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Original Article

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Influence of Age on the Discrimination of *Tectona grandis* by **VIS/NIR Spectroscopy**

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ABSTRACT

Wood color and properties are variables among and within species, and fast and non-destructive techniques can be applied to their characterization, being also important in wood marketing and quality control. This paper evaluated the influence of age on the discrimination of Tectona grandis L.F. (teak) wood by VIS/NIR spectroscopy. Wood from three ages, with heartwood and sapwood, were studied, totaling 36 samples per age. Quantitative colorimetric data, based on CIELAB 1976, visible and NIR infrared spectra were collected from radial and tangential surfaces in five positions of each sample, in a total of 540 spectra. Both techniques were adequate for age discrimination in teak wood. Statistical differences were observed in the chromatic coordinates in heartwood and sapwood for each age. VIS/NIR spectroscopy can be applied for age discrimination based on teak solid samples and for wood quality control.

Keywords: spectroscopy, species characterization, wood discrimination.

1. INTRODUCTION

Wood and its products present technological properties determined by different analytical tests. In general, tests are performed using samples and traditional methods that can be destructive, expensive and hard-working. For forest-based industries, the characterization and performance of a detailed quality control of products is not always an easy task (Muñiz et al., 2012). So, it is necessary the implementation of new technologies for the previous knowledge of wood properties (Amorim et al., 2013).

One characterization technique that can be applied is the quantitative or qualitative colorimetric analysis, which is performed on colorimeters or spectrophotometers, where reflectance curves of samples are analyzed based on wavelengths (Gonçalez et al., 2001). CIELAB 1976 system is the most widely applied technique for color characterization because it presents uniform space for color distribution based on three axis: L*, a* and b* (Gonçalez et al., 2001). Luminosity value (L*) is approximately the luminance value (Y) from white to black; chromatic coordinate a* varies from green to red, and chromatic coordinate b* varies from blue to yellow, based on the human brain perceptions of opposite colors.

In the wood sector, color is an important feature to determine the final use in large scale of a particular species, and this characteristic can increase the commercial value of some species based on patterns such as "mahogany" (Camargos & Gonçalez, 2001). In addition, some properties can be evaluated based on colorimetric parameters (Amorim et al., 2013) and color can be applied on pre-classification and qualification of wood logs (Barros et al., 2014). For species recognition, color is generally presented in studies with images and classification methods like artificial neural networks and other recognition patterns (Bombardier & Schmitt, 2010; Peng, 2013).

Another non-destructive technique is near infrared spectroscopy (NIR), which applies energy of 750-2500 nm (Pasquini, 2003) and information can be directly collected from material surface. In forest industry, NIR has been used online for the detection of chemical, physical and mechanical properties of some lignocellulosic materials (Tsuchikawa & Schwanninger, 2013). For wood discrimination, studies have shown the efficiency of NIR for different particle size of materials (Braga et al., 2011; Pastore et al., 2011; Sandak et al., 2011; Nisgoski et al., 2015a) and diverse pretreatment and classification methods (Brunner et al., 1996, Oliveira et al., 2015).

Tectona grandis (teak) wood is well-known by its decorative effect and resistance, so it is widely applied in naval construction, civil construction, floor and decks, also in furniture, decorative veneers and ornament in general, and it is mostly planted in the Midwestern region of Brazil (ABRAF, 2013). Teak wood also presents adequate relation between strength and specific gravity, tension and static bending, and high natural durability (Crespo et al., 2008). In 2015, the total Teak planted area in Brazil was 87410 hectares, having increased 33% in relation to 2010 (IBÁ, 2016).

Tectona grandis has been widely reforested in Brazil and its wood properties present high technological potential. However, further studies about this specie should be carried out using non-destructive and quick methods. The possibility of determining the wood age may help and stimulate its use, once wood properties present differences throughout its life cycle. Some of these chronological changes are related to characteristics such as specific mass and chemical, mechanical, physical, anatomical and biological properties. Consequently, the aim of the present paper was to evaluate a simple method to determine the age of *Tectona grandis* wood through VIS/NIR spectrometry and to increase the database of VIS/NIR spectra of solid samples.

2. MATERIAL AND METHODS

Tectona grandis trees came from São José do Rio Claro, state of Mato Grosso, Brazil, with 10, 13 and 17 years of age. For each age, boards were produced, obtaining six samples with dimensions of 2x2x3 cm in three positions: 0%, 50% and 100% of board, with heartwood and sapwood, in a total of 36 samples per age. VIS/NIR data were collected in radial and tangential surface of five points, in a total of 540 spectra. For posterior analysis, an average of five data were applied, namely 36 spectra per age. Samples were dried in kiln to reach 12% moisture content and remained in climatic room until analysis.

Colorimetric evaluation was performed with Konica Minolta CM-5 spectrophotometer, with spectral range from 360 to 750 nm, D65 light source and 10° observation angle (CIELAB standard). Five measurements of each sample were performed from radial and tangential surfaces, from which lightness (L*), green-red chromatic coordinate (a*) and blue-yellow chromatic coordinate (b*) were obtained. Data were analyzed using descriptive statistics and regression analysis. Chroma (C) and hue angle (h*) were calculated by Equations 1 and 2.

$$C = \sqrt{(a^{*2} + b^{*2})} \tag{1}$$

$$h^* = tan^{-1}(b^*/a^*)$$
 (2)

Where: C = chroma; a^* = green-red coordinate; b^* = blue-yellow coordinate; h^* = hue angle.

Infrared analyses were performed with Bruker Tensor 37 spectrometer (Bruker Optics, Ettlingen, Germany) equipped with an integrating sphere and operating in reflectance mode; 64 scans were averaged with resolution of 4 cm⁻¹ and spectral range from 10,000 to 4,000 cm⁻¹. In room at temperature of 23 ± 2 °C and relative humidity of 60%, wood samples were placed on top of the integrating sphere and five spectra were obtained from radial and tangential surfaces.

The Unscrambler X chemometric software (version 10.1, from CAMO Software AS) was used to analyze data. Exploratory modeling was performed by analyzing the score and loading graphs obtained by principal component analysis (PCA) to verify possible differences based on sample ages. Second derivative of Savitzy-Golay (polynomial order = 2, smoothing point = 3) was applied to raw data. Spectral analysis was based on ASTM E1655-05 (ASTM, 2000).

The statistical analysis of results was performed in a completely randomized design. When differences among treatments were significant, the Tukey test was applied at 5% significance level for the comparison of means.

3. RESULTS AND DISCUSSION

3.1. Colorimetry

Reflectance curves in visible spectra (Figure 1) of teak at three ages present distinction in two groups: heartwood and sapwood. It was expected because samples are visually darker and lighter, respectively. Also it is observed that reflection decrease with increasing age in both groups.

Color variation can be related to density differences in teak and other wood species (Garcia et al., 2014; Garcia & Marinonio, 2016). Cremonez et al. (2015) analyzing the same teak samples of this study obtained apparent density of 0.54, 0.62 and 0.65 g/cm³ for ages 10, 13 and 17 years, respectively. So, differences in reflectance curves can be related to density.



Figure 1. Reflectance in visible spectra for teak heartwood and sapwood at different ages.

The colorimetric parameters of teak wood for three ages are in Table 1. Based on color classification reported by Camargos & Gonçalez (2001), wood from sapwood is white-gray and from heartwood in brown-olive, for all ages. The same color pattern was obtained for teak sapwood (Lopes et al., 2014) and heartwood (Queiroz et al., 2016) in other studies.

The results showed a decrease in luminosity (L^*) as a function of age for sapwood and heartwood, resulting in darker material. Chromatic coordinate a^{*} increased with age, and as a result, red pigment is predominant in color formation. Chromatic coordinate b^{*} increased in sapwood, representing a more yellow tone; however, this value decreased in heartwood as a function of age. Chroma (C^{*}) presents more influence on sapwood, showing more intense color as a function of age.

Chemical composition, mainly extractives, which in function of tree age are accumulated on cell walls, are responsible for wood color alterations, which selectively absorb light from different origin. Garcia & Marinonio (2016) reported that teak heartwood presents more extractives and is darker. The authors studied 12-year-old wood and obtained extractive percentage from 1.2 to 4.35%. Another study on teak at 4, 6 and 12 years showed extractive percentages of 4.39-3.59-4.76% respectively (Chagas et al., 2014). In older trees, 50-70 years, Miranda et al. (2011) obtained 9.2% of extractives, and Haupt et al. (2003) showed results of 8.8-9.4% for 29-year-old wood. For trees old as 35 years, Thulasidas & Bhat (2009) presented 13% of extractives. This result confirms that higher extractive content in older trees contributes do darkening.

Another characteristic that presents influence on color parameters is density. Garcia & Marinonio (2016) concluded that luminosity decreased as a function of an increase in sapwood density; and chromatic coordinates a* and b* presented positive correlation with sapwood density, but neither are directly related to heartwood. This behavior support results obtained for ages of 10-13-17 years in this study.

To illustrate the distribution of samples at different ages, a graph of the principal component analysis of the reflectance curve is shown in Figure 2. For heartwood and sapwood, there is a distinction from samples in each age, and it is possible to apply visible spectroscopy to discriminate teak based on age information. Visible spectroscopy was also efficient in the discrimination of pine species based on needles (Nisgoski et al., 2015b).

3.2. NIR spectroscopy

Mean spectra in NIR infrared from teak at different ages (Figure 3) show the same pattern for heartwood and sapwood. When analysis is made separately, it is possible to verify that there is an increase in absorbance values for higher ages.

Some irregularities in heartwood spectra may be the result of humidity in samples or equipment, especially near 7300 and 5200 cm⁻¹ (Figure 3). Other bands are related to the cell wall composition and presence of extractives (Schwanninger et al., 2011). To eliminate noise or other effects in spectra, second derivative pretreatment was applied. This preprocessing has already been applied in literature (Sandak et al.,

Position	Age	L*	a*	b*	С	h*
Sapwood	10	77.41 E (3.01)	4.55 A (0.83)	21.14 A (2.20)	21.64 A (1.82)	77.86 E (1.82)
	13	74.39 D (2.79)	5.03 A (1.01)	21.91 A (1.01)	22.49 A (1.86)	77.15 DE (1.86)
	17	73.48 D (3.10)	5.64 B (0.81)	23.52 B (1.14)	24.20 B (1.53)	76.54 D (1.53)
Heartwood	10	62.35 C (3.36)	8.99 C (1.12)	25.59 C (2.60)	27.14 D (2.04)	70.60 C (2.04)
	13	58.20 B (5.31)	9.74 D (1.80)	23.09 B (2.50)	25.08 BC (2.53)	67.19 B (2.53)
	17	52.94 A (3.96)	10.65 E (1.62)	23.51 B (2.62)	25.83 C (2.33)	65.63 A (2.33)

Table 1. Mean colorimetric parameters (CIELAB 1976) for teak wood at different ages and (standard variation).

Where: $L^* =$ luminosity; $a^* =$ green-red coordinate; $b^* =$ blue-yellow coordinate; C = chroma and $h^* =$ hue angle. Different letter in same column indicate statistical differences based on the Tukey test at 5% error probability.



Figure 2. Principal component analysis (PCA) with visible spectra of teak at different ages.



Figure 3. NIR infrared from teak heartwood and sapwood at different ages.

2011). PCA was carried out with second derivative data to verify the distribution of wood samples and influence of age. Regions with humidity influence $(5300-5500 \text{ cm}^{-1} \text{ and } 6900-7300 \text{ cm}^{-1})$ were eliminated from the analysis (Figure 4).

The discrimination of teak wood based on age is evident. Changes in wood properties as a function of age, trunk position and tree origin were reported in literature by Thulasidas & Bhat (2009). These alterations can contribute to distinguish samples by near infrared



Figure 4. Principal component analysis (PCA) from second derivative spectra of teak at different ages. Scores graph (A) and Loadings graph (B).

spectroscopy. The influence of tree age in spectra was also reported by Milagres et al. (2013) in studies with eucalyptus, and some genetic influence can also occur (Hein & Chaix, 2014). For heartwood, the influence of trunk position on also appear, and samples at position of 0% and 13 years are distinct from the other groups.

4. CONCLUSION

The chromatic coordinates of teak wood are influenced by age in heartwood and sapwood. VIS/NIR spectrometry was effective to discriminate the age of *Tectona grandis* wood. This non-destructive and quick technique can be applied on industrial processes to evaluate and monitor wood quality, as well to define its final use according to wood age. In addition, manual spectrometers are also available, and based on these results, it could be concluded that this method is useful to classify wood from the log yard to processing in industrial plants.

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