ACTA
AMAZONICA | http://dx.doi.org/10.1590/1809-4392202301753

MATERIALS TECHNOLOGY | ORIGINAL ARTICLE

Colorimetry and chemical properties of thermally modified *Parkia pendula* and *Simarouba amara* wood

Leonardo Vinícius de SOUZA[1], Diego Martins STANGERLIN[1], Rafael Rodolfo de MELO[2*], Elaine Cristina LENGOWSKI[1], Eraldo Antonio BONFATTI JÚNIOR[1], Leonardo Gomes de VASCONCELOS[1], Alexandre Santos PIMENTA[3]

[1] Universidade Federal de Mato Grosso – UFMT, Av. Fernando Corrêa da Costa 2367, 78060-900 Cuiabá, Mato Grosso, Brazil

[2] Universidade Federal do Semi-árido – UFERSA, Av. Francisco Mota 572, 59625-900 Mossoró-RN, Brazil

[3] Universidade Federal do Rio Grande do Norte – UFRN, RN 160, km 03, s/n, Distrito de Jundiaí, 59280-000 Macaíba-RN, Brazil

*Corresponding author: rafael.melo@ufersa.edu.br

ABSTRACT

The thermal modification of wood is a process that alters its chemical composition, aiming to improve technological properties such as dimensional stability, reduction of equilibrium moisture content, homogeneity, or color. In this context, this study aimed to evaluate the colorimetry and chemical properties of the wood of *Parkia pendula* (*angelim-saia*) and *Simarouba amara* (*marupá*) that was thermally modified by different methods. Three treatments were evaluated: T1 (pre-treatment in an oven for one hour at 120 °C and treatment in an oven at 180 °C for three hours); T2 (pre-treatment in an autoclave at 125 °C for three hours under 1.2 kgf cm^{-2} and treatment in an oven at 180 °C for three hours); and T3 (without pre-treatment, with sample treatment in vegetable oil). The thermally modified wood was evaluated in relation to untreated wood for structural chemical composition (extractives, lignin, holocellulose and ash content), colorimetric parameters defined through the CIELAB (Commission Internationale de l´Eclairage) system and total color variation. We detected a significant increase in extractives content and a decrease in holocellulose and lignin content in T3 for both species, which can be explained by the impregnation of oil in the wood samples. The thermal modification caused the surface darkening of the wood of both species, which was more pronounced in *P. pendula*. Despite the colorimetric change, there was no chemical damage to the wood.

KEYWORDS: Amazonia, color variation; tropical timber; wood composition; holocellulose

Colorimetria e propriedades químicas da madeira de *Parkia pendula* e *Simarouba amara* modificada termicamente

RESUMO

A modificação térmica da madeira é um processo que altera sua composição química, visando melhorar propriedades tecnológicas como estabilidade dimensional, redução do teor de umidade de equilíbrio, homogeneidade ou cor. Neste contexto, este estudo teve como objetivo avaliar a colorimetria e as propriedades químicas da madeira de *Parkia pendula* (angelim-saia) e *Simarouba amara* (marupá) modificada termicamente por diferentes métodos. Foram avaliados três tratamentos: T1 (pré-tratamento em estufa por uma hora a 120 °C e tratamento em estufa a 180 °C por três horas); T2 (pré-tratamento em autoclave a 125 °C por três horas sob 1,2 kgf cm^{-2} e tratamento em estufa a 180 °C por três horas); T3 (sem pré-tratamento, com tratamento da amostra em óleo vegetal). A madeira termicamente modificada foi avaliada em relação à madeira não tratada quanto à composição química estrutural (extrativos, lignina, holocelulose e teor de cinzas), parâmetros colorimétricos definidos através do sistema CIELAB (Commission Internationale de l´Eclairage) e variação total de cores. Detectamos um aumento significativo no teor de extrativos e uma diminuição no teor de holocelulose e lignina em T3 para ambas espécies, o que pode ser explicado pela impregnação de óleo nas amostras de madeira. A modificação térmica provocou o escurecimento superficial da madeira de ambas espécies, que foi mais pronunciado em *P. pendula*. Apesar da alteração colorimétrica, quimicamente não houve prejuízos para as madeiras.

PALAVRAS-CHAVE: Amazônia; variação da cor; madeiras tropicais; composição da madeira; holocelulose

CITE AS: Souza, L.V. de; Stangerlin, D.M.; Melo, R.R. de; Lengowski, E.C.; Bonfatti Júnior, E.A.; Vasconcelos, L.G. de; Pimenta, A.S. 2024. Colorimetry and chemical properties of thermally modified *Parkia pendula* and *Simarouba amara* wood. *Acta Amazonica* 54: e54mt23175.

INTRODUCTION

Thermal modification consists of exposing a given material to high temperatures with different equipment and ways of transferring heat to the material. Regarding wood, heat transfer can be achieved using water, steam, nitrogen, vegetable oil, and melted metals (Batista 2019). Usually, the thermal treatment aims to change the chemical composition of wood to improve some of its technological properties, such as color, dimensional stability, equilibrium moisture, volumetric swelling, and resistance to biodeterioration, among others (Bal *et al.* 2015; Xu *et al.* 2019).

The technological behavior of thermally modified wood is mainly associated with altering the chemical constituents of the cell wall and extractives (Esteves and Pereira 2009). This change begins with the degradation of hemicelluloses, cellulose, and lignin (Borges and Quirino 2004). Among these constituents, hemicelluloses are hydrophilic polymers with the highest contribution to wood hygroscopicity. Their reduction and degradation is desirable (Ferreira *et al.* 2019), as it allows the material to acquire better volumetric stability (Abreu Neto *et al.* 2021). The degree of modification of technological properties intended to occur during thermal modification depends on the species being treated, the final temperature of the process, treatment time, and the medium employed for heat transfer (Sikora *et al.* 2018; Abreu Neto *et al.* 2021). Changes in the physical properties of thermally modified woods, as in the case of color, are strongly related to the type of method employed for thermal modification (Hill *et al.* 2021).

The first characteristic of wood that is perceptible to the human eye is its color, which plays a role in determining its use (Bonfatti Jr. and Lengowski 2018). Dark-colored species are generally preferred for more noble purposes such as decorative panels and furniture. Light-colored wood is usually employed to manufacture low value-added products (Conte *et al.* 2014). From an aesthetic point of view, thermally modified woods are more superficially darkened, have greater homogeneity, and are visually more attractive to the consumer market (Dzurenda 2018; Pelosi *et al.* 2021).

Among the timber species traded in Brazilian Amazonia, and other countries of South America, *Parkia pendula* (Willd.) and *Simarouba amara* (Aubl.) are used for lower value-added purposes such as wooden linings and boxwork (Souza *et al.* 2023). Thermally modified processes aiming at color darkening can increase the potential of these species for use for more noble purposes, contributing to reduce the impact on more traditional timber species and avoid their overexploitation. Therefore, the objective of this study was to evaluate the colorimetry and changes in the chemical properties of *P. pendula* and *S. amara* wood after exposure to different types of thermal treatment, with the aim of achievinng darker wood color without negatively affecting structural chemical properties of the treated wood.

MATERIAL AND METHODS

Sample preparation

To conduct this study, we used boards of *P. pendula* and *S. amara*, popularly known in Brazil as *angelim-saia* and *marupá*, respectively. Boards were obtained from six trees of each species, originating from two forest management areas (three trees of each species per area), in Garantã do Norte, northern region of the state of Mato Grosso, in the Amazon region of Brazil. Eight samples measuring 2.5 cm x 2.5 cm x 12 cm (radial x tangential x longitudinal length) were obtained from each tree, totaling 48 samples per species. After drying at 60 °C in an oven, the samples were kept in an environmental chamber at 20 °C and 65% relative humidity until reaching constant weight.

Thermal treatments

The samples were submitted to three thermal treatments using 12 samples per treatment and a control for each species (two from each tree). Treatments were as follows: T1 - the test specimens were placed in an oven with forced air circulation, first heated for one hour at 120 °C, then the temperature was increased to 180 °C ± 2 °C min-1 for 3 hours; T2 - the test specimens were placed in an autoclave at 125 °C for 3 hours under 1.2 kgf cm-2 aiming to simulate wet conditions and indirect steaming, and next were placed in an oven with forced air circulation to 180 °C ± 2 °C min-1 for 3 hours; T3 - the specimens were submerged in soybean oil for 3 hours at 180 °C using deep metallic trays sealed with thermal insulating paper and were subsequently subjected to one hour at 120 °C, then the temperature was increased to 180 °C ± 2 °C min-1 for 3 hours. In each thermal treatments, the samples were only exposed when the nominal temperature was reached. The treatments are summarized in Table 1. The control (C) consisted of samples not subjected to any thermal modification, which remained stored in the environmental chamber until the beginning of the tests.

Colorimetric determination

Colorimetric analyses of the test specimens were carried out for each treatment and the control. The colorimetry was determined using a spectrophotometer equipped with an illuminant D65 and an observation angle of 10°, according

Table 1. Experimental thermal treatments (ambient and conditions) applied to *Parkia pendula* (*angelin-saia*) and *Simarouba amara* (*marupá*) wood samples.

Treatment code	Wood pre-treatment	Wood treatment
T1	Oven at 120 °C for 1h	Oven at 180 °C for 3h
T2	Autoclave at 125 °C for 3h	Oven at 180 °C for 3h
T3	No pre-tratment	Soybean oil at 180 °C for 3h

to the procedures standardized by the CIELab system (Commission Internationale de l´Eclairage). For each sample, five measures were acquired, and the mean values of the following parameters were determined: L* = brightness; a* = chromatic coordinate on the green-red axis; b* = chromatic coordinate on the yellow-blue axis; C* = saturation or chromaticity; h* (hue or ink angle). The total color variation (ΔE) was determined using equation 1, according to the standard ASTM D 2244 (ASTM 2021).

$$\Delta E* = \sqrt{(\Delta L^2 + \Delta a^2 + \Delta b^2)} \quad [1]$$

where: ΔE = full-color variation; ΔL* = variation of the L* parameter; Δa* = variation of the a* parameter; Δb* = variation of the parameter b*.

The total color variation was classified according to Cividini (2007), and the wood color was defined by the parameters established by Camargos and Gonçalez (2001).

Chemical composition

This analysis was carried out to assess whether changes in the color of wood, though aesthetically desireable, can cause chemical changes that reduce the quality of the treated wood. After the colorimetric analysis, all samples were ground to sawdust for chemical analysis, following the recommendations of the TAPPI Standards (TAPPI 2017, 2021, 2022). The samples were ground using a Wiley-type mill and then sieved. The fraction collected between the 40 and 60 mesh sieves was used to determine the chemical composition. The analysis was carried out following the TAPPI standards: T204-om17 – total extractives content (TAPPI 2017); T222-om15 – lignin content (TAPPI 2021); and T211-om22 – ash content (TAPPI 2022). The holocellulose content was calculated as the difference between the total sample weight and the sum of extractives, lignin, and ash content.

Statistical analyses

The experimental design adopted was completely randomized for within-tree samples. Distribution normality and homogeneity of variance of the data were ascertained with the Shapiro-Wilk test and Levene test, respectively. Statistical differences among treatments and the control were determined separately for each species using a Tukey test for chemical (total extractives, lignin, holocellulose and ash) and colorimetric (L*, a*, b*, C* and h) parameters, with a 5% significance level. All the statistical procedures were performed with the R software (R Core Team 2023), using the Experimental Design package (version 1.2.2).

RESULTS

Colorimetry

The colorimetric characteristics of both species differed significantly from the control showing darker colors for all treatments (Tables 2; Figure 1). The color classification of the control wood was white-gray for *S. amara* and brownish-yellow for *P. pendula*, and total color variation was higher for *S. amara* (Table 3; Figure 1).

Relative to the control, luminosity decreased from 22% (T1) to 36% (T2) in *P. pendula*, and from 21% (T1) to 32% (T2, T3) in *S. amara*; the a* parameter, which is related to the intensity of the red color, decreased significantly up to 16% (T2) in *P. pendula*, and increased over100% for all treatments in *S. amara*; the b* parameter, which relates to the intensity of the yellow color, decreased up to 38% (T2) in *P. pendula*, and increased up to 77% (T3) in *S. amara* (Table 2). Both a* and b* were associated with surface darkening after thermal modification in both species (Figure 1).

In *P. pendula*, saturation decreased significantly in all treatments relative to the control (Table 2), with average values from 12.4% (T3) to 32.3% (T2) lower than the control. In *S.*

Table 2. Colorimetric parameters determined for *Parkia pendula* (angelin-saia) and *Simarouba amara* (marupá) wood as a function of different thermal treatments. Values are the mean ± standard deviation of 12 samples.

Species	Treatment	Colorimetric parameter				
		L*	a*	b*	C*	h
P. pendula	C	59.73 ± 3.01 a	14.10 ± 0.45 a	24.21 ± 1.52 a	28.27 ± 0.85 a	59.88 ± 1.69 a
	T1	42.11 ± 1.24 b	13.05 ± 0.37 b	17.10 ± 0.75 c	21.56 ± 0.80 c	52.66 ± 0.73 c
	T2	37.97 ± 1.43 c	11.84 ± 0.57 c	15.01 ± 0.80 d	19.13 ± 0.91 d	51.69 ± 1.11 c
	T3	41.24 ± 2.65 b	13.26 ± 0.65 b	20.81 ± 1.75 b	24.76 ± 1.34 b	57.35 ± 3.15 b
S. amara	C	85.95 ± 1.11 a	2.06 ± 0.15 d	19.90 ± 0.83 d	20.13 ± 0.94 d	84.09 ± 0.34 a
	T1	67.88 ± 1.63 b	8.43 ± 0.57 c	26.79 ± 0.59 c	28.10 ± 0.73 c	72.49 ± 0.87 b
	T2	58.33 ± 1.65 c	10.79 ± 1.02 b	27.97 ± 0.91 b	29.91 ± 0.97 b	69.33 ± 0.84 c
	T3	58.32 ± 2.76 c	13.06 ± 1.29 a	35.18 ± 2.69 a	37.56 ± 2.74 a	69.63 ± 1.84 c

C = control (untreated wood); T1 = pre-treatment in oven at 120 ºC for 1h+ treatment in oven at 180 ºC for 3h; T2 = pre-treatment in autoclave at 125 ºC for 3h + treatment in oven at 180 ºC for 3h; T3 = without pre-treatment, treatment in soybean oil at 180 ºC for 3h. Means followed by the same letter in the same column within species are statistically equal according to a Tukey test at a 5% significance level.

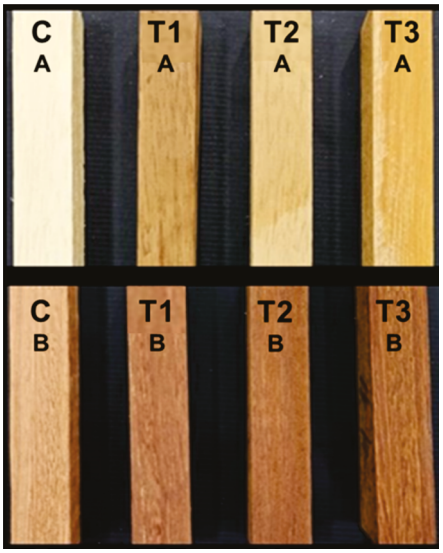

Figure 1. Examples of color variation in *Simarouba amara* (*marupá*) (A) and *Parkia pendula* (*angelin-saia*) (B). C = untreated control wood; T1, T2, T3 = experimental thermal treatments.

Table 3. Total color variation (ΔE) and color classification according to Camargos and Gonçalez (2001) of *Parkia pendula* (*angelin-saia*) and *Simarouba amara* (*marupá*) subjected to three experimental thermal treatments (T1, T2, T3) relative to untreated wood (control, C). Values are the mean ± standard deviation of 12 samples.

Treat	ΔE	Color
Parkia pendula		
C	-	Brownish-yellow
T1	19.0 ± 2.9	Dark-brown
T2	23.7 ± 3.5	Dark-brown
T3	18.8 ± 3.7	Reddish-brown
Simarouba amara		
C	-	Gray-white
T1	20.4 ± 2.8	Light-yellow
T2	30.1 ± 4.7	Olive-brown
T3	33.4 ± 5.4	Yellow-orange

Treat = treatment; C = control (untreated wood); T1 = pre-treatment in oven at 120 ºC for 1h+ treatment in oven at 180 ºC for 3h; T2 = pre-treatment in autoclave at 125 ºC for 3h + treatment in oven at 180 ºC for 3h; T3 = without pre-treatment, treatment in soybean oil at 180 ºC for 3h.

amara, saturation increased significantly from 39.6% (T1) to 86.6% (T3) relative to the control. The ink angle decreased significantly in all treatments relative to the control for both species (Table 2), varying from 4.2% (T3) to 13.7% (T1) lower in *P. pendula*, and from 13.8% (T1) to 17.6% (T2) lower in *S. amara*.

Chemical composition

The relative content of total extractives differed significantly from the control in T2 and T3 of *P. pendula* (Table 4), decreasing 16.7% in T2 and increasing 133.8% in T3. In *S. amara*, total extractives increased significantly in all treatments.

In *P. pendula*, relative lignin content only differed significantly from the control in T3, decreasing by 10.2% (Table 4). In *S. amara*, there was a significant increase of 6.3% in the relative lignin content in T1 and a significant decrease of 23.9% in T3.

Relative holocellulose content decreased significantly by 13.9% in T3 in *P. pendula* relative to the control, and significantly decreased in T1 and T3, by 4.4% and 27.9%, respectively, in *S. amara* (Table 4).

In *P. pendula*, relative ash content was significantly higher than the control in T1, and differed significantly from the control in all treatments in *S. amara*, increasing in T1 and T2, and decreasing by 87% in T3.

DISCUSSION

Colorimetry

A decrease in luminosity after thermal treatment of wood has also been reported in other studies for *Simarouba amara* Aubl., *Indosasa angustata* McClure and *Toona sinensis* (Juss.) M. Roem. (Freitas *et al.* 2016; Nguyen *et al.* 2019; Xie *et al.* 2021). The reduction in luminosity is associated with the volatilization and oxidation of extractives and the degradation of hemicelluloses (Charrier *et al.* 2002; Lengowski *et al.* 2018).

Table 4. Changes in the relative content (%) of chemical components of *Parkia pendula* (*angelin-saia*) and *Simarouba amara* (*marupá*) wood as a function of three experimental thermal treatments (T1, T2, T3). Values are the mean ± standard deviation of 12 amples taken from six trees of each species.

Species	Treatment	Total extractives	Lignin	Holocellulose	Ash
P. pendula	C	8.69 ± 0.15 b	29.55 ± 0.43 a	61.74 ± 0.35 a	0.04 ± 0.17 b
	T1	8.50 ± 0.18 b	29.05 ± 0.50 a	62.43 ± 0.28 a	0.19 ± 0.16 a
	T2	7.24 ± 0.04 c	30.20 ± 0.46 a	62.54 ± 0.47 a	0.04 ± 0.03 b
	T3	20.32 ± 0.34 a	26.53 ± 0.23 b	53.13 ± 1.12 b	0.04 ± 0.14 b
S. amara	C	3.29 ± 0.07 c	30.12 ± 0.43 b	66.57 ± 0.66 a	0.69 ± 0.21 c
	T1	4.33 ± 0.04 b	32.03 ± 0.30 a	63.63 ± 0.37 b	0.74 ± 0.17 b
	T2	4.39 ± 0.05 b	30.45 ± 0.14 b	65.15 ± 0.14 a	0.79 ± 0.03 a
	T3	29.02 ± 0.19 a	22.93 ± 0.07 c	48.03 ± 0.05 c	0.09 ± 0.10 d

C = control (untreated wood); T1 = pre-treatment in oven at 120 ºC for 1h+ treatment in oven at 180 ºC for 3h; T2 = pre-treatment in autoclave at 125 ºC for 3h + treatment in oven at 180 ºC for 3h; T3 = without pre-treatment, treatment in soybean oil at 180 ºC for 3h. Means followed by the same letter in the same column within species are statistically equal according to a Tukey test at a 5% significance level.

As observed in this study for *P. pendula*, a decrease in parameters a* and b* was also reported for thermally modified wood of *Carpinus betulus* L. and *Guarea trichilioides* L. (Gunduz and Aydemir 2009; Santos *et al.* 2014). On the other hand, as in our study, Freitas et al. (2016) also observed an increase in a* and b* for thermally treated wood of *S. amara*, as did Aydemir *et al.* (2012) for *Cupressus sempervirens* L.

C* and h depend on variations in a* and b* (Lazarotto *et al.* 2016; Bonfatti Jr. *et al.* 2018). As the values of b* were generally higher than those of a* for both species, the yellow matrix had more influence on saturation and ink angle (Zanuncio *et al.* 2014). This variation evidences a species-dependent chemical modification of the chromophores (mainly the extractive content) responsible for the wood color (Ahajji *et al.* 2009).

According to the color change criteria proposed by Cividini (2007), the total color variation (ΔE) for all experimental treatments were classified with "different color" – ΔE > 12. This classification indicates that the heat treatment caused significant changes in the colors of the wood evaluated. The color of the wood of *P. pendula* before treatment indicates a more intense presence of red pigment, which caused a reduction in the a* parameter after thermal modification.

Chemical composition

The general increase observed in relative extractives content may be related to the condensation of volatile extractives and water during thermal modification of the wood, due to the degradation of hemicellulose and cellulose (Hill *et al.* 202). This effect was also observed by Čabalová *et al.* (2018) and Gasparik *et al.* (2019). The significant increase in total extractive content observed in T3 resulted from the residual soybean oil used in this treatment that remained in the wood after thermal modification (unpublished data). Thus, heat treatment did not increase the amount of extractives. This finding is further supported by the similar behavior observed in thermal modification studies by Todaro et al. (2015), Gasparik et al. (2019), and Meca et al. (2019).

Lignin has higher thermal degradation resistance than polysaccharides (Nair *et al.* 2017; Jiang *et al.* 2020). There could not have occurred any reduction in lignin in our study because its thermal degradation only occurs above 300 oC (Borges and Quirino 2004) and the maximum temperature used in our study was 180 °C. The decrease in relative lignin content in T3 in both species is likely due to the immobilization of part of the soybean oil used in the treatment in the ultrastructure of the wood (Lengowski *et al.* 2018). Some increase in the lignin content, such as observed in T1 for *S. amara*, was expected to occur due to the increase of cross-linking with furfural, which is a component derived from the thermal degradation of hemicelluloses (Jaludin *et al.* 2010; Wang *et al.* 2018).

The holocellulose content represents the total fraction of wood polysaccharides, consisting of cellulose and hemicelluloses, with hemicellulose being the chemical compound with the lowest thermal stability (Stangerlin *et al.* 2013), therefore the decrease in holocellulose in modified wood may be associated with hemicellulose degradation.

Relative ash content increased after heat treatment of wood in several species, however, this modification may be owed to a reduction in other components, such as extractives, which are volatilized (Todaro *et al.* 2015). Ash represents the mineral fraction of the cell wall and, eventually, the mineral content of the parenchyma lumen, and it is not possible to change its amount in absolute terms (Souza *et al.* 2023). Therefore, the variation in relative ash content observed in our study in both species is owed to absolute variation in other components. The large decrease in ash content in T3 in *S. amara* was probably owed to the immersion in soybean oil, as the amount of extractable substances from the wood increased with oil impregnation.

CONCLUSIONS

The tested thermal treatments significantly changed the colorimetry and the color classification of *Parkia pendula* and *Simarouba amara* wood to darker hues. However, for *P. pendula*, only the treatment that involved boiling the wood in soybean oil (T3) promoted a sufficiently expressive change in the original color to enhance its potential for nobler uses of the wood where the aesthetical aspect is valued. The other treatments did not promote enough darkening relative to the original wood color. The color of *S. amara* wood only slightly changed compared to the untreated wood in treatments T2 and T3. Only T3 caused some alteration in the chemical composition of the untreated wood by partially incorporating the triglycerides of the soybean oil into the wood structure. Despite the incrustation of soybean oil in wood, this aspect is not expected to reduce the quality or mechanical properties of the wood.

ACKNOWLEDGMENTS

The study was financed by Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), and Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq).

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RECEIVED: 14/06/2023
ACCEPTED: 31/07/2024
ASSOCIATE EDITOR: João Paulo Silva
DATA AVAILABILITY: The data that support the findings of this study are available from the corresponding author [Rafael Rodolfo de Melo] upon reasonable request.