

## Thermal Modification's Influence on the Color of *Tectona grandis* L.f. Sapwood to Resemble Heartwood

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The commercial value of teak wood is associated with its heartwood, which has a naturally darker color than sapwood. To reduce this color disparity, thermal modification can be used to homogenize the color between sapwood and heartwood by minimizing color differences. This research aimed to assess the effectiveness of thermal modification in making the color of teak sapwood similar to that of heartwood. Thermal modification was carried out at 160, 180, 200, and 220 °C for 150 min. Thermal modification of teak sapwood at 180 °C ensured greater color similarity to the heartwood of a fast-growing 16-year-old plantation. The material darkened with increasing process temperature. There was a change in the content of soluble substances, and hot water extraction had greater correlations with hue angle, lightness, and yellowness. Thermal modification also reduced the number of hydroxyl groups and improved the wood's thermal stability.

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### INTRODUCTION

Teak wood is valued on the international market because of its golden-brown color, high dimensional stability, and ease of drying and machining. Additionally, the high natural durability of heartwood is due to the presence of tectoquinone and lapachol extractives (Brocco *et al.* 2017; Silva *et al.* 2023). Natural teak forests, especially high-quality old-growth stands, are in decline. The production of teak logs from natural forests is also decreasing because of overexploitation, deforestation, land-use conversion, and the growing demand for forest environmental services (Kollert and Kleine 2017). However, because of their valuable wood properties and adaptability, teak plantations have been established in more than 70 countries, especially in tropical regions with fast-growing plantations (Ramasamy *et al.* 2021).

However, the teak wood from these plantations is lighter than that from natural forests (Moya and Tenorio 2021). The same authors state that young teak wood from plantations is classified in the general market of tropical wood as “white color”, and there

is also wide color variation. Color is a key feature that influences the commercialization of teak wood, which should be considered when producing wood in fast-growing plantations.

Thinnings in teak plantations are essential for obtaining wood with greater volumetric increases by reducing the competition between trees. Additionally, it provides an income alternative for producers in the early years (Chagas *et al.* 2014). However, teak wood at young ages, especially from thinnings, represents a challenge for the producers because of the consumer market demand for quality (Silva *et al.* 2023).

Studies indicate that wood from intermediate thinnings has satisfactory physical and mechanical properties and is suitable for furniture production and light civil construction (Albuês *et al.* 2024; Souza *et al.* 2024). The logs resulting from the first thinning or the upper section of the trees, characterized by smaller diameters and a greater proportion of sapwood, have lower commercial value. For this reason, these materials are generally used in domestic markets, where color and other wood properties are less valued (Moya and Tenorio 2021). The percentage of sapwood ranges from 30 to 70%, decreasing with age (Berrocal *et al.* 2020; Curvo *et al.* 2024). This devaluation is related to the low natural durability, lighter heartwood color, and greater color heterogeneity, which affect both in-service performance and aesthetics, reducing the possibility of its use (Garcia *et al.* 2014; Gašparík *et al.* 2019). The sapwood color of *Tectona grandis* is generally yellow or light gray (Sreekumar and Sanil 2021). Additionally, teak sapwood has a greater tendency to be attacked by biological agents than heartwood, which can be explained by its lower extractive content, which is essential for wood durability (Niamké *et al.* 2021). Because of these characteristics, it is necessary to apply technologies to add value to juvenile wood from thinnings (Chagas *et al.* 2014).

Thermal modification is a viable option because it ensures color uniformity in juvenile teak wood, providing technological and economic benefits (Lopes *et al.* 2014a; Méndez-Mejías and Moya 2016; Lengowski *et al.* 2020). This process involves changing the chemical composition of the cell wall by exposing the wood to high temperatures, usually between 140 and 220 °C, resulting in changes in the physical and mechanical properties, especially in the colorimetric parameters, making the wood darker (Candelier *et al.* 2016; Jones and Sandberg 2020).

The darkening of thermally modified wood can be controlled to achieve new color patterns, depending on the schedule and type of process, where time and temperature are key factors (Lopes *et al.* 2014b). In addition, thermally modified sapwood can attain a color similar to that of heartwood, reducing heterogeneity within batches of juvenile teak wood and increasing commercial viability (Sousa and Castro 2021). Another advantage of thermal modification is the increased resistance of wood, mainly against rot fungi, because of the change in the chemical composition of wood and the reduction in hygroscopicity, which disfavors the development of these microorganisms (Moya *et al.* 2017; Batista 2019). This advantage is especially important in woods having a high percentage of sapwood, which have low natural durability, such as juvenile teak wood from thinnings.

Additionally, thermal modification is an eco-friendly process worthy of further study since wood species respond differently to it (Pratiwi *et al.* 2019; Mendoza *et al.* 2020). Thus, this research aimed to evaluate thermal modification for enhancing the value of teak sapwood by changing its color to achieve colorimetric characteristics similar to those of heartwood. In addition, the study aimed to evaluate the chemical changes in thermally modified wood by analyzing soluble substances in different solvents and using Fourier transform infrared (FTIR) and thermogravimetric (TG) techniques.

## EXPERIMENTAL

### Materials

The material for this study was from thinned trees of a 16-year-old commercial plantation of *Tectona grandis* L.f. (teak) located in the municipality of Nova Maringá, Mato Grosso, Brazil. After commercial heartwood lumber was produced, the remaining, predominantly sapwood, was used to make battens measuring  $25 \times 50 \times 370$  mm<sup>3</sup> (radial  $\times$  tangential  $\times$  longitudinal). Sixty battens with at least 80% sapwood were selected, and they were equally divided into five groups, represented by untreated wood (control) and four target temperatures of thermal modification. Twelve additional battens made entirely of heartwood from the same logs were sampled and used as color reference (not thermally modified).

### Thermal Modification

Initially, the sapwood samples were air-dried and then placed in a climate-controlled room ( $20 \pm 3$  °C and  $65 \pm 2\%$  relative humidity) until they reached the equilibrium moisture content (*ca.* 12%). Their tops were sealed with a high-temperature-resistant silicone-based adhesive (ThreeBond, 12B-12).

The thermal modification of teak sapwood was carried out according to the schedule proposed by Severo and Calonego (2009), which includes the adopted target temperatures. First, the samples were dried at  $100 \pm 5$  °C in an oven with forced air circulation until a constant mass was reached. Immediately, the samples were transferred to a Linn Elektro Therm oven, model KK260, equipped with a sidewall electrical resistance heating system.

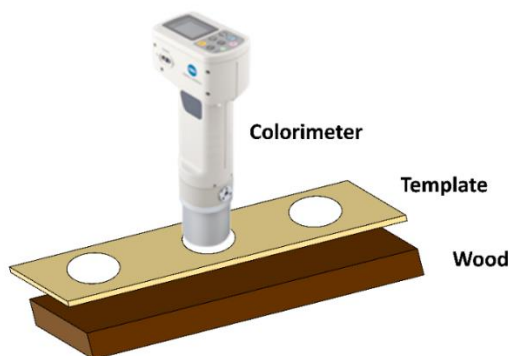
The thermal modification was performed at atmospheric pressure (open system) and followed a three-step schedule controlled by the equipment's programmable time and temperature controller: i) initial heating from 100 °C to the target temperature (160, 180, 200, or 220 °C) at a rate of 4.23 °C per minute; ii) maintenance of the target temperature for 150 min; and iii) shutting down the equipment, allowing the samples to cool naturally to 25 °C (room temperature). The temperatures cited above were defined on the basis of the methodology of Severo and Calonego (2009) and because thermal modification processing occurs for treatments between 160 °C and 240 °C (Militz and Altgen 2014). Additionally, at the industrial scale, thermal modification is typically conducted at temperatures between 180 °C and 220 °C (Brischke 2019).

The mass loss (mean  $\pm$  standard deviation) for each target temperature was as follows:  $5.87 \pm 0.72$  (160 °C),  $7.01 \pm 0.74$  (180 °C),  $10.55 \pm 0.70$  (200 °C), and  $16.22 \pm 1.53$  (220 °C).

### Colorimetric Analyses

A portable colorimeter (Konica Minolta, model CR-410) was used to determine the colorimetric parameters of the CIE  $L^*a^*b^*$  color space. The sensor had a 5-mm aperture, and the illuminant was D<sub>65</sub>, which included a xenon lamp, a 10° supplementary standard observer, and an illumination area of 50 mm diameter.

The lightness ( $L^*$ ), redness ( $a^*$ ), and yellowness ( $b^*$ ) were measured at three different measurement points on the tangential surface of each sample. The thermally modified samples were evaluated before (Untreated) and after thermal modification. To ensure consistent measurement locations, a  $50 \times 370$  mm<sup>2</sup> template with three equidistant 50 mm diameter holes was used (Fig. 1).



**Fig. 1.** Use of a template for the acquisition of colorimetric parameters

Chroma ( $C^*$ ) and the hue angle ( $h^\circ$ ) were calculated *via* Eqs. 1 and 2:

$$C^* = [(a^*)^2 + (b^*)^2]^{0.5} \quad (1)$$

$$h^\circ = \arctang(b^*/a^*) \quad (2)$$

where  $C^*$  is the chroma;  $h^\circ$  is the hue angle;  $a^*$  is redness; and  $b^*$  is yellowness.

The colorimetric parameters variations between the heartwood (reference) and thermally modified sapwood were calculated according to Eqs. 3 to 8, as specified in the standard ASTM D2244-23 (2023).

$$\Delta L^* = L^*_1 - L^*_0 \quad (3)$$

$$\Delta a^* = a^*_1 - a^*_0 \quad (4)$$

$$\Delta b^* = b^*_1 - b^*_0 \quad (5)$$

$$\Delta C^* = C^*_1 - C^*_0 \quad (6)$$

$$\Delta H^* = s [2 (C^*_1 C^*_0 - a^*_1 a^*_0 - b^*_1 b^*_0)]^{0.5} \quad (7)$$

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (8)$$

where  $\Delta L^*$  is the lightness difference;  $\Delta a^*$  is the redness difference;  $\Delta b^*$  is the yellowness difference;  $\Delta C^*$  is the chroma difference;  $\Delta H^*$  is the metric hue difference;  $\Delta E$  is the total color difference;  $L^*_0$ ,  $a^*_0$ ,  $b^*_0$ , and  $C^*_0$  are the colorimetric parameters of heartwood;  $L^*_1$ ,  $a^*_1$ ,  $b^*_1$ , and  $C^*_1$  are the colorimetric parameters of thermally modified wood; and  $s$  is the constant, where  $s = 1$  if  $a^*_0 b^*_1 > a^*_1 b^*_0$  or  $s = -1$  if  $a^*_0 b^*_1 < a^*_1 b^*_0$ .

### Solubility, Near-infrared Spectroscopy by Fourier Transform (FTIR), and Thermogravimetric (TG) Analysis

For the untreated and thermally modified sapwood, six samples were randomly selected from each treatment. These samples were halved in length, and one part was used to assess solubility, FTIR, and TG analysis. The samples were ground in a Wiley-type knife mill (Marconi, model MA304) and sifted through 40- and 60-mesh sieves.

The soluble wood substances were quantified in triplicate per treatment following ASTM standards D1110-84 (2013) and D1107-96 (2013) for hot water and organic solvents (ethanol, acetone, and ethanol/toluene – 2:1, v:v) solubility, respectively.

FTIR analyses were performed using a Shimadzu IRAffinity-1 spectrophotometer. The infrared spectrum was obtained *via* the attenuated total reflectance (ATR) method with

an AC-2098 accessory. Spectrum acquisition was carried out with IRSolution software (Version: 1.50). The samples were scanned over the spectral range of 550 to 4000  $\text{cm}^{-1}$ , with 50 scans and a resolution of 4  $\text{cm}^{-1}$ . Transmittance measurements, Happ-Genzel apodization, and a gain setting of 1 were used.

For thermogravimetric analysis, the Shimadzu DTG-60H and SDT Q-600 thermal analyzers (TA Instruments) was used. The analyses were carried out under dry atmospheric air, with a constant flow rate of 100  $\text{mL}\cdot\text{min}^{-1}$ , using approximately 8 mg of sample in an open alumina capsule, from room temperature to a maximum temperature of 600  $^{\circ}\text{C}$ , with a heating rate of 20  $^{\circ}\text{C}\cdot\text{min}^{-1}$ . Thermogravimetric (TG) curves were obtained to evaluate mass loss (%) as a function of temperature.

### Statistical Analyses

The experiment was carried out in a completely randomized design with five treatments (untreated and four thermally modified temperatures). Statistical analyses were performed using the R software (R Core Team 2021, R Foundation for Statistical Computing, version 4.4.1; Vienna, Austria).

The data were analyzed *via* analysis of variance (ANOVA), followed by Tukey's test if significant ( $p < 0.05$ ). To compare the colorimetric parameters of the treatments with those of heartwood (reference), Dunnett's test was employed ( $p < 0.05$ ). The colorimetric parameters differences ( $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ ,  $\Delta C^*$ ,  $\Delta h^*$ , and  $\Delta E$ ) were assessed *via* linear regression models. When applicable, the critical temperature values were determined by setting the first derivative of the function to zero.

Pearson's correlation was calculated between colorimetric parameters and solubilities measured in different solvents, with significance tested *via* t tests ( $p < 0.05$ ).

## RESULTS AND DISCUSSION

### Colorimetric Analyses

For lightness ( $L^*$ ), there was no significant difference between untreated wood and thermally modified sapwood (TMW) at 160  $^{\circ}\text{C}$ , indicating that the wood retained its light appearance similar to that of sapwood (Table 1). However, there was gradual darkening with further increasing temperature.

**Table 1.** Colorimetric Parameters of Untreated and Thermally Modified Teak Sapwood Compared to Heartwood (Reference)

| Treatment              | $L^*$                    | $a^*$                   | $b^*$                    | $C^*$                    | $h^{\circ}$              |
|------------------------|--------------------------|-------------------------|--------------------------|--------------------------|--------------------------|
| Untreated              | 72.4 <sup>(3.5)</sup> a* | 1.5 <sup>(1.4)</sup> e* | 20.1 <sup>(1.5)</sup> a  | 20.2 <sup>(1.3)</sup> a* | 85.5 <sup>(4.4)</sup> a* |
| 160 $^{\circ}\text{C}$ | 71.7 <sup>(0.9)</sup> a* | 3.6 <sup>(0.5)</sup> c* | 19.3 <sup>(0.8)</sup> a  | 19.7 <sup>(0.9)</sup> a* | 79.4 <sup>(3.1)</sup> b* |
| 180 $^{\circ}\text{C}$ | 63.6 <sup>(3.7)</sup> b* | 6.0 <sup>(0.6)</sup> b* | 20.6 <sup>(2.9)</sup> a  | 21.5 <sup>(2.8)</sup> a  | 73.7 <sup>(3.1)</sup> c* |
| 200 $^{\circ}\text{C}$ | 49.2 <sup>(3.6)</sup> c* | 7.1 <sup>(0.5)</sup> a* | 13.9 <sup>(0.5)</sup> b* | 15.6 <sup>(0.5)</sup> b* | 62.3 <sup>(1.5)</sup> d* |
| 220 $^{\circ}\text{C}$ | 38.9 <sup>(1.9)</sup> d* | 2.7 <sup>(0.5)</sup> d* | 3.3 <sup>(1.1)</sup> c*  | 4.3 <sup>(1.1)</sup> c*  | 50.8 <sup>(1.2)</sup> e* |
| Heartwood              | 58.8 <sup>(2.7)</sup>    | 8.0 <sup>(0.6)</sup>    | 20.5 <sup>(1.4)</sup>    | 22.0 <sup>(1.3)</sup>    | 68.6 <sup>(1.7)</sup>    |

$L^*$ : lightness;  $a^*$ : redness;  $b^*$ : yellowness;  $C^*$ : chroma;  $h^{\circ}$ : hue angle; Standard deviations are in parentheses. Means followed by the same letter in the same column do not differ according to the Tukey test ( $p < 0.05$ ). An asterisk (\*) indicates a significant difference from the heartwood according to Dunnett's test ( $p < 0.05$ ).



The  $L^*$  of TMW at 180 °C (63.6) was the closest to that of the heartwood (58.8). This finding aligns with the results reported by Cuccui *et al.* (2017), who thermally modified teak sapwood under vacuum conditions at 180 °C (3 h and 5 h) and 190 °C (3 h), achieving  $L^*$  values close to 40 for all three processes, whereas heartwood was 52.

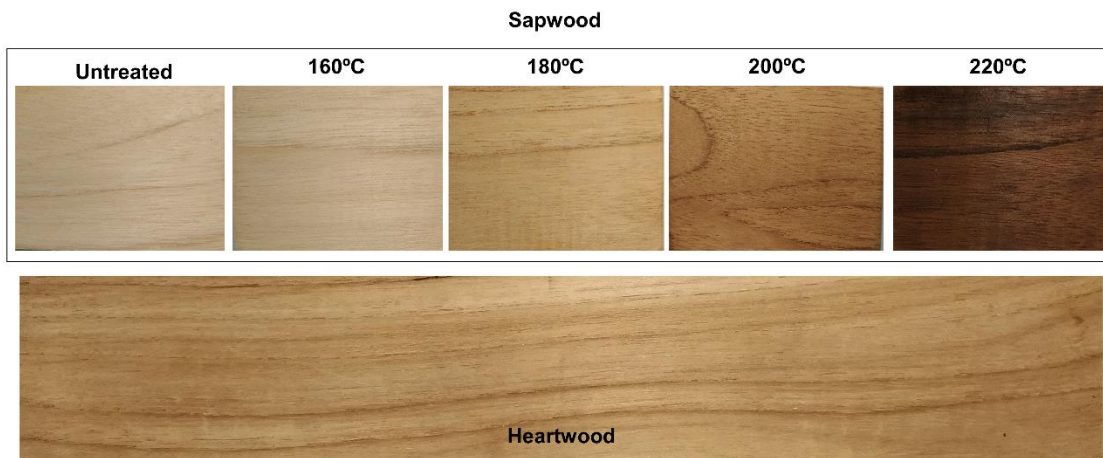
There was a significant difference in redness ( $a^*$ ) among all the treatments (Table 1). All means, including those of the heartwood, were between 0 (achromatic) and 10, corresponding to the lower end of the positive scale indicating the presence of red pigment. The value of  $a^*$  increased up to 200 °C and then decreased at 220 °C. In absolute terms, the  $a^*$  of TMW at 200 °C (7.1) was closer to that of heartwood (8.0); however, there was a significant difference according to Dunnett's test ( $p < 0.05$ ).

The yellowness ( $b^*$ ) and chroma ( $C^*$ ) of untreated wood, TMW at 160 °C, and TMW at 180 °C did not significantly differ. However, treatments at 200 and 220 °C resulted in lower levels of  $b^*$  and reduced  $C^*$ . This decrease suggests thermal degradation of the chemical components responsible for wood color. Previous studies have identified the quinone group as a major contributor to the color of teak wood (Qiu *et al.* 2019; Silva *et al.* 2023), which could explain the observed changes in chromatic characteristics with thermal modification.

All the treatments presented  $C^*$  values lower than 30, indicating a greater presence of gray color.  $C^*$  is correlated with  $b^*$ , as it has relatively higher values than  $a^*$ . Compared with the heartwood samples, the untreated wood and TMW at 160 and 180 °C presented similar  $b^*$  values, with a minimal 0.1% difference observed at 180 °C. The  $C^*$  of the TMW at 180 °C was 0.5% greater than that of the heartwood.

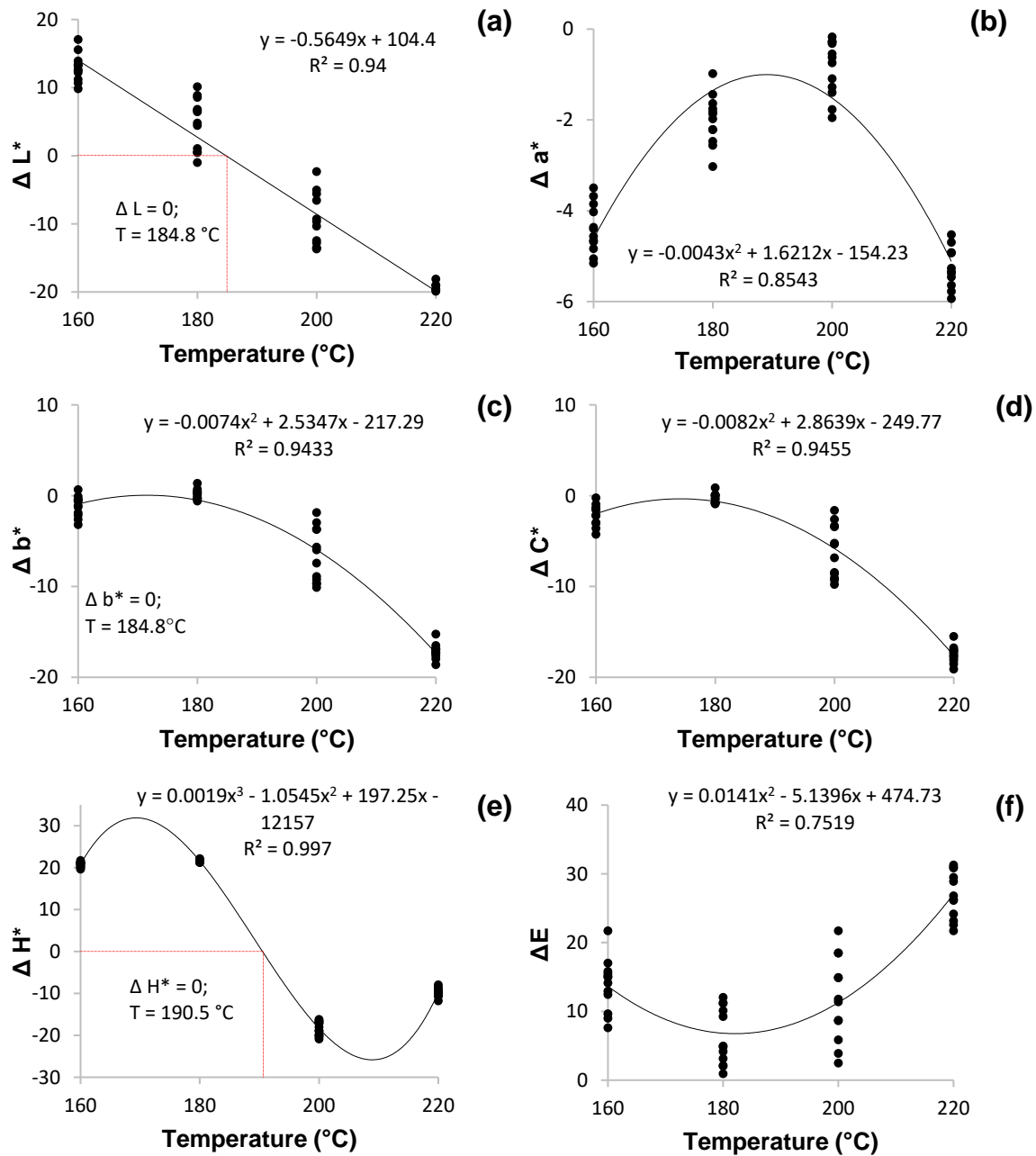
There was a significant difference in the hue angle ( $h^\circ$ ) among all the treatments. As the temperature increased,  $h^\circ$  decreased, which was influenced by changes in the  $a^*$  and  $b^*$  coordinates. A significant difference of 5% in  $h^\circ$  was observed between TMW and heartwood at 180 °C, the smallest difference found.

The results obtained in this study corroborate the observations of previous studies, such as Lengowski *et al.* (2020), who reported significant reductions in the parameters  $L^*$ ,  $b^*$ ,  $C^*$ , and  $h^\circ$  after the thermal modification of teak wood at 160 °C for 45 min. Similarly, Menezes (2017) reported the darkening of teak wood (lower  $L^*$ ), a greater presence of red ( $a^*$ ) and yellow ( $b^*$ ) pigments, and a reduction in the  $C^*$  and  $h^\circ$  parameters after thermal modification at 160 °C for 1 h. Méndez-Mejías and Moya (2016) reported that color changes were most pronounced in  $L^*$ , followed by  $a^*$ , whereas  $b^*$  showed minor changes.



**Fig. 2.** Untreated and thermally modified teak sapwood at different temperatures were compared with heartwood (reference)

In addition to the results presented in Table 1, in Fig. 2 it is possible to see pictures of the five treatments and the heartwood (reference). Untreated wood and TMW at 160 °C were similar and lighter than heartwood; on the other hand, TMW at 200 °C was darker than heartwood. This is likely that the thermal modification at 160 °C was too mild to cause significant changes, not only in color, but also in other wood properties, due to its low mass loss (5.87%). TMW at 180 and 200 °C visually resembled heartwood from the 16-year-old thinned plantation.



**Fig. 3.** Color difference between heartwood (reference) and thermally modified teak sapwood at different temperatures: (a)  $\Delta L^*$  = lightness difference; (b)  $\Delta a^*$  = redness difference; (c)  $\Delta b^*$  = yellowness difference; (d)  $\Delta C^*$  = chroma difference; (e)  $\Delta H^*$  = metric hue difference; and (f)  $\Delta E$  = total color difference.

In Fig. 3, “0” in all the graphs represents heartwood (reference). The  $\Delta L^*$  exhibited a negative linear relationship with increasing temperature (Fig. 3a). According to the regression analysis, TMW achieved the same  $L^*$  as heartwood at 184.8 °C. Under the experimental conditions, the TMW at 180 °C was the one that most closely matched the reference value for  $L^*$  (Table 1). According to Cuccui *et al.* (2017),  $L^*$  is the most significant colorimetric parameter affected by thermal modification.

The  $\Delta a^*$  (Fig. 3b) showed a parabolic trend with negative values, indicating a reduction in red character. The temperature at which  $\Delta a^*$  approached zero was estimated to be 188.5 °C. There was also a reduction in yellow coloration ( $\Delta b^*$ ) (Fig. 3c) and chroma ( $\Delta C^*$ ) (Fig. 3d), which approached zero at temperatures of 171.3 °C and 174.6 °C, respectively.

The  $\Delta H^*$  (Fig. 3e) exhibited a cubic model trend. The ideal temperature for sapwood to achieve  $h^*$ , similar to that of heartwood, was estimated to be 190.5 °C. These findings confirm that the thermal modification of teak sapwood can homogenize the color of sapwood and heartwood.

The  $\Delta E$  (Fig. 3f) corroborated the results obtained from individual colorimetric parameters. TMW at 180 °C presented the smallest color variation compared with that of heartwood, approaching the 0 axis, which represents the color of heartwood. The  $\Delta E$  was smaller than that reported by Gašparík *et al.* (2019), who achieved  $\Delta E = 27.0$  at 210 °C for 3 h during the thermal modification of teak wood, and that reported by Lengowski *et al.* (2020), who reported  $\Delta E = 24.1$  at 160 °C for 45 min.

Regarding the homogeneity between the sapwood and heartwood samples, the results align with those of Lopes *et al.* (2014a), who reported that temperatures of 180 and 200 °C for 2.5 h reduced the visual difference between heartwood and sapwood. According to Fig. 3f, the wood exhibited darker tones at 200 °C, whereas at 180 °C, homogenization occurred more gently, resulting in lighter tones. Similarly, Cuccui *et al.* (2017) noted that thermal modification minimized the color disparity between teak sapwood and heartwood.

Buchelt and Wagenführ (2011) reported that  $\Delta E > 5$  indicates a perceptibly different color. The TMW at 180 °C had the lowest mean ( $\Delta E = 7.9$ ). However, in some samples, the variation was less than 2, which, according to the authors, is considered a common and accepted color variation. Further studies should be carried out at temperatures ranging from 180 to 200 °C to explore different durations and heating rates. Additionally, teak heartwood from older plantations and natural forests should be tested as reference materials.

Thus, thermal modification was effective in the homogenization of teak sapwood, potentially increasing its value, since some markets prefer darker and more homogeneous wood. Teak wood from natural forests is darker and more resistant to biodeterioration (Moya and Berrocal 2010; Niamké *et al.* 2021). Some studies have also shown that thermally modified short-rotation teak wood can be more resistant to subterranean termites (Brito *et al.* 2022) and exhibit increased decay resistance against white rot fungi (Martha *et al.* 2023).

### Solubility, FTIR, and TG analysis

Changes in colorimetric parameters occur due to the degradation of extractives, the formation of oxidative products, and the degradation of hemicelluloses (Lopes *et al.* 2014a; Menezes 2017; Lengowski *et al.* 2020). The solubility was significantly affected by the thermal modification temperature (Table 2).



**Table 2.** Soluble Substances in Untreated and Thermally Modified Teak Sapwood in Different Solvents

| Treatment | Hot Water (%)            | Ethanol:Toluene (%)     | Ethanol (%)             | Acetone (%)             |
|-----------|--------------------------|-------------------------|-------------------------|-------------------------|
| Untreated | 11.4 <sup>(0.07)</sup> a | 9.6 <sup>(0.2)</sup> b  | 9.5 <sup>(0.2)</sup> b  | 8.2 <sup>(0.08)</sup> b |
| 160 °C    | 11.2 <sup>(0.22)</sup> a | 11.2 <sup>(0.2)</sup> a | 10.7 <sup>(0.2)</sup> a | 9.2 <sup>(0.2)</sup> a  |
| 180 °C    | 10.0 <sup>(0.9)</sup> b  | 8.6 <sup>(0.8)</sup> c  | 9.4 <sup>(0.5)</sup> b  | 7.7 <sup>(0.2)</sup> b  |
| 200 °C    | 9.7 <sup>(0.3)</sup> b   | 9.5 <sup>(0.4)</sup> c  | 8.5 <sup>(0.1)</sup> c  | 8.0 <sup>(0.3)</sup> b  |
| 220 °C    | 7.7 <sup>(0.2)</sup> c   | 10.5 <sup>(0.1)</sup> b | 8.9 <sup>(0.3)</sup> c  | 9.0 <sup>(0.4)</sup> a  |

The standard deviations are in parentheses. Means followed by the same letter in the same column do not differ according to the Tukey test ( $p < 0.05$ ).

In general, the content of soluble substances in hot water decreased with increasing thermal modification temperature. This is attributed to the continual volatilization of low-molecular-weight soluble substances due to heat exposure during the process.

Another pattern was verified in ethanol:toluene and acetone, where the soluble substances increased from untreated to TMW at 160 °C, decreased from it until 180 °C, followed by another increase from 180 °C until 220 °C. A third pattern was observed in ethanol, which was slightly different from the previous pattern, where after the first increase at 160 °C, the soluble substances decreased until 200 °C, followed by an increase from 200 °C to 220 °C.

These patterns are related to the balance of volatilized substances and the formation of new compounds resulting from the degradation of structural components (Juízo *et al.* 2018; Lengowski *et al.* 2020). Mass loss is a critical parameter for assessing thermal modification quality and serves as an indicator of the effectiveness of the process employed (Candelier *et al.* 2016). The higher the thermal modification temperature was, the greater the mass loss, where a continual increase was observed from 160 °C (5.87%) to 220 °C (16.22%).

With respect to TMW, in general, the highest levels of soluble substances in all the solvents were detected at 160 °C, even with the lowest mass loss. This is attributed to the combination of two factors: the accumulation of soluble degradation products, mainly from extractives and hemicelluloses (Brito *et al.* 2008; Mohareb *et al.* 2012; Juízo *et al.* 2018), and the lowest temperature of thermal modification, with a lower capacity to volatilize these degradation products. At higher temperatures (180, 200, and 220 °C), both capacities (production and volatilization) were potentialized, leading to different results according to the nature of the extractable substances, which is dependent on the solubilities of the different solvents. Terpenes, aldehydes, fatty acids, and alcohols are among the volatilized organic compounds derived from thermal degradation (Xu *et al.* 2019).

Among the solvents used, hot water and the ethanol:toluene mixture removed the highest amounts of substances (total means of 10.0 and 9.9%, respectively), whereas ethanol and acetone had the lowest abilities to solubilize wood components (9.4 and 8.4%, respectively). As previously discussed, the soluble substances in TMW may vary from those in untreated wood, either increasing or decreasing, depending on the specific process and variables such as time and temperature. These factors influence chemical reactions during thermal modification, leading to the formation of different types of substances (Gomes *et al.* 2023).

Hot water can remove tannins, gums, sugars, starch, and dyes (ABNT 14570 2017), which explains the greater solubility of this solvent. Qiu *et al.* (2019) verified the presence of water-soluble compounds, such as tannins, carbohydrates, alkaloids, and

cyclitol, in teak wood. Extraction in ethanol/toluene removes waxes, fats, resins, gums, and some water-soluble substances (ABNT 14660 2004). Acetone extracts compounds such as fatty acids and resins, sterols, waxes, and nonvolatile hydrocarbons, whereas pure ethanol extracts waxes, fats, and some resins (ABNT 2010).

Compared with other solvents, hot water-soluble substances had a significant influence (Table 3), indicating a greater effect on colorimetric parameters. Although the  $\Delta E$  was not significant for any of the solvents, acetone and ethanol:toluene were positively correlated, whereas hot water was negatively correlated.

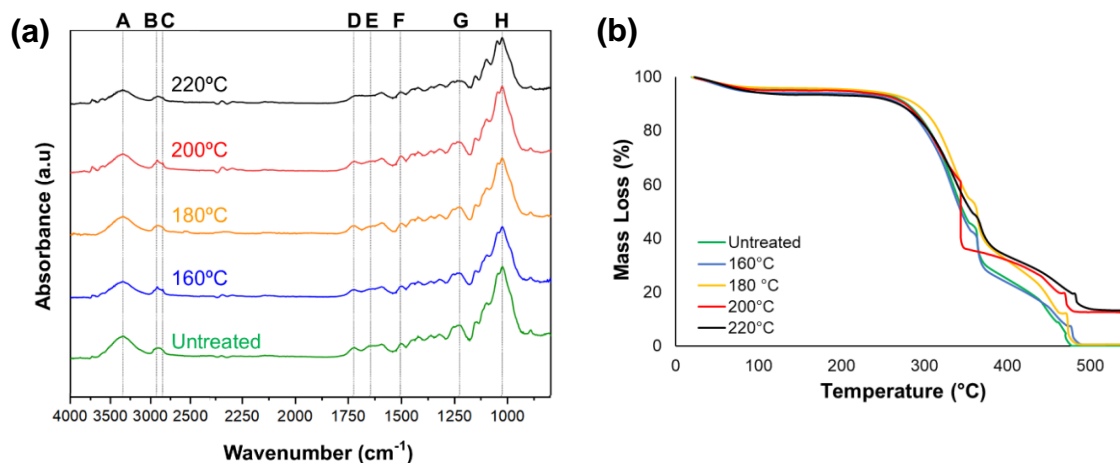
**Table 3.** Correlation between the Colorimetric Parameters and Soluble Substances in Different Solvents in Untreated and Thermally Modified Teak Sapwood

| Parameters      | $L^*$               | $a^*$              | $b^*$              | $C^*$              | $h^\circ$          | $\Delta E$         |
|-----------------|---------------------|--------------------|--------------------|--------------------|--------------------|--------------------|
| Ethanol         | 0.7 <sup>ns</sup>   | -0.4 <sup>ns</sup> | 0.5 <sup>ns</sup>  | 0.5 <sup>ns</sup>  | 0.7 <sup>ns</sup>  | -0.2 <sup>ns</sup> |
| Acetone         | -0.1 <sup>ns</sup>  | -0.5 <sup>ns</sup> | -0.4 <sup>ns</sup> | -0.4 <sup>ns</sup> | -0.2 <sup>ns</sup> | 0.6 <sup>ns</sup>  |
| Ethanol/toluene | -0.03 <sup>ns</sup> | -0.5 <sup>ns</sup> | -0.3 <sup>ns</sup> | -0.4 <sup>ns</sup> | -0.1 <sup>ns</sup> | 0.6 <sup>ns</sup>  |
| Hot water       | 0.9 <sup>*</sup>    | -0.1 <sup>ns</sup> | 0.9 <sup>*</sup>   | 0.9 <sup>*</sup>   | 1.0 <sup>*</sup>   | -0.8 <sup>ns</sup> |

$L^*$ : Lightness;  $a^*$ : redness;  $b^*$ : yellowness;  $C^*$ : chroma;  $h^\circ$ : hue angle;  $\Delta E$ : total color difference; \*: significant ( $p < 0.05$ )

Compared with other solvents, the pronounced effect of hot water-soluble substances on colorimetric parameters is crucial for ensuring the quality of TMW. Because colorimetric analyses are quicker and nondestructive, they could reduce costs and delays in laboratory analyses, making them useful for estimating properties during TMW production.

Figure 4a presents the variation in the FTIR spectrum between the untreated sapwood and TMW. There were no consistent changes in the TMW spectra, possibly because several reactions occurred at the same time. Overall, there seems to have been a decrease in the intensity of the hydroxyl band at approximately  $3350\text{ cm}^{-1}$  (A), especially at  $220\text{ }^\circ\text{C}$ . The bands at approximately  $2800$  to  $2950\text{ cm}^{-1}$  were the result of overlapping asymmetric stretching at  $2930\text{ cm}^{-1}$  (B) and symmetric vibrations at  $2850\text{ cm}^{-1}$  (C) from  $\text{CH}_2$  and  $\text{CH}_3$ . Typically, the asymmetric band presents a higher absorptivity, which was only true for TMW at  $160$  and  $200\text{ }^\circ\text{C}$ , where the peaks were sharper, with maxima at approximately  $2850$  and  $2915\text{ cm}^{-1}$ . Overall, there seems to have been a decrease in the peak at  $1650\text{ cm}^{-1}$ , which was attributable mainly to conjugated  $\text{C}=\text{O}$  bonds. However, there was an increase at higher wavenumbers, resulting in no clear difference between the  $1650\text{ cm}^{-1}$  band (E) and the  $1730\text{ cm}^{-1}$  band (D) at  $220\text{ }^\circ\text{C}$ , the latter corresponding mainly to nonconjugated  $\text{C}=\text{O}$  bonds. This suggests that the chemical environment around these functional groups differed from that of the untreated material. There seems to have been a decrease followed by an increase in the peak at  $1510\text{ cm}^{-1}$  (F), which was associated with benzene ring stretching vibrations. The  $1230\text{ cm}^{-1}$  (G) peak decreased with increasing treatment temperature, which is associated with the stretching vibrations of the  $\text{C}-\text{O}$  bonds of lignin and xylans. Although there was an apparent decrease in the  $1030\text{ cm}^{-1}$  (H) peak, this decrease might be due to the  $3350\text{ cm}^{-1}$  (A) band, which proportionally decreases the remaining peaks. The characteristic IR bands of wood and their assignments are based on Esteves *et al.* (2013) and Csanády *et al.* (2015).



**Fig. 4.** a) Spectra in the near-infrared region by Fourier transform infrared spectroscopy (FTIR) of untreated and thermally modified teak sapwood: A – 3350  $\text{cm}^{-1}$ ; B – 2930  $\text{cm}^{-1}$ ; C – 2850  $\text{cm}^{-1}$ ; D – 1730  $\text{cm}^{-1}$ ; E – 1650  $\text{cm}^{-1}$ ; F – 1510  $\text{cm}^{-1}$ ; G – 1230  $\text{cm}^{-1}$ ; H – 1030  $\text{cm}^{-1}$ ; b) Thermogravimetric (TG) curves of untreated and thermally modified teak sapwood

Figure 4b presents the thermogravimetric (TG) curves of the untreated and TMW samples. The mass loss up to 100 °C was similar for untreated and TMW at all temperatures, with a reduction of approximately 5%. The mass loss was minimal between 100 and 200 °C, at approximately 0.5% for all the treatments.

In the 200 °C to 300 °C range, the reduction was approximately 12%, except for TMW at 180 °C, which was 8%. Between 300 and 400 °C, while untreated and TMW at 160 and 180 °C had mass losses close to 60%, TMW at 200 and 220 °C had lower values, approximately 50%. It was also observed that in this temperature range, for TMW at 200 °C, the loss was more pronounced between 300 and 350 °C, as shown in Fig. 4b. The first stage of thermal degradation by combustion occurred between temperatures of approximately 270 and 350 °C for all the treatments. This process involves the depolymerization of hemicelluloses, which are more liable because of their lower molecular weight, followed by the degradation of cellulose (Moon *et al.* 2013). The lower mass loss in the TG analysis for TMW at 200 and 220 °C can be attributed to the prior degradation of hemicelluloses and amorphous cellulose caused by thermal modification, expressed by the mass loss caused by the process (10.6 and 16.2%, respectively). The hydrolysis of hemicelluloses and cellulose led to a reduction in the number of sorption sites for water molecules, which is consistent with the FTIR analysis (Fig. 4a).

Between 400 and 500 °C, the mass loss was lower for TMW at 200 and 220 °C, approximately 19%, than for the untreated samples and TMW at 160 °C, which accounted for a mean of 24%. In this TG temperature range, TMW at 180 °C had the greatest mass loss, reaching 32%. This TG temperature range corresponds to the second stage of combustion, which is characterized by the thermal decomposition of lignin and the fixed carbon synthesized in the first stage (Moon *et al.* 2013). Lignin degrades more slowly than other components do, decomposing over a temperature range of 150 to 900 °C, highlighting its stability and heat resistance (López-González *et al.* 2013; Pereira *et al.* 2013). The lower thermal degradation observed for TMW at 200 and 220 °C in this TG temperature range indicates the greater thermal stability of thermally modified wood at higher temperatures. Additionally, the residual mass was 0.5% for untreated and TMW at 160 and 180 °C, and 13% for TMW at 200 and 220 °C.

## CONCLUSIONS

1. Thermal modification of teak sapwood at 180 °C ensured greater color similarity to the heartwood of a fast-growing 16-year-old plantation. The ideal temperature for homogenizing the color of sapwood with that of heartwood, based on the process variables of this study, ranged from 171.3 to 190.5 °C, depending on the colorimetric parameters considered.
2. Increasing the thermal modification temperature gradually caused the teak sapwood to darken and reduced the yellowness, chroma, and hue angle. Compared with untreated wood, TMW at 160 °C did not significantly change wood color, whereas the most substantial variations occurred at 220 °C.
3. The highest content of soluble substances was detected at 160 °C. Hot water-soluble substances significantly influenced lightness, yellowness, chroma, and hue angle.
4. Thermal modification altered the chemical structure of the wood, leading to a reduction in the number of hydroxyl groups and an increase in the thermal stability of the wood.
5. Thermal modification is a viable process for achieving color homogenization between teak sapwood and heartwood. This process can add value to a low-cost material in a reduced amount of time, and it is also eco-friendly.

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