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Estimation of *Acacia melanoxylon* unbleached Kraft pulp brightness by NIR spectroscopy

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Abstract

Aim of the study: The ability of NIR spectroscopy for predicting the ISO brightness was studied on unbleached Kraft pulps of *Acacia melanoxylon* R. Br.

Area of study: Sites covering littoral north, mid interior north and centre interior of Portugal.

Materials and methods: The samples were Kraft pulped in standard identical conditions targeted to a kappa number of 15. A Near Infrared (NIR) partial least squares regression (PLSR) model was developed for the ISO brightness prediction using 75 pulp samples with a variation range of 18.9 to 47.9 %.

Main results: Very good correlations between NIR spectra and ISO brightness were obtained. Ten methods were used for PLS analysis (cross validation with 48 samples), and a test set validation was made with 27 samples. The 1stDer pre-processed spectra coupling two wavenumber ranges from 9404 to 7498 cm^{-1} and 4605 to 4243 cm^{-1} allowed the best model with a root mean square error of ISO brightness prediction of 0.5 % (RMSEP), a r^2 of 99.5 % with a RPD of 14.7.

Research highlights: According to AACC Method 39-00, the present model is sufficiently accurate to be used for process control (RPD ≥ 8)

Keywords: Acacia *melanoxylon*; unbleached Kraft pulps; ISO Brightness; NIR; RPD.

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Introduction

Pulps produced from *Acacia* species are competitive in the world market of hardwood pulps now dominated by *Eucalyptus*. However, pulps produced from *Acacia* species, namely from *Acacia mangium*, are emerging in the hardwood pulps market and an increasing number of commercial plantations is developing in Asia (Griffin *et al.*, 2014).

A. melanoxylon was also the focus of research for the wood technological quality, e.g. wood volume (Rucha *et al*., 2011), heartwood variation (Knapic *et al.*, 2006), basic density (Santos *et al.,* 2012a; Igartúa & Monteoliva, 2009), wood macroscopic properties and fiber characteristics (Santos *et al.* 2013; Tavares *et al.,* 2011) and mechanical properties (Machado *et al.,* 2014). *A. melanoxylon* is also interesting for pulping as evaluated by different authors regarding pulp yields and properties (Santos *et al.*, 2012b; Anjos *et al.,* 2011; Lourenço *et al.*, 2008; Santos *et al.* 2007; Santos *et al.*, 2006; Santos *et al.,* 2005).

One important pulp quality parameter is the ISO brightness, which is correlated with the pulp kappa number, i.e. pulp brightness can sometimes increase with the decrease in kappa number.

Brightness of pulp is the numerical value of the reflectance factor of a sample with respect to blue light of specific spectral and geometric characteristics. It is one of the most important characteristics in pulp characterization given their relation to the bleaching process, the kinds of fibre used in the pulp produced and also correlated with same important chemical characteristics of the pulp, namely kappa number (Santos *et al.*, 2012b; Santos *et al.,* 2008a; Santos *et al.,* 2008b). Given this correlation existing with the chemical pulp composition and brightness is also possible use Near-infrared (NIR) spectroscopy with PLS calibration to predict the brightness.

In addition unbleached Kraft pulps, brightness information is required for planning of bleaching sequences and consumption of bleaching chemicals which are related with the initial brightness of the pulp.

Given Near-infrared (NIR) spectroscopy it is one of the most powerful non-destructive techniques, has been applied to the determination of various properties in numerous materials, such as food (Wang *et al.,* 2014; Fu *et al.*, 2012), ethanol (Xu *et al.,* 2013), and wood lignin (Zhang *et al.,* 2011; Yao *et al*., 2010; Alves *et al.,* 2006; Hodge & Woodbridge, 2004; Poke *et al.,* 2004; Schwanninger & Hinterstoisser, 2001) and cellulose (Tyson *et al.*, 2010; Hein *et al.*, 2009; Poke *et al.*, 2006), wood properties and basic density (Santos *et al.,* 2012a; Alves *et al.,* 2012; Sykes *et al.,* 2005; Kelley *et al.,* 2004; Hauksson *et al*., 2001), pulp yield, kappa number and mechanical and optical properties of pulps (Santos *et al.*, 2014; Downes *et al.*, 2009; Schimleck *et al.,* 2006; Fardim *et al.,* 2005; Schimleck *et al.,* 2005; Fardim *et al.,* 2002).

NIR spectroscopic procedures are well suited to an automated on-line application for continuous control of various properties of the material, and the same collected spectral information may be used to estimate different parameters, if the specific models have been developed and validated. This paper focuses on the development of a NIR-PLSR model for the prediction of the ISO brightness of unbleached Kraft pulps produced under identical pulping conditions, as it would be the case in pulp mill operation, using *A. melanoxylon* pulps as case study.

Moreover the brightness analysis in pulp is already made with a not painful methodology this research could improve the work in a laboratory or in factory because many other parameters are available to be analysed with NIR infrared spectroscopy and all analyses could be performed in the same equipment.

Materials and methods

Sample preparation

A total of 75 wood discs from *Acacia melanoxylon R. Br.*, belonging to 20 trees from four sites in Portugal and collected at different stem height levels were used in this study. Detailed information on samples, sites and stands is available elsewhere (Santos *et al.*, 2013). Samples of 25 g oven-dry wood were milled using a knife mill (Retsch) with a 1 mm output screen and the fraction coarser than 0.25 mm was selected to Kraft pulping using a multi-batch digester system under the following reaction conditions: active alkali charge 21.3 % (as NaOH); sulfidity 30 %; liquor/wood ratio 4/1; time to temperature of 160 ºC, 90 min; time at temperature of 160 ºC, 90 min. These conditions were set to obtain a target kappa number of 15. The pulped chips were disintegrated, washed, and screened. Under these conditions the pulp yields ranged from 47.0 to 58.2 % (Santos *et al.,* 2012b). The pulp yield range was wide corresponding to the large variation of heartwood proportion in the wood of the different discs, as shown in the same samples by Santos *et al.* (2013) e.g. 38 to 45 % at 65 % height level and 2 to 15 % at the tree top. The 75 pulp samples had variation of kappa number from 10 to 17.

Pulps discs were produced using a Glass Vacuum Filter Holder 16307 - Sartorius with 0.2 μm membrane porosity, and a diameter matching the diameter of the spinning cup module of the near infrared spectrometer for collecting spectra and determined ISO brightness.

The brightness of the pulps was measured by the reflectance in pulp discs compared to the reflectance of known standards using a standard instrument (ISO standard 2470).

Spectra collection and data processing

The pulp disc samples were conditioned in a climatic chamber at 60 ºC for a period of 48 hours before spectral acquisition. NIR spectra were collected in the wavenumber range from 12000 to 3800 cm^{-1} with a near infrared spectrometer (BRUKER, model Vector 22/N) in diffuse reflectance mode, using a spinning cup module. Each spectrum was obtained with 100 scans at a spectral resolution of 16 cm^{-1}.

The samples were randomly divided into a calibration set containing 48 samples and a validation set (test set) containing 27 samples. The processing was done in two steps. First, the infrared data from the calibration samples were regressed against the measured ISO brightness, and by means of full cross validation with one sample omitted a significant number of PLS components (rank) was obtained using OPUS/Quant 2 software (version 7.2 BRUKER). Besides the raw spectra, also pre-processed spectra with ten methods were used for PLS analysis (Alves *et al*., 2007).

The quality of the calibration models was assessed by means of cross validation and by using the test set validation results by determining their coefficient of determination (r^2), root mean square error of cross validation (RMSECV), root mean square error of prediction (RMSEP) and the residual prediction deviation or ratio of performance to deviation (RPD).

The selection of the final model was based on its predictive ability assessed by the number of samples classified as outsiders and/or outliers that should be as less as possible.

Results and discussion

The ISO brightness of the unbleached pulps of *A. melanoxylon* obtained in this study ranged from 18.9 to 47.9 %, with an average of 28.0 % and a standard deviation of 6.1% (Table 1). This variability represents a good data scattering and covered a wide range of levels of pulp brightness that improve the accuracy of the final model proposed. The two sets showed similar statistics.

Table 1. Number of samples and range of ISO brightness in the calibration set and test set (SD-standard deviation)

	Total Samples	Calibration set	Test set
Nº of samples	75	48	27
Maximum	47.9	43.4	47.9
Minimum	18.9	19.3	18.9
Average	28.0	27.4	29.1
SD	6.1	5.2	7.4

The coupled spectral information from two wavenumber ranges from 9404 to 7498 cm^{-1} and 4605 to 4243 cm^{-1} was used for calibration, as found by automated optimization. The near-infrared data from the 48 samples of the calibration set were regressed against their experimentally determined ISO brightness, and the results obtained with the various pre-processing of the raw spectral data are summarized in Table 2. The obtained rank ranged from two (MinMax, VecNor, 1stDer+VecNor) to six (no spectral pre-processing), while the coefficients of determination (r^2) ranged from 88.1 % to 99.4 %, and RMSECV from 0.4 to 1.5 %. When using the test set validation, the coefficients of determination ranged from 90.6 % to 99.5 %.

The best model was selected by using the 1stDer (first derivative) of the spectral data and the corresponding plot of NIR-PLSR predicted versus the laboratorial determined ISO brightness is shown in Figure 1. Both the cross-validation and the validation showed high correlation between predicted and determined values, with a RMSEP of 0.5 %, a rank of five and no outliers.

In this study, the ISO brightness of hardwood unbleached kraft pulps is predicted with a very high accuracy and precision (0.5 %) and with RPD = 14.7. These results compare very favourably with the few data available, as shown in Table 3 that summarizes the models published in the bibliography regarding estimation of pulp brightness using NIR spectroscopy. Fardim *et al.,* (2002), using unbleached and unrefined kraft pulp, reported a root mean square error of prediction of 1.3 % for ISO brightness, with 3 PLS components and 1stDer as pre-processing.

The second model published by Fardim *et al.,* (2005), was based on one sample of *Eucalyptus grandis* wood that was pulped and refined with different conditions, presented a RMSECV = 1.2 %, with a Rank = 6.

Table 2. Results of the cross-validation of the calibration set and of the test set validation as well as the percentage of outsiders (OS) and outliers (OL) of the test set obtained during prediction of the samples with unknown ISO brightness, using various preprocessing methods of the raw spectral data

Preprocessing	Nº of samples	Rank	r^2 (%)	RMSECV	RPD	Rank	r^2 (%)	RMSEP	RPD	OS (%)	OL (%)
MinMax	46	2	97.2	0.9	6.0	6	98.0	1.0	7.2	0.0	0.0
1stDer+MSC	47	4	94.8	1.2	4.4	5	97.4	1.2	6.2	0.0	0.0
MSC	42	4	94.9	0.9	4.4	8	95.5	1.2	4.8	0.0	0.1
ConOff	46	5	99.2	0.5	11.5	5	99.5	0.5	14.1	0.0	0.0
None	46	6	99.4	0.4	12.7	6	99.5	0.5	15.0	0.0	0.0
2ndDer	46	3	88.1	1.5	2.9	6	90.6	1.7	3.7	0.0	0.1
1stDer	**48**	**5**	**98.9**	**0.6**	**9.5**	**5**	**99.5**	**0.5**	**14.7**	**0.0**	**0.0**
1stDer+SLS	46	5	98.0	0.7	7.2	4	96.6	1.4	5.4	0.0	0.0
VecNor	47	2	96.2	1.0	5.2	5	97.0	1.3	5.9	0.0	0.0
1stDer+VecNor	46	2	95.0	1.1	4.5	4	97.6	1.1	6.5	0.0	0.0
SLS	48	4	96.6	1.0	5.4	7	98.9	0.8	9.8	0.0	0.0

MinMax – minimum-maximum normalization; 1stDer+MSC – first derivative + multiplicative scatter correction; MSC – multiplicative scatter correction; ConOff – constant offset elimination; None – no spectral data processing; 2ndDer – seconded derivative; 1stDer – first derivative; 1stDer + SLS – first derivative + straight line subtraction; VecNor – vector normalization; 1stDer + VecNor – first derivative + vector normalization; SLS – straight line elimination; r^2 – coefficient of determination; RPD – ratios of performance to deviation; RMSECV – root mean square error of cross validation; RMSEP – root mean square error of prediction.

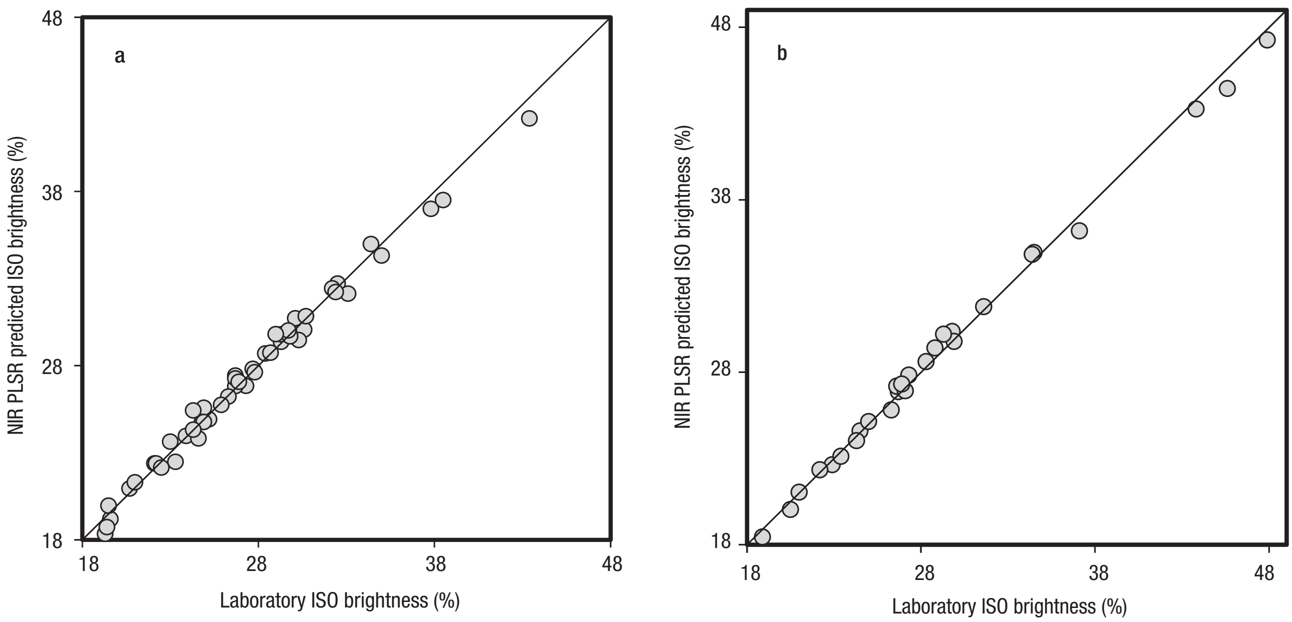

Figure 1. NIR PLSR predicted versus laboratory determined ISO brightness for blackwood pulps: a) cross validation (47 samples); b) test set validation (27 samples).

Table 3. Models published in the bibliography

Species	References	Type of pulping	Pre-processing	Rank	RMSECV	RMSEP
Eucalyptus grandis	Fardim *et al.* 2002	kraft	1stDer	3	1.1	1.3
*Eucalyptus grandis**	Fardim *et al.* 2005	kraft	1stDer	6	1.2	-
Picea abies	Nilsson *et al.* 2005	Thermo mechanical	1stDer	3	0.8	0.8
			MSC	2	0.5	1.0

* Calibration for light absorption, equivalent to ISO brightness.

Nilsson *et al*. (2005) proposed two models for prediction of ISO brightness in thermomechanical pulps of Norway spruce bleached with hydrogen peroxide (45 samples) and sodium dithionite (45 samples). In both cases it was possible to predict the ISO brightness of the bleached thermomechanical pulps from near-infrared spectra on pulp with lower errors of prediction (RMSEP).

The ratios of performance to deviation (RPD) may be used to evaluate if the prediction models fulfill the requirements of the AACC Method 39-00 for process control that require a RPD ≥ 8 (AACC Method 39-00:15). The RPD was introduced by Williams and Norris (2004) as the ratio between the standard deviation of the reference data of the validation set and the standard error of prediction of a cross-validation or of the test set validation.

In the present case, the RPD for the validation of the NIR PLS-R model was 14.7 (Table 2), thereby allowing the conclusion that it is applicable for process control.

Therefore pulp NIR spectra may be used in prediction of ISO brightness. It is noteworthy that once NIR spectra are obtained, they may provide additional information e.g. kappa number of the pulp.

Conclusions

The results demonstrated the potential of using NIR spectra for prediction of ISO brightness of unbleached hardwood pulps in a variation range of 18.9 to 47.9 %, with an accuracy that fulfils the requirements to be used in industrial control processes.

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