Impact of Thermal Modification on Swelling and Mechanical Behavior of *Couratari* spp.

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Thermal modification mechanisms and their effects on physical and mechanical properties of native Amazon hardwoods are not yet completely understood. It is expected that such treatments can improve the properties of low-value Amazonian woods and sapwood residues. This study aimed to investigate the impact of heat treatment on the swelling and mechanical properties (strength and stiffness to Static Bending and Janka hardness) of *tauari* wood (*Couratari* spp.), a low-value Amazonian hardwood. For this, *tauari* wood samples were thermally modified in an electric oven under hot air irradiation at final temperatures of 160 °C, 170 °C, 180 °C, 190 °C, 200 °C, and 210 °C for 2.5 h. The main results showed that thermal modification increased the hydrophobicity of *tauari* wood without any noticeable effects on the mechanical behavior of the wood up to 200 °C. It was stated that up to 200 °C thermal modification is beneficial in terms of gains in hydrophobicity. In contrast, above 200 °C, despite an increase in hydrophobicity, consistent decreases in strength (MOR) and hardness were observed.

DOI: 10.15376/biores.18.3.5242-5252

Keywords: Thermally modified wood; Amazon wood; MOR; Swelling coefficients

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INTRODUCTION

The supply of traditional hardwoods (*e.g*., Ipe, Brazilian teak, mahogany, cedar) from forests managed in the Amazon is declining. Alternatives include other species (*e.g*., *Pseudopiptadenia psilostachya* and *Eschweilera ovata*) among the more than 250 species with logging potential, technological characterization of second forest management cycle species, and use of modification technologies for non-durable woods (ter Steege *et al.* 2013; Santos *et al.* 2014; Fauset *et al.* 2015; Balboni *et al.* 2018).

Among non-traditional woods, the white woods (wood of a lighter color) are present in great abundance in the Amazon rainforest but are negatively associated with low natural durability. The white wood "*Tauari*" is actually one of the most harvested and exported woods in Brazil, and includes mainly the species *Couratari guianensis*, *Couratari oblongifolia*, and *Couratari stellata* (Okino *et al.* 2015). It is a hardwood having a density of 550 to 600 $kg \cdot m^{-3}$, with heartwood and sapwood indistinct by color and with low durability (class 5 and S, according EN standards); it is widely used in industry for construction (frames, ceilings, and wainscoting), manufacturing floors, furniture, and for

domestic utensils (Gerard *et al.* 2011; Okino *et al.* 2015; Cruz *et al.* 2019; Laskowska 2020).

The improvement of the characteristics of woods with lower quality and/or durability can increase the service life of the product, optimize its use, and diversify the options of managed species, contributing to the conservation of the Amazon forest. In recent decades, one of the technological alternatives of great interest to wood improvement is the process of thermal modification (Kesik *et al.* 2014). Thermally modified timber (TMT) is the product of a partial pyrolysis process at temperatures between 160 and 260 °C, with changes in the chemical composition of its cell wall due to the degradation of its polymers (Kollmann and Fengel 1965; Esteves and Pereira 2009; De Cademartori *et al.* 2013). The TMT was developed for external applications, substituting high-value tropical species, an ecological alternative to wood preserved with biocides, and as a tool for improving durability, hygroscopicity, and wood darkening (González-Penã and Hale 2009; Kesik *et al.* 2014; Kozakiewicz *et al.* 2018).

These modifications alter the color of the wood, increase dimensional stability, reduce hygroscopicity, and increase resistance to microbiological attack (Severo *et al.* 2016). The positive effect of thermal modification on wood decay is evident in the literature (Altgen *et al.* 2019; Brito *et al.* 2019), as well the changes on chemical composition (Esteves and Pereira 2009; Srinivas and Pandey 2012; Batista *et al.* 2016; Severo *et al.* 2016). However, there may be a reduction in the strength properties (Korkut 2012) and reversibility in hydrophobicity gains (Čermák *et al.* 2016).

The mechanisms of thermal modification are well established for softwoods or planted hardwoods but how this process affects the properties of Amazon native hardwood is not clear. Here, the authors study the effects of thermal modification temperature (160 to 210 ºC) in some physical and mechanical properties of *Tauari* wood and the reversibility of anti-swelling performance gains between various drying-soaking cycles. The result of this study will help establish the fundamental knowledge for thermal modification of low value-added native woods like *Tauari* and sapwood residues, increasing the yield on lumber and providing evidence on thermally modified *Tauari* in industrial applications for higher value-added products such as floors, decks, and external coatings.

EXPERIMENTAL

Materials

The sample trees of *Tauari* (C*ouratari* spp., unknown age) used in this study were harvested from managed native forests certified by the Forest Stewardship Council (FSC) located in western part of Para state, Brazil. Defect-free wood samples of dimensions 50 × 50 × 600 mm³ (width × thickness × length) were cut from radial boards of ten logs. The used species had heartwood indistinct from sapwood. However, the researchers were careful to use only the most central wood, discarding material close to the pith and close to the bark.

Wood samples were randomly divided into seven groups, one control and six for final temperatures – and had their ends sealed with red silicone polymeric adhesive (Tekbond, Embu das Artes, Brazil), which is resistant up to 316 °C, to avoid the presence of a thermal modification gradient along the piece and ensuring homogeneity of modification, as described in the technique patent (Severo and Calonego 2011). The wood samples were divided into seven groups, one control and six for final temperatures.

Twenty-five wood samples per treatment (50 × 50 × 600 mm³) were thermally modified in a high-temperature 1.0 m³ electric oven (Fanem, Model 315 SE, São Paulo, Brazil) at atmospheric pressure with heat transfer by irradiation. Heating was performed in two stages: (i) drying at 100 °C for 24 h and (ii) thermal modification from 100 °C until the final temperatures at a rate of 1.3 °C/min, following Severo *et al.* (2016). The final temperatures of 160, 170, 180, 190, 200, and 210 °C were maintained for 2.5 h and then the oven was turned off. After that, the samples were taken from the system when the oven temperature reached 40 °C.

After thermal modification, all wood samples (including the control group) were conditioned at 21 °C temperature and 65% relative humidity (ABNT 1997), until they were ready for test specimens' preparation.

Methods

The dimensional change behavior in tangential and radial directions on control and thermally modified specimens (20 × 20 × 70 mm³) extracted from static bending specimens was evaluated as described in Kozakiewicz *et al.* (2018). The total volumetric swelling was obtained from the sum of swellings in both directions. The wood specimens were oven-dried and then saturated in distilled water at 20 °C for 24 h, repeating the drying-saturation cycle three times, to test the hypothesis that the improvement of hygroscopicity due to thermal modification is reversible. After saturation phases, the careful oven-drying process was adopted (40 °C 24 h + 103 ± 2 °C 24 h) (to avoid cracks in the cell wall, due to moisture-temperature gradients). Specimens' dimensions were measured between all phases.

The linear and volumetric swelling values were calculated by Eq. 1, anti-swelling efficiency (ASE) by Eq. 2, water absorption (WA) by Eq. 3, and water absorption between 1º and 3º cycles (WR) using Eq. 4,

$$\alpha = \frac{s - s_d}{s_d} x\ 100 \quad (1)$$

$$ASE = \frac{\alpha_c - \alpha_h}{\alpha_c} x\ 100 \quad (2)$$

$$WA = \frac{w_{sat} - w_d}{w_d} x\ 100 \quad (3)$$

$$WR = \frac{WA_{3^\circ} - WA_{1^\circ}}{WA_{1^\circ}} \quad (4)$$

where α is the swelling (%), s is the tangential, radial, or volumetric dimensions (mm) of the specimen after 24 h of immersion, s_d is the dimension of the specimen in the oven-dried condition, *ASE* is the anti-swelling efficiency (%), α_c and α_h are the swelling values (%) of the control and thermally modified specimens, respectively, *WA* is the rate of water absorption (%), w_{sat} and w_d are the weights (g) in the soaking condition, *WR* is the rate of water absorption (%), WA_{3° and WA_{1° are the water absorption values in the 1st and 3rd cycles.

The modulus of rupture (MOR, MPa) and modulus of elasticity (MOE, MPa) in bending and hardness were evaluated using an electromechanical servo-controlled machine (EMIC DL30000, Instron, Curitiba, Brazil). The MOR and MOE were determined in three-point static bending tests with loading in the longitudinal-tangential planes of each forty specimens (20 × 20 × 460 mm³) per treatment. The authors also calculated the characteristic value of bending strength (f_{Mk}) for each treatment, as detailed in Eufrade Junior *et al.*

(2015). The Janka hardness of treatments was determined by partial penetration (up to radius) of steel sphere with 1.0 cm² in diameter in the period of at least 1.0 min using ten replicates per treatment with dimensions of 50 × 50 × 150 mm³. Two penetrations were made in both tangential (f_{Ht}) and radial (f_{Hr}) directions and the average achieved from both was expressed as "side hardness (f_{Hs})". All tests were conducted according to NBR 7190/97 (ABNT 1997) standard with specimens conditioned at 21 °C and 65% relative humidity.

The authors assessed the data using descriptive statistics and analysis of variance ($p < 0.05$) with seven treatments (control and six temperature levels). The average values were compared with the Tukey test at the significance level of 5%. Statistical analyses were performed in Minitab software, version 19.2020.1 (Minitab Inc., State College, PA, USA).

RESULTS AND DISCUSSION

Based on the results shown in Table 1, the swelling decreased significantly with increasing final temperature of wood modification. In all directions and temperatures, the control group had higher swelling values compared to that of heat-treated samples. At maximum temperature level (210 °C), the ASE in all directions increased by more than 50%.

Čermák *et al.* (2016) obtained similar results for *Pinus sylvestris* L. wood (oven-dry density of 530 kg.m^{-3}) with an ASE increase of 42.7% following thermal treatment at 220 °C. Thermal modification induced a decrease in the content of free hydroxyl groups and an increase in cellulose crystallinity, which restricts the absorption of water within the cell wall (Esteves and Pereira 2009), and thus the treatment results in decrease in the swelling (Biziks *et al.* 2015). With the increase in heat, the lignin polymer undergoes several cleavage reactions, self-condensation, polycondensation, and cross-linking to form lignin-lignin and lignin-carbohydrates complexes (surrounding the cellulose microfibrils). These lead to a relative increase in wood content, making the wood more dimensionally stable, and less hygroscopic (Sivonen *et al.* 2002; Bekhta and Niemz 2003).

The swelling also decreased in both tangential and radial directions with increasing heat treatment, as shown in Fig. 1. After thermal modification at 160 °C, the swelling decreased 15% in tangential and 37% in radial directions. At 200 °C, the radial ASE values dropped 39%, while tangential ASE decreased 29%. The tangential ASE was equal to radial ASE only at 210 °C. Korkut (2012) and Tiryaki *et al.* (2016) reported similar results for different species heat-treated up to 190 °C.

These results indicate distinct thermal behaviors for tangential and radial planes with a more intense degradation of hydrophilic chemical components or a higher formation of hydrophobic compounds in the radial direction at temperatures up to 200 °C. Baas (1982) stated that the radial wall has a higher lignin proportion, while the tangential wall has much more hydrophilic components. The author also reported that lignin occupies cleavages between microfibrils in the radial direction, and that in this regard it acts as a bulking agent, restricting movement. It seems that up to 200 °C, the proportion of lignin in the radial direction increases, making the gains in ASE higher. Above 200 °C, both directions have similar lignin proportions, due to the almost complete degradation of the hemicelluloses, equaling the ASE values.

Table 1. Effect of Heat Treatment at Different Temperatures on Swelling Coefficients

Heat Treatment	Swelling (%)		
	Tangential	Radial	Volumetric
Control	8.7 (± 0.7) a	6.91 (± 0.9) a	17.08 (± 1.7) a
160 °C	7.34 (± 0.8) b	4.33 (± 1.0) c	12.62 (± 1.5) c
170 °C	7.29 (± 0.6) b	5.29 (± 0.7) b	13.97 (± 0.7) b
180 °C	6.97 (± 1.2) b	5.18 (± 1.3) b	13.25 (± 1.9) b
190 °C	7.45 (± 0.5) b	5.01 (± 0.5) b	13.74 (± 0.7) b
200 °C	6.2 (± 0.8) c	4.21 (± 1.1) c	11.49 (± 2.0) c
210 °C	4.05 (± 1.0) d	3.25 (± 1.1) d	8.05 (± 2.2) d

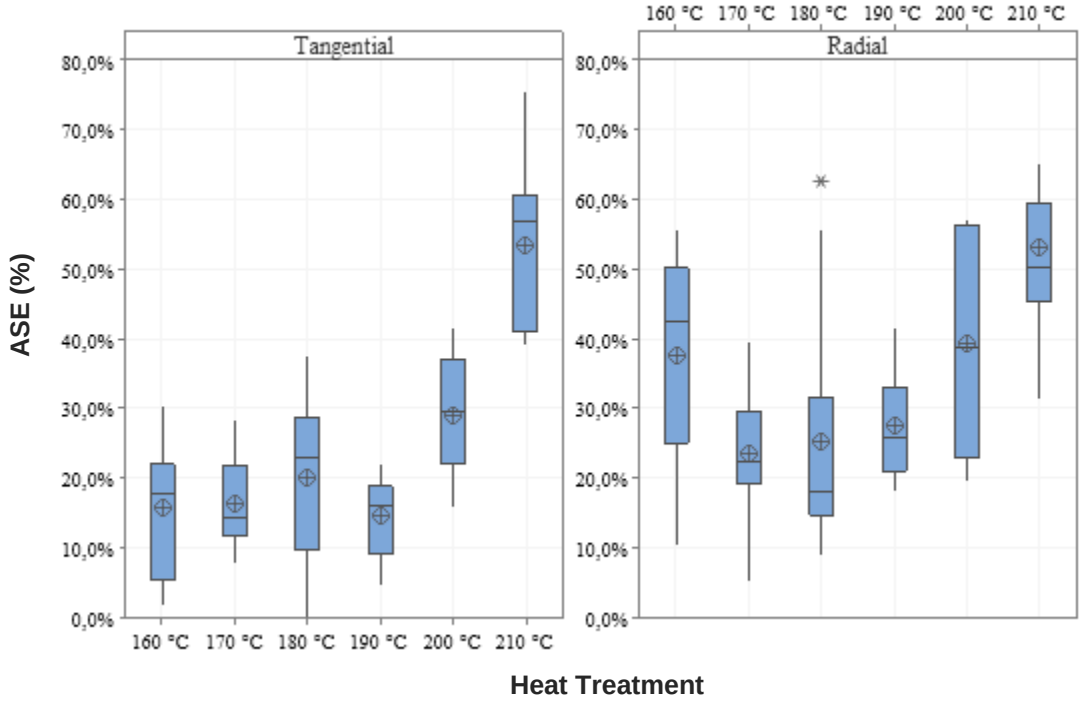

Fig. 1. ASE in tangential and radial directions as function of heat treatment

Figure 2 shows that after consecutive drying-soaking cycles, the ASE of wood thermally modified above 180 °C was decreased. The maximum ASE decrease was at 190 °C with 80%, 59%, and 71% in tangential, radial, and volumetric directions, respectively. The TMT at 180 °C recorded the 2nd largest decrease in ASE, at approximately 50% for all directions.

Biziks *et al.* (2015) also observed a decreasing trend in ASE of thermally modified woods with an increase in the number of sorption cycles. The main reasons for the decrease in ASE between cycles reported in the literature are the relaxing of internal drying stresses created during thermal modification (Hill *et al.* 2012) and leaching of secondary products from thermal modification that had blocked the penetration of water molecules between microfibrillar regions (micropores) immediately after thermal treatment (Biziks *et al.* 2015). No significant variation in dry mass in any treatment or the control group after each drying-soaking cycle was observed in this study. In contrast, after the 3rd cycle of drying and soaking there was a significant change in the rate of water absorption (+ 10%) at 180

°C and 190 °C, which had the highest ASE variations. These results illustrate that the decrease in the dimensional stability of TMT between water sorption cycles does not occur due to the leaching of co-products of thermal degradation. The most likely hypothesis is that there will be a relaxation of the stresses generated during the wood drying at high temperatures, allowing the re-entry of water into new sorption sites.

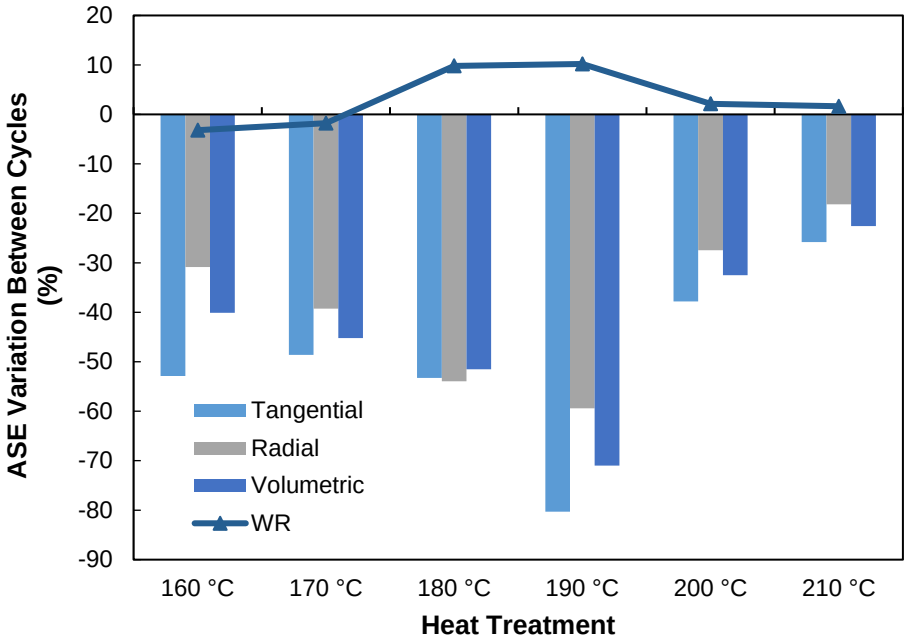

Fig. 2. ASE and water absorption rate (WR) of thermally modified wood as a function of thermal modification and variation between the 1st and 3rd drying-soaking cycles

As shown in Tables 2 and 3, the mechanical behavior of *Tauari* wood was not affected significantly by thermal treatment up to 200 °C.

The changes were significant for MOR only at 210 °C. The MOR for untreated wood was 122.1 MPa, while that for heat-treated wood increased to 10.5% at 160 °C and 9% at 180 °C. However, over 180 °C, the MOR started to decrease, with a significant drop of 32% at 210 °C. The thermal modification did not affect the MOE, which was experimented at insignificant variations (from 6% to 10%). The characteristic strength (f_{Mk}) that accounts the dispersion of results within treatment besides average values had an increase of approximately 15% at 160 °C because of the lower dispersion results of this treatment – and began to decrease from 190 °C, with a drop of 37% in f_{Mk} at 210 °C.

The MOR in static bending test is one of the properties most negatively affected by the thermal modification process, restricting this modification for structural applications (Esteves and Pereira 2009). Several studies report significant reductions in MOR, from 44% to 50% for treatments between 170 and 210 °C (Bekhta and Niemz 2003; Calonego *et al.* 2012) and an increase in wood stiffness (MOE) values with increasing modification temperature (Boonstra *et al.* 2007; Esteves *et al.* 2007).

It was evident that up to 200 °C, thermal modification increases the strength and stiffness of *Tauari* wood. This behavior is related to (i) lower equilibrium moisture content, (ii) increased stiffness around the microfibrils due to lignin crosslinking, (iii) formation of chemical bonds with higher energy than OH bonds, and (iv) increased wood crystallinity

(Kubojima *et al.* 1998; Esteves *et al.* 2007; Windeisen *et al.* 2009). These conditions would mitigate thermal degradation at softer temperatures (Bayani *et al.* 2019). However, over 200 °C, degradation of wood components surpassed the positive effects. At this level, there was a severe decrease in xylose content and breakdown of the lignin-hemicellulose matrix reinforcing the microfibrils, thereby compromising the load-sharing capacity (Windeisen *et al.* 2009). Kubojima *et al.* (2000) found that even in the smooth phase (< 200 °C) of modification, the behavior of the wood could change from gain to loss depending on the duration of the modification process used.

In the literature for TMT, the average strength values are typically used to compare treatment levels. However, it was observed that the characteristic strength values were more indicated for characterization of thermally modified wood because it allows eliminating extreme values and gives higher safety for decision making in the use of this type of wood in structural applications. To the authors' knowledge, this is the first work that brings this approach in the classification of TMT.

A significant effect of temperature was observed in f_{HS} and f_{HR} only in the intense phase (210 °C). The average values of f_{HT} were comparatively lower than f_{HR} in all treatments evaluated. Thermal modification promoted a slight increase in radial and tangential hardness. Above 180 °C, the f_{HS} begins to decrease progressively, with a maximum decrease of 27% for f_{HS} and f_{HR} and 28% for f_{HT} at 210 °C compared to control group. No significant differences were observed between tangential and radial hardness, as seen for swelling behavior, in any of the treatments evaluated in the present study.

The effect of thermal modification on wood hardness remains unclear and depends on the species and process type. Wentzel *et al.* (2019) did not observe any effect of thermal treatment up to 200 °C on Brinell hardness for different *Eucalyptus* species. Priadi and Hiziroglu (2013) observed a reduction in hardness for several wood species modified at 200 °C for 2 h and concluded that thermal modification causes damage to cell walls, compromising wood strength. Leitch (2009) observed an increase in hardness values for wood modified at 200 °C by Thermowood ®. The lower moisture-balancing of wood and surface hardening caused by drying and approximation of cellulose microfibrils has a positive effect on resistance (Boonstra *et al.* 2007), which could justify the increase in hardness in the smooth phase of modification (160 to 170 °C).

Table 2. Strength and Stiffness to Static Bending of *Tauari* Wood at Various Thermal Modification Temperatures

Treatment	Bending Strength - MOR (MPa)		f_{Mk} (MPa)	Bending Stiffness - MOE (MPa)	
	Average	Δ (%)		Average	Δ (%)
Control	127.8 (± 22.9)	-	97	16552 (± 3364)	-
160 °C	141.3 (± 25.3)	10.5%	112	17614 (± 2613)	6.4%
170 °C	134.6 (± 31.9)	5.3%	94	17755 (± 2433)	7.3%
180 °C	139.4 (± 29.5)	9.0%	97	17676 (± 2356)	6.8%
190 °C	125.3 (± 34.1)	-2.0%	88	18086 (± 2782)	9.3%
200 °C	123.3 (± 31.1)	-3.5%	85	18264 (± 2507)	10.3%
210 °C	86.4 (±29.1) *	-32.4%	60	16535 (± 3266)	-0.1%

Δ Denotes the changes compared to control samples, f_{Mk} is the characteristic strength of in static bending for the treatment; standard deviations are shown in parentheses, Significant differences ($p<0.05$) were marked with an asterisk (*).

These results demonstrate that by heat treatment it is possible to improve the wood-water relationship, which can also increase the natural durability of woods, such as *Tauari*,

and take advantage of sapwood residues discarded in sawmills, increasing the diversity of harvested species in suitable forests, and the yield in sawn timber. The use of thermally modified *Tauari* can be relevant to improve value for specific end-uses and access new markets.

Table 3. Janka Hardness at Different Thermal Modification Temperatures

Treatment	Hardness (MPa)					
	Side (f_{Hs})	Δ (%)	Radial (f_{Hr})	Δ (%)	Tangential (f_{Ht})	Δ (%)
Control	44.5 (± 4.5) a	-	46.4 (± 9.4) ab	-	42.7 (± 9.8) a	-
160 °C	45.9 (± 8.0) a	2.9%	49.5 (± 9.6) a	6.7%	42.2 (± 12.4) a	-1.2%
170 °C	45.8 (± 9.4) a	2.9%	46.0 (± 8.4) ab	-0.8%	45.7 (± 14.4) a	7.0%
180 °C	42.7 (± 5.6) ab	-4.2%	44.1 (± 12.1) ab	-4.9%	41.2 (± 11.4) a	-3.5%
190 °C	42.5 (± 8.3) ab	-8.4%	44.4 (± 10.3) ab	-4.4%	40.7 (± 13.1) a	-4.7%
200 °C	41.1 (± 6.6) ab	-7.8%	43.4 (± 8.7) ab	-6.6%	38.8 (± 10.0) a	-9.1%
210 °C	32.3 (± 10.3) b	-27.5%	33.8 (± 11.4) b	-27.1%	30.7 (± 10.1) a	-28.1%

Δ Denotes the change compared to control samples; standard deviation is shown in parentheses; Means followed by the same lower-case letter per column do not differ between them by Tukey test at the 0.05 probability level

CONCLUSIONS

1. Thermal modification is an effective method to reduce swelling of *Couratari* spp. wood, and it does not affect its mechanical behavior up to 200 °C.
2. The swelling decreased with increasing temperature. It was confirmed that radial swelling is most affected by heat treatment up to 200 °C. Above this, the gains in swelling in all directions are equal.
3. The ASE in all directions increases above 50% at 210 °C; however, these improvements are reversible after consecutive drying-soaking cycles and may even decrease by up to 80%.
4. The mechanical properties of *Tauari* wood depend on the intensity of thermal modification used, increasing at softer temperatures (up to 180 °C) and decreasing at more severe temperatures (above 200 °C). Above 200 °C, although swelling behavior improved, consistent reductions in wood strength (MOR) and hardness were observed.

ACKNOWLEDGMENTS

The major portion of the study was carried out as part of the PhD thesis of Andrade. The authors are grateful for the support of the Western Pará Federal University (UFOPA) and São Paulo State University (Brazil). The wood material was provided by Tramontina SA (Brazil) and the study was financed in part by the *Coordenação de Aperfeiçoamento de Pessoal de Nível Superior – Brazil* (CAPES) – Finance Code 001.

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Article submitted: March 28, 2022; Peer review completed: June 29, 2022; Revised version received and accepted: May 25, 2023; Published: June 15, 2023.
DOI: 10.15376/biores.18.3.5242-5252