Kappa Number Prediction of *Acacia melanoxylon* Unbleached Kraft Pulps using NIR-PLSR Models with a Narrow Interval of Variation

António J. A. Santos,[a,b,c] Ofélia Anjos,[a,d] Rogério Simões,[b] José Rodrigues,[c] and Helena Pereira [a]*

A total of 120 *Acacia melanoxylon* R. Br. (Australian blackwood) stem discs, belonging to 20 trees from four sites in Portugal, were used in this study. The samples were kraft pulped under standard identical conditions targeted to a Kappa number of 15. A Near Infrared (NIR) partial least squares regression (PLSR) model was developed for the Kappa number prediction using 75 pulp samples with a narrow Kappa number variation range of 10 to 17. Very good correlations between NIR spectra of *A. melanoxylon* pulps and Kappa numbers were obtained. Besides the raw spectra, also pre-processed spectra with ten methods were used for PLS analysis (cross validation with 48 samples), and a test set validation was made with 27 samples. The first derivative spectra in the wavenumber range from 6110 to 5440 cm^{-1} yielded the best model with a root mean square error of prediction of 0.4 units of Kappa number, a coefficient of determination of 92.1%, and two PLS components, with the ratios of performance to deviation (RPD) of 3.6 and zero outliers. The obtained NIR-PLSR model for Kappa number determination is sufficiently accurate to be used in screening programs and in quality control.

Keywords: Acacia melanoxylon; Kappa number; NIR; RPD

*Contact information: a: Centro de Estudos Florestais, Instituto Superior de Agronomia, Universidade de Lisboa, Tapada da Ajuda, 1349-017 Lisboa, Portugal; b: Universidade da Beira Interior, Unidade de Materiais Têxteis e Papeleiros, 6201-001 Covilhã, Portugal; c: Tropical Research Institute of Portugal (IICT), Forest and Forest Products Centre, Tapada da Ajuda, 1349-017 Lisboa, Portugal; d: Instituto Politécnico de Castelo Branco, Escola Superior Agrária, Apartado 119, 6001-909 Castelo Branco, Portugal; * Corresponding author: hpereira@isa.ulisboa.pt*

INTRODUCTION

Acacia melanoxylon R. Br. (blackwood) is one of the main wattle species valued for its wood in its natural region of Australia. In Europe, where it was introduced as an ornamental tree and for stabilization of sand dunes (Humphries 1996), the species has not yet found its place in the wood processing industry. However recent studies researched the technological quality of European grown *A. melanoxylon* regarding *e.g.* heartwood variation (Knapic *et al.* 2006), wood basic density (Igartúa and Monteoliva 2009; Santos *et al.* 2012), wood macroscopic properties (Santos *et al.* 2013), and mechanical properties (Santos *et al.* 2007; Machado *et al.* 2014).

A. melanoxylon is also an interesting species for pulping, and pulp yields and properties were already evaluated (Santos *et al.* 2005, 2006, 2012; Anjos *et al.* 2011; Lourenço *et al.* 2008). One important pulping quality parameter is the delignification degree, measured by the residual lignin content in the pulp. Usually it is determined as the

Kappa number, which is used in the pulp industry as a process control tool to maximize yield and quality while minimizing consumption of energy and chemicals.

The determination of Kappa number relies on the oxidation of the residual lignin in the pulp and is a standard method (TAPPI T 236 om-13 1999). This classical wet-chemistry method is, however, tedious and impractical when a large number of samples has to be evaluated, for instance in screening programs. This has led to the search for fast, non-destructive, reproducible, and accurate determination methods.

Near-infrared (NIR) spectroscopy is a method that complies with such requirements and has been applied to determination of various properties in numerous materials. For instance, it has been used to estimate wood chemical components including lignin (Schwanninger and Hinterstoisser 2001; Kelley *et al.* 2004; Alves *et al*. 2006; Yao *et al.* 2010; Zhang *et al.* 2011) and cellulose (Wright *et al.* 1990; Schimleck *et al.* 1997; Poke and Raymond 2006; Hein *et al.* 2009), as well as for the determination of wood properties (Hauksson *et al.* 2001; Kelley *et al.* 2004; Sykes *et al.* 2005) and pulp yield (Michell 1995; Hodge and Woodbridge 2004; Schimleck *et al.* 2005; Downes *et al.* 2009). NIR spectroscopy was also applied for the determination of the Kappa number of hardwood pulps (Easty *et al.* 1990; Michell 1995; Malkavaara and Alen 1998; Fardim *et al.* 2002; Terdwongworakul *et al.* 2005) and of softwood pulps (Malkavaara and Alén 1998; Alves *et al.* 2007). However, in most cases, the models were developed using samples in which the range of Kappa numbers was controlled by varying the pulping conditions, *e.g*. the wood chips were delignified to different degrees targeted to achieve a suitable variation of Kappa numbers *e.g*. 58 to 100 in pine pulps (Alves *et al.* 2007), 30 to 90 in mixed pulps (Antti *et al.* 2000); 3 to 34 in hardwood softwood mixed pulps (Easty 1990), and 16 to 27 in eucalypt pulps (Birkett and Gambino 1989). This does not correspond to normal pulping situations where the pulping conditions are constant and the Kappa numbers may fall within a narrow range of 13 to 20.

This paper focuses on the development of a NIR-PLSR model for the prediction of the Kappa number of Kraft pulps obtained under such conditions, *e.g*. identical pulping conditions and a narrow variation range, using *A. melanoxylon* pulping as a case study. It was aimed at obtaining models performing better than those found in the literature, which are not precise enough for screening purposes, according to AACC Method 39-00 (1999), due to failing the ratios of performance to deviation (RPD) criteria.

EXPERIMENTAL

Sample Preparation

A total of 120 *A. melanoxylon* wood discs, belonging to 20 trees from four sites in Portugal, were used in this study. Detailed information on the samples, sites, and stands is available elsewhere (Santos *et al.* 2013). Samples of 25 g oven-dry wood 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 after a few preliminary experiments to obtain the target Kappa number of 15, as needed for papermaking applications. The pulp was washed according to Santos *et al.* (2012). Under these conditions the pulp yield ranged from 47.0 to 58.2%. 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 Kappa number of the pulp samples was determined according to the standard method TAPPI 236 om-13 (1999). A selection of 75 samples was made from the total samples, based on those with Kappa numbers between 10 and 17.

Pulps discs were produced with the washed and screened pulps, in Glass Vacuum Filter Holder 16307 - Sartorius with 0.2 µm membrane filter, and a diameter matching the diameter of the spinning cup module of the near infrared spectrometer.

Spectra Collection

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

Data Processing

The samples were randomly divided into a calibration set containing 48 samples and a validation set (test set) containing 27 samples by means of a principal component analysis. The processing was done according to Alves *et al.* (2007) in two steps. First, the infrared data from the calibration samples were regressed against the Kappa number, 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 (BRUKER). Besides using the raw spectra, the following pre-processing methods were applied prior to calculation of the PLSR models: multiplicative scatter correction (MSC), first derivative (1stDer), second derivative (2ndDer), vector normalization (VecNor), straight line subtraction (SLS), minimum maximum normalization (MinMax), constant offset (ConOff), and combinations of them. In a second step, the validation of the PLSR models was performed using the independent test set.

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 (Gierlinger *et al.* 2002; Alves *et al.* 2007).

RESULTS AND DISCUSSION

The Kappa number of the unbleached pulps of *A. melanoxylon* obtained under the same pulping conditions ranged from 10.0 to 16.8, with an average of 13.6 and a standard deviation of 1.3 (Table 1), corresponding to a sample with a narrow range of Kappa numbers, as it was the objective of this work. The two sets showed similar statistics.

The spectral range from 6110 to 5440 cm^{-1} was used for calibration. This range was found by automated optimization and has also already used by Alves *et al.* (2007). The NIR based data from the 48 samples of the calibration set were regressed against their experimentally determined Kappa number, and the results obtained with the various pre-processing of the raw spectral data are summarized in Table 2. The obtained rank ranged from one (2ndDer) to five (no spectral pre-processing), while the coefficients of determination (R^2) ranged from 76.4% to 87.9%, and the root mean square error of cross validation (RMSECV) from 0.5 to 0.6. When using the test set validation, the coefficients of determination ranged from 85.7% to 92.1%.

Table 1. Number of Samples and Range of Kappa Number in the Calibration Set and Test Set (SD is standard deviation)

	Calibration set	Test set
Nº of samples	48	27
Maximum	16.8	15.7
Minimum	10.0	10.3
Average	13.6	13.7
SD	1.3	1.4

The best model using the first derivative (1stDer) of the spectral data was selected, and the NIR-PLSR predicted versus the laboratorial determined Kappa numbers are shown in Fig. 1. Both the cross-validation and the validation show high correlation between the predicted and the determined Kappa number values, with an RMSEP of 0.4, a rank of three, and no outliers.

Table 2. Results for Cross-validation and Test Set Validation*

Pre processing	Nº of samples	Rank	R^2 (%)	RMSECV	RPD	Rank	R^2 (%)	RMSEP	RPD	OS (%)	OL (%)
MinMax	48	3	78.9	0.6	2.2	4	86.7	0.5	2.8	0.0	0.0
1stDer+MSC	48	2	81.6	0.6	2.3	2	89.4	0.4	3.3	0.0	0.0
MSC	47	3	87.9	0.5	2.9	3	85.7	0.5	2.7	0.0	0.0
ConOff	47	4	76.4	0.6	2.1	3	90.6	0.4	3.3	0.0	0.0
None	46	5	86.3	0.5	2.7	5	88.3	0.5	3.0	0.0	0.0
2ndDer	46	1	81.5	0.5	2.3	1	89.2	0.4	3.2	0.0	1.4
1stDer	**47**	**3**	**79.0**	**0.6**	**2.2**	**3**	**92.1**	**0.4**	**3.6**	**0.0**	**0.0**
1stDer+SLS	48	3	79.6	0.6	2.2	2	90.4	0.4	3.3	0.0	0.0
VecNor	47	3	78.5	0.6	2.2	3	85.7	0.5	2.7	0.0	0.0
1stDer+VecNor	48	2	81.7	0.6	2.3	3	88.0	0.5	2.9	0.0	1.4
SLS	46	3	82.4	0.5	2.4	3	91.2	0.4	3.6	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: second derivative; 1stDer: first derivative; 1stDer+SLS: first derivative + straight line subtraction; VecNor: vector normalization; 1stDer+VecNor: first derivative + vector normalization; SLS - straight line elimination. Rank: number of PLS components; RPD: residual prediction deviation; RMSECV: root mean square error of cross validation; RMSEP: root mean square error of prediction.
* Also shown are percentage of outsiders (OS) and outliers (OL) of the test set obtained during prediction of samples with unknown Kappa number, using various preprocessing methods

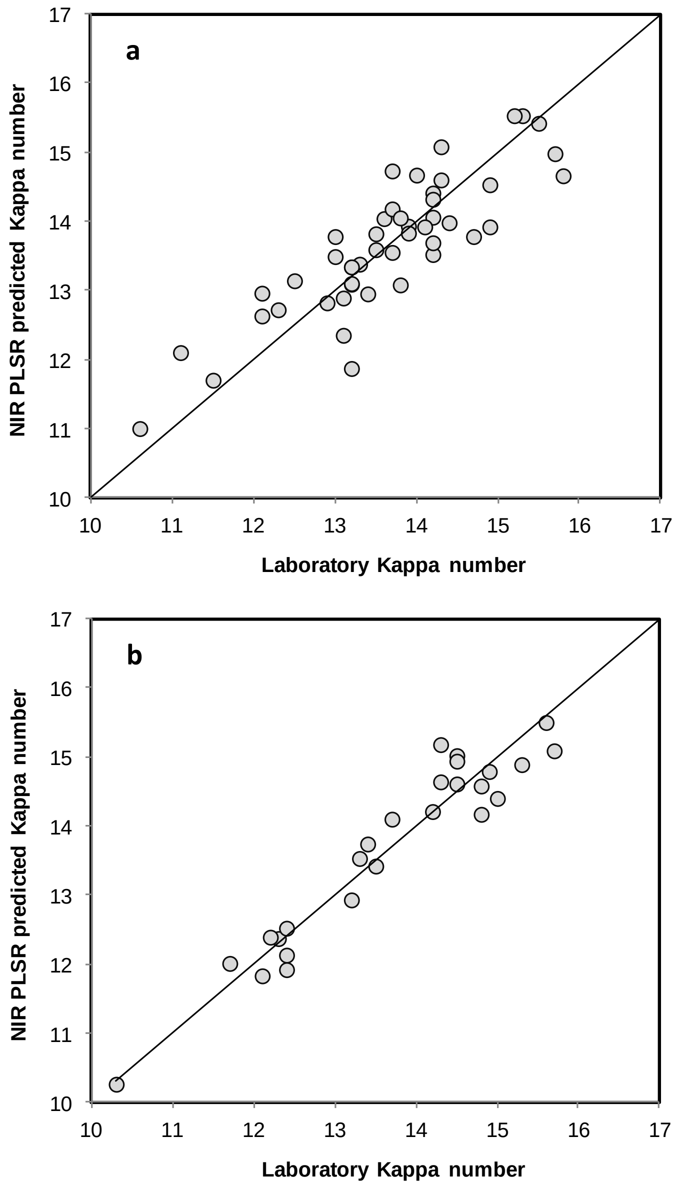

Fig. 1. NIR PLSR predicted versus laboratory determined Kappa number for blackwood pulps: a) cross validation (47 samples), b) test set validation (27 samples).

This is the first time that the Kappa number of hardwood unbleached kraft pulps could be predicted with such high accuracy and precision (0.6 Kappa units). In fact, previous models for Kappa number determination using NIR spectral data showed higher prediction errors, as summarized in Table 3.

Zhang *et al.* (2011) developed a model for *Acacia* spp. with root mean square error of cross validation (RMSECV) of 1.0 units of Kappa number and predictive ability corresponding to a RMSEP of 0.8 units of Kappa number, while Monrroy *et al*. (2008) reported 3.6 and 2.9 units of Kappa, respectively for *Eucalyptus globulus*. Alves *et al.* (2007) reported a root mean square error of prediction of 2.3 units of Kappa number for *Pinus pinaster* kraft pulps. Antti *et al*. (2000) developed a model with a predictive ability corresponding to a RMSEP of 1.7. Easty *et al.* (1990) calibrated the Kappa number of hardwood-softwood blends using the second derivative of the NIR spectra at 5952 cm^{-1} with an R^2 of 0.99, but did not validate the result.

Birkett and Gambino (1989) determined multiple linear regression calibration models for Kappa number with 95% confidence limits of ± 1.4 for *Eucalyptus grandis* pulp and of ± 2.1 for mixed pine pulps. The best model so far published presents a root mean square error of prediction of 0.8 units of Kappa number, a coefficient of determination of 80.0 % within a range of variation of 14 Kappa units. The model developed with the largest Kappa units range (71) has a root mean square error of prediction of cross validation of 3.2.

Table 3. Kappa Range of the Different Models Published in the Bibliography

Species	References	Kappa range	R^2	RMSECV	RMSEP
Acacia spp.	Zhang *et al*. 2011	12.5 - 26.3	85.0 / 80.0	1.0	0.8
Eucalyptus globulus	Monrroy *et al.* 2008	8.5 – 62.4	97.0 / 93.0	3.6	2.9
Pinus pinaster	Alves *et al.* 2007	58.1 - 100.3	95.0 / 95.9	2.4	2.3
Mixed kraft pulp	Antti *et al.* 2000	30 – 90	98.2	-	1.7
hardwood-softwood mixtures	Easty *et al.* 1990	3.4 - 33.9	99.0	-	-
Eucalyptus grandis kraft pulp	Birkett and Gambino 1989	16 – 27	96.3	1.4	-
mixed-eucalyptus pulp		16 – 27	93.1	1.9	-
mixed-pine kraft pulp		24.7 - 87.8	99.8	2.1	-
all pulp samples		16 - 87.8	99.4	3.2	-

The ratios of performance to deviation (RPD) may be used to evaluate whether the prediction models fulfill the requirements of AACC Method 39-00 for screening in breeding programs or for quality control that require a RPD ≥ 2.5 or RPD ≤ 5, respectively (AACC 1999). 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 this case, the RPD for the validation of the NIR PLS-R model was 3.6 (Table 2), thereby allowing the conclusion that the method is applicable for screening in breeding programs.

The use of this statistical tool to assess near infrared-based partial least squares regression (NIR PLS-R) models has been very infrequent for forest products. The only published use of RPDs was dedicated to wood density (Santos *et al.* 2012; Alves *et al.* 2012; Inagaki *et al.* 2012).

A reliable model will be obtained only if the model is able to predict the test samples in a proper way (Antti *et al.* 2000; 1996), as it is commonly agreed. But according to the results presented here, this is not sufficient, because complementary data of an external validation is needed, stressing the importance of an additional step (evaluation) before making the decision in model selection. Following that, the 1stDer spectra gave the best model, with the highest coefficient of determination (R^2), lowest root mean square error of prediction (RMSEP), and highest residual prediction deviation or ratio of performance to deviation (RPD).

CONCLUSIONS

1. The results demonstrated the potential of using NIR spectra for prediction of Kappa numbers in well delignified unbleached hardwood pulps with an accuracy that fulfils the requirements to be used in screening programs and in quality control processes.
2. The developed NIR-PLSR models improved the prediction ability in relation to previously published models and showed the possibility of using a narrow range of Kappa number values to perform high quality calibration and validation processes.

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