PREDICTION OF RADIAL AND TANGENTIAL SHRINKAGES BY NEAR-INFRARED SPECTROSCOPY: AN EXAMPLE FOR *Tectona grandis* FROM TOGO AND FOR *Liquidambar styraciflua* FROM MADAGASCAR

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ABSTRACT: Dimensional stability is one of the most important properties of wood used as timber. However, it is very time-consuming to measure this trait. Near-infrared spectroscopy is a tool adapted for prediction of many properties of wood, as chemical contents, basic density, and the time needed for the analysis is highly reduced. The issue here is to check the effectiveness of NIRS tool to build models and to predict the radial and tangential shrinkages of Teak (Togo) and Liquidambar (Madagascar) wood both from plantations. It was possible to predict the dimensional stability by means of NIR spectroscopy: For Teak, the transversal cross-section spectra yielded better NIRS-based models while for Liquidambar, the tangential surface gave the best statistics model. For the two species, the prediction models for tangential shrinkage showed similar results in term of efficiency. After independent validation or cross-validation, respectively for Teak, and for Liquidambar wood, we assume that NIRS can be used to predict radial and tangential shrinkages for screening.

Key words: Dimensional stability, Liquidambar, Teak, NIRS.

PREDIÇÃO DA RETRATIBILIDADE RADIAL E TANGENCIAL POR ESPECTROSCOPIA NO INFRAVERMELHO PRÓXIMO: UM EXEMPLO DE *Tectona grandis* DO TOGO E DE *Liquidambar styraciflua* DE MADAGASCAR

RESUMO*: A estabilidade dimensional é uma das mais importantes propriedades físicas da madeira como material estrutural. No entanto, a medição dessa propriedade consome tempo. A espectroscopia no infravermelho próximo é uma ferramenta adaptada para predição de muitas propriedades da madeira, como as químicas e a densidade básica e o tempo requerido para análise é altamente reduzido. A questão aqui é checar a efetividade da ferramenta NIRS para a construção de modelos e para a predição da retratibilidade radial e tangencial na madeira de teca (Togo) e âmbar (Madagascar) de plantações. O resultado mostrou o possível uso do NIRS para a predição da estabilidade dimensional; para Teca, os espectros medidos na face transversal renderam os melhores modelos, enquanto em Âmbar, os espectros tangenciais produziram as melhores estatísticas. Para as duas espécies, os modelos de predição para retratibilidade tangencial mostraram resultados similares em termos de eficiência. Após validação independente e validação cruzada, respectivamente, para a madeira de Teca e de Âmbar, assume-se que o NIRS pode ser utilizado para predizer a retratibilidade tangencial e radial para análises preliminares.*

Palavras-chave: Estabilidade dimensional, Âmbar, Teca, NIRS.

1 INTRODUCTION

The main wood quality factors related to dimensional characteristics are tangential and radial shrinkages. Shrinkage is affected by several properties, extractives contents especially (HERNANDEZ, 2007; STAMM, 1971; TAYLOR, 2008). Teak has been reported as presenting shrinkage of 2.5 to 3.0% in the radial direction and 3.4 to 5.8% in the tangential direction (SIMATUPANG; YAMAMOTO, 2000; TROKENBRODT; JOSUE, 1999). The good dimensional stability of Teak wood is mainly due to the bulking effect of the ethanol and hot water soluble wood extractives located in the cell wall (SIMATUPANG; YAMAMOTO, 2000). Some results have been published

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on the shrinkage of Teak wood (BHAT, 1998; SANWO, 1987; SIMATUPANG; YAMAMOTO, 2000). Shrinkage of *Liquidambar stryraciflua* (Sweetgum), which is less studied, has been reported as presenting shrinkage of 7% in the radial direction and 13% in the tangential one (RAKOTONDRAOELINA; RAKOTOVAO, 2005).

Determination of shrinkage is based on tests that require destructive sampling and extensive sample preparation. Breeders and industries would benefit from employing a more rapid, non-destructive technique for the estimation of these properties. An option for the estimation of these wood properties is near infrared spectroscopy (NIRS). The potential application of NIRS to predict wood characteristics has been reported in the literature (KELLEY et al., 2004; SO et al. 2004; TSUCHIKAWA, 2007). For studies concerning forest product, there is few references in which NIRS is used to assess shrinkage (BAILLÈRES; DAVRIEUX; HAM-PICHAVANT, 2002; TAYLOR, 2008).

This paper evaluates the potential of NIR spectroscopy for the assessment of shrinkages for *Tectona grandis* and *Liquidambar styraciflua* woods both from plantations.

2 MATERIAL AND METHODS

2.1 Wood samples

Twenty trees of Teak were select and cut for analysis from two plots in Togo, namely Tchorogo (34 years old) and Oyou (40 years old). Twenty seven trees of Liquidambar (3 by 9 provenances) were select and cut in provenance trial (23 years old), namely Mandraka in Madagascar. Planks were cut radially (500 mm in the longitudinal axis and 50mm in the tangential axis) between a height of 1 and 1.5 meters. Four beams were cut per plank along the diameter (20 x 20 x 500 mm) and lastly, 5 (Teak) and 2 (Liquidambar) wood samples (20 x 20 x 10 mm) were cut per beam. The samples were stabilized to a theoretical MC of 12% in a climate chamber at 20°C and 65% humidity. A total of 393 and 35 samples were used for measuring dimensions, and NIR absorbance for Teak and Liquidambar respectively. For Teak, 2 NIR measurements were recorded (LR and RT surfaces) per sample, for Liquidambar one additional was recorded (LT surface).

2.2 Determination of shrinkages

First, the samples were immersed in water until saturation after several sequences of vacuum and pressure. Then, the samples were dried to the anhydrous state, by oven drying at 103 ± 2°C. For the two states, all samples were weighed and radial and tangential dimensions were measured with a displacement transducer. Radial shrinkage (RS) and tangential shrinkage (TS) were obtained using the equation (1) between the saturation state and the final oven dried state:

$$S = \frac{D_{SAT} - D_{0\%}}{D_{SAT}} \quad (1)$$

where S: shrinkage, D_{SAT}: saturated dimension, $D_{0\%}$: dried dimension.

2.3 Near infrared spectra collection

Near-infrared spectra were collected in the NIR region from 12,500 to 3,800 cm^{-1} (800-2,850 nm) with an NIR spectrometer (Bruker model Vector 22/NI) in diffuse reflectance mode at a spectral resolution of 8 cm^{-1}. Spectra taken from tangential-radial (TR) and longitudinal-radial (LR) surfaces (sample used for dimensional stability) and longitudinal-tangential (LT for Liquidambar only) were used in the calibration modelling. Each spectrum was obtained with 32 scans, and means were calculated and compared to the gold standard in order to obtain the absorption spectrum. Temperature and relative humidity were kept constant (20°C, 65%) throughout the NIR processing.

2.4 Data processing

For Teak, a validation set was established by a selection of 100 samples among the 393 samples based on the sample distances calculated by five components after a principal component analysis on the spectral data. We assumed that these covered the shrinkage ranges. Then, the spectra data from 293 remaining samples were regressed against the TS and RS, and using 5 random cross validation groups, the number of Partial Least Squares (PLS) components (rank) was obtained using Unscrambler 9.8 (Camo). The PLS models were then used to predict data of the validation set, evaluating their predictive ability. For Liquidambar, we used full cross-validation process because the low number of sample didn't allow us to use the Teak approach. According to Kokutse et al. (2010), we selected SNV (standard normal variate) and derivative 2, for which effects were explained, as NIR spectra pre-processing.

We detected and discarded outliers samples whose the predicted value lies outside the calibration range, and samples whose Mahalanobis distance were too large, meaning that the similarity of the spectra compared to the calibration spectra was too big. The quality and the final selection of the models were assessed by coefficient of determination (R^2) from reference values and predicted values by the models, standard error of calibration (SEC) and standard error of cross-validation (SECV).

$$SEC = \sqrt{\frac{\sum_{i=1}^{N_C}(Y_i - \hat{Y}_i)^2}{N_C - k - 1}}.$$

(2) Where Y_i is the known value of the constituent of interest of sample i, $\hat{Y}_i$ is the predicted value, N_C is the number of samples used to obtain the calibration, and k is the number of factors used to obtain the calibration.

Validation set for Teak was used to test the model's power. The performance of models was evaluated by the coefficient of determination (R^2) from reference values and predicted values and the standard error of prediction (SEP, Eq. 3).

$$SEP = \sqrt{\frac{\sum_{j=1}^{N_P}(Y_j - \hat{Y}_j)^2}{N_P - 1}}.$$

(3) Where Y_j is the known value of the constituent of interest of sample j, $\hat{Y}_j$ is the predicted value, and Np is the number of samples in the prediction set.

Models were tested by the Ratio of Performance Deviation (RPD) which is the ratio of the standard error SD (deviation for the reference method values) of sample validation divided by SEP. RPD_{CV} for Liquidambar is the ratio of the standard error SD (deviation for the reference method values) of sample divided by SECV.

3 RESULTS AND DISCUSSION

3.1 Calibration – Teak

For all samples, the radial and tangential shrinkages varied from 1.7% to 5.4% and from 2.3% to 9.3% respectively, with a standard deviation of 0.7 and 1.3 (Table 1). Tangential shrinkage is more variable than radial shrinkage. The coefficient of variation for radial and tangential shrinkage is 22% and 24%.

The calibration results are given in Table 2. All the models are highly significant with determination coefficients greater or equal to 0.74. The models calculated on the TR surface absorbance perform better than the model based on LR surface. The r^2 are systematically higher. In addition, the TS models are better than the RS models, not taking in consideration the TR surface. For the shrinkage properties, the SEC is more than 10 times higher than the SEL which is equal to approximately 0.026.

3.2 Validation – Teak

The population used to validate the models has statistical characteristics similar to those of the calibration population (Table 3). The variability percentages explained by the validation models are lower than those for calibration but the difference is slight (from to 7 - 14 points for RS). The findings for calibration are also found in the validation population in Figure 1 and Table 3; i.e. (a) the models for the TR surface are higher than the models on the LR surface, (b) the TS models are better than the RS models. The values found for the RPD show that these models which RPD>2 may be used for rough screening according to the reference given by Williams and Sobering (1993).

3.3 Calibration and full cross-validation – Liquidambar

The radial and tangential shrinkages varied from 4.4% to 8.8% and from 10.6% to 16.2% respectively, with a standard deviation of 1.0 and 1.2% (Table 1). Tangential shrinkage is slightly more variable than radial shrinkage. The coefficient of variation for radial and tangential shrinkage is 17% and 10%.

The models have a determination coefficient greater or equal to 0.52 (Table 4). The performances of models decrease from LR, LT and TR surfaces according to NIR

Table 1 – Descriptive statistics of reference values for Teak and Liquidambar.

Tabela 1 – *Estatística descritiva dos valores de referência para Teca e Âmbar.*

Species	Shrinkage	N	M	SD	CV	Min	Max	SEL
Tectona grandis	Radial	393	3.2	0.7	22	1.7	5.4	0.026
	Tangential	393	5.5	1.3	24	2.3	9.3	0.026
Liquidambar styraciflua	Radial	35	5.6	1.0	17	4.4	8.8	-
	Tangential	35	12.5	1.2	10	10.6	16.2	-

N: Number of samples, M: mean (%). SD: standard deviation (%). CV: coefficient of variation (%). Min: minimum value (%). Max: maximum value (%). SEL: standard error laboratory (KOKUTSE; BRANCHERIAU; CHAIX, 2010)

Table 2 – Results of the PLS calibration and cross-validation on SNV and 2nd Derivate pre-processing spectral data for Teak.

Tabela 2 *– Resultados da calibração e validação cruzada de PLS para os espectros tratados por SNV e segunda derivada para Teca.*

NIR surface	Shrinkage	N	Outliers	M	SD	Rank	SEC	SECV	r^2
LR	Radial	293	10	3.1	0.68	4	0.35	0.39	0.74
	Tangential	293	10	5.5	1.24	4	0.52	0.59	0.83
TR	Radial	293	10	3.1	0.7	5	0.26	0.30	0.86
	Tangential	293	12	5.5	1.23	5	0.43	0.48	0.88

N: number of sample. M: mean. SD: standard deviation. SEC: standard error of calibration. SECV: standard error of cross validation.

Table 3 – Results of PLS test-validation of shrinkage for Teak.

Tabela 3 *– Resultados para teste de validação PLS da retratibilidade para Teca.*

NIR surface	Shrinkage	N	Outliers	M	SD	SEP	r^2	RPD
LR	Radial	100	1	3.21	0.65	0.37	0.67	1.8
	Tangential	100	3	5.68	1.27	0.60	0.78	2.1
TR	Radial	100	3	3.21	0.65	0.36	0.72	1.8
	Tangential	100	4	5.64	1.27	0.52	0.83	2.4

N: number of sample. M: mean. SD: standard deviation. SEP: standard error of prediction. RPD: ratio performance to deviation.

measurement. The r^2 are systematically higher for tangential shrinkage models.

In spite of the difference of sample number because the work began for Liquidambar recently, the results showed the same tendency. Increasing number of Liquidambar wood samples would confirm results obtained here and allow using NIRS for shrinkage prediction. The models developed for the TR section are better than the models developed for the LR section (Tables 3 and 4) for both species and for LT surface (for Liquidambar). It is thought that this observation may be explained, firstly by the fact that the width of the LR surface of the sample corresponds to the dimension of the infrared beam (10mm). Operational errors in positioning the samples may therefore have added noise to the experiment measurements. Furthermore, the infrared beam touches all the anatomical elements directly on the TR surface while the same elements are measured by diffuse reflection inside the material in the LR and LT surfaces.

With respect to the shrinkage properties, the models for TS are systematically better than the RS models both for the two species (Tables 2, 3, and 4). However, for radial shrinkage there is a higher measurement error than for tangential shrinkage, but according to the two species, the coefficient of variation for TS compared to RS is higher for Teak and lower for Liquidambar. Radial shrinkage closely depends on anatomic factors above and beyond individual cell structure and composition. Among them, the major factor that affects shrinkage is the restraint of radial shrinkage by rays because of the low shrinkage potential and high stiffness as compared to tissues of longitudinally aligned cells (BAILLÈRES; DAVRIEUX; HAM-PICHAVANT, 2002). RS is therefore probably more dependent on the cellular organization, which does not influence spectra measured by the NIRS technique (HERNANDEZ, 2007). Other explanation is the fact that the measurement error for TS (0.9%) is lower than the RS measurement error (1.6%). It is also thought that there is an operational effect, the measurements were always taken in the same order: tangential then radial. It is possible therefore that additional humidity between the two measurements may have affected the radial measurements.

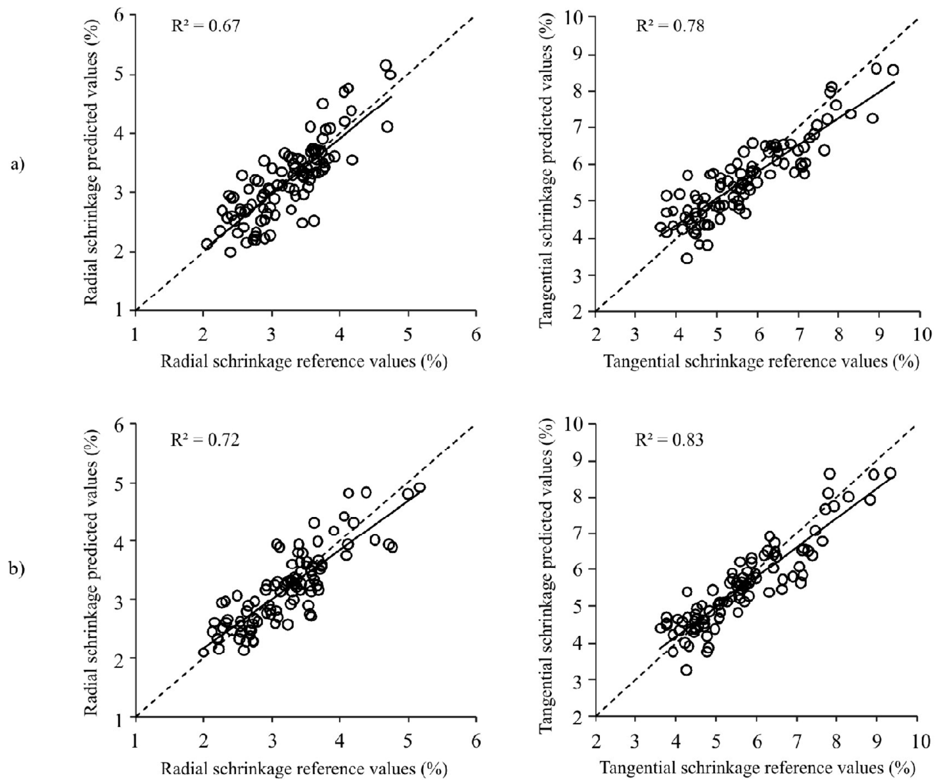

Figure 1 – Validation results of PLS models: NIR-predicted versus laboratory determined for radial and tangential shrinkage in *Tectona grandis* (A: NIR data from Longitudinal-Radial surface, B: NIR data from Tangential-Radial surface).

Figura 1 *– Resultados de validação dos modelos PLS: valores preditos por NIR e determinados em laboratório para retratibilidade radial e tangencial em Tectona grandis (A: NIR a partir da face longitudinal-radial, B: NIR a partir da face radial-tangencial).*

Table 4 – Results of the PLS calibration and cross-validation on SNV and 2nd Derivate pre-processing spectral data for shrinkage of Liquidambar.

Tabela 4 *– Resultados da calibração e validação cruzada PLS para os espectros tratados por SNV e segunda derivada para retratibilidade do âmbar.*

NIR surface	Shrinkage	N	Outliers	M	SD	Rank	SEC	SECV	r^2	RPD_{cv}
LR	Radial	35	3	5.4	0.64	3	0.30	0.38	0.67	1.7
	Tangential	35	2	12.5	1.30	5	0.22	0.42	0.89	3.1
TR	Radial	35	3	5.4	0.65	4	0.19	0.35	0.71	1.9
	Tangential	35	0	12.5	1.30	2	0.57	0.63	0.76	2.1
LT	Radial	35	1	5.4	0.79	3	0.44	0.56	0.52	1.4
	Tangential	35	1	12.4	1.20	4	0.28	0.48	0.85	2.5

N: number of sample. M: mean. SD: standard deviation. SEC: standard error of calibration. SECV: standard error of cross validation. RPD_{cv}: ratio performance to deviation.

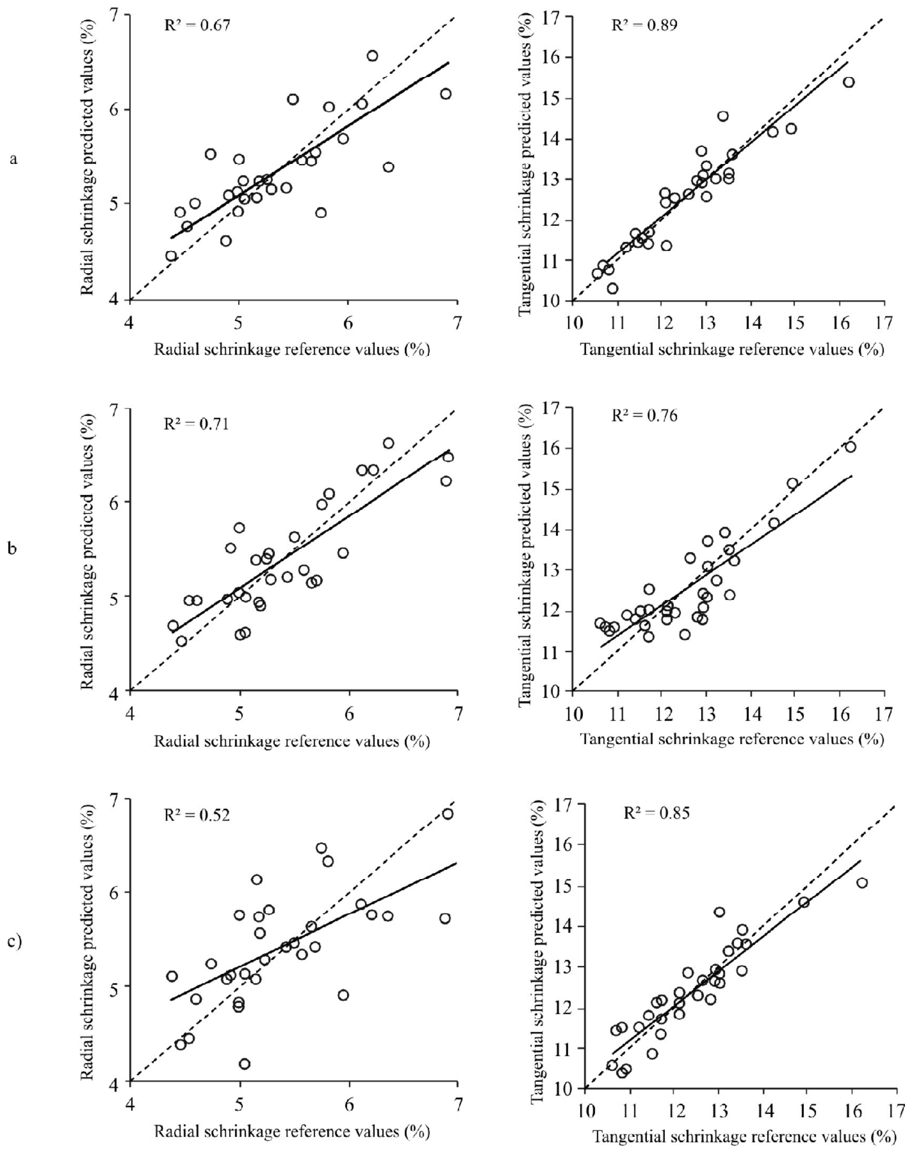

Figure 2 – Cross-validation results of PLS models: NIR-predicted versus laboratory values determined for radial and tangential shrinkage in *Liquidambar styraciflua* (A: NIR data from Longitudinal-Radial surface, B: NIR data from Tangential-Radial surface, C: NIR data from Longitudinal-Tangential surface).

Figura 2 *– Resultados de validação cruzada dos modelos PLS: valores preditos por NIR versus valores determinados em laboratório para retratibilidade radial e tangencial em Liquidambar styraciflua (A: NIR a partir da face longitudinal-radial, B: NIR a partir da face radial-tangencial, C: NIR a partir da face longitudinal-tangential).*

The statistical models obtained by NIRS calibration demonstrate highly significant levels of correlation between the predicted values and the reference values. Shrinkage is affected by the extractives (AREVALO FUENTES, 2002; BREMAUD, 2006; CHAFE, 1987; HERNANDEZ, 2007; TAYLOR, 2008). Because of the relationship of density and extractive content to wood shrinkage, it is perhaps not surprising that NIR spectra could also provide good predictions of total volumetric shrinkage. In the transverse direction, the cellular organization and cross-sectional shape of the cells play an important role in shrinkage. But the anatomy of the material does not however appear to play a determining role in adsorption.

4 CONCLUSION

The findings of this study demonstrate the possible use of NIRS to estimate the dimensional stability of Teak and Liquidambar woods. The precision of the models developed is lower than the reference measurement but its value and the rapidity of measurement undisputedly show advantages for this approach. In accordance with the reference list commonly adopted, the RPD values obtained after validation for the best prediction models, whether from ranges of solid woods, are sufficient for screening.

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