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# ESTIMATION OF MOISTURE IN WOOD CHIPS BY NEAR INFRARED SPECTROSCOPY

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## ABSTRACT

In order to assess the moisture content of wood chips on an industrial scale, readily applicable techniques are required. Thus, near infrared (NIR) spectroscopy was used to estimate moisture in wood chips by means of partial least squares regressions. NIR spectra were obtained in spectrometer with an integrating sphere and optical fiber probe, on the longitudinal and transverse surface of *Eucalyptus* wood chips. The specimens had their masses and NIR spectra measured in 10 steps during drying from saturated to anhydrous condition. Principal Component Analysis was performed to explore the effect of moisture of wood chip on NIR signatures. The values of moisture content of chips were associated with the respective NIR spectra by Partial Least Squares Regression (PLS-R) and Partial Least Squares Discriminant Analysis (PLS-DA) to estimate the moisture content of wood chips and its moisture classes, respectively. Model developed from spectra recorded on the longitudinal face by the integrating sphere method presented statistics slightly better ($R^2cv$ = 0,96; RMSEcv = 7,15 %) than model based on optical fiber probe ($R^2cv$ = 0,90; RMSEcv = 11,86 %). This study suggests that for calibration of robust predictive model for estimating moisture content in chips the spectra should be recorded on the longitudinal surface of wood using the integrating sphere acquisition method.

**Keywords:** Cellulose, integrating sphere, optical fiber, paper, physical properties,

**RESUMEN**

Para evaluar el contenido de humedad de las astillas de madera a escala industrial, se requieren técnicas fácilmente aplicables. Por lo tanto, se utilizó la espectroscopía de infrarrojo cercano (NIR) para estimar la humedad en astillas de madera mediante regresiones parciales de mínimos cuadrados. Los espectros NIR se obtuvieron en un espectrómetro con una esfera de integración y una sonda de fibra óptica, en la superficie longitudinal y transversal de las astillas de madera de *Eucalyptus*. Las muestras tenían sus masas y espectros NIR medidos en 10 pasos durante el secado de condición saturada a anhidra. Los valores del contenido de humedad de las astillas se asociaron con los espectros NIR respectivos mediante Regresión Parcial de Mínimos Cuadrados (PLS-R) y Análisis Discriminante de Mínimos Cuadrados Parciales (PLS-DA) para estimar el contenido de humedad de las astillas de madera y sus clases de humedad, respectivamente. El modelo desarrollado a partir de espectros registrados en la cara longitudinal por el método de la esfera integradora presentó estadísticas ligeramente mejores ($R^2cv$ = 0,96; RMSEcv = 7,15 %) que el modelo basado en una sonda de fibra óptica ($R^2cv$ = 0,90; RMSEcv = 11,86 %). Este estudio sugiere que para la calibración de un modelo predictivo robusto para estimar el contenido de humedad en las astillas, los espectros deben registrarse en la superficie longitudinal de la madera utilizando el método de adquisición de esfera integradora.

**Palabras clave:** Celulosa, esfera integradora, fibra óptica, Papel, propiedades físicas.

## INTRODUCTION

Although moisture is not an intrinsic characteristic of wood, it is among its most important properties, because its variation affects the behavior of the material during the industrial processing and application phases (Tsuchikawa and Schwanninger 2013). In industries that use wood chips as raw material, the knowledge of moisture is an important parameter of quality, since in addition to guaranteeing the quality of the final product, it reduces losses and costs with reagents (Fardim 2005).

In the cellulose and paper industry, although it is not a limiting factor in the Kraft pulping process, the knowledge of the moisture of the chips is essential to make adjustment calculations in the process. Moisture values are important to determine the dry mass of the chips correctly and to calculate the quantity of the cooking reagents, and their correct ratio of wood liquor (Gomide and Fantuzzi Netto 2000).

Biermann (1996) also emphasizes the importance of knowledge and the control of moisture in the costs of transportation and commercialization of raw material. The influence is observed in situations where the purchase of chips is carried out by weight, in this way, the greater the moisture of the material, the lower the amount of raw material purchased.

To perform the monitoring of water contents in the wood, it is necessary to adopt techniques that are fast, efficient and inexpensive, in order to obtain improvements in the quality of the final product (Muñiz *et al.* 2012). The methods currently available are time-consuming, making it difficult to control the drying process for a large quantity of raw material.

The principle of operation of the NIR technique consists of exposing a specimen to the near infrared region spectrum, the generated spectra contain data of the chemical constituents of the material that, when related to the results of conventional analyzes, generate statistical models that explain most this information in the spectra (Price *et al.* 2001; Pavia *et al.* 2010; Pasquini 2018). Thus, it is possible to estimate several properties contained in biological materials, such as wood (Dahlbacka and Lillhonga 2010; Arriel *et al.* 2019; Tyson *et al.* 2012; Tsuchikawa and Schwanninger 2013; Tsuchikawa and Kobori 2015).

Some studies were carried out using near infrared spectroscopy (NIR) to estimate wood moisture. Thygesen and Lundqvist (2000) have investigated the thermal effects on NIR spectra for estimating moisture content in *Picea abies* wood under temperature conditions varying between -20 °C and +25 °C. Eom *et al.* (2013) applied the NIRS technique to measure the surface moisture of poplar wood of *Populus* specie during desorption conditions. Fujimoto *et al.* (2012) evaluated the NIR spectra obtained from specimen *Larix kaempferi* containing different amounts of water were used to verify the effect of moisture conditions on the accuracy of the estimated density of the wood. Watanabe *et al.* (2011) applied the NIR technique for classification based on moisture from green spruce wood. The authors have shown that NIR spectroscopy has the potential to estimate the mean green wood moisture, although it only provides values of surface moisture content. Karttunen *et al.* (2008) reported a survey of the moisture distribution in two sets of wild pine trunks using NIR spectroscopy. Moisture variation among trees was detected with high precision. Tham *et al.* (2018) carried out a study applying the capacitive method and the NIR spectroscopy together to simultaneously predict the density and moisture of wood specimens. The results suggest the possibility of a new device combining the capacitive method and the NIR spectroscopy to predict density and moisture with greater accuracy.

These studies have pointed to NIR spectroscopy as a promising alternative in the estimation of wood moisture. However, the influence of the anisotropy of the material and the path of spectral acquisition in the characterization of the wood chips via spectroscopy in the NIR is not yet fully understood. Therefore, it is necessary to know these parameters in order to develop predictive models based on moisture in wood chips in order to maintain the quality of the raw material and contribute to the industries that use wood chips in their production, reducing costs with reagent and reducing water consumption.

## EXPERIMENTAL SECTION

### Wood chips and water desorption monitoring

Forty (40) wood chips from *Eucalyptus urophylla* and *Eucalyptus grandis* hybrids of different ages and sizes were used. The chips present, in average, the following dimensions: 35 mm wide, 25 mm long (longitudinal direction) and 3 mm - 4 mm thick. The selection was performed according to the wood chips that presented better conditions on their surfaces for the acquisition of the spectra.

The specimens were identified and submitted to saturation in a vessel with water, which was changed periodically for 30 days until complete saturation. The moisture of the specimens was performed in10 steps during drying of according to the gravimetric method described in NBR 14929 (ABNT 2017).

In the first phase, the saturated test specimens were submitted to natural drying until reaching equilibrium moisture (~12 %). Mass measurements and spectral acquisition were performed when the control specimens lost about 10 % of the mass as a function of the pre-determined anhydrous mass. After reaching equilibrium moisture, the test specimens were subjected to drying in an oven at 50 °C ± 2 °C until the control specimen lost approximately 10 % of the mass in relation to anhydrous mass, according to the procedure described in Santos (2017).

### Recording NIR spectra

The spectral acquisition was performed in a diffuse reflection mode using a Fourier transform spectrometer. The spectrometer has two acquisition paths: integrating sphere and optical fiber probe. The spectra were captured in the near infrared region, opening the range of 12500 $cm^{-1}$ to 4000 $cm^{-1}$, with spectral resolution of 3845 $cm^{-1}$ and 32 scans for reading according to Costa *et al*. (2018).

The spectra were captured during the 10 drying steps, at every 10 % mass loss of water using the fiber optical probe on longitudinal and transverse surface of the material. For acquisitions based on integrating sphere, NIR spectra were taken only on longitudinal surface of wood. It was not possible to record NIR spectra on the transverse surface of chips due to the difficulty of positioning the specimen on scanner window.

Individual chip specimens were investigated instead to analyze chip batches in order to reduce the possible noise level in the signal. NIR were recorded from an optical fiber probe or integrating sphere directly on the single chip surface. When using a portion of chips, there is a lot of empty space between the chips and between the sensor and the wood surface, generating noise in the signal.

**Multivariate statistics**

Principal Component Analysis (PCA), Partial Least Squares Regression (PLS-R) and the Partial Least Squares Discriminant Analysis (PLS-DA) were developed in the free software Chemoface version 1.61 (Nunes *et al.* 2012).

PCA was used to evaluate the effect of the presence of water in the wood chips are in their spectral signature. PLS-R was developed to associate spectra with the chip moisture values determined by gravimetric method and generate a regression capable of estimating continuous values of moisture based on the NIR spectra recorded on the chips. PLS-DA model was held in order to classify their moisture in three (3) categories of moisture (up to 40 %, between 40 % and 80 %, and above 80 % moisture content) based on NIR spectrum signature.

Analyzes were performed separately for the spectra obtained in the longitudinal and transverse surfaces of the specimens and by two methods of spectral acquisition: integrating sphere and fiber optic probe.

After adjusting several preliminary models, six latent variables (LV) calculated from 1300 spectroscopic variables were used for all models. Thus, the presented models were developed with these six (6) latent variables for calibrations and validations. To select the best predictive models the following criteria were adopted: coefficient of determination of the cross-validation model ($R^2CV$), root mean standard error of cross-validation (RMSECV) and the ratio of performance to deviation (RPD), as described in Rosado et al. (2019). The independent and cross-validation methods were used to test the robustness of the estimates. Leave one out method was used for full cross validations while for independent validation was done using 2/3 of samples chosen at random for calibrations and 1/3 of remaining specimens for test set validation.

The calibrations were performed from the original (untreated) spectra and the mathematically treated spectra by the first derivative method using Savitzky–Golay algorithm with 13-point filter and a second-order polynomial, as described in Costa *et al*. (2018). Moreover, the wavenumbers from 12000 $cm^{-1}$ to 9000 $cm^{-1}$ were not considered. That process had the purpose eliminate noise and improve the quality of the calibration signal.

## RESULTS AND DISCUSSION

### Effect of moisture on spectral signature

Figure 1 shows diffuse reflectance spectra of wood chips obtained on the longitudinal face using the integrator sphere acquisition path in different moisture classes, with the original data and after the mathematical treatment of the first derivative. The first derivative is able to identify differences in moisture classes in wood chips.

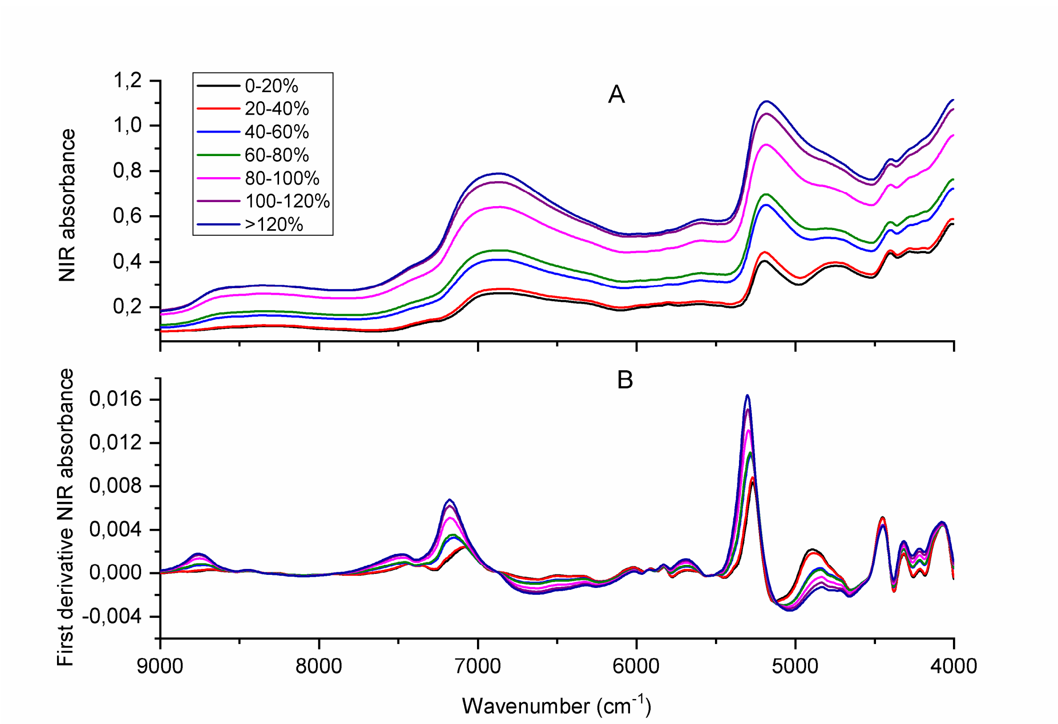


**Figure 1:** Diffuse reflection spectra obtained with the original (untreated) data (A) and with the treatment of the first derivative (B).

Absorption peaks can be observed at wavelengths of approximately 7000 $cm^{-1}$ and 5100 $cm^{-1}$ or (1428 and 1960) nm. These values are consistent with the results obtained by Watanabe *et al.* (2011) that found greater absorption at the wavelength of 1430 nm and 1910 nm. The variation in these absorption peaks can be associated with variation in moisture content, since they indicate vibrations characteristic of hydroxyl groups – OH present in water. These peaks increase with increasing chip moisture.

According to Karttunen *et al*. (2008) water absorption bands occurs mainly due to changes in the free water content in capillaries, because different water levels can modify the NIR spectrum when incident light is spread on the surface of the specimen.

According to Adedipe and Dawson-Andoh (2008) a higher or lower spectral range has no significant influence on moisture prediction, from which the range encompasses water absorption bands. The same authors, when limiting the spectral range from 1400 nm to 1940 nm, predicted the water content in the wood with similar precision when they used a range 800 nm to 2500 nm.

**Principal component analysis**

The principal component analyzes (PCA) were carried out with original spectra obtained through the two acquisition pathways (integrator sphere and optical fiber) in the longitudinal and transverse faces of the wood chips, to carry out a preliminary evaluation of the behavior of the spectra and possible separation of the specimens according to the 10 moisture steps ranging from 1 (saturated condition) to 10 (anhydrous condition) Figure 2.

The two main components together account for approximately 100 % of the variability of the analyzed data on the longitudinal side of both acquisition pathways, 99,35 % are explained by the main component 1 (PC1) and 0,50 % is explained by the main component 2 (PC2) in the fiber optic acquisition pathway. with regards to the NIR spectra taken by the integrating sphere, 99,40 % of variance was explained by PC1 and 0,48 % by PC2.

The integrating sphere in the longitudinal face was able to differentiate better the specimen with different moisture, generating less overlaps. The wettest specimens were more dispersed in relation to the drier specimens. This was due to the existence of similar moisture in these measurement steps.

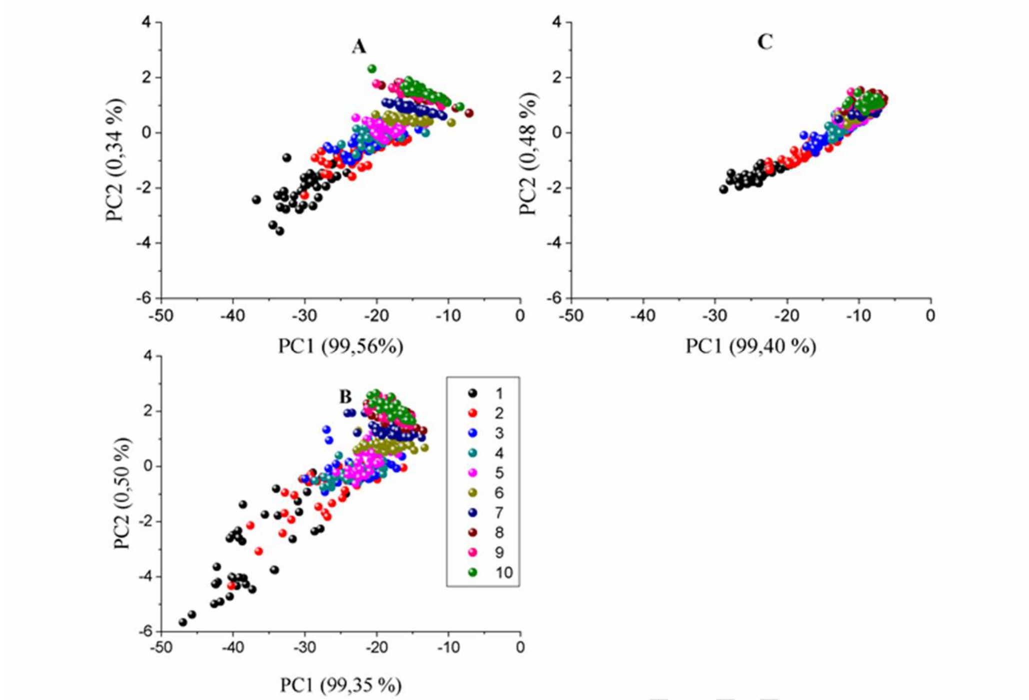


**Figure 2:** Graphic of scores obtained by PCA applied to the spectral information measured in the wood chips from the optical fiber probe acquisition path on the longitudinal face (A) transverse surface (B) and from the acquisition path acquisition integrating sphere longitudinal surface (C).

**Global model for estimating chip moisture**

Table 1 presents the statistics associated to calibrations and cross validations for estimating the wood chip moisture from the original spectra and treated with the first derivative.

The spectra-based models in the NIR were efficient for estimating moisture in wood chips with errors between 7,15 % and 11,86 %. The longitudinal face is the most suitable for spectral acquisition, since the estimation error is smaller when compared to the transverse face, besides the operational ease of measurement. However, the transverse face can also be used in the estimation of moisture, since they presented acceptable RPD of 3,16. According to Sobering and Williams (1993) with calibrations with RPD values between 2 and 3 indicate that the predictions are approximate and values between 3 and 5 indicate that the calibrations are satisfactory for the predictions.

**Table 1:** Calibrations and cross-validations for moisture estimation in wood chips.

| **Model** | **Via of acquisition** | **Surface** | **Treat.** | **$R^2$cal** | **RMSEc %** | **$R^2$cv** | **RMSEcv %** | **RPD** |
|---|---|---|---|---|---|---|---|---|
| 1 | Sphere | Long | - | 0,95 | 7,54 | 0,95 | 7,78 | 4,82 |
| 2 | | | 1d | 0,96 | 6,79 | 0,96 | 7,15 | 5,24 |
| 3 | Fiber | Long | - | 0,94 | 9,08 | 0,93 | 9,38 | 4,00 |
| 4 | | | 1d | 0,94 | 9,17 | 0,93 | 9,61 | 3,90 |
| 5 | | Trans | - | 0,90 | 11,30 | 0,89 | 11,60 | 3,23 |
| 6 | | | 1d | 0,90 | 11,29 | 0,90 | 11,86 | 3,16 |

Treat - mathematical treatment; 1d - first derivative; $R^2$c - coefficient of determination of the calibration; RMSEc - Root mean square error of calibration; $R^2$cv - coefficient of determination of the cross-validation; RMSEcv - Root mean square error of cross-validation; RPD - ratio performance to deviation; Long - longitudinal surface and Trans - transverse surface.

Regarding the spectral acquisition method, the calibrations developed from the two types of spectra in the NIR (integrator sphere and fiber optic) have the potential to satisfactorily estimate the moisture of the wood. However, the models generated from the spectra obtained by integrator sphere (models 1 and 2) presented more satisfactory statistical results ($R^2$CV higher than 0,95 and RMSECV lower than 7,77 %).

The models generated from the first derivative of the spectra (models 2, 4 and 6) provided better estimates. Martens and Naes (1991) argue that mathematical treatments aim to improve signal quality and reduce noise. However, it is observed that there was no significant improvement in the spectra of the wood chips via optical fiber treated with the first derivative.

In general, the model generated by the spectra of the integrating sphere presented better statistics than those generated by optical fiber probe. According to Costa (2018) this difference between the models can be explained from the comparison between the areas of the acquisition path ways. The integrating sphere acquisition pathway has a circular area with a diameter of 10 mm, while the fiber optic acquisition pathway has a circular area of approximately 1 mm in diameter. The higher value area allows better representation of the surface of the wood chips. In this way, this path becomes better suited to acquire spectra in order to estimate moisture in wood chips.

The wood surface that presented the best results, in both acquisition pathways, was longitudinal. This result differed from some authors, such as Defo *et al.* (2007) who used near-infrared spectroscopy to determine the moisture of *Quercus* spp. (red oak) by means of spectra collected on the radial, tangential and transverse face. When comparing the prediction of the models generated in the different faces, the authors realized that the transversal face was the one that obtained the best performance. This difference in results may have occurred due to the raw material of the authors being lumber, whereas the one used in the study is wood in the form of wood chips.

Figure 3 shows the relationship between moisture estimated by the NIR and determined in the laboratory from the optical fiber probe, on both surfaces (longitudinal and transverse) of the wood chips. The prediction accuracy is higher in specimens with moisture content lower than 30 % (Figure 3). Yang *et al.* (2014) have reported fiber saturation point of 29 % for *Eucalyptus urophylla* wood. Thus, we supposed that the spectral change in lower moisture content is mainly due to the decrement of absorbed water whereas in higher moisture content spectral change is resulted in the change of free water.

The cross-validation values obtained by the acquisition of spectra in the integrating sphere presented values similar to the measured values of moisture in the laboratory (Figure 3). However, it was observed that the cross-validation performed by the longitudinal face presented a better distribution of the data ($R^2$ = 0,93), when compared with the transversal face that presented the lowest performance ($R^2$ = 0,89). This result may have occurred due to the fact that the longitudinal face presents a rough surface in relation to the transverse face, since the roughness may have facilitated the penetration of light in the chips.

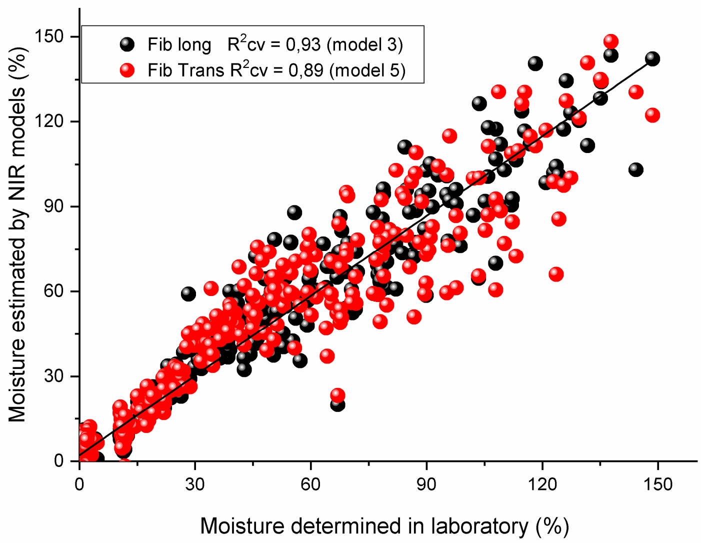


**Figure 3:** Moisture of the wood chips determined in the laboratory and estimated in the NIR from the optical fiber according to models 3 and 5 of Table 1.

Zhang *et al.* (2015) studied the correlation between NIR spectroscopy and the surface roughness of the wood. The authors used optical fiber to obtain the spectra. The results showed that the roughness of the surface of the wood can influence in the statistics to estimate the properties of the wood from NIR spectroscopy. Greater surface roughness may be associated with more pronounced diffuse reflection.

The longitudinal face through the integrating sphere presented the best model in the PLS-R analysis, so it was divided into three ranges of moisture (0 % to 40 %, 40 % to 80 %, and > 80 % moisture) for to generate models capable of predicting wood moisture by classification using PLS-DA.

Model 2 of Table 1 was applied in the NIR spectra of specimens to generate estimates. Specimens were separated into 3 classes (0 % to 40 %, 40 % to 80 %, and > 80 %) based on the predicted moisture content by model 2 and a confusion matrix was presented in Table 2 to evaluate the correct classification ratio.

**Table 2:** Confusion matrix of predictions of chip moisture based on NIR spectra through model 2 (Table 1).

| Nominal Moisture classes (%) | Moisture estimated by model 2 (%) | | | Correct classification | | Total specimens |
|---|---|---|---|---|---|---|
| | 0 - 40 | 40 - 80 | > 80 | No. | % | No. |
| 0 - 40 | 203 | 6 | 6 | 203 | 94,4 | 215 |
| 40 - 80 | 12 | 71 | 12 | 71 | 84,5 | 84 |
| > 80 | | 7 | 89 | 89 | 88,1 | 101 |
| Total | 215 | 83 | 107 | 363 | 90,7 | 400 |

The confusion matrix (Table 2) shows that 363 from 400 (90,7 %) of specimens were correctly classified based on PLS-R model 2 (Table 1). 203 from 215 chips had their estimated moisture value correctly classified within the class of drier samples (0 to 40) %. In the class of samples with intermediate humidity (between 40 and 80), 84 % of the samples were correctly classified by model 2 of Table 1. Finally, in the class of the most humid samples, 89 of 101 samples were correctly classified and only 7 samples (6,9 %) had moisture estimates that classified them as intermediate samples.

This approach is very useful for pulp and paper companies that need to have a tool that allows them to separate chips into batches of different moisture quickly and reliably.

**Model for estimating the moisture of the wood chips per class**

Table 3 shows the regression models obtained by calibration and cross-validation from the spectra with and without first derivative treatment.

Table 3 shows that the first moisture class of (0 to 40) % was the one that presented the best estimates of wood moisture, especially when submitted to the treatment of the first one derivative, resulting in $R^2$CV of 0,96 and RMSECV of 2,15 % and RPD of 5,33 (model 8) which indicates that this model is suitable for estimating the moisture of the wood.

**Table 3:** Calibrations and cross-validations for the estimation of moisture in each class by PLS-R.

| Model | Moisture | Treat | $R^2c$ | RMSEc (%) | $R^2CV$ | RMSECV (%) | RPD |
|---|---|---|---|---|---|---|---|
| 7 | 0 - 40 | - | 0,96 | 2,22 | 0,95 | 2,34 | 4,80 |
| 8 | | 1d | 0,96 | 2,00 | 0,96 | 2,15 | 5,33 |
| 9 | 40 - 80 | - | 0,65 | 6,93 | 0,50 | 8,39 | 1,40 |
| 10 | | 1d | 0,71 | 6,27 | 0,46 | 8,99 | 1,31 |
| 11 | > 80 | - | 0,81 | 7,54 | 0,69 | 9,55 | 1,82 |
| 12 | | 1d | 0,87 | 6,16 | 0,76 | 8,43 | 2,07 |

Treat - mathematical treatment; 1d - first derivative; $R^2c$ - coefficient of determination of the calibration; RMSEc - Root mean square error of calibration; $R^2CV$ - coefficient of determination of the cross validation; RMSECV - Root mean square error of cross-validation and RPD - ratio performance to deviation.

The moisture range of 40 % to 80 % was the one that showed the lowest performance with $R^2CV$ of 0,46 and RMSCV of 8,99 % and RPD of 1,40 (model 9), being considered unsatisfactory. RPD values greater than 1,5 are considered satisfactory in studies on forest sciences (Schimleck *et al*. 2003).

The third moisture class (> 80 %) provided a model with $R^2CV$ of 0,76 and RMSECV of 8,43 and RPD of 2,07, presenting better estimates than the second class of moisture, however, the error found is considered high, even though the RPD is indicating that the model is satisfactory. The best estimate found in this class was the treatment of the first derivative as well as in the first class of moisture. However, the second class of moisture that presented the lowest performance did not improve the model when performing the first derivative treatment in the spectra.

Figure 4 shows the plots made from the PLS-R in the three moisture ranges of original spectra and mathematically treated by the first derivative, collected from the longitudinal face through the integrator sphere acquisition path.

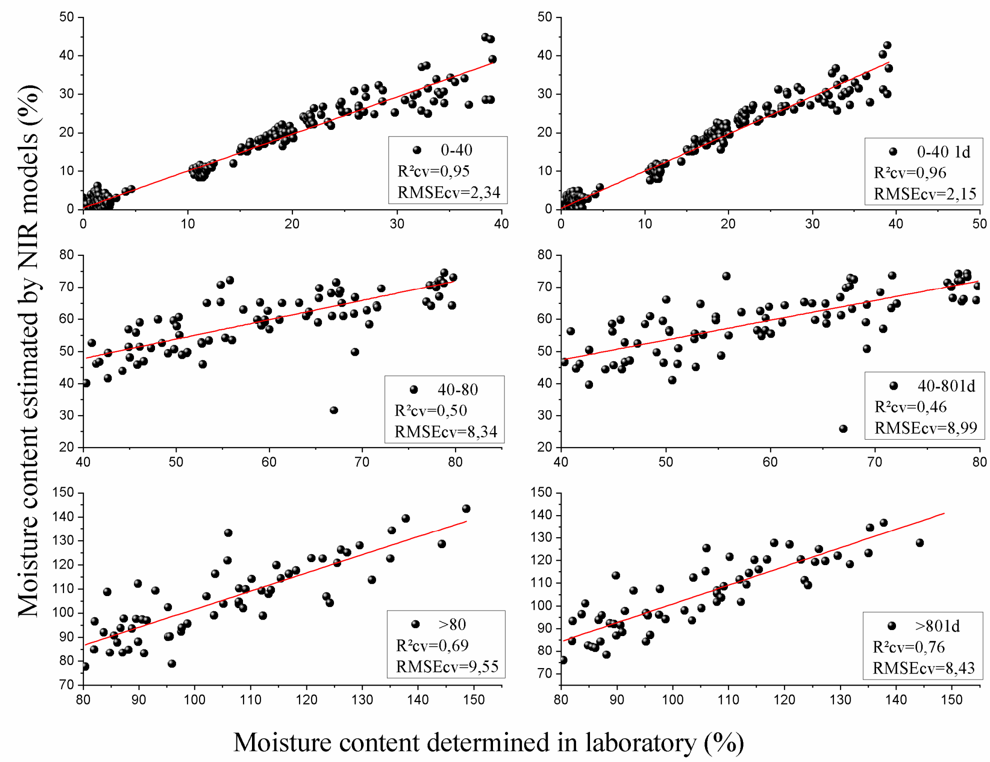


**Figure 4:** Moisture of the wood chips determined in laboratory and estimated from NIR by integrating sphere of according to Table 3.

Figure 4 shows that the calibration values obtained from the spectra measured in the 0 % to 40 % moisture range were more similar to those measured in the laboratory. In this moisture range the spectra treated with the first derivative were the ones that indicated the best model.

The moisture of 40 % to 80 % presented a lower adjustment of the data when compared to the first and third moisture range. The third range of moisture also showed a certain dispersion of the specimens when related to the actual values, however it showed improvement when the first derivative treatment was performed.

**Test set validation of models for moisture estimation by class**

According to Pasquini (2003), external validation is recommended because it presents results that are closer to the real ones. Therefore, the models of the three moisture ranges were validated according to this method (Table 4).

**Table 4:** Cross- and test set validations for the estimation of moisture in each class.

| Moisture range (%) | $R^2CV$ | RMSECV (%) | $R^2p$ | RMSEP (%) | RPD |
|---|---|---|---|---|---|
| 0-40 | 0,97 | 2,51 | 0,96 | 2,16 | 3,90 |
| 40-80 | 0,64 | 7,10 | 0,42 | 10,49 | 1,27 |
| > 80 | 0,79 | 9,93 | 0,56 | 12,50 | 2,04 |

$R^2CV$ - coefficient of determination of the cross-validation; RMSECV - Root mean square error of cross validation; $R^2p$ - coefficient of determination of external validation; RMSEP – Root mean square error of external validation; RPD - standard deviation performance ratio.

From Table 3 and Table 4 it is possible to notice that the external validation values were similar to the values obtained through cross validation. However, most of the external validation values were inferior to those obtained in the cross validation.

Figure 5 shows the values obtained in the laboratory and predicted by the NIR, showing the distribution of the calibration points and the validation of the best model for estimating moisture in wood chips.

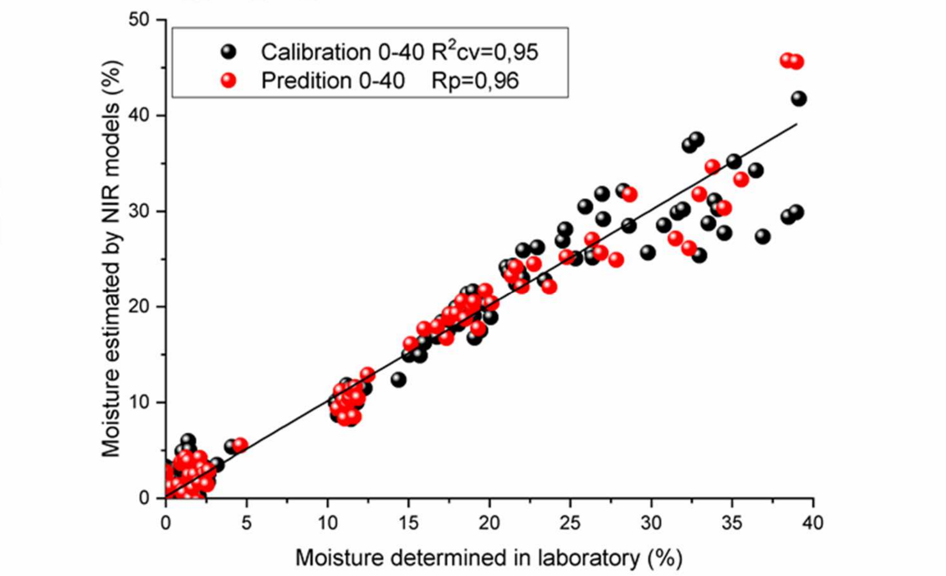


**Figure 5:** Regression of wood chips moisture values obtained in the laboratory and estimated in the NIR.

In order to improve the models, the initial specimens were separated according to the moisture of the wood and the wave numbers of 9000 cm$^{-1}$ to 12000 cm$^{-1}$ were excluded due to the occurrence of noise. However, as can be seen in Figure 5, only the first moisture range of 0 % to 40 % showed a strong correlation between the measured values and the predicted values, especially in moisture up to 25 %. This value is desirable for the pulp and paper industries, since the moisture in the wood chips should be above 25 %, but below 55 % for better use of the raw material in pulping and lower consumption of reagents.

**Partial Least Squares - Discriminant Analysis**

Table 5 lists the PLS-DA classifications, including the number of correct and incorrect classifications and the correct classification percentage by means of cross-validations. The confusion matrix (Table 5) shows that 343 from 400 (85,75 %) of specimens were correctly classified based on PLS-DA model.

**Table 5:** Confusion matrix of predictions of chip moisture through PLS-DA analysis.

| **Nominal Moisture classes (%)** | **Moisture estimated by NIR (%)** | | | **Correct classification** | | **Total specimens** |
|---|---|---|---|---|---|---|
| | 0 - 40 | 40 - 80 | > 80 | No. | % | No. |
| 0 - 40 | 253 | 3 | 0 | 253 | 98,83 | 256 |
| 40 - 80 | 26 | 43 | 14 | 43 | 51,81 | 83 |
| > 80 | 0 | 14 | 47 | 47 | 77,05 | 61 |
| Total | 279 | 60 | 61 | 343 | 85,75 | 400 |

Table 5 shows that in the first moisture class (0 to 40) %, composed of 256 specimens, three of these specimens were incorrectly classified as belonging to the second moisture class, corresponding to 1,18 % of incorrect specimens. In the second class of moisture (40 to 80) %, 40 from 83 specimens were classified as incorrect; 26 specimens were classified in the first moisture class and 14 specimens as the third moisture class, corresponding to 48,20 % of specimens misclassified. In the third moisture range (> 80 %), of the only 14 specimens were misclassified, which represents 22,96 % of incorrect specimens.

The class that classified the most specimens incorrectly was 40 % to 80 % of moisture, while the class that obtained the most correct classifications was 0 % to 40 % of moisture, presenting 98,82 % of correct classifications. Also, it is verified that none of the specimens of the first class of moisture was classified as being of the third, and the opposite also occurred. This can be explained by the large difference between these two classes of moisture. Therefore, the specimens that were classified as incorrect could present similar moisture in the classes that were assigned.

This study was carried out with the objective of verifying the feasibility of this technique for rapid, immediate estimate of the moisture content in wood chips. The promising findings of this approach open up new possibilities for applying NIR spectroscopy in real situations, in which it is necessary to know the raw material properties in real time for to optimizing the production process. One of the potential applications would be on conveyors that take the chip from the pile to the digester in pulp and paper mills. In this situation, the challenges are even greater, as chips with different moisture, wood density and lignin content are mixed in the digester and the resultant pulp must be as uniform as possible. Thus, more comprehensive studies including chips with varying wood density and lignin content should be carried out to reduce the distance from what is done under laboratory conditions and to real situations in the pulp companies.

## CONCLUSIONS

This study indicates that NIR spectroscopy associated with multivariate analysis has the potential to estimate wood moisture in *Eucalyptus* chips. The model can be generated from NIR spectral signatures obtained by integrator sphere and optical fiber. The longitudinal face of the chips was shown to be more suitable for recording NIR spectra and estimating the moisture in wood chips when compared to the transverse face.

PLS-DA was able to correctly classified 85,75 % of the specimens in three moisture classes. In each class, 98,82 % of specimens were correctly classified into the group of drier specimens (0 to 40) % and 77,04 % of specimens were correctly grouped in the class of wetter specimens (moisture > 80 %). PLS-DA models misclassified 48,20 % of specimens with moisture varying from 40 % to 80 %.

For PLS-R models, the estimates used for classifications of moisture classes yielded better results. The percentage of correct classifications was 91 % when chips were grouped into the three moisture classes based on the estimates originated from PLS-R model.

This approach can be useful for the pulp and paper industries as it provides accurate estimates of the moisture content of chips, assisting in the definition of cooking parameters and optimizing industrial processes and the consumption of raw material and reagents.

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