POTENTIAL USE OF NIR AND VISIBLE SPECTROSCOPY TO ANALYZE CHEMICAL PROPERTIES OF THERMALLY TREATED WOOD

Elaine Cristina Lengowski1*, Graciela Inês Bolzon de Muñiz2, Umberto Klock2, Silvana Nisgoski1

1Contestado University, Brazil
2Federal University of Paraná, Laboratory of Wood Chemistry, Department of Forest Engineering and Technology, Federal University of Parana, Av. Lothario Meissner, 900, Jardim Botânico, 80210-170, Curitiba-PR, Brazil

* Corresponding author: elainelengowski@yahoo.com.br

ABSTRACT

The modification of wood by thermal treatments produces characteristic changes in its chemical composition and surface color and can improve the structural and esthetic quality. The objective of this paper is to evaluate the potential of near infrared and visible spectroscopy to predict the modifications in chemical composition of Eucalyptus grandis and Pinus taeda after thermal treatment. Near infrared and visible spectra were collected directly on the longitudinal surface of wood samples. The thermally modified wood of both species showed higher content of lignin and extractives than the untreated wood. There was darkening of thermally modified wood, as a result of chemical modification caused by heat. The Near infrared/visible technique identified the groups that were modified by treatment. NIR and visible spectroscopy can be used to predict chemical composition of natural or thermally treated Eucalyptus grandis and Pinus taeda.

Keywords: Chemical analysis, Eucalyptus grandis, Pinus taeda, thermal modification, wood color, wood composition.

INTRODUCTION

The application of thermal treatment in the wood industry has been growing in recent years. Studies have reported that heat process can modify some properties of wood as natural durability, dimensional stability, hygroscopicity (Olarescu et al. 2014, Militz and Altgen 2016, Pertuzatti et al. 2016, Huang et al. 2018) and mechanical properties (Kuzman et al. 2015, Militz and Altgen 2016). Differences have also been verified in the equilibrium moisture content, volumetric swelling and chemical composition of juvenile and mature wood (Gonzáles-Peña et al. 2009, Esteves et al. 2011, Severo et al. 2012). The review of Esteves and Pereira (2009) reported changes in mass loss, chemical composition, anatomical
structures, equilibrium moisture, dimensional stability, durability, mechanical properties, wettability, weathering, finishing, gluing, color and quality control. These results were confirmed in *Pinus elliottii* (Conte et al. 2014, Pertuzzatti et al. 2016), in eucalyptus species (Garcia et al., 2014; Zanuncio et al. 2014a, 2014b, 2014c), and other commercial wood as *Lecythis pisonis* (De Paula et al. 2016), *Simarouba amara* (Freitas et al. 2016) and *Tectona grandis* (Méndez-Mejías and Moya 2016).

One important change in heat-treated wood is its generally darker appearance, which can be explained by the combination of several factors: (a) the dissolution, oxidation and decomposition of the extractives and other chemical components; (b) formation of oxidative products, such as quinones and products of degradation of hemicelluloses and lignin, which migrate to the wood surfaces; (c) the removal or migration of extractives and nutritive compounds such as low molecular weight sugars and amino acids towards the wood surfaces; and (d) the relative increase in the lignin content after the treatment, mainly due to the degradation of the hemicelluloses (Torres et al. 2012, Cademartori et al. 2013, Stangerlin et al. 2013, Bekhta et al. 2014). The wood with large quantities of extractives soluble in water present more intense changes of color after the treatment (Varga and Van der Zee 2008). Some literature report that in hardwoods, color modification is less pronounced than in softwoods in function of different lignin content because this component is responsible for 80-95% of light absorbance in wood (Mitsui 2004, Moura e Brito 2011). Higher extractive contents and higher reactivity of hemicelluloses and lignin can accelerate the degradation process at low temperature, on the other hand, organized cellulose regions may promote a slow degradation process (Poletto et al. 2012).

The natural pine wood color (pale yellow) is not stable to light exposure, becoming grayish after exposure to sunlight, and when exposed in air it becomes whitish. Thermal treatment confers a reddish brown color, which is appreciated by most consumers (Esteves et al. 2008, Zanuncio et al. 2014b). Thermal modification has potential to improve the structural and esthetic quality of wood, in particular that from *Pinus* spp. and *Eucalyptus* spp. species with good properties, fast growing, but sometimes with unattractive coloration. In addition, the use of cultivated species to replace native wood species is necessary to preserve and reduce the pressure on the native forest.

Application of near-infrared spectroscopy to study thermally treated wood has revealed a close relationship between the chemical changes of thermally modified *Fagus sylvatica* samples and the corresponding NIR spectra (Schwanninger et al. 2004), alterations on phenolic, carbonyl, carboxyl, acetyl and hydroxyl groups (Mitsui et al. 2008, Chen et al.
2014, Li et al. 2015, Méndez-Mejías and Moya 2016), and changes in pine and eucalyptus properties (Esteves and Pereira, 2008). It has potential for use in quality control of thermally modified wood, because it allows evaluation of several wood properties by obtaining a single spectrum (Bächle et al. 2010, Méndez-Mejías and Moya 2016).

Therefore, the objective of this paper is to evaluate the modifications in chemical composition of Eucalyptus grandis and Pinus taeda based on near infrared and visible spectroscopy of solid samples. This paper is the first to evaluate the spectral analyses from visible spectroscopy to predict changes on chemical composition after the thermal treatment of wood. The consolidate technical to analyze this is NIR spectroscopy, however, if we can use the visible spectroscopy we can evaluate two properties in one equipment, color parameters and quality of treatment applied on the wood, and also the colorimeter is more cheaper.

**MATERIALS AND METHODS**

**Materials**

Trees of Eucalyptus grandis W. Hill and Pinus taeda L. came from plantations with age of 18 years. Boards with dimensions of 110 x 20 x 3 cm respectively in length, width and thickness were thermally treated. Wood untreated and thermally modified (Figure 1), came from the company TWBrazil, located in Ponta Grossa, Parana state, Brazil. The thermal modification was performed by the VAP HolzSysteme® process, applying heat of 160 °C in a saturated steam atmosphere with efficient elimination of oxygen. The treatment was done in five stages: an initial heating ramp of 0,76 °C·min⁻¹ until constant temperature (110 °C), 25 minutes at this temperature; a second ramp of 0,20 °C·min⁻¹ until the final temperature (160 °C); 45 minutes at maximum temperature; and natural cooling. The total time of the process was about 16 hours, carried out in two 8-hour heating-cooling cycles. Four boards per treatment were analyzed. One sample of each board, with dimensions of 20 cm (length) x 8 cm (width) and 3 cm (thickness), were extracted on the middle of board and applied for color and spectroscopy analysis.
Methods

Near Infrared spectroscopy

In each sample, eight spectra were collected directly on the longitudinal surface, four in radial section and four in tangential section, in a room with temperature of 23 ± 2 °C and relative humidity of 60%. A total of 128 spectra were obtained, 32 by treatment. The infrared analyses were performed with a Bruker Tensor 37 spectrometer (Bruker Optics, Ettlingen, Germany) equipped with an integrating sphere and operating in reflectance mode. Spectra were obtained at 64 scans with resolution of 4 cm⁻¹ and a spectral range of 10,000–4,000 cm⁻¹ transformed to nm in Opus version 6.5 program (Bruker Optics). For analysis, the spectra were averaged for each surface, i.e., 2 in each radial and 2 in each tangential section in one sample, and 16 were evaluated for each treatment.

Color measurements

Wood color was measured with a Konica Minolta CM-5 spectrophotometer coupled to a computer, with adjustment to a D65 light source and 10° observation angle (CIE-Lab standard). The reflectance data were obtained with a spectral range from 400 – 750 nm. Eight spectra were collected per sample directly on the longitudinal surface (4 in radial and 4 in
A total of 128 spectra were obtained, 32 for each species and treatment, and analysis was performed with all spectra.

The following color parameters were measured according to the CIELAB: color space: lightness (L*), green-red coordinate (a*), blue-yellow coordinate (b*), color saturation (C*), and hue angle (h). A colorimetric scan of the surface of these samples was performed randomly, with a total of 10 readings per sample (radial/tangential section). The values of color saturation (C) and hue angle (h) were calculated by equations 1 and 2:

\[ C = \sqrt{a'^2 + b'^2} \]  
\[ h = \arctan \left( \frac{b'}{a'} \right) \]

The changes between natural and thermally modified wood were analyzed based on \( \Delta E \) (total color variation), calculated by equation 3:

\[ \Delta E = \sqrt{\Delta L^2 + \Delta a'^2 + \Delta b'^2} \]

Spectral analysis

The Unscrambler X chemometric program (version 10.1, from CAMO Software AS) was used to analyze the data. Exploratory modeling was done by analyzing the score and loading graphs obtained by principal component analysis (PCA) to verify possible differences in natural and thermally modified wood. PCA was based on algorithm NIPALS and cross validation with centered data. PCA is widely applied for analysis of spectroscopy data as a preliminary step to observe variance structure of data and visualization of its natural clustering, followed by multivariate methods (Acquah et al. 2016).

Partial least squares (PLS) with cross validation were applied to build models for chemical property prediction. The adequate number of factors was evaluated based on lowest error of prediction and highest correlation coefficient, without eliminating samples in building the models. The main aim of PLS is analysis of numerous X-variables (spectra data) to predict response variables (chemical composition). The most important information is the number of principal components (PCs) selected, which will be included in regression models to result in a lower prediction error, that is, to present the lowest difference between references and estimated values (Kumar et al. 2014).

In spectral analysis, pretreatment of data is important to eliminate noise and remove physical phenomena in order to improve the subsequent analysis. Multiplicative scatter correction (MSC) pretreatment was applied to the data, and in this case, the mean of the calibration set was used as the reference for the test set. MSC is a scatter-corrective
pretreatment technique applied to reduce the variability between samples due to scattering and also to adjust for baseline shifts (Fearn et al. 2009).

Samples were randomly divided into 96 for calibration (75% of spectra) and 32 for external prediction (25% of spectra). Spectral analysis was based on ASTM E1655-05 (ASTM 2012). Although number of samples is few, the objective of the manuscript is to test the potential of technique to predict chemical changes. For NIR, literature report good results, but for reflectance spectra of visible light available data are related to physical properties.

Chemical analysis

Samples of natural and thermally modified wood were ground in a Wiley mill (De Leo, Porto Alegre, Brazil) and classified. The material between 40-60 mesh was analyzed. Chemical characterization was done in triplicate in accordance with TAPPI methods (TAPPI 2004): T204-om97 for total extractives; T222-om02 for lignin content; T211-om02 for ash content. Holocellulose content was calculated by difference from total extractives, insoluble lignin and ash content.

Statistical analysis

Statistical analysis was applied to identify differences in chemical composition and color between natural and thermally modified wood. Comparisons of means were performed by the Tukey test at 5% significance level.

RESULTS AND DISCUSSION

Chemical composition

Thermal treatment resulted in an increase in extractives (44.61% and 27.56%) and lignin content (36.18% and 9.08%), but reduced holocellulose content (15.68% and 4.41%), respectively for Eucalyptus grandis and Pinus taeda (Table 1). Heat treatment causes degradation of hemicelluloses (Brito et al., 2008), while solubilization of water extractives can contribute to increase these compound’s content (Esteves et al. 2011, Tong and Zhang 2016). Lignin condensation reactions with other cell wall components results in crosslinking and contributes to an apparent increase in its content. On the other hand, extractives can be degraded or leave the wood and new compounds can be formed (Esteves and Pereira 2009, Tong and Zhang 2016).
Table 1. Mean (standard deviation) of wood chemical composition.

<table>
<thead>
<tr>
<th>Material</th>
<th>Extractives (%)</th>
<th>Lignin (%)</th>
<th>Holocellulose (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>Eucalyptus grandis</em> – natural</td>
<td>6.77 a (0.90)</td>
<td>22.06 a (5.05)</td>
<td>71.17 a (1.54)</td>
</tr>
<tr>
<td><em>Eucalyptus grandis</em> – 160 °C</td>
<td>9.79 b (0.66)</td>
<td>30.07 b (3.46)</td>
<td>60.15 b (1.66)</td>
</tr>
<tr>
<td><em>Pinus taeda</em> – natural</td>
<td>2.54 c (0.99)</td>
<td>27.97 b (3.94)</td>
<td>69.49 a (1.61)</td>
</tr>
<tr>
<td><em>Pinus taeda</em> - 160 °C</td>
<td>3.24 d (1.08)</td>
<td>30.50 b (2.68)</td>
<td>66.26 c (1.29)</td>
</tr>
</tbody>
</table>

*Equal letters in a column indicate no statistical difference by the Tukey test at 5% probability.

The increase in lignin content has also been observed in *Eucalyptus citriodora* (Silva, 2012), *Eucalyptus saligna* and *Pinus caribaea* var. *hondurensis* (Brito et al. 2008), *Pinus taeda* (Silva 2012), *Pinus elliottii* (Severo et al. 2012) and *Pinus* spp. (Tong and Zhang 2016), while it remained equal for *Pinus caribaea* with treatment of 200 °C (Poubel et al. 2013). For extractives, reduction in *Eucalyptus saligna* (Brito et al., 2008), *Eucalyptus citriodora* (Silva 2012) and *Eucalyptus grandis* (Zanuncio et al., 2014b), and increase in *Pinus caribaea* var. *hondurensis* (Brito et al. 2008), *Pinus caribaea* (Poubel et al. 2013), *Pinus elliottii* (Severo et al. 2012) and *Pinus* spp. (Tong and Zhang 2016) have been reported. Esteves et al. (2008) studied extractives content in *Eucalyptus globulus* and observed an increase at the start of treatment. Reduction in holocellulose content has also been observed in *Eucalyptus grandis* (Zanuncio et al., 2014b), *Pinus caribaea* (Poubel et al. 2013), *Pinus elliottii* (Severo et al. 2012) and *Pinus* spp. (Tong and Zhang 2016). Also differences between juvenile and mature wood were reported (Severo et al. 2012). Negative correlation between Klason lignin and luminosity was found for *Eucalyptus grandis* and *Pinus caribaea* var. *hondurensis* (Moura and Brito 2011).

Colorimetry

The results of color measurements as well as multiple comparisons between means by the Tukey test are reported in Table 2.
Table 2. Colorimetric parameters of *Eucalyptus grandis* and *Pinus taeda* with and without thermal modification.

<table>
<thead>
<tr>
<th>Material</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
<th>C*</th>
<th>h</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>Eucalyptus grandis</em> – natural</td>
<td>65.75a</td>
<td>13.03a</td>
<td>20.46a</td>
<td>24.27a</td>
<td>57.62a</td>
</tr>
<tr>
<td><em>Eucalyptus grandis</em> – 160 °C</td>
<td>36.43b</td>
<td>8.64b</td>
<td>13.07b</td>
<td>15.68b</td>
<td>56.40b</td>
</tr>
<tr>
<td><em>Pinus taeda</em> – natural</td>
<td>77.32c</td>
<td>6.71c</td>
<td>30.05c</td>
<td>30.81c</td>
<td>77.53c</td>
</tr>
<tr>
<td><em>Pinus taeda</em> – 160 °C</td>
<td>52.64d</td>
<td>9.51d</td>
<td>23.71d</td>
<td>25.56a</td>
<td>68.18d</td>
</tr>
</tbody>
</table>

Lightness (L*), green-red coordinate (a*), blue-yellow coordinate (b*), saturation (C*) and hue angle (h). Equal letters in a column indicate no statistical difference by the Tukey test at 5% probability.

The results indicate a significant reduction in lightness (L*) in both species after thermal treatment. In *Eucalyptus grandis*, the decrease was more pronounced (44.6%) than in *Pinus taeda* (31.9%), probably in function of chemical groups of extractives and its volatilization. The same behavior has been observed in other studies with *Eucalyptus grandis* (Moura and Brito, 2011, Griebeler, 2013, Cademartori et al. 2013), *Eucalyptus saligna* (Pincelli et al. 2012), *Eucalyptus globulus* (Esteves et al. 2008), *Pinus caribaea* (Pincelli et al. 2012), *Pinus caribaea* var. *hondurensis* (Moura and Brito 2011) and *Pinus pinaster* (Esteves et al. 2008).

According to Varga and Van-Den Zee (2008), dissolution, oxidization and decomposition of extractives are the main reasons for color changes and modification of surface chemistry. Components that absorb visible light are formed during heat treatment. Examples are the formation of condensation products, low molecular-weight phenolic substances and oxidation products such as quinine like substances from lignin. Splitting of aliphatic side chains in lignin and condensation reactions (Chen et al. 2012a), percentage decline of hydroxyl groups and percentage increase of acid groups and esterified structure were found after heat treatment and caused darker color (Chen et al. 2012b, Yildiz et al. 2013).

For the chromatic coordinate a*, positive values are related to high levels of red color. In eucalyptus wood, a reduction of this parameter was observed, but in pinus wood an increase was observed. Pincelli et al. (2012) observed the same behavior in *Eucalyptus saligna* and *Pinus caribaea* wood, at different temperatures (120-180 °C). This change depends on the species and final temperature. This is related to volatilization of some chemical compounds that give a red color (Moura and Brito 2011, Zanuncio et al. 2014b) and is strongly correlated with the concentration of phenolic extractives (Gierlinger et al. 2004,
Zanuncio et al. 2014b). The presence of polar extractives is reported to influence color variations during thermal modification (Fan et al. 2010).

For the chromatic coordinate $b^*$, positive values are related to high levels of yellow color. In both species, a decrease in these values was observed. The behavior found for pine wood is not in agreement with the findings of Pincelli et al. (2012), who observed a maximum gain in yellow color with heating to 160 °C. Griebeler (2013) did not observe variation in $b^*$ in *Eucalyptus grandis* thermally modification at 160 °C.

Color saturation ($C$) was greater in pine than eucalyptus. Treated pine wood had similar saturation as natural eucalyptus wood. $C$ is calculated as a function of chromatic coordinates $a^*$ and $b^*$, and is dependent on the pattern of their variation. In both species, color saturation decreased after modification. An increase in this parameter was observed in *Pinus caribaea* (Pincelli et al. 2012) and in *Eucalyptus grandis* (Griebeler 2013) until 160 °C. Fan et al. (2010) found that decreases are mainly influenced by $b^*$ values, while changes in $a^*$ values have less effect on color saturation.

For hue angle ($h^*$), both species had the same behavior, a decrease in the parameter. Other studies have obtained the same results (Pincelli et al. 2012, Griebeler 2013). The reduction of tonality differences between species by heat treatment is reportedly a tool for homogenizing wood tonality and adding value to non-traditional species (Esteves et al. 2008).

The colorimetric parameters of the species studied, based on the classification table reported by Camargos and Gonçalez (2001), show the difference in wood color: natural eucalyptus was classified as rose and thermally modified as dark brown; natural pine was classified as light yellow and thermally modified as brown olive.

Color variation between natural and thermal treated wood (Table 3) shows reduction of luminosity (negative $\Delta L$) for both species; reduction of parameters $a^*$ and $b^*$ for *Eucalyptus grandis*, and reduction of parameter $b^*$ and increase of parameter $a^*$ for *Pinus taeda*, resulting in an appreciable change in total color ($\Delta E$). Values higher than 12 in ($\Delta E$) are classified as very appreciable by Hikita et al. (2001).

**Table 3**: Variation in colorimetric parameters in samples of *Eucalyptus grandis* and *Pinus taeda* after thermal modification, in relation to natural wood.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>$\Delta L^*$</th>
<th>$\Delta a^*$</th>
<th>$\Delta b^*$</th>
<th>$\Delta E$</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>E. grandis x E. grandis</em> 160 °C</td>
<td>-29.32</td>
<td>-4.39</td>
<td>-7.39</td>
<td>30.77</td>
</tr>
<tr>
<td><em>P. taeda x P. taeda</em> 160 °C</td>
<td>-24.68</td>
<td>3.04</td>
<td>-14.08</td>
<td>28.93</td>
</tr>
</tbody>
</table>
Changes in wood color after heating are the result of several reactions (dehydration, hydrolysis, oxidation), principally involving extractives, and contribute to formation of double bonds, carbonyl functionalities and quinoid structures, but lignin and hemicellulose can also play important roles in formation of color substances during heat treatment (Chen et al. 2014). The extractives can form new color substances during heat treatment. The possible condensation of tannins and the oxidation of the hydroxyl groups in flavone molecules can be a reason for formation of new color substances (Fan et al. 2010).

Heat modification has a negative effect on light reflection (Figure 2). Low reflection rate of violet, blue and green light, between 400-520 nm, contribute to a dark brown color (Fan et al. 2010). The reflectance of thermally treated wood increased with, a maximum in the red region (620-740 nm). Esteves et al. (2008) observed that in pine wood, reflectance increased more in radial planes, and in eucalyptus wood in tangential sections, and that the relationship between chemical composition and lightness decrease for pine with a strong correlation ($R^2 = 0.96$).

![Reflectance curves of visible spectra of studied species.](image)

The better photostability of thermally treated wood is partially explained by the increase of lignin stability by condensation and increase in phenol content during the heat treatment. Persze and Tolvaj (2012) presented results about photodegradation of wood and summarized the primary changes in the decomposition of lignin, with the liberation of free phenoxy
radicals. These radicals react with oxygen to produce carbonyl chromophoric groups, which are responsible for discoloration.

**Near Infrared spectroscopy**

The mean spectral patterns of natural and thermally modified wood (Figure 3) show the same main lignocellulosic composition (Schwanninger et al. 2011, Tsuchikawa and Schwanninger 2013) and some baseline influence. According to Schwanninger et al. (2011), the region near 2334, 227, 1471-1473 nm is related to cellulose and hemicelluloses; wavelengths near 2134, 1440, 1447-1448 nm are related to lignin and extractives; bands at 1927 nm and region from 1435 to 1438 nm are attributed to water content. Changes in these regions after thermal treatment are attributed to changes on OH groups (Mitsui et al. 2008).

![Figure 3: Mean NIR spectra of studied species.](image)

Wavenumbers between 1500-1900 nm are related to first combinations of harmonics from CH, CH₂, CH₃, and ArCH, forming the first harmonic region of RCO₂H and RCO₂-R’ and H₂O and the second harmonic of C=O (hemicelluloses). These regions, present in hemicellulose and amorphous cellulose, are more sensitive to temperature, resulting in changes after thermal treatment. Changes in the 1705-1724 nm region show degradation of carbohydrates and in acetylating reactions in hemicelluloses, while at 1674 nm, alterations are related to lignin (Michell and Schimleck 1996, Fackler and Schwanninger 2010).

The behavior of wood components due to thermal degradation are different in function of polymer composition and whether they are isolated or combined in the wood cell matrix...
(Popescu et al. 2011). Also evaluated species and final temperature and heating rate present influence in final spectra (Mehrotra et al. 2010).

**Spectral analysis**

Principal component analysis (PCA) of near-infrared (Figure 4a) and visible spectra (Figure 4b) shows a better distinction of species and treatment in the visible range. In NIR analysis, MSC pretreatment was applied and natural wood (*E. grandis* and *P. taeda*) were grouped and distinct from the treated wood. Also, the influence of surface color is evident in distinguishing samples after thermal treatment. In the visible range, all samples are distinct (natural and treated wood and both species). Bächle et al. (2012) also reported the possibility of distinguishing thermally treated beech, spruce and ash, and recommended the application of PCA in quality control.

In NIR spectra (Figure 5a), wavelength at 1316, 1406, 1921, 2120, 2175 and 2269 nm present more influence in discrimination of species and treatments. This bands are related to cell wall components, cellulose, hemicelluloses and lignin, also extractives present in species. Some influence of moisture content is visible in region near 1921 nm, showing that degradation process of wood components is influenced by species in the same heat conditions. In visible spectra (Figure 5b), almost all wavelengths present influence in samples distinction, because natural reflectance of species is based on color pigments of each one: i) region from 495-589 nm are related to green and yellow color and region from 670-700 nm is attributed to red color. *Eucalyptus* wood and samples treated are red to brown, and pinus wood is pale yellow.
Figure 4: Principal component analysis of NIR spectra (A) and visible spectra (B).
Prediction of chemical composition of samples was done based on spectra pretreated with MSC. Different species (*Eucalyptus* and *Pinus*) and treatment (natural and thermally treated) were evaluated from visible and near spectra by partial least square (PLS) analysis. Error of prediction and correlation coefficients of models based on natural and thermally treated wood (Table 4) showed the potential application of NIR and visible spectra to predict lignin, holocellulose and total extractives of *Eucalyptus grandis* and *Pinus taeda* wood, both natural and thermally modified at 160 °C.
Table 4: PLS model for chemical composition prediction studied species.

<table>
<thead>
<tr>
<th></th>
<th>Calibration</th>
<th></th>
<th>External prediction</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PC number</td>
<td>$R^2$</td>
<td>RMSEC</td>
<td>$R^2$</td>
</tr>
<tr>
<td><strong>NIR spectra</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lignin</td>
<td>3</td>
<td>0.92</td>
<td>0.96</td>
<td>0.87</td>
</tr>
<tr>
<td>Holocellulose</td>
<td>3</td>
<td>0.92</td>
<td>1.18</td>
<td>0.93</td>
</tr>
<tr>
<td>Total Extractives</td>
<td>3</td>
<td>0.89</td>
<td>0.94</td>
<td>0.86</td>
</tr>
<tr>
<td><strong>Visible spectra</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lignin</td>
<td>2</td>
<td>0.92</td>
<td>0.93</td>
<td>0.92</td>
</tr>
<tr>
<td>Holocellulose</td>
<td>2</td>
<td>0.93</td>
<td>1.10</td>
<td>0.88</td>
</tr>
<tr>
<td>Total Extractives</td>
<td>2</td>
<td>0.90</td>
<td>0.90</td>
<td>0.76</td>
</tr>
</tbody>
</table>

Literature report prediction of chemical properties based on near infrared spectra (Karlinasari et al. 2014, Via et al. 2014) and the influence of sample preparation (Hein et al. 2009), but analyses of visible spectra are scarce (Prades et al. 2014).

These results indicate the possibility of applying VIS/NIR on production lines in industries making solid wood products and where the final quality depends on chemical composition of raw material. The possibility of using color measurements in industry to predict chemical properties can be an innovation for quality control in the furniture and flooring industry, because the components can influence external aspects by reacting with varnish and paints, for example. Our results shows the potential of color parameters, for its application in industry, other analysis with more samples, species and thermal treatments must be done.

CONCLUSIONS

Thermal treatment changed chemical composition and color of wood treated at 160 °C in a saturated steam atmosphere with efficient elimination of oxygen. Extractives and lignin content increased and holocellulose content decreased in *Eucalyptus grandis* and *Pinus taeda*.

An appreciable change in total color between natural and thermal treated wood was observed, with reduction in luminosity for both species. A decrease in $a^*$ and $b^*$ chromatic coordinate was verified for *Eucalyptus grandis* and reduction of parameter $b^*$ and increase of parameter $a^*$ for *Pinus taeda*. Visible spectra were able to distinguish all samples and treatments.
NIR spectra showed changes in chemical composition of cell wall after thermal treatment and the influence of species on wood degradation process. Data were efficient in distinction of natural and treated wood.

NIR and visible spectra were used to predict chemical composition of *Eucalyptus grandis* and *Pinus taeda* in natural condition and after thermal treatment with $R^2$ from 0.76 to 0.92, showing its potential, and must be tested on line in industry.

REFERENCES


