLS-DA model was validated by a test set represented by the spectra recorded on press line face of the same samples that were not used to build the PLS-DA models.

RESULTS AND DISCUSSION

NIR Spectra

Figure 2 shows the raw NIR spectra measured on the rough surface of the solid specimens from 9,000 to 4,000 cm^{-1}, the most informative range. Variation in NIR absorbance values between the MDF samples could be observed within this NIR range.

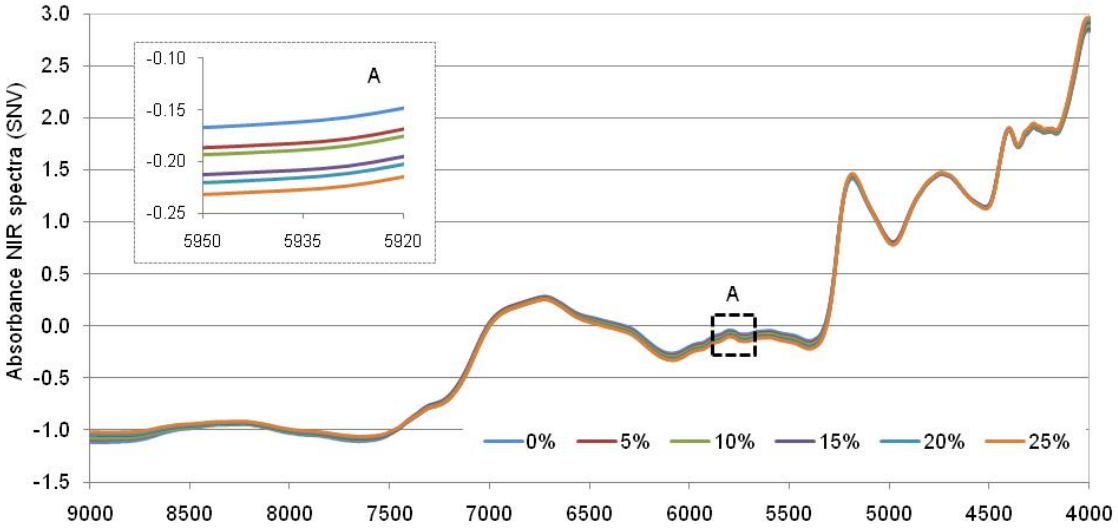

Fig. 2. NIR spectra after standard normal variate (SNV) transformation from 9,000 to 4,000 cm^{-1} obtained from solid panels.

The NIR spectra from the panels with low sugarcane (SC) bagasse showed low NIR absorbance values, while samples with high SC exhibited high absorbance along many parts of the NIR range. For instance, at the 5,920-5,950 cm^{-1}, band assigned to lignin content variations (Workman and Weywer 2007; chap. 10), the NIR spectra showed clear differences in their absorbance values according to the SC content (Fig. 2A). However, many ranges of the NIR spectra (for instance, at 4,600-4,900 cm^{-1}) showed contrasting trends. These differences reflect the variations of chemical composition of these panels, as well as the interaction of sugarcane bagasse, *Eucalyptus* fibers, and resin contents.

Principal Component Analysis

The complex information contained in the NIR spectra (Fig. 2) requires statistical tools to allow proper interpretations and enhance data applications. Principal Component Analysis is the most common and versatile method to analyze NIR spectra information (Rodrigues 2007). Differences between particleboards can be simply recognized by the application of principal component analysis in their NIR spectra (Fujimoto et al. 2008). Campos et al. (2009) applied PCA in order to discriminate composition classes in particleboards manufactured from particles of *Eucalyptus* and *Pinus* wood and SC bagasse. Here, the same approach was performed in medium density fiberboards produced with *Eucalyptus* fibers and bagasse particles.

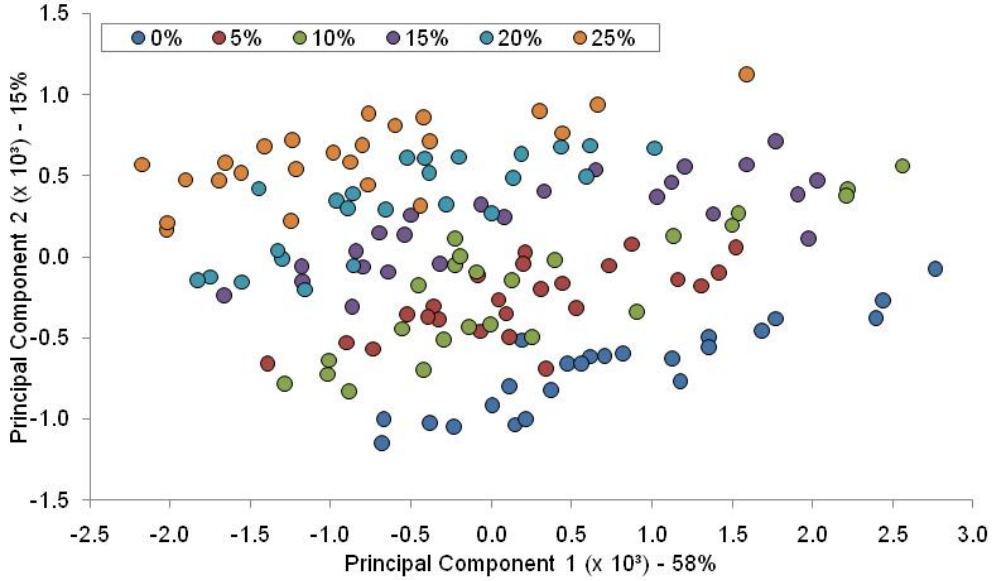

Fig. 3. Two dimensional scatter plots of scores for PC 1 and PC 2 from principal component analyses of the snv+d2 NIR spectra data obtained from solid panels (sanded lateral face) showing the six SC contents by colour. The sugarcane bagasse content % is indicated in the legend.

Figure 3 shows the two-dimensional scatter plots of scores for PC1 and PC2 from PCA of the snv+d2 NIR spectra data obtained from solid panels showing the six SC contents by colour. The principal component PC1 and PC2 accounted for 73% of the NIR spectra variability. Each panel composition (distribution of *Eucalyptus* and bagasse contents) was made using different resin content (13% or 16%). However, it was not

possible to distinguish differences between the classes of composition by means of the PCA analysis.

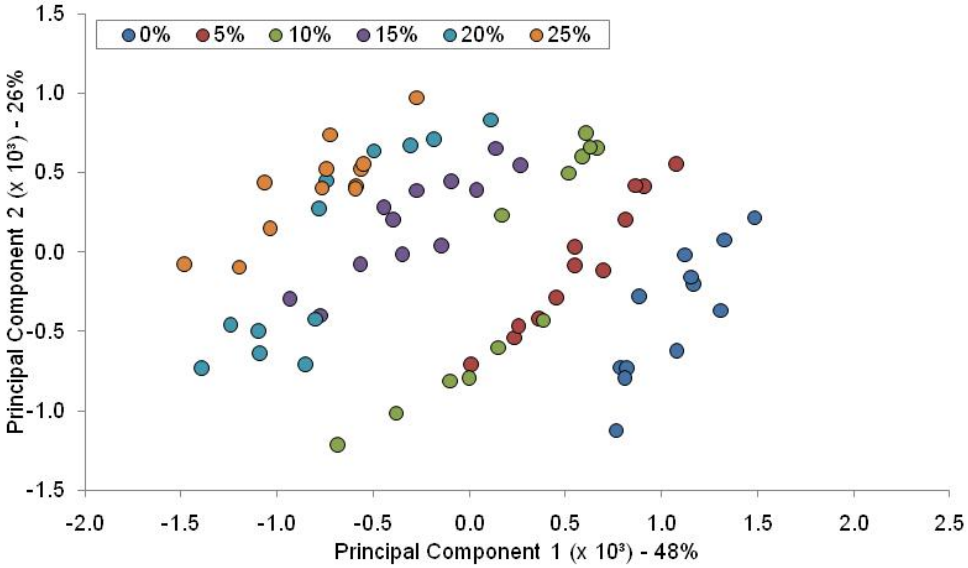

Fig. 4. Two dimensional scatter plots of scores for PC1 and PC2 from principal component analyses of the snv+d2 NIR spectra data obtained from ground panels showing the six SC contents by colour. The sugarcane bagasse content % is indicated in the legend.

Figure 4 shows the scores plot for PC1 (48%) and PC2 (26%) from the NIR spectra of the ground samples, in which the classes of MDF panels (1 to 6) were easily distinguished from each other with clearly different patterns of response. The plots of PC scores of the NIR spectra measured from both solid and ground MDF panel presented distinguishable clusters, but the partition among the MDF samples was clearer for the ground panels, likely due to the homogenization of their physical structure. In this study, the first three principal components (PC1, PC2, and PC3) accounted for 78% of the total NIR spectra variability for ground MDF samples.

Partial Least Regression Analysis

PLS-R models were developed in order to estimate the SC content of MDF panels from a range of NIR spectra measurements (press line, rough face, sanded face, and ground NIR spectra). The statistics associated with these cross-validated PLS-R models (Table 2) are promising.

The SC content in MDF panels presented strong coefficients of determination between NIR-predicted and laboratory-determined values. Based on the NIR spectra, the PLS-R models were able to estimate the bagasse content with a prediction error of approximately 1.5%. The sanded face NIR spectra yielded the best model statistics (R^2_{cv}=0.96 and RMSECV=0.97) due to its very low RMSECV; however, this model requires 4 latent variables. The reason for this result can be attributed to the physical effects due to the sanding operation. Perhaps the fibres become more exposed to NIR radiation after sanding. Such a supposed phenomenon would produce lower prediction deviations, but the coefficients of determination were similar for all types of NIR measurement. The influence of the roughness on the spectra is important issue because

the signal/noise quality increases as the roughness decreases. Figure 5 (A) shows the NIR-predicted versus Laboratory-determined plot for SC content for solid panels. While the press line NIR spectra yielded a higher error than sanded and a lower R-square than the ground samples, it still had a strong model; additionally, the higher error in measuring an individual sample can be offset by the ability to represent the high throughput of MDF panels in the industrial process with NIR. The error of prediction is for an individual sample, but in process control you are more interested in the population parameters. As such, using the press line spectra for industrial process control looks very promising. Moreover, the press line NIR spectrum is the type of NIR information more easily and quickly obtained.

Table 2. Cross-validated PLS-R Models for Sugar Cane Content Using a Range of NIR Spectral Measurements

NIR Spectral Measurement	R^2_{cv}	RMSECV	BIAS	LV	N
Press line face NIR spectra	0.96	1.59	0.082	5	72
Rough face NIR spectra	0.96	1.67	0.023	3	72
Sanded face NIR spectra	0.96	0.97	0.019	4	72
Ground MDF NIR spectra	0.97	1.58	0.011	3	72

For practical applications, NIR spectra could be directly measured in the MDF in order to verify the homogeneity of the products in an online process. The NIR spectra measured on the ground MDF samples gave slightly higher R^2_{cv} and lower RMSECV, and the NIR-predicted versus Laboratory-determined plot for SC content is presented in Fig. 5 (B).

The analysis of the loading plots of PLS-R calibrations is useful to investigate the underlying relationships that have made the estimation of bagasse content in MDF panels possible by NIR spectroscopy. The assignments of absorption bands (Fig. 6) are useful to identify which wood components were important for the modelling. It helps to understanding how NIR spectroscopy can evaluate MDF composition.

The bands at 5,300 cm^{-1} and 3,200 cm^{-1} yielded high regression coefficients. These bands are associated to O-H hydrogen bonding and were assigned to the moisture content of the MDF panels. The band at 4,970 cm^{-1} is associated to N-H/C=O combination from polyamide, while the band at 4,820 cm^{-1} is assigned to N-H/C-N combination band from the urea contained in the panel samples. The relatively high regression coefficient at 4,820 cm^{-1} indicates that resin also played an important role on PLS-R models for SG content. These findings confirm that at least a part of the variability of the MDF composition (and their NIR spectra) is attributed to the resin content.

The band at 4,412 cm^{-1} is associated to O-H/C-H bonds of cellulose (O-H and C-O) and represents the stretching and C-O stretching combination (Workman and Weyer 2007). Simultaneously, variations in this specific band can be attributed to variations in lignin content, as this band is associated with CHO bonds. The lignin and cellulose contents of wood were very important for the PLS-R models for SC content in MDF panels, since the band at 4,412 cm^{-1} yielded the highest regression coefficients.

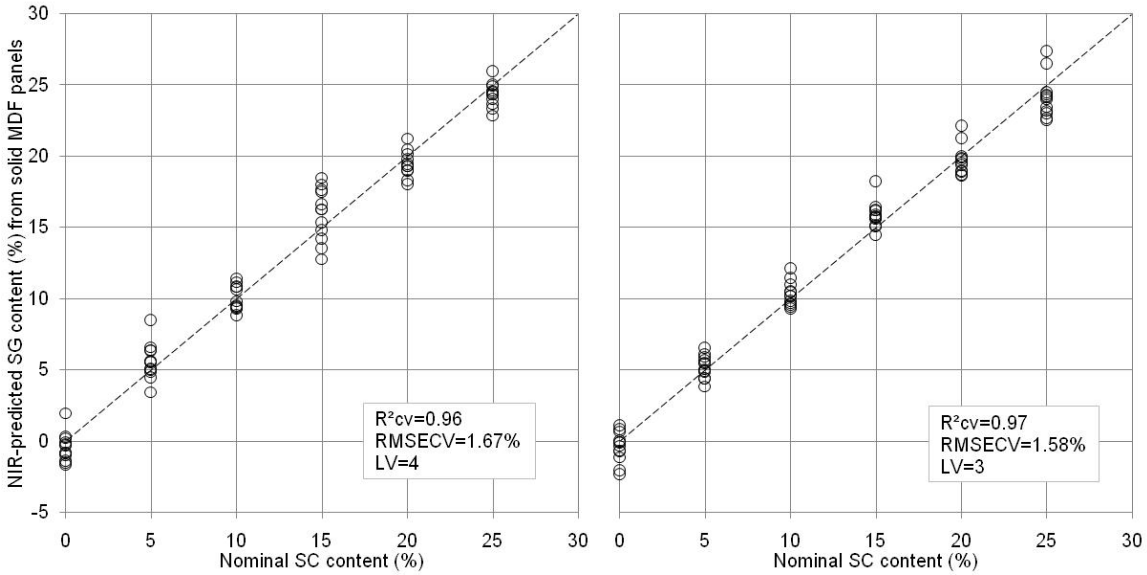

Fig. 5. NIR predicted versus Laboratory determined plot for SC content by partial least square regression using snv+d2 NIR spectra of solid (left) and ground (right) MDF panels.

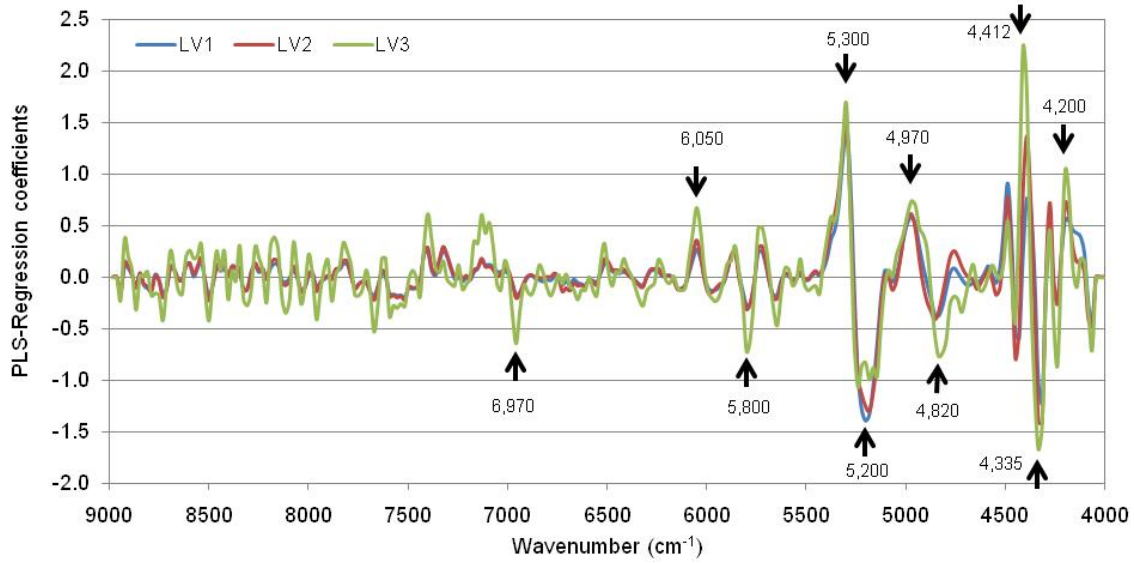

Fig. 6. Regression coefficients of the PLS-R models to predict SC content from snv+d2 NIR spectra measured on ground panels. Bands assigned to chemical compounds are represented by arrows.

The regression coefficients also were high at 4,335 cm^{-1}. This band is associated with C-H methylene C-H, as in linear aliphatic $R(CH_2)NR$ (Workman and Weyer 2007). Although this band is assigned to variations in hydrocarbons, the high regression coefficients at this band seem to be related to the second derivative NIR spectra effects. The band at 4,200 cm^{-1} showed a significant regression coefficient and represents the C-H stretching and C-C stretching combination (Workman and Weyer 2007). The band at 5,800 is associated with C-H stretch of the first overtone and may be related to variations

in hemicelluloses. The band at 6,970 represents the OH groups with H-bonds of intermediate strength. Krongtaew et al. (2010) presented a table containing the band assignments of lignins and polysaccharides molecules presents in wood fibres and cane sugar bagasse. Here, the focus is just on the assignment of the bands having high PLS-regression coefficients.

In short, the regression coefficients at these bands indicate by means of the NIR spectra the variations in the MDF panel properties due to the different combinations of lignin and cellulose contents, as well as the resin contents of the samples.

Partial Least Regression - Discriminant Analysis (PLS-DA)

As the SC content of these MDF panels is a discrete variable, Partial Least Regression - Discriminant Analysis (PLS-DA) were performed to separate the sample classes according to their NIR spectra.

Cross-validation results

The cross-validations of the PLS-DA regressions for the composition classes using NIR spectra from solid panels had promising results, allowing fine discrimination of the six classes of SC content of the MDF samples. The results of classification obtained from this cross-validated PLS-DA model (Table 3) show the efficiency of the method in distinguishing samples.

Table 3. Classification of the Cross-Validated PLS-DA Model for SC Content Classes Based on NIR Spectra Measured on Solid Samples

		NIR-predicted SC (%) classes						Incorrect classification		Correct classification	
		0	5	10	15	20	25	No.	%	No.	%
Nominal SC (%) classes	0	24						0	0	24	100
	5		23	1				1	0.7	23	99.3
	10		2	19	2	1		5	3.5	19	96.5
	15				24			0	0	24	100
	20				1	22	1	2	4.2	22	98.6
	25						24	0	0	24	100
							Total	8	5.6	207	94.4

This model correctly classified ninety-four percent of samples. One (1) sample containing 5% of SC, five (5) samples with 10%, and two (2) samples containing 20% SC (8 of 215 samples) were misclassified. The misclassified samples were grouped generally in the neighbouring classes. For example, if they were supposed to be in the 10% class, they were grouped normally in the 5 or 15 percent class. That knowledge is useful, since misclassifying a 10% as a 15% is better than misclassifying a 10% as a 25%.

The two-dimensional scatter plots of scores for PC 1 and PC 2 were developed from the NIR spectra after mathematical transformations (snv+detrend+d2). The scores of samples obtained by PLS-DA approach are shown in the Fig. 7. The PC 1 and 2 accounted for 81% of the NIR spectra variability, and the samples with different classes of SC could be reasonably distinguished from each other.

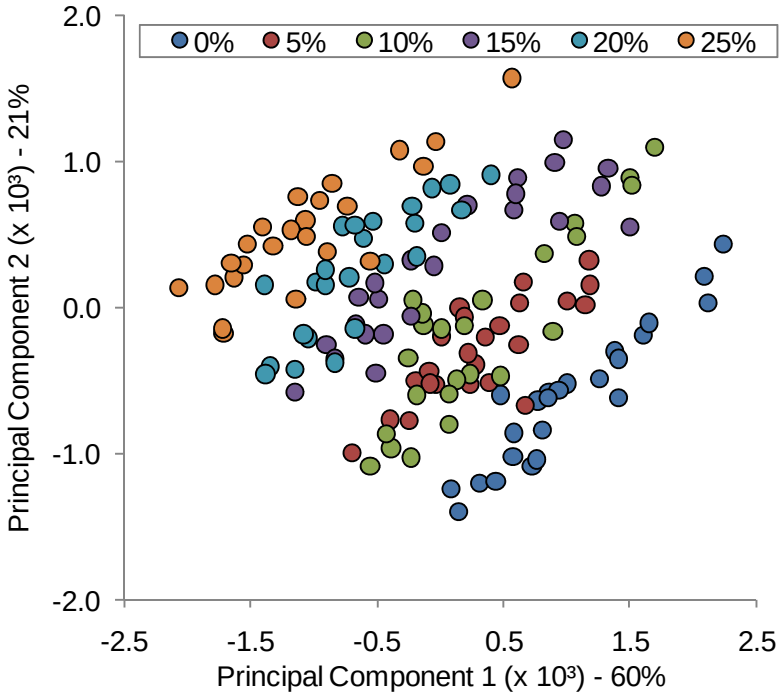

Fig. 7. Two-dimensional scatter plots of scores for PC 1 and PC 2 from PLS-DA of the snv+detrend+d2 NIR spectra obtained from solid panels showing the six SC contents by colour

Independent validation results

The NIR spectra of the samples used for validations were not used for PLS-DA development. The previous PLS-DA model was applied on the NIR spectra of the independent set samples. Table 4 presents the results of classification obtained from the PLS-DA models validated by this independent test set (press line surface NIR spectra) for SC content classes.

Table 4. Classification of the Validated PLS-DA Models by Independent Test Set for SC Content Classes Based on NIR Spectra (solid sample).

		NIR-predicted SC (%) classes						Incorrect classification		Correct classification	
		0	5	10	15	20	25	No.	%	No.	%
Nominal SC (%) classes	0	12						0	0	12	100
	5		12					0	0	12	100
	10			11	1			1	0.7	11	99.3
	15			1	10	1		2	1.4	10	98.6
	20					12		0	0	12	100
	25						12	0	0	12	100
							Total	3	2.1	69	97.9

Despite the difference of surface, and surface quality, the PLS-DA model allowed good discrimination between the six classes of SC content based on their NIR spectra in this sample set of MDF panels. Table 4 shows that this validated PLS-DA model

correctly classified ninety-eight percent of samples of the test set. Only two samples containing 15% of SC and one sample presenting 10% SC (3 of 72 samples) were misclassified.

CONCLUSIONS

The MDF panels having different SC contents could be distinguished from each other by the principal component analysis of their NIR spectra with clearly different patterns of discrimination. The PLS-R models were able to estimate the SC content of the MDF samples, presenting a strong coefficient of determinations (0.96) between the NIR-predicted and Lab-determined values and low standard error of prediction (~1.5%) in the cross-validations. The NIR spectra measured in the ground MDF panels yielded the best statistic models (R^2_{cv}=0.97 and RMSECV=1.6%). However, for practical applications, the PLS-R models developed with NIR spectra from solid panels are more useful than NIR-based models from ground samples, because it provides immediate results. The resin, cellulose, and lignin content played a key role on the PLS-R calibrations for SC bagasse content. The PLS-DA model correctly classified ninety-four percent of MDF samples by cross-validations and ninety-eight percent of the panels by independent test set. These NIR-based models can be useful to quickly verify SC bagasse content in unknown MDF panels and to control the composition of these engineered wood products in an online process after climatization of the boards.

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