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# Impact of thermal treatment on the properties of assacú (*Hura crepitans* L.) and murici (*Byrsonima crispa* A.Juss.) Amazon woods

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## Abstract

**Background:** White and low-density Amazon woods, such as assacú (*Hura crepitans* L.) and murici (*Byrsonima crispa* A.Juss.), have restricted use, given their low physical-mechanical strength and low natural durability. Physicochemical changes caused by heat treatment can improve different quality traits of these woods, such as changing the visual appearance from white to brown tones, improving dimensional stability, moisture control, and resistance to attack from biological organisms.

**Methods:** Assacú and murici wood samples were heat treated (180 and 220°C) for 60 minutes in a muffle kiln. The physical (moisture, mass loss, density, shrinkage, swelling), mechanical (dynamic modulus of elasticity), and chemical (extractives, solubility in hot water, lignin, and holocellulose) properties were evaluated following treatment and compared with those measured before treatment.

**Results:** The heat treatments T2 (180°C) and T3 (220°C) reduced the moisture content; however, the T3 treatment caused a more intense mass loss. For murici wood, this mass loss was 14%. The anisotropy coefficient was reduced for assacú wood by 1.30 and 1.03% for the T2 and T3 treatments, respectively, improving the dimensional stability of this wood. The density and modulus of elasticity were affected by the treatment, thus reducing the strength of the wood. The extractive content of the woods increased by 4.99 and 7.49% for sampled of assacú and murici, respectively, that were treated at 220°C. For the primary metabolites, holocellulose and lignin, degradation of these compounds occurred due to the decrease in their concentrations. The linear analysis of the studied variables indicated a high correlation between the physical properties and the chemical components of the woods (e.g., anisotropy coefficient *x* lignin and holocellulose *x* apparent density, $r > 0.95$).

**Conclusions:** The heat treatment of Amazon woods directly influenced their physical-mechanical and chemical properties. In general, the higher temperature treatment caused the greatest changes in the studied species, and the exposure to heat caused noticeable changes in their colour. Heat treatment is a useful process for the forestry sector since heat-treated wood can be used to manufacture high-value-added products, such as fine furniture, residential floors, musical instruments, and non-structural components.

**Keywords:** Tropical woods; heat treatment; extractives; density; dimensional stability; wood quality.

## Introduction

The forestry industry consumes a large volume of wood in its various market segments, which is supplied from native forest management areas or plantations. However, many commercial species grown in plantations lack adequate properties to yield high added-value products from sawn wood (Souza et al. 2020). Traditional woods can be replaced by other native species that are little known, such as small-diameter woods, or even species without technological characterization (Nascimento et al. 2023).

Low-density, white-coloured Amazon woods such as *Didymopanax morototoni, Hura crepitans, Ochroma pyramidalis, Simarouba amara*, and *Virola* sp., among others, are relegated to the background, given their low biological and physical-mechanical resistance and unattractive colouring for various manufacturers; however, technological processes applied to these woods can facilitate their integration into the market as "alternative woods", contributing to a more sustainable production chain (Freitas et al. 2016; Araújo et al. 2019; Costa et al. 2022).

In recent times, modifying wood properties has been one of the main objectives of research in wood-based product technology. Fundamentally, these modifications aim to increase the durability of wood and reduce the issues associated with the dimensional instability of lumber and engineered products (Delucis et al. 2014). Heat treatment or thermal modification is used commercially in some countries, such as France, the United Kingdom, and the United States, and this technique consists of heating wood in the temperature range of 120-260°C for a fixed exposure time (Conte et al. 2014; De Paula et al. 2016).

The modification of wood by heat treatment produces changes in its characteristics, such as increased surface hardness, bonding with hydrophobic adhesives, and improved weather resistance (Lakreb et al. 2022). This technique also results in a decrease in the equilibrium moisture content and improves dimensional stability. For light coloured woods with low natural durability, thermal modification can result in a commercially attractive dark colour and enhanced durability. This adds value to the product, allowing the use of previously disregarded species (Modes et al. 2017; Lengowski et al. 2018; Samani et al. 2021).

In Brazil, *H. crepitans* (Euphorbiaceae) is known as assacú, a large tree species whose wood is easy to work with, and of low density (< 0.40 g/cm$^3$). It has indistinct heartwood and sapwood, white with yellowish reflections, straight grain, medium texture, smooth to the touch and low gloss surface. The blue spots that are sometimes observed are due to the development of fungi. The anatomical traits of this species are apotracheal axial parenchyma with diffuse and medium to large vessels, mostly obstructed by shiny tyloses (LPF 2021). Another species occurs widely in the Brazilian Amazon is *Byrsonima crispa* (Malpighiaceae), popularly known as murici, which has a medium-sized arboreal habit and is classified as a small-diameter tree. Its wood is of medium density (< 0.60 g/cm$^3$), indistinct heartwood, light brown colour, straight grain and medium texture, diffuse porosity, large vessels, solitary and obstructed by tylosis, indistinct apotracheal parenchyma, and distinct rays. It is easy to work, able to be planed and sanded (Nascimento et al. 2023). However, these two species have limited utilisation due to the inadequacy of one or more of their properties for a specific purpose. The objective of this study is to fill in gaps in the knowledge of the properties of assacú and murici woods, and to evaluate the effects of heat treatment on their physical, mechanical, and chemical properties.

## Methods

### Study site

The was conducted in the secondary forest area of terra firme of the Experimental Tropical Silviculture Station of the Instituto Nacional de Pesquisas da Amazônia – EEST/INPA (02°37'55.5'' S and 60°09'11" W). The station is located 60 km from the capital of Amazonas and occupies an area of 21,000 hectares. The climate of the region is of the Köppen Afi type, with an average temperature of 26°C (19–39°C). The average annual rainfall is 2407.6 mm, with a rainy season between November and May (average monthly rainfall of 288 ± 99 mm), and a dry season between June and October (average monthly rainfall of 136 mm. ± 64 mm) that includes months with less than 100 mm rainfall (Tanaka et al. 2014; PELD 2015).

The study area has vegetation classified as a humid forest, with three distinct vertical strata, known as the understorey, grove, and canopy, with an average canopy height of 30 m and emergent trees that can reach up to 45 m. The basal area varies from 28 to 30 m$^2$/ha, and the soil in this area is classified as latosol with low nutrient concentration, high acidity, and high aluminium concentration (Quesada et al. 2010; Aleixo et al. 2019).

### Tree selection and sample preparation

Six trees were randomly selected for further study based on their diameter at breast height (DBH) and commercial height. Boards measuring 2.54 cm thick and 3 m long were taken from three individuals of each species, *Hura crepitans* L. (assacú) and *Byrsonima crispa* A.Juss. (murici). Samples taken from every tree were sent to the Wood Anatomy Laboratory - COTEI/INPA/MCTI/Brazil for confirmation of the identification made in the field.

The specimens used in the tests to determine the physical properties of the wood had dimensions of 20×20×30 mm in the radial, tangential, and longitudinal directions, respectively. Thirty specimens of each species were prepared, and were allocated to the following groups: 10 untreated specimens (T1), 10 specimens treated at 180°C (T2), and 10 specimens treated at 220°C (T3). In the mechanical properties test, specimens had dimensions of 20×20×300 mm, for the radial, tangential, and longitudinal directions. To determine the chemical properties of each species, ground wood was used and passed through a 60-mesh sieve. For all treatments, a sample of 100 g was used.

Prior to treatment, the specimens were acclimatised under controlled temperature (20±2°C) and humidity (65±5%), until reaching equilibrium moisture content, weighed on an analytical balance with a precision of 0.01 g and measured with a digital calliper with a precision of 0.01 mm. Subsequently, the specimens were exposed to heat in a muffle kiln at temperatures of 180°C and 220°C for 60 minutes. After thermal modification, the specimens were placed in a climatic chamber, weighed again, and measured with a digital calliper. To characterise the changes caused in the treated specimens, their physical, mechanical, and chemical properties were determined.

The physical-mechanical tests performed were moisture, shrinkage, swelling, loss of mass, density, and modulus of elasticity, and the experimental design consisted of a factorial scheme with n = 180 (2 species x 3 treatments x 6 tests x 5 replicate). For the chemical analysis, the tests performed were total extractives, solubility in hot water, lignin and holocellulose, with factorial n = 72 (2 species x 3 treatments x 4 tests x 3 replicates).

**Physical-mechanical tests**

The apparent density was determined by the stoichiometric method using a 0.01 g precision digital scale and digital calliper in a room with temperature (20±2°C) and humidity (65±5%) (NBR 7190 - ABNT 2022). From measurements of mass and volume, apparent density was calculated as:

$$\rho_{12\%} = m/V \quad (1)$$

where: $\rho_{12\%}$ is the apparent density (g/cm$^3$), m is the specimen mass (g) and V is the specimen volume (cm$^3$).

The maximum linear shrinkage ($\beta$) was calculated from the dimensions in the green and dry state of the radial ($r$), tangential ($t$), and longitudinal ($l$) directions, and maximum volumetric shrinkage through the volume using the approach described by Samani et al. (2021):

$$\beta\,(r,t,l) = (D_v - D_s)/Dv*100 \quad (2)$$

where: $\beta$ ($r$, $t$, $l$) is the maximum shrinkage in each orthogonal direction (%), $D_v$ is the green dimension in each orthogonal direction (mm), and $D_s$ is the dry dimension in each structural direction (mm).

$$\beta_v = (V_v - V_s)/V_v*100 \quad (3)$$

where $\beta_v$ is the maximum volumetric shrinkage (%), $V_v$ is the green volume of the specimen (cm$^3$); and $V_s$ is the dry volume of the specimen (cm$^3$).

Swelling was calculated based on the dimensions ($r$, $t$, $l$) of the dry and saturated samples using the approach described in Samani et al. (2021):

$$\alpha v = (V_{sat} - V_{sec})/V_{sec}*100 \quad (4)$$

where: $\alpha v$ is the maximum volumetric swelling (%), $V_{sat}$ is the saturated volume of the specimen (cm$^3$), and $V_{sec}$ is the dry volume of the specimen (cm$^3$).

The anisotropy coefficient is the ratio of the tangential shrinkage to the radial shrinkage (Araújo CSF et al. 2022), and was calculated using the following equation:

$$CA = \beta_t/\beta_r \quad (5)$$

where CA is the anisotropy coefficient (%), $\beta_t$ is the maximum tangential shrinkage (%), and $\beta_r$ is the maximum radial shrinkage (%). Values of CA were compared against reference values (Table 1) to determine wood quality.

Equilibrium moisture content was determined following the procedures described in ASTM D2016-74 (ASTM 2013). All specimens were weighed at room temperature, and the controls were then subjected to drying in an oven at 100±3°C (T1). The treated species were subjected to thermal modification, either T2 (180°C) or T3 (220°C). Finally, the dried or thermally modified specimens were weighed, and the moisture is calculated as follows:

$$EM = (P_u - P_s)/P_s*100 \quad (6)$$

where EM is the equilibrium moisture content (%), $P_u$ is the initial mass (g), and $P_s$ is the final dry mass (g).

An important macroscopic change to be analysed is the mass loss of heat-treated wood, as it will have as a direct consequence a change in wood density (Samani et al. 2021). To evaluate the mass loss, first it is necessary to calculate the dry mass based on its moisture content:

$$M_{dry} = 100\times M_i/U+100 \quad (7)$$

where $M_{dry}$ is the dry mass (g), $M_i$ is the initial mass (g) and $U$ is the wood moisture content (%).

Using the result obtained for the dry mass, the mass loss after heat treatment is calculated as follows:

$$ML = M_{dry} - M_1/\,M_{dry}*100 \quad (8)$$

where ML is the mass loss- (%); $M_{dry}$ is the dry mass (g); and $M_1$ is the mass after heat treatment (g).

The dynamic modulus of elasticity was calculated from information on stress wave velocity and density of each specimen using Eq. (9) (Araújo RD et al. 2022). A stress wave timer (Metriguard Model 238A, Pullman, WA, USA) was used to measure stress wave propagation time in each specimen.

$$MOE_D = (L/t)^2*D/g*10^{-5} \quad (9)$$

TABLE 1: Wood quality based on the anisotropy coefficient.

| Anisotropy coefficient (%) | Quality class |
|---|---|
| ≤1.54 | Good |
| 1.54 ≤ CA ≤ 2.10 | Normal |
| > 2.10 | Poor |

where $MOE_D$ is the dynamic modulus of elasticity (MPa), L is the length of the specimen (m), t is the stress wave propagation time (s), D is the density of the specimen ($kg/m^3$), and g is acceleration due to gravity ($m/s^2$).

**Chemical analysis**

The extractives content was determined from samples collected from each treatment using the methods described in ASTM D1108-96 (ASTM 2013). From each treatment, 2.00 g of sawdust was collected, weighed and then placed in a paper filter, dried and reweighed. The sawdust was placed in a Soxhlet extractor for 8 hours using hexane solvent. The obtained extract was solubilised and weighed, and the extractives content calculated using the following equation:

$$EC = P_{ext}/P_s*100 \quad (10)$$

where EC is the extractives content (%), $P_{ext}$: mass of the final extract (g), and $P_s$ is the sample weight on a dry basis (g).

Determinations of materials soluble in hot water were performed on samples that underwent extraction with hexane following the procedures described in ASTM D1110-8 (ASTM 2013). A second extraction was carried out in hot water for approximately 4 h. The samples were then dried in an oven for 24 h and weighed until they achieved constant mass. Hot water soluble extractives content was calculated using the following equation:

$$HWE = (P_1-P_2)/P_1*100 \quad (11)$$

where HWE is the hot water soluble extractives content (%), $P_1$ is the dry sample weight before extraction (g), and $P_2$ is the dry sample weight after extraction (g).

After the samples were free of extractives (extraction in hexane and water), 1.00 g of this material was hydrolysed with 72% $H_2SO_4$ for approximately 6 h to determine lignin content according to ASTM D1106-69 (ASTM 2013). The hydrolysed material was washed with hot water, filtered, dried in an oven at 100°C, and then weighed. The lignin content was calculated using the following equation:

$$Lignin = (P_{lig}/P_s)*100 \quad (12)$$

where Lignin is the lignin content (%), $P_{lig}$ is the lignin weight obtained from the hydrolysed material (g); and $P_s$ is the weight of the dry base sample (g).

Holocellulose content was determined by treating 1.00 g of extractive-free sawdust with nitric acid (3%) in a reflux system in a water bath (80°C) for 30 minutes (Ramadan & Nasser 2008). This material was then treated with sodium hydroxide (3%) under heating for further digestion. Finally, the residue was washed, dried, and weighed. Holocellulose content was calculated using the following equation:

$$Holocellulose = P_2/P_1*100 \quad (13)$$

where Holocellulose is the hemicellulose + cellulose content (%), $P_1$ is the initial dry weight of the sample (g), and $P_2$ is the dry weight of the crude pulp obtained (g).

**Data analysis**

The raw data were analysed to determine that they met the assumptions of normality, homogeneity, and independence of the residuals. Analysis of variance (ANOVA) and Tukey's test were used to compare treatments to assess the effect of thermal treatment (5% significance level). These analyses were done using Minitab® v21.1. Finally, a Pearson's correlation matrix was estimated from the variables that make up the properties evaluated, and correlation was classified according to Callegari-Jacques et al. (2003) using the PAST 4.08 program.

## Results and Discussion

**Physical-mechanical properties**

Heat treatment reduced the moisture in the wood in both the T2 (180°C) and T3 (220°C) processes (Figure 1A). However, the T3 treatment caused a more intense mass loss, with an index of 14% for the murici wood (Figure 1B). Unfortunately, the assacú wood sample was damaged during the treatment as it burned. Therefore, it appears that heat treatment above 180°C can cause deep degradation in the wood cell wall, where structural compounds such as lignin and cellulose are affected. Kumar et al. (2020) state that the energy value of biomass depends on its biochemical composition, being influenced by the types of compounds in the wood since certain extractives have fats, waxes, some resins, and oils in their composition that influence the calorific value of the species.

Carvalho et al. (2015) applied heat treatment (180 to 220°C) to *Pinus* spp. wood, reducing the moisture content from 14.7% to 11.4% with a mass loss of 4.3%. Anjos (2014), using the same process for Amazon woods (*Goupia glabra, Manilkara huberi*, and *Peltogyne recifencis*), found values of mass loss that ranged from 3 to 12%, values close to those of the present study. The loss of mass is associated with chemical degradation and with a decrease in hygroscopic water inside the wood (Borrega & Kärenlampi 2008). Batista (2019), in a review on wood heat treatment, states that exposure of wood to temperatures above 60°C causes possible structural and chemical changes in the cell wall that is, higher temperatures cause degradation of the hygroscopic components of the cell wall wood, generating a greater decrease in equilibrium moisture. In this sense, the thermal treatments (T2 and T3) promoted lower equilibrium moisture in the wood samples, improving their physical and technological properties, as well as reducing their hygroscopicity and improving their dimensional stability.

One of the explanations for the reduced hygroscopicity of heat-treated wood is linked to the decrease in hemicellulose. By reducing the wood's ability to

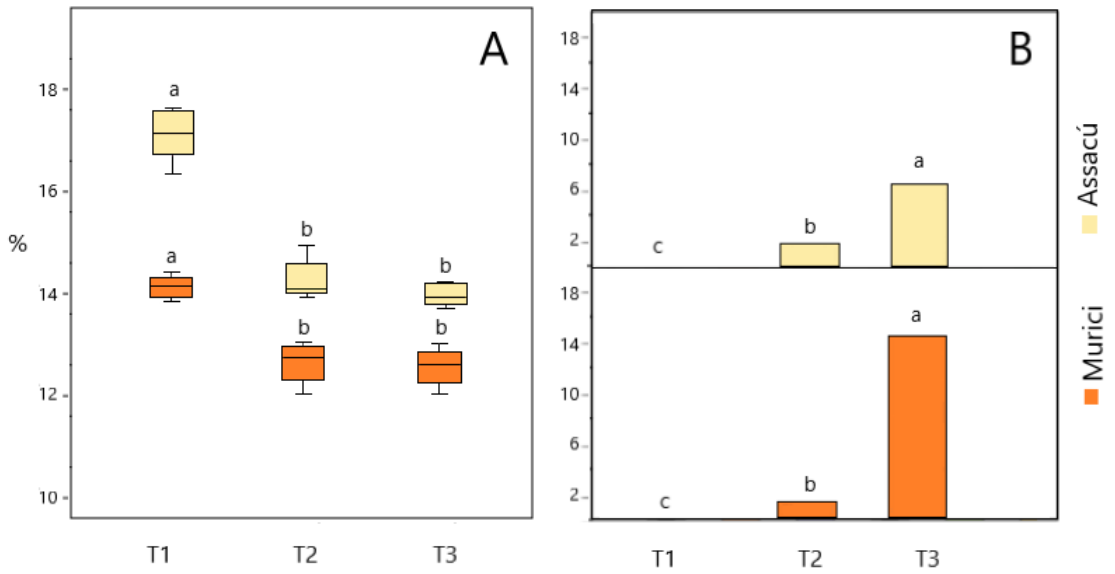



FIGURE 1: Comparison of equilibrium moisture (A) and mass loss (B) among treated and untreated samples. Samples with the same letter do not differ from each other according to Tukey's test at 95% probability ($p < 0.05$).

exchange water with the environment, shrinkage and swelling problems are minimized (De Paula et al. 2016). Table 2 shows that linear shrinkage was higher in murici wood, with the highest value in a specimen of this species subjected to the T3 treatment ($\beta_t$ 9.09%). This large change in dimensions appears to be associated with the higher mass loss that was also observed in this species. In general, the greatest retractability is observed in the tangential plane and the least (often almost imperceptible) in the longitudinal plane (Araújo CSF et al. 2022; Lakreb et al. 2022), a result also observed in the current study. The values of linear shrinkage ($\beta_{r,t,l}$) for a species increased after treatments T2 and T3, compared with the untreated (T1) samples. The same behaviour was observed for volumetric shrinkage. Swelling of specimens receiving the highest temperature treatment (T3) was less than that observed in specimens receiving the T1 and T2 treatments (Figure 2). Similar swelling behaviour was observed in studies by Poubel et al. (2013), who found a decrease in swelling from 18.83% to 9.31% for heat-treated (220°C) pine wood. The volumetric swelling values found in the present study on Amazon woods are consistent with those found in other studies (Anjos & Sousa 2015; De Paula et al. 2016).

The ratio between tangential and radial linear shrinkage is designated as the coefficient of anisotropy (CA). The quality of the wood can be measured by its dimensional stability, and CA < 2.00% is recommended since woods with higher values have internal stresses causing various defects, such as splitting and warping (Fróes et al. 2019). The heat-treated woods in this study generally had good dimensional stability, which adds to their commercial value (Borges & Quirino 2004). Assacú had lower values of CA than murici (Table 2) and heat treatment also reduced CA in this species. Under the T3 treatment, CA decreased to 1.03% compared to 1.40% in the untreated specimens (T1).

The exposure of xylem tissue to elevated temperatures causes loss of mass and consequently changes in physical-mechanical properties. In general, it is believed that the higher the temperature and treatment time, the greater the decrease in wood resistance (Borrega & Kärenlampi 2008; Vollbrecht et al. 2022). The apparent density ($\rho_{12\%}$) and modulus of elasticity (MOED) of heat-treated wood are shown in Figure 3. This shows that $\rho_{12\%}$ of untreated assacú wood (0.47 g/cm$^3$) is lower than untreated murici (0.68 g/cm$^3$) (Araújo et al. 2019; LPF 2021). Heat treatments (T2 and T3) caused decreases

TABLE 2: Results of the linear shrinkage test of heat-treated wood.

| **Linear shrinkage, $\beta$ (%)** | **Species** | | | | | |
|---|---|---|---|---|---|---|
| | **Assacú** | | | **Murici** | | |
| | **T1 (100 °C)** | **T2 (180 °C)** | **T3 (220 °C)** | **T1 (100 °C)** | **T2 (180 °C)** | **T3 (220 °C)** |
| Tangential ($t$) | 3.42 | 3.59 | 4.80 | 4.49 | 5.16 | 9.09 |
| Radial ($r$) | 2.44 | 2.76 | 4.67 | 2.82 | 3.30 | 5.52 |
| Longitudinal ($l$) | 0.22 | 0.25 | 0.18 | 0.25 | 0.29 | 0.33 |
| CA ($t/r$) | 1.40A | 1.30A | 1.03B | 1.59a | 1.56a | 1.65a |

CA: anisotropy coefficient.
Means followed by the same letter on the line do not differ from each other by Tukey's test at 95% probability ($p < 0.05$).

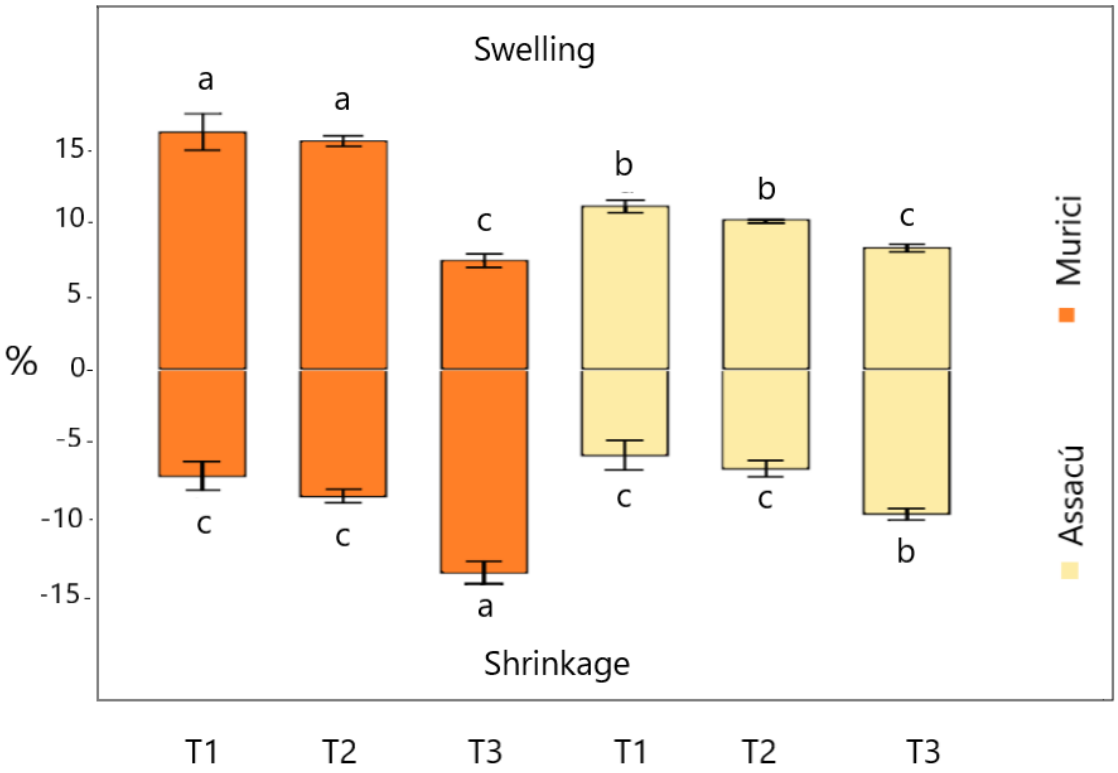



FIGURE 2: Volumetric effect of heat-treated wood (swelling and shrinkage). The same letter in the column does not differ by Tukey's test at 95% probability ($p < 0.05$).

in density in these two species (Figure 3A). For assacú, this decrease was approximately 15%, considering temperatures from 180 to 220°C, and for murici, the decrease was smaller (from 1.5 to 10.3%). In other thermal studies of *Fagus orientalis* wood treated at 160°C, a decrease in $\rho_{12\%}$ of up to 5.17% was observed (Charani et al. 2007), while *Eucalyptus cloeziana* wood treated at 210°C this decrease was 12.37% (Huller et al. 2017). Future studies will be carried out to verify the influence of heat treatment on basic density. This variable must be differentiated from $\rho_{12\%}$, which has a positive correlation with wood moisture (Costa et al. 2014; Vollbrecht et al. 2022).

The average values obtained for the dynamic modulus of elasticity (MOED) of the species studied are shown in Figure 3B. Values ranged from 5,765 to 8,128 MPa for

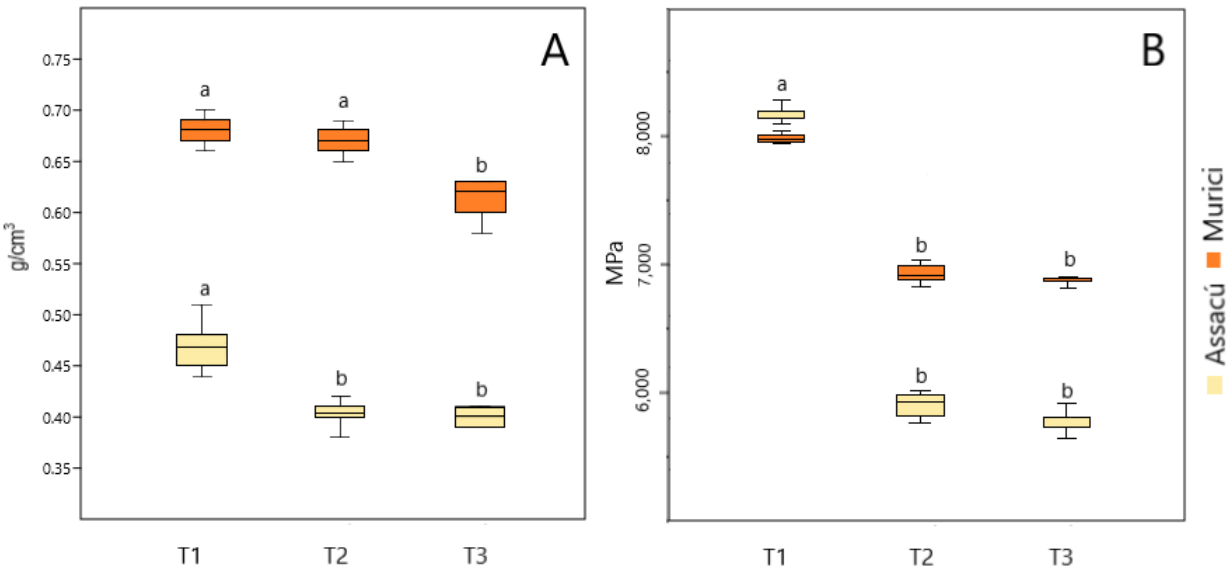



FIGURE 3: Results of physical-mechanical tests of heat-treated wood: A – apparent density; B – dynamic modulus of elasticity. The same letter in the sample does not differ from each other by Tukey's test at 95% probability ($p < 0.05$).

assacú and from 6,790 to 8,001 MPa for murici. Thus, while both species had similar mean values of MOED, greater variation was observed in assacú. The greatest reduction in elastic modulus was observed for assacu wood (180 and 220°C). This behaviour may be related to the physical and chemical properties of each species, with assacu wood having a lower apparent density and higher moisture content. According to INPA (1991) and Senalik & Farber (2021), moisture is a factor that reduces the mechanical resistance of wood. No significant differences in MOED were observed between T2 and T3 treated specimens for either, assacú or murici.

Garcia et al. (2012) heat treated *Eucalyptus grandis* wood (180-230°C) and concluded that the MOED decreased by approximately 13% relative to the untreated samples. Evaluating the impact resistance of heat-treated wood, Huller et al. (2017) observed resistance variation in the tested section, that is, there was a decrease in resistance in the treated woods at 210°C, but these values were different for each plane. In the radial section, the decrease was ~40%, and in the tangential section, the decrease was ~52%. Although the equilibrium moisture is reduced in the heat treatment, which would theoretically potentiate a greater mechanical resistance to wood, since the dry mass has greater resistance, the loss of mass can explain the decrease in the mechanical properties of the treated wood since the process heat causes degradation of the chemical components of the cell wall.

**Chemical properties**

The results of the chemical analyses of the heat-treated woods are presented in Table 3. The extractive content of the woods increased considerably for the treated woods, mainly for the process at 220°C (4.99 and 7.49%). However, the hot-water-soluble extractives content did not show any apparent trend with heat treatment. For the primary metabolites, holocellulose and lignin, there was evidence of their degradation (hypothetically due to weight loss) with heat treatment. There was also evidence that the lignin content of murici wood decreased as the treatment temperature increased (c.f. 30.35% under T1 and 23.40% under T3). Similar behaviour was observed for the concentration of holocellulose in the assacú wood, which decreased from 57.25% to 42.93% under the higher temperature treatment.

Lengowski & Muniz (2016) found a significant increase in the concentration of these metabolites when evaluating the content of total extractives from treated (160°C) and untreated specimens from *Pinus taeda,* (27.56%), *Eucalyptus grandis* (44.61%), and *Tectona grandis* (48.59%). Carvalho et al. (2015) found similar results for heat-treated *Pinus* spp. wood, where extractives increased by more than 50%, and holocellulose a 9% decrease for temperatures of 220°C. In the present study, the extractive content was also increased by more than 50% after exposure of the wood to heat, and for lignin, the decrease was 3.01-23.15% and holocellulose 14.22-25.99%.

Results from the chemical analysis of the heat-treated wood offer a likely explanation for some of the results obtained in the physical tests, such as the loss of mass, density, and dimensional instability. These appear to be linked to the process of free water evaporation and impregnation (water in the form of vapour and/or droplets), and the degradation of primary wood metabolites, mainly certain polyoses that are easily degraded, as well as smaller portions of lignin and cellulose. These observations are confirmed in studies by Barroco et al. (2020), who used infrared spectroscopy to evaluate wood treated at 160-200°C. In other studies, on heat treatment, Yeo et al. (2017) confirmed that hemicelluloses are the first wood carbohydrates to degrade with heat treatment due to their heterogeneous, non-crystalline structure and low molecular weight compared to other wood polymers. Lignin and mainly cellulose undergo progressive degradation that includes depolymerization and dehydration.

For extractives, these compounds tend to increase in concentration when the wood is exposed to heat. At first, compounds of the terpene class are volatilized, and when quantification occurs, after treatment, a higher concentration of polar compounds is observed, as well as aldehydes and acetic acid, products of the degradation of wood polymers (Kačik et al. 2012; Carvalho et al. 2015).

A Pearson's correlation was performed from the results of the properties evaluated, totalling 55 correlations (Table 4), where eight correlations showed greater significance ($p < 0.05$): holocellulose x ρ12% (apparent density), extractives x EM (equilibrium moisture); extractives x lignin; lignin x ρ12%, lignin x CA (anisotropy coefficient); hot water x ρ12%, hot

TABLE 3: Comparison of chemical components among wood specimens subjected to different levels of heat treatment. The same letter in the sample does not differ from each other by Tukey's test at 95% probability ($p < 0.05$).

| Species | Treatment | Moisture (%) | Extractives (%) | Hot water (%) | Lignin (%) | Holocellulose (%) |
|---|---|---|---|---|---|---|
| Assacú | T1 (untreated) | 17.00a | 1.99d | 8.64b | 28.90a | 57.25a |
| | T2 (180 °C); | 14.32b | 2.74c | 7.86b | 28.40a | 44.40b |
| | T3 (220 °C) | 14.03b | 4.99b | 8.65b | 28.00a | 42.93c |
| Murici | T1 (untreated) | 14.00b | 3,92b | 10.53a | 30.45a | 53,73a |
| | T2 (180 °C); | 12.60c | 7.24a | 6.67c | 24.90b | 46.59b |
| | T3 (220 °C) | 12.50c | 7.49a | 7,60b | 23.40b | 46.09b |

TABLE 4: Pearson's correlation coefficient, obtained from the correlations between the physical-mechanical and chemical variables. Values in bold are significant at a 5% probability.

| | $\rho_{12\%}$ | WL | β | α | EM | MOED | Extract | Hot water | Lignin | Holocellulose |
|---|---|---|---|---|---|---|---|---|---|---|
| WL | 0.0214 | | 0.1691 | 0.1137 | 0.2846 | 0.4019 | 0.3924 | 0.5352 | 0.3019 | 0.9098 |
| β | 0.0422 | -0.6421 | | 0.5713 | 0.1302 | 0.2012 | 0.1956 | 0.7891 | 0.4628 | 0.8120 |
| α | 0.6839 | 0.7103 | -0.2942 | | 0.8923 | 0.2445 | 0.5488 | 0.0254 | 0.7061 | 0.1501 |
| EM | -0.5117 | 0.5251 | -0.6887 | -0.0718 | | 0.3991 | 0.0264 | 0.6219 | 0.0688 | 0.2953 |
| MOED | 0.5495 | 0.4241 | -0.6070 | 0.5631 | 0.4264 | | 0.9722 | 0.1284 | 0.7510 | 0.3187 |
| Extract | 0.7987 | -0.4319 | 0.6130 | 0.3107 | **-0.8640** | 0.0184 | | 0.1984 | 0.0114 | 0.1221 |
| Hot water | **-0.9186** | -0.3208 | 0.1415 | **-0.8667** | 0.2577 | -0.6910 | -0.6100 | | 0.2339 | 0.0274 |
| Lignin | **-0.8352** | 0.5094 | -0.3758 | -0.1985 | 0.7773 | -0.1675 | **-0.9113** | 0.5736 | | 0.0552 |
| Holocellulose | **-0.9509** | 0.0601 | -0.1259 | 0.6642 | -0.5154 | 0.4944 | 0.6991 | **-0.8614** | -0.8014 | |
| CA | -0.7222 | 0.5852 | -0.2303 | -0.0293 | 0.6961 | -0.1485 | -0.7663 | 0.4268 | **0.9570** | -0.7544 |

water x α (swelling), and hot water x holocellulose. More contributions to form the combinations are associated with ρ12%, CA, holocellulose, extractives, and hot water, and 25.45% are low correlations (r < 0.30), 29.09% moderate (0.30 > r < 0.60), and 46.46% high (r > 0.60) according to Callegari-Jacques et al. (2003).

Wood characterisation studies developed by Lobão et al. (2011) and Silva et al. (2014) found correlations above 0.70 between basic density (Db) and the chemical components of wood. Silva et al. (2020), in recent studies with Amazon woods, found significant correlations between extractives and Db (r = 0.56). Fernandes et al. (2017) found a correlation for extractives/hot water (r = 0.55), while Soares et al. (2018), working with *Eucalyptus* sp. observed significant extractives/cellulose correlations (r = -0.56).

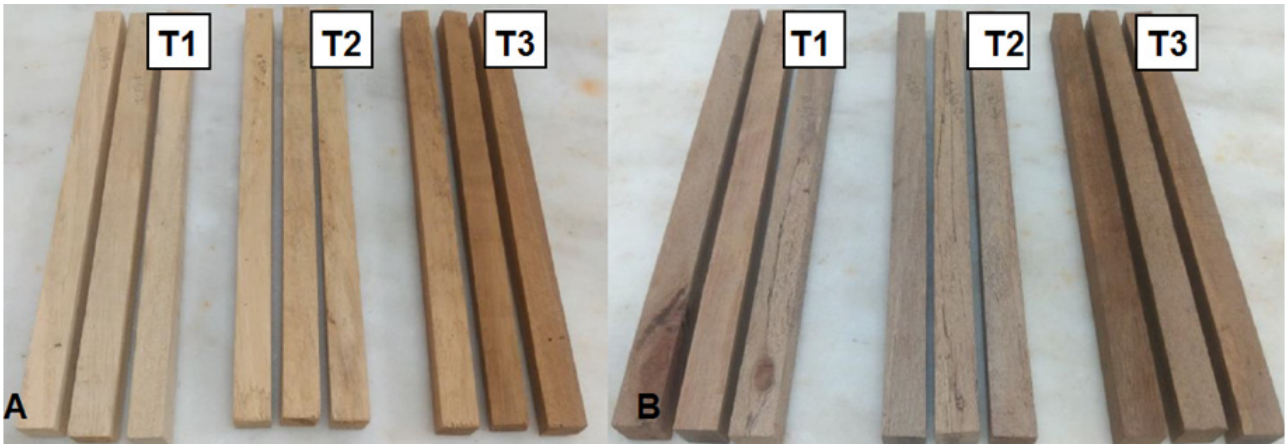
Extractives are found in the middle lamella, parenchyma cells, and vessels in xylem tissue (Nascimento et al. 2021), and their concentration is closely related to density, dimensional stability, and moisture; hence, there are high correlations with these variables. In addition to wood density, which is considered a response variable in several studies, whether technological or ecological (Kollmann and Cotê Junior 1968; Zieminska et al. 2013), there were several high correlations found for swelling (α), hot water, lignin, holocellulose, extractives, and anisotropy coefficient (CA).

**Visual aspects**

An important change in heat-treated wood is its generally darker appearance, and exposure of the wood to heat alters the colourimetric pattern of the wood according to the treatment temperature. Figure 4 shows the colour of the untreated wood (T1) and the thermally altered wood (T2 and T3). There was a colour change as a function of the heat treatments for both assacú and murici. Visual differences in murici wood were barely perceptible for T2, becoming more evident in T3. The colour changes in assacú wood can be considered more linear, with a colour change being perceptible with each treatment applied, T2 and T3.

Changes in the colour of heat-treated wood were observed by Moura & Brito (2011); however, the colour changes did not show linearity as a function of the temperatures applied to the treatments, but in general, the higher temperature (180°C) resulted in a more

FIGURE 4: General aspects of heat-treated woods: A – assacú; B – murici (T1: untreated samples; T2: treated samples (180 °C); T3: treated samples (220 °C).



intense colour. Colourimetry analysis is an efficient tool to confirm colour changes in heat-treated wood. Lengowski et al. (2018) confirmed a significant decrease in the luminosity of *Eucalyptus grandis* and *Pinus taeda* wood exposed to a temperature of 160°C. The colour change can be explained by the combination of several factors, such as migration, solubility, oxidation, and decomposition of extractives. There is also the decomposition of hemicelluloses and lignin, which produce various compounds such as quinoids and other metabolites such as sugars and low molecular weight amino acids, which migrate towards wood surfaces (Bekhta et al. 2014; De Paula et al. 2016).

## Conclusions

The heat treatment (180 and 220°C) of assacú (*Hura crepitans*) and murici (*Byrsonima crispa*) wood directly influenced the physical-mechanical and chemical properties, with a decrease in equilibrium moisture, anisotropy coefficient, apparent density, modulus of elasticity, lignin and holocellulose content, and increased mass loss, linear shrinkage, and extractives.

The analysis of the tested properties indicated a linear relationship between the physical variables and the chemical components of the wood, where the greatest contributions to form the combinations were associated with density, anisotropy coefficient, holocellulose, extractives, and hot water.

In general, the higher temperature treatment caused more changes in the studied woods, and the exposure to heat caused noticeable changes in its colour, emphasizing that the assacú wood presented more linear changes.

The thermal treatment provides a decrease in water in the wood, thus enhancing the resistance to xylophagous organisms as well as dimensional stability, improving the structural and aesthetic quality of the wood that adds value to little-known species, without the market, of low density, unattractive colouring despite great occurrences in tropical forests, and indicated for the practice of sustainable forest management.

## List of abbreviations

| | |
|---|---|
| ρ12% | apparent density |
| α | swelling |
| β | shrinkage |
| CA | anisotropy coefficient |
| EM | equilibrium moisture |
| MOED | dynamic modulus of elasticity |
| WL | mass loss |

## Competing interests

The authors here by declare that there is no conflict of interest associated with this manuscript to the best of their knowledge.

## Authors' contributions

ASL and CSN designed the study. RDA and CSN analysed the data and wrote the manuscript. NH, IAC, and MSO participated in the experimental part and analysed the data. CSN and CCN reviewed the manuscript. NH Funding acquisition. All authors read and approved the final manuscript.

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