INFLUENCE OF COMMERCIAL THERMAL TREATMENT ON *Eucalyptus grandis Hill ex Maiden* WOOD PROPERTIES

Carolina Aparecida Barros Oliveira[2*], Karina Aparecida de Oliveira[2], Julio Cesar Molina[3], Vinicius Borges de Moura Aquino[4] and André Luis Christoforo[5]

[1] Received on 10.02.2021 accepted for publication on 17.05.2021.
[2] Universidade Estadual Paulista Júlio de Mesquita Filho, Programa de Pós-Graduação em Engenharia Mecânica, Guaratinguetá, SP - Brasil. E-mail: <carolina.barros@outlook.com> and <kari.oliveira@outlook.com>.
[3] Universidade Estadual Paulista Júlio de Mesquita Filho, Departamento de Engenharia, Itapeva, SP - Brasil. E-mail: <julio.molina@unesp.br>.
[4] Universidade Federal do Sul e Suldeste do Pará, Departamento de Engenharia Civil, Santana do Araguaia, PA - Brasil. E-mail: <aquino.vini@hotmail.com>.
[5] Universidade Federal de São Carlos, Departamento de Engenharia civil, São Carlos, SP - Brasil. E-mail: <christoforoal@yahoo.com.br>.
*Corresponding author.

ABSTRACT – This study aimed to evaluate the influence of commercial thermal treatment on Eucalyptus grandis considering its physical, chemical, and mechanical properties. The wood samples were heat-treated in an autoclave with saturated steam and pressure application at four different temperatures: 155, 165, 175, and 185 °C. The physical, chemical, and mechanical properties were altered due to the heat treatment. The extractives content varied between 6.06% and 28.75%; lignin between 28.93% and 37.96%; holocellulose between 65.01% and 38.12%. The mechanical properties reduced significantly with the increase of the heat treatment temperature. Through the set of data obtained, it was possible to generate significant and high precision regression models capable of estimating such properties for heat treatment temperatures not studied experimentally, enabling the determination of the most suitable temperature of heat treatment to achieve a certain property value of the treated wood.

Keywords: Chemical properties; Mechanical properties; Regression models..

INFLUÊNCIA DO TRATAMENTO TÉRMICO COMERCIAL NAS PROPRIEDADES DA MADEIRA DE Eucalyptus grandis Hill ex Maiden

RESUMO – Este estudo teve como objetivo avaliar a influência do tratamento térmico comercial no Eucalyptus grandis considerando suas propriedades fisicas, químicas e mecânicas. As amostras de madeira foram tratadas termicamente em autoclave com vapor saturado e aplicação de pressão em quatro diferentes temperaturas: 155, 165, 175 e 185 ° C. As propriedades fisicas, químicas e mecânicas foram alteradas devido ao tratamento térmico. O teor de extrativos variou entre 6,06% e 28,75%; lignina entre 28,93% e 37,96%; holocelulose entre 65,01% e 38,12%. As propriedades mecânicas reduziram significativamente com o aumento da temperatura do tratamento térmico. Com o conjunto de dados obtidos, foi possível gerar modelos de regressão significativos e de alta precisão capazes de estimar tais propriedades para temperaturas de tratamento térmico não estudadas experimentalmente, possibilitando a determinação da temperatura de tratamento térmico mais adequada para atingir determinado valor de propriedade do tratado madeira.

Palavras-Chave: Propriedades químicas; Propriedades mecânicas; Modelos de regressão.

Revista Árvore 2021;45:e4527
http://dx.doi.org/10.1590/1806-908820210000027

1. INTRODUCTION

The use of thermal modified wood on civil construction, industry and furniture has arisen as an alternative of preserved wood for outdoor utilization, not using chemical preservatives such Chromated copper arsenate (CCA), Chromated copper borate (CCB) and creosote, reinforcing their properties against biological attacks and extreme weathering conditions (Almeida et al., 2019; Herzog et al., 2004; Kačíková et al., 2013; Lahtela and Kärki, 2014).

Wood thermally treated can be described as a physical-chemical process that change wood anatomy and wood constituents, such cellulose, hemicellulose, lignin and extractives, performing this process under temperature varying between 150 and 280 °C on controlled atmosphere, which can be vacuum, steam or under heated oil (Batista et al., 2016b; Lee et al., 2018; Wikberg and Maunu, 2004), and for a controlled time, improving physical properties reducing shrinkage, permeability and hygroscopicity, increasing dimensional stability (Barboutis and Kamperido, 2019; Batista et al., 2018; Ribeiro et al., 2019). Otherwise, with wood constituents degradation, such as cellulose and hemicellulose, mechanical properties are reduced proportionally with thermal treatment (Chen et al., 2016; Kariz et al., 2016; Kubovský et al., 2020; Lahtela and Kärki, 2014).

Related to the behavior of wood constituents after thermal treatment, it is noted that the index of cellulose crystallinity increases due pyrolysis of amorphous cellulose, avoiding hydroxyls groups to reach water, diminishing moisture content (Kačíková et al., 2013; Kubovský et al., 2020). Hemicellulose starts to degrade at lower temperatures, close to 150 °C, due its lower thermal stability (Ahmadi et al., 2019; Pertuzzatti et al., 2018; Severo et al., 2012; Sikora et al., 2018). Lignin displays variation of its content in relation of wood constituents, reducing in temperatures close to 150 °C and increasing on 200 °C. It occurs due to its thermal stability along thermal treatment and process of degradation and repolymerization, decreasing hygroscopicity on wood (Kačíková et al., 2013; Sikora et al., 2018; Tjeerdsma and Militz, 2005).

After the thermal treatment, the wood presented a darker shade, an important characteristic that increases its market value (Baysal et al., 2014; Soltani et al., 2016).

For commercial use, several wood thermal treatment process are available, such as PlatoWood®, RetiWood®, ThermoWood® and Le Bois Perdure® (Gašparík et al., 2019; Gurleyen et al., 2019; Jirouš-Rajković and Miklečić, 2019; Platowood, 2020). One of the most used process, ThermoWood®, is divided in three phases: drying and heating the wood until 130 °C, then the thermal treatment, elevating the temperature until 200 °C and cooling and stabilizing wood, controlling humidity content (International Thermowood Association, 2003; Gurleyen et al., 2019; Kubovský et al., 2020; Sikora et al., 2018).

Considering the use of thermal modified wood on industry, furniture, civil and rural construction for structural and non-structural purpose, *Eucalyptus grandis* Hill ex Maiden wood species is an alternative, demanding to evaluate the effect of thermal treatment on physical and mechanical properties.

On the literature, several studies analyzed the influence of thermal treatment on *Eucalyptus grandis*, considering colorimetric factors (Cademartori et al., 2013; Lazarotto et al., 2016; Moura and Brito, 2011; Zanuncio et al., 2014), chemical factors (Moura et al., 2012b; Zanuncio et al., 2014), anatomical factors (Cheng et al., 2017; Modes et al., 2013), physical factors (Bal and Bektaş, 2012) and mechanical factors (Cademartori et al., 2012; Moura et al., 2012a).

However, no researches on the literature present the full characterization of *Eucalyptus grandis* of physical, mechanical and chemical properties using commercial thermally treated wood, performed on industry, and the possibility to estimate physical, chemical and mechanical properties of thermal modified wood in function of thermal treatment temperature.

For this reason, the present assignment aimed to analyze the performance of *Eucalyptus grandis* submitted to different temperatures of heat treatment, observing its physical, chemical, and mechanical properties, besides verifying the possibility of estimating such properties for temperatures not analyzed experimentally, through regression models.

2. MATERIAL AND METHODS

2.1 Wood

The logs of *Eucalyptus grandis* Hill ex Maiden used in the present research were provided by 10-year-old-planted-forestry, located in Ribeirão Branco, São Paulo, Brazil (24°10' 26.2"S latitude, 48°48' 06.4"W longitude, 900 to 1000 m altitude), in predominant Cambisol Haplic soil, argisolic with rugged relief. The logs had average diameters of 32 cm and length of 3 m were sawn on boards with a transversal dimension of 6 cm x 16 cm x 3 m and subsequently air-dried until reaching a moisture content of 12 ± 2%.

2.2 Thermal treatment

The thermal treatment of wood was performed on industrial scale, heating wood using autoclave (Krupp mechanical industries), with 2 meters in diameter and 20 meters in length, pressure (maximum working pressure of 1569 kPa) and controlled temperature and saturated steam. The heating rate used by the company was 1.66 $°C.min^{-1}$. The process used on thermal treatment can be described in five stages: Initial heating, autoclave loading, heating, thermal treatment and cooling, as shown in Figure 1. The heating rate used by the company was 1.66 $°C.min^{-1}$. The process used on thermal treatment can be described in five stages: Initial heating, autoclave loading, heating, thermal treatment and cooling, as shown in Figure 1.

Initially, the empty autoclave at room temperature, approximately 20 ºC, was heated with saturated steam to 100 ºC, lasting about one hour (t0 to t1) (step 1). Then, the autoclave door was opened to load wood in its interior. This process reduced the temperature from 100 °C to approximately 40 °C (step 2). The wood in the autoclave was heated to the desired thermal treatment temperature (step 3). On the present research, four temperatures were considered on thermal treatment: 155, 165, 175 and 185 °C. When the desired heat treatment temperature was reached, it was kept constant for two hours (t3 to t4), with pressure of 735 kPa. During the cooling stage, the pressure was relieved until the inner temperature on the autoclave reach room temperature (approximately 20 °C). The time from t2 to t3 and t4 to t5 varies depending on the temperature to be reached.

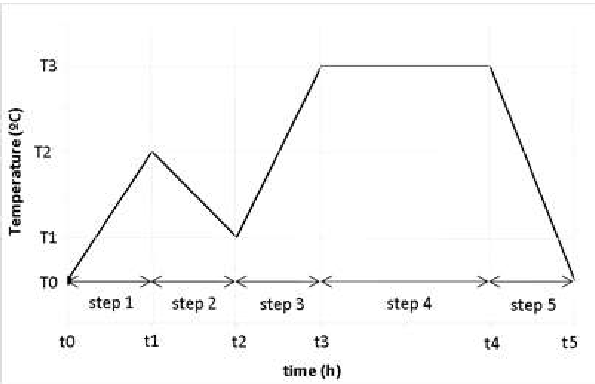

Figure 1 – Thermal treatment program
Figura 1 *– Programa de tratamento térmico.*

After thermal treatment, the boards was sawn to produce test samples to characterize *Eucalyptus grandis* considering physical, mechanical and chemical properties.

2.3 Characterization

The samples for physical and mechanical propoeties were following the disposed on the Brazilian Standard – NBR 7190, Brazilian Association of Technical Standards - ABNT (1997).

The following physical and mechanical properties were determined by: apparent density (ρap,12%), modulus of elasticity in compression parallel to the grain (E_{c0}), compressive strength parallel to the grain (f_{c0}), modulus of elasticity in tension parallel to the grain (E_{t0}), tensile strength parallel to the grain (f_{t0}), modulus of elasticity on static bending (E_{M0}), strength on static bending (f_{M0}), shear strength parallel to the grain (f_{v0}), hardness parallel to the grain (f_{H0}), hardness normal to the grain (f_{H90}) and toughness (f_{bw}). Twelve boards of each temperature were separated after the thermal treatment, and a sample was extracted from each board for each property, totaling twelve samples for each property and thermal treatment temperature.

For the treatment control (without thermal treatment) and for all four thermal treatment temperatures, *Eucalyptus grandis* were classified into strength classes, determining the characteristic strength value (Equation 1), as NBR 7190, ABNT (1997).

$$fk = \left(2.\frac{f1 + f2 + f3 + \cdots + f\left(\frac{n}{2}\right) - 1}{\left(\frac{n}{2}\right) - 1} - f\frac{n}{2} \right).1.10 \quad \text{Eq.1}$$

Where: f_k is the characteristic strength value and n is the number of test pieces used. It should be noted that the results of strength should be placed in ascending order $f_1 \leq f_2 \leq \ldots \leq f_n$, ignoring the highest value if the number of samples is odd, and not taking f_k below f_1 nor 0,70 of the average value.

Figure 2 – Control and thermal treated samples. From left to right: control, 155, 165, 175 and 185 °C.
Figura 2 *– Amostras de controle e tratadas termicamente. Da esquerda para a direita: controle, 155, 165, 175 e 185 °C.*

For chemical analysis, six replications were performed for each thermal treatment temperature, the samples of wood were obtained according the Technical Association of the Pulp and Paper Industry - TAPPI Standard (TAPPI, 1985; TAPPI, 1997). The wood was crushed to reach small particles passing a 42 mesh (0.355 mm). The total extractive were evaluated by standard TAPPI 204 cm-97 (TAPPI, 1997), checking the volume of extractives on the samples. These samples were extracted in phases in a soxhlet with a mix toluene/ethanol for 6 hours (1:1 v/v); ethanol 95% pure for 5 hours and boiling distilled water for 30 minutes. After removing extractives, the samples were washed with distilled water and dried in oven at 103 ± 2 °C for 24 hours. The extractive content was calculated by difference in mass.

The result of extractive-free wood was used to determine total lignin content by modified Klason method (Gomide and Memuner, 1986), by the sum of insoluble and soluble lignin content. The holocellulose content was determined by difference between lignin content and extractive-free wood mass (Tjeerdsma and Militz, 2005).

2.4 Statistical analysis

The influence of thermal treatment temperature was evaluated by Tukey test ($p < 0.05$). Moreover, it was performed checking the possibility to estimate physical, chemical and mechanical properties in function of thermal treatment properties using linear regression model. The regression models were generated considering different in natura temperatures of 25 °C and 30 °C.

To validate the models, the analysis of variance (ANOVA), F test ($p < 0.05$), the formulated null hypothesis consisted by the non-representativeness of

Table 1 – Results of density, mass loss, mechanical and chemical properties of *Eucalyptus grandis* wood for different thermal treatment temperatures.
Tabela 1 *– Resultados da densidade, perda de massa, propriedades mecânicas e químicas da madeira de* ***Eucalyptus grandis*** *em diferentes temperaturas de tratamento térmico.*

Properties	Thermal Treatment Temperatures (°C)					CV (%)	
	IN	155	165	175	185	Min	Max
ρ_{ap},12% (g cm^{-3})	0.537 A	0.520 A	0.502 A	0.457 A	0.434 A	8.39	22.66
mass loss (%)	-	3.17	6.52	14.90	19.18	-	-
E_{c0} (GPa)	9.96 A	6.46 B	5.90 B	5.48 B	5.04 B	4.72	18.76
f_{c0} (MPa)	36.73 A	11.91 B	10.51 B	10.46 B	7.29 C	5.04	24.42
E_{t0} (GPa)	12.48 A	9.05 B	8.69 B	8.06 B	7.36 B	14.51	24.39
f_{t0} (MPa)	43.98 A	35.56 B	28.36 B	19.11 C	13.75 C	6.16	27.49
E_{M0} (GPa)	9.74 A	5.80 B	4.42 BC	3.29 B	2.83 C	17.11	28.02
f_{M0} (MPa)	43.31 A	24.47 B	14.21 C	11.93 C	7.80 D	6.03	28.48
f_{v0} (MPa)	3.89 A	3.50 AB	2.56 B	2.18 B	2.16 C	8.53	29.39
f_{H0} (MPa)	34.18 A	23.68 B	17.55 BC	15.64 C	13.65 C	4.86	29.08
f_{H90} (MPa)	30.73 A	18.94 B	8.85 C	7.92 C	6.54 C	6.1	28.33
f_{bw} (MPa)	105.81 A	41.00 B	26.71 BC	16.00 C	13.12 C	11.79	25.08
Extractive (%)	6.06 E	21.49 D	23.49 C	25.61 B	28.75 A	1.77	3.47
Insoluble lignin (%)	25.78 D	31.62 C	37.28 A	35.17 AB	32.59 BC	0.93	6.29
Soluble lignin (%)	3.15 A	0.72 B	0.68 B	0.59 B	0.55 B	2.41	5.28
Total lignin (%)	28.93 D	32.34 C	37.96 A	35.77 AB	33.14 BC	0.71	6.25

Averages followed by the same letter mean that they do not differ statistically at 5% probability by Tukey's test. CV refer to the extreme values of coefficient of variation. Apparent density ($\rho_{ap,12\%}$), modulus of elasticity in compression (E_{c0}), compressive strength (f_{c0}), modulus of elasticity in tension (E_{t0}), tensile strength (f_{t0}), modulus of elasticity on static bending (E_{M0}), strength on static bending (f_{M0}), shear strength (f_{v0}) and hardness (f_{H0}) parallel to the grain, hardness normal to the grain (f_{H90}) and toughness (fbw).
Médias seguidas da mesma letra significam que não diferem estatisticamente a 5% de probabilidade pelo teste de Tukey. CV referem-se aos valores extremos do coeficiente de variação. Densidade aparente (pap, 12%), módulo de elasticidade na compressão (E_{c0}), resistência à compressão (f_{c0}), módulo de elasticidade na tração (Et0), resistência à tração (f_{t0}), módulo de elasticidade na flexão (E_{M0}), resistência à flexão (f_{M0}), resistência ao cisalhamento (f_{v0}) e dureza (f_{H0}) paralela às fibras, dureza normal às fibras (f_{H90}) e resistência ao impacto na flexão (f_{bw}).

Table 2 – Results of characteristic values of strength properties of *Eucalyptus grandis* wood for different thermal treatment temperatures.
Tabela 2 *– Resultados dos valores característicos das propriedades de resistência da madeira de **Eucalyptus grandis** para diferentes temperaturas de tratamento térmico.*

Properties	Thermal Treatment Temperatures (oC)				
	IN	155	165	175	185
$f_{c0,k}$ (MPa)	21.43	13.1	12.73	12.45	13.19
$f_{t0,k}$ (MPa)	30.41	14.81	11.39	6.5	4.37
$f_{M0,k}$ (MPa)	34.46	12.93	12.38	8.51	5.8
$f_{v0,k}$ (MPa)	6.65	3.46	3.61	2.84	1.59
$f_{H0,k}$ (MPa)	18.3	19.54	14.47	18.59	18.47
$f_{H90,k}$ (MPa)	11.66	9.83	8.04	5.33	6.09
$f^{bw,k}$ (MPa)	30.13	33.92	19.97	14.34	12.06

Characteristic value of: compressive strength ($f_{c0,k}$), tensile strength ($f_{t0,k}$) , strength on static bending (fM0,k), shear strength ($f_{v0,k}$) and hardness ($f_{H0,k}$) parallel to the grain, hardness normal to the grain ($f_{H90,k}$) and toughness ($f_{bw,k}$).
Valor característico de: resistência à compressão ($f_{c0,k}$), resistência à tração ($f_{t0,k}$), resistência à flexão ($f_{M0,k}$), resistência ao cisalhamento ($f_{v0,k}$) e dureza ($f_{H0,k}$) paralela às fibras, dureza normal às fibras ($f_{H90,k}$) e resistência ao impacto na flexão ($f_{bw,k}$).

the tested models (H_0: $\beta = 0$), and the representativeness as an alternative hypothesis (H_1: $\beta \neq 0$). P-value greater than the significance level implies in the accepting H_0 (the model tested is not representative), refuting it otherwise (the model tested is representative).

It is important to highlight that twelve samples were used to determine physical and mechanical properties for each temperature levels, including the reference temperature (control) and six samples for thermal treatment temperature for chemical properties, resulting in 810 determination at all.

Regression models were generated considering different in natura temperatures of 25 °C and 30 °C.

3. RESULTS

Figure 2 illustrates the coloring change of the heat-treated samples at the four treatment temperatures studied (155, 165, 175, and 185 ºC) in relation to the control sample. The thermal treatment considerably changed the color of the wood, changing it from a light color (control) to a brown color that gradually increased as the treatment temperature got higher. The

Table 3 –Results of regression models.
Tabela 3 *–Resultados dos modelos de regressão.*

Model	R² (%)	p-value
$\rho_{ap,12\%} = 0.5537 - 0.000441\ T$	14.96	0.042
$E_{c0} = 10.591 - 0.02863\ T$	71.98	0.000
$f_{c0} = 40.165 - 0.17713\ T$	98.17	0.000
$E_{t0} = 13.145 - 0.02941\ T$	64.83	0.000
$f_{t0} = 48.24 - 0.1494\ T$	67.29	0.000
$E_{M0} = 10.696 - 0.0392\ T$	78.53	0.000
$f_{M0} = 48.21 - 0.1994\ T$	86.67	0.000
$f_{v0} = 4.168 - 0.00954\ T$	46.35	0.000
$f_{H0} = 37.71 - 0.1199\ T$	71.79	0.000
$f_{H90} = 34.35 - 0.1417\ T$	82.25	0.000
$f_{bw} = 117.84 - 0.5551\ T$	92.86	0.000
Extractives = 3.124 + 0.12825 T	97.33	0.000
Insoluble lignin = 24.85 + 0.0546 T	68.59	0.000
Soluble lignin = 3.4445 – 0.016471 T	98.55	0.000
Total lignin = 28.29 + 0.0381 T	51.83	0.002
Holocellulose = 68.59 – 0.16637 T	95.81	0.000

From ANOVA (F; $p < 0.05$), p-value < 0.05 implies on significance of regression model determined (temperature variation explain effectively variations on dependent variables), and non-significant otherwise (p-value ≥ 0.05). Apparent density ($\rho_{ap,12\%}$); Modulus of elasticity in compression (E_{c0}), compressive strength (f_{c0}), modulus of elasticity in tension (E_{t0}), tensile strength (f_{t0}), modulus of elasticity on static bending (E_{M0}), strength on static bending (f_{M0}), shear strength (f_{v0}) and hardness (f_{H0}) parallel to the grain, hardness normal to the grain (f_{H90}) and toughness (f_{bw}).
A partir da ANOVA (F; p <0,05), o valor de p < 0,05 implica na significância do modelo de regressão determinado (variação de temperatura explica efetivamente variações na dependência variáveis), e não significativas caso p-valor ≥ 0,05. Densidade aparente (pap, 12%); Módulo de elasticidade na compressão (E_{c0}), resistência à compressão (f_{c0}), módulo de elasticidade na tração (E_{t0}), resistência à tração (f_{t0}), módulo de elasticidade na flexão (E_{M0}), resistência à flexão (f_{M0}), resistência ao cisalhamento (f_{v0}) e dureza (f_{H0}) paralela às fibras, dureza normal às fibras (f_{H90}) e resistência ao impacto na flexão (f_{bw}).

coloring change occurred uniformly throughout the thickness of the wood, it is also possible to observe that the higher the temperature of heating treatment, the greater was the occurrence of cracks in the wood.

Table 1 lists the mean values, extreme values of coefficient of variation (CV) and the results of Tukey test ($p < 0.05$) of the density and average loss of mass in function of thermal treatment temperature.

Table 2 lists the characteristic values of *Eucalyptus grandis* strength properties, following the disposed on NBR 7190 (ABNT, 1997).

Considering the characteristic value of compressive strength parallel to the grain, *Eucalyptus grandis* can be classified on C20 strength class, following the NBR 7190 (ABNT, 1997).

Table 3 lists the regression models of physical, mechanical and chemical properties estimated by thermal treatment temperatures, being underlined the models considered significant by ANOVA.

4. DISCUSSION

Considering the apparent density results on Table 1, the values on this research were similar to the reached by Bal and Bektaş (2012) with 20-year-old trees, which found density values of 0.554, 0.548, and 0.545 g cm^{-3} for temperatures of control, 150 °C and 180 °C, respectively, with coefficient of variation (CV) ranging between 11 and 14%. Calonego et al. (2012) found for 30-year-old trees *Eucalyptus grandis* density values varying between 0.445 and 0.477 g cm^{-3} and CV between 5.17 and 7.81% analyzing temperatures of control, 140, 160 and 180 °C. Batista et. al. (2018) encountered density varying between 0.41 and 0.49 g cm^{-3}.

Considering all the results, the present study demonstrated a maximum mass loss of 19%, when Calonego et. al. (2012) reached a maximum mass loss of 7%, Bal and Bektaş (2012), 0.9% and Batista et. al. (2018), 16%. This can be explained due to the age of the trees when harvested, demanding further researches for more significant results. The major variation of CV can be explained due to degradation of wood constituents and wood hysteresis (Calonego et al., 2012; Herrera-Díaz et al., 2019; Kačíková et al., 2013).

For mechanical properties, the NBR 7190 (ABNT, 1997) establishes for characterization to be considered adequate, i.e., the statistical significance of results is guaranteed with no further evaluation, the CV must be lower than 18% for normal efforts and 28% for tangential efforts. Then, checking the results, some properties (f_{c0}, E_{t0}, f_{t0}, f_{v0}) did not meet this requirement. A possible explanation is the intrinsic variability of wood considering edaphoclimatic factors, anatomy and modifications during thermal treatment (Calonego et al., 2012; Christoforo et al., 2020; Kačíková et al., 2013; Lahtela and Kärki, 2014).

On compressive strength parallel to the grain, the results on this study demonstrated two groups of values considering Tukey test result, with 185 °C temperature reducing significantly fc0, with a maximum reduction of 80% when compared with control results. For E_{c0}, thermally treated the results were equivalent according Tukey test, with a maximum reduction of 49%. In general, for mechanical properties, such severe worsening after thermal treatment may be explained by degree reduction of celulose crystallinity after more than 2 hrs, thermal treatment temperature above 170 °C and a great degradation of hemicellulose, reducing material elasticity (Bhuiyan et al., 2000; Čabalová et al., 2018; Cheng et al., 2017; Durmaz et al., 2019; Severo et al., 2012; Silva et al., 2013).

For thermally modified *Eucalyptus grandis*, Calonego et al. (2012) found f_{c0} values ranging between 39.8 MPa (control) and 41.5 MPa (160 °C) (CV between 7.5 and 9.7%); for E_{c0}, values varying between 12.8 GPa (control) and 11.9 GPa (180 °C) (CV varying between 13.2 and 25.2%); Compressive strength parallel to the grain increased with thermal treatment, differently from what occurred on this study and modulus of elasticity on compression parallel to the grain presented a maximum reduction of 7%, seven times lower than the obtained on this research.

For modulus of resistance (f_{M0}) and modulus of elasticity (E_{M0}) on static bending test for thermally treated *Eucalyptus grandis*, Cademartori et al. (2012) found f_{M0} ranged between 63.2 MPa (180 °C) and 82.77 MPa (control) (CV varying between 14.6 and 17.8%). For E_{M0}, 11.2 GPa (180 °C) and 11.5 GPa (control) (CV ranging between 15 and 18%). Moreover, Calonego et al. (2012) encountered values for thermally treated *Eucalyptus grandis* for

f_{M0} ranging between 52.5 MPa (180 °C) and 69 MPa (control) (CV varying between 5 and 24 %); for E_{M0}, 10.7 GPa (control) and 11.3 GPa (160 °C) (CV varying between 5.5 and 11.2%).

In this research, the maximum reduction on conventional strength on static bending test was 82% when comparing with value control, much higher than Calonego et al. (2012) and Cademartori et al. (2012) (decrease of 24% on both studies). For conventional modulus of elasticity on static bending test, the maximum reduction was 71%, while on Cademartori et al. (2012) the decrease on E_{M0} was 2% and on Calonego et al. (2012), there was an maximum increase of 5.6%.

Extractives content increased 374% comparing control and 185 °C treatments, with an increase being equivalent with thermal temperature raise. For thermally treated *Eucalyptus grandis*, Zanuncio et al. (2014) found similar results, but a minor increase on extractives content, rising from 6.0% (control) to 6.8% (200 °C), an increment of 13%. On the study of Moura et al. (2012b), the increase on extractive content was 27%, expanding from 7.8% (control) to 10% (180 °C). Batista et al. (2016a) encountered extractive content ranging between 2.2% (control) and 15.85% (180 °C), an increase of 613%.

On total lignin content, the results on this study demonstrated a proportional increase with thermal treatment temperature until 165 °C, being a maximum rise of 31% when comparing with control and then, a progressive reduction with temperature rise, reaching an increase of 15% to 185 °C temperature results with control results. This increase on lignin content may be explained by the thermal decomposition of carbohydrates, hemicellulose degradation and condensation reactions due to hemicellulose depolymerization (Čabalová et al., 2018; Kačíková et al., 2013; Yildiz and Gümüşkaya, 2007).

Zanuncio et al. (2014) reached different results for total content, with 28.8% (control), 29.3% (170 °C) and 30.4% (200 °C), an increase of 5% at 200 °C temperature when compared with control *Eucalyptus grandis*. Moreover, for thermally treated *Eucalyptus grandis*, Moura et. al. (2012b) found progressive increments on total lignin content with proportional increases on treatment temperature, varying from 31.9% (control) to 35.2% (180 °C), a variation of 10%.

For holocellulose content, there was a great reduction from control content, with 65,18% and 185 °C temperature, with 38,12%, being a decrease of 41%. Zanuncio et al. (2014) encountered a minor reduction of 2%, ranging from 65.2% (control) to 63.9% (200 °C). Moura et al. (2012b) also found similar results to treated *Eucalyptus grandis*, with a reduction of 9%, varying from 60.22% (control) to 54.9% (180 °C). Batista et al. (2016a) met a reduction of 24%, ranging from 69.4% (control) to 52.8% (180 °C) for thermally treated *Eucalyptus grandis*. Such behavior of holocellulose content may be major explained by hemicellulose degradation, due to low amount of cellulose that can be degraded at temperatures below 200 °C (Tjeerdsma and Militz, 2005).

From Table 3, it is important to point out that regression models were generated based on mean values for each thermal treatment temperature, and for control. The precision (measured by coefficient of determination – R^2) and significance remained the same of the models disposed on Table 3, fact that can be explained by the minor temperature of thermal treatment is seven times more than the temperature considered on condition control.

All regression models were considered significant by ANOVA. Considering the precision on adjustment, nine models (E_{c0}, f_{c0}, E_{M0}, f_{M0}, f_{H0}, f_{H90}, f_{bw}, Extractives, Soluble lignin and Holocellulose) disposed R^2 above 70 % (Montgomery, 2012), indicating good precision on adjustment, being possible to estimate such properties for intermediate values of temperature not explored. Kačiková et al. (2013) developed the only one study in the literature estimating wood physical, chemical and mechanical properties in function of thermal treatment temperature. This study showed the difference on color properties, hemicellulose, holocellulose, lignin, extractives, degree of polymerization of cellulose, total crystallinity index of cellulose, f_{M0} and E_{M0} properties with temperature using exponential regression models, differently of the present research, which used linear models, with elevated R^2, ranging between 72 % and 99 %, indicating the possibility of their use for estimate such properties on Norway spruce wood.

According to the statistical analysis carried out, *Eucalyptus grandis* wood, which was heat-treated by the process described in this study, it showed a significant reduction in mechanical properties by gradually increasing the treatment temperature. As reported by several authors, the loss of mechanical strength is the greatest disadvantage observed in heat-treated wood, which impairs its use in applications that demand greater wood strength (Hill, 2006; Moura et al., 2012a).

On the other hand, the heat treatment gave the wood a darker color, making it more attractive to the market and adding value to the final product (Esteves et al., 2008; Conte et al., 2014). Despite the impossibility of use in applications that demand greater mechanical resistance, the heat-treated wood has great decorative potential and can be used in other applications where there is less stress on the wood.

With the generated regression models, it is possible to estimate the properties of *Eucalyptus grandis* wood heat-treated for intermediate temperatures not explored experimentally, making it possible to find the ideal treatment temperature to result in a heat-treated wood with the best set of mechanical properties desirable for a particular use.

5. CONCLUSIONS

The heat treatment significantly influenced the physical, chemical, and mechanical properties of *Eucalyptus grandis*. The mechanical properties were reduced, being mainly affected by the modifications of the chemical components of the wood;

The reduction in the mechanical properties of heat-treated wood makes it impossible to use it in applications that demand greater resistance; the attribution of color adds value to the wood market and makes it possible to use it mainly in decorative applications;

The regression models were significant and some showed good quality in the adjustment, making it possible to estimate the properties of the wood as a function of the treatment temperature without the need for experimental studies.

6. AUTHOR CONTRIBUTIONS

Carolina A. B. Oliveira and Julio C. Molina: Study conception and design; Carolina A. B. Oliveira and Karina A. Oliveira: Material preparation and data collection. Vinicius B. M. Aquino and André L. Christoforo: Data analyze. Carolina A. B. Oliveira and Vinicius B. M. Aquino: Text written and translation. All authors read and approved the final manuscript.

7. REFERENCES

Associação Brasileira de Normas Técnicas – ABNT. NBR 7190: Projeto de estruturas de madeira. Rio de Janeiro: 1997.

Ahmadi M, Moezzipour B, Moezzipour A. Thermal stability of wood fibers produced from recycled medium density fiberboards. Drvna Industrija. 2019;70(2):149–55. doi:10.5552/drvind.2019.1833

Almeida AS, Criscuolo G, Almeida TH, Christoforo AL, Chahud E, Branco LAMN et al. Influence of CCA-A preservative on physical-mechanical properties of Brazilian tropical woods. BioResources. 2019;14(2):3030–41. doi:10.15376/biores.14.2.3030-3041

Bal BC, Bektaş I. The effects of heat treatment on some mechanical properties of juvenile wood and mature wood of *Eucalyptus grandis*. BioResources. 2012;7(2):5117–27. doi:10.15376/biores.7.4.5117-5127

Barboutis I, Kamperido V. Impact of heat treatment on the quality of tree-of-heaven wood. Drvna Industrija. 2019;70(4):351–58. doi:10.5552/drvind.2019.1842

Batista DC, Muniz GIB, Oliveira JTS, Paes JB, Nisgoski S. Effect of the Brazilian thermal modification process on the chemical composition of *Eucalyptus grandis* juvenile wood - part 1: Cell wall polymers and extractives contents. Maderas: Ciencia y Tecnologia. 2016a;18(2):273–84. doi:10.4067/S0718-221X2016005000025

Batista DC, Nisgoski S, Oliveira JTS, Muñiz GIB, Paes JB. Resistance of thermally modified *Eucalyptus grandis* W. Hill ex Maiden wood to deterioration by dry-wood termites (*Cryptotermes* sp.). Ciência Florestal. 2016b;26(2):671–78. doi:10.5902/1980509822766

Batista DC, Oliveira JTS, Paes JB, Nisgoski S, Muñiz GIB. Effect of the Brazilian process of thermal modification on the physical properties of *Eucalyptus grandis* juvenile wood. Maderas: Ciencia y Tecnologia. 2018;20(4):715–24. doi:10.4067/

S0718-221X2018005041701

Baysal E, Degirmentepe S, Simsek H. Some surface properties of thermally modified scots pine after artificial weathering. Maderas: Ciencia y Tecnologia. 2014;16(3):355–364. doi:10.4067/S0718-221X2014005000028

Bhuiyan MTR, Hirai N, Sobue N. Changes of crystallinity in wood cellulose by heat treatment under dried and moist conditions. Journal of Wood Science. 2000;46(6):431–436. doi:10.1007/BF00765800

Čabalová I, Kacík F, Lagaňa R, Výbohová E, Bubeníková T, Čaňová I, et al. Effect of thermal treatment on the chemical, physical, and mechanical properties of pedunculate oak (*Quercus robur* L.) wood. BioResources. 2018;13(1):157–170. doi:10.15376/biores.13.1.157-170

Cademartori PHG, Schneid E, Gatto DA, Beltrame R, Stangerlin DM. Modification of static bending strength properties of *Eucalyptus grandis* heat-treated wood. Materials Research. 2012;15(6):922–927. doi:10.1590/S1516-14392012005000136

Cademartori PH, Schneid E, Gatto DA, Martins Stangerlin D, Beltrame R. Thermal modification of *Eucalyptus grandis* wood: variation of colorimetric parameters. Maderas Ciencia y Tecnología. 2013;15(1):57–64. doi:10.4067/S0718-221X2013005000005

Calonego FW, Severo ETD, Ballarin AW. Physical and mechanical properties of thermally modified wood from *E. grandis*. European Journal of Wood and Wood Products. 2012;70(4):453–460. doi:10.1007/s00107-011-0568-5

Chen C, Wang W, Cao J, Qi Q, Ma W. Properties of thermally modified southern pine wood pretreated with alkylalkoxysilanes. BioResources. 2016;11(2):5285–98. doi:10.15376/biores.11.2.5285-5298

Cheng XY, Li XJ, Xu K, Huang QT, Sun HN, Wu YQ. Effect of thermal treatment on functional groups and degree of cellulose crystallinity of eucalyptus wood (*Eucalyptus grandis* × *Eucalyptus urophylla*). Forest Products Journal. 2017;67(1-2):135–140. doi:10.13073/FPJ-D-15-00075

Christoforo AL, Couto NG, Almeida JPB, Aquino VBM, Lahr FAR. Apparent density as an estimator of wood properties obtained in tests where failure is fragile. Engenharia Agrícola. 2020;40(1):105–112. doi:10.1590/1809-4430-eng.agric.v40n1p105-112/2020

Conte B, Missio AL, Pertuzzatti A, Cademrtori PHG, Gatto DA. Physical and colorimetric properties of *Pinus elliottii* var. *elliottii* thermally treated wood. Scientia Forestalis. 2014; 42(104): 555-563.

Durmaz E, Ucuncu T, Karamanoglu M, Kaymakci A. Effects of heat treatment on some characteristics of Scots pine (*Pinus sylvestris* L.) wood. BioResources. 2019;14(4):9531–43. doi: 10.15376/biores.14.4.9531-43

Esteves B, Graça J, Pereira H. Extractive composition and summative chemical analysis of thermally treated eucalypt wood. Holzforschung. 2008;62(3):344-351. doi:10.1515/HF.2008.057

Gašparík M, Gaff M, Kačík F, Sikora A. Color and chemical changes in teak (*Tectona grandis* L. f.) and meranti (*Shorea* spp.) wood after thermal treatment. BioResources. 2019;14(2):2667–83. doi: 10.15376/biores.14.2.2667-2683

Gomide JL, Memuner BJ. Determinação do teor de lignina em material lenhoso: método Klason modificado. O Papel. 1986;47(8):36–38.

Gurleyen L, Ayata U, Esteves B, Gurleyen T, Cakicier N. Effects of thermal modification of oak wood upon selected properties of coating systems. BioResources. 2019;14(1):1838-49. doi:10.15376/biores.14.1.1838-1849

Herrera-Díaz R, Sepúlveda-Villarroel V, Torres-Mella J, Salvo-Sepúlveda L, Llano-Ponte R, Salinas-Lira C, et al. Ananías et al. Influence of the wood quality and treatment temperature on the physical and mechanical properties of thermally modified radiata pine. European Journal of Wood and Wood Products. 2019;77(4):661–671. doi:10.1007/s00107-019-01424-9

Herzog B, Goodell B, Lopez-Anido R, Muszynski L, Gardner D, Tascioglu C. Effect of creosote and copper naphthenate preservative treatments on properties of FRP composite materials used for wood reinforcement. Journal of Advanced Materials. 2004;36(4):25–33.

Hill CAS. Wood modification: chemical, thermal and other processes. Chichester: John Wiley & Sons, 2006. ISBN: 978-0-470-02172-9.

International Thermowood Association. Thermowood® Handbook. 2003. [cited 2020 October 15]. Available from: https://asiakas.kotisivukone.com/files/en.thermowood.palvelee.fi/downloads/tw_handbook_080813.pdf.

Jirouš-Rajković V, Miklečić J. Heat-treated wood as a substrate for coatings, weathering of heat-treated wood, and coating performance on heat-treated wood. Advances in Materials Science and Engineering. 2019;2019:1–9. doi:10.1155/2019/8621486

Kačíková D, Kačík F, Čabalová I, Ďurkovič J. Effects of thermal treatment on chemical, mechanical and colour traits in Norway spruce wood. Bioresource Technology. 2013;144:669–674. doi:10.1016/j.biortech.2013.06.110

Kariz M, Kuzman MK, Sernek M, Hughes M, Rautkari L, Kamke FA, et al. Influence of temperature of thermal treatment on surface densification of spruce. European Journal of Wood and Wood Products. 2016;75(1):113–123. doi:10.1007/s00107-016-1052-z

Kubovský I, Kačíková D, Kačík F. Structural changes of oak wood main components caused by thermal modification. Polymers. 2020;12(2):485-496. doi:10.3390/polym12020485

Lahtela V, Kärki T. Effects of impregnation and heat treatment on the physical and mechanical properties of Scots pine (*Pinus sylvestris*) wood. Wood Material Science and Engineering. 2014;11(4):217–227. doi:10.1080/17480272.2014.971428

Lazarotto M, Cava SS, Beltrame R, Gatto DA, Missio AL, Gomes LG, et al. Biological resistance and colorimetry of heat treated wood of two eucalyptus species. Revista Arvore. 2016;40(1):135–145. doi:10.1590/0100-67622016000100015

Lee SH, Ashaari Z, Lum WC, Halip JA, Ang AF, Tan LP et al. Thermal treatment of wood using vegetable oils: a review. Construction and Building Materials. 2018;181(2018):408–419. doi:10.1016/j.conbuildmat.2018.06.058

Modes KS, Santini EJ, Vivian MA. Hygroscopicity of wood from *Eucalyptus grandis* and *Pinus taeda* subjected to thermal treatment. Cerne. 2013;19(1):19–25. doi: 10.1590/S0104-77602013000100003

Montgomery DC. Design and analysis of experiments. Nova Jersey: John Wiley & Sons; 2012. ISBN 978-1118-14692-7.

Moura LF, Brito JO. Effect of thermal rectification on colorimetric properties of *Eucalyptus grandis* and *Pinus caribaea* var. *hondurensis* woods. Scientia Forestalis. 2011;39(89):69–76.

Moura LF, Brito JO, Bortoletto Júnior G. Efeitos da termorretificação na perda de massa e propriedades mecânicas de *Eucalyptus grandis* e *Pinus caribaea* var. *hondurensis*. Floresta. 2012a;42(2):305–314. doi:10.5380/rf.v42i2.17635

Moura LF, Brito JO, Silva Junior FG. Effect of thermal treatment on the chemical characteristics of wood from *Eucalyptus grandis* W. Hill ex Maiden under different Atmospheric Conditions. Cerne. 2012b;18(3):449–455. doi: 10.1590/S0104-77602012000300012

Pertuzzatti A, Missio AL, Cademartori PHG, et al. Effect of process parameters in the thermomechanical densification of *Pinus elliottii* and *Eucalyptus grandis* fast-growing wood. BioResources. 2018;13(1):1576–90. doi:10.15376/biores.13.1.1576-1590

Platowood. The Platowood® process. 2020. [cited 2020 October 16]. Available from: https://www.platowood.com/upload_directory/uploads/the-platowood-process.pdf

Ribeiro DP, Vilela AP, Silva DW, Napoli A, Mendes RF. Effect of heat treatment on the properties of sugarcane bagasse medium density particleboard (MDP) panels. Waste and Biomass Valorization. 2019; 11:6429–41. doi:10.1007/s12649-019-00882-9

Severo ETD, Calonego FW, Sansígolo CA. Physical and chemical changes in juvenile and mature woods of *Pinus elliottii* var. elliottii by thermal modification. European Journal of Wood and Wood Products. 2012;70(5):741–747. doi:10.1007/s00107-012-0611-1

Sikora A, Kačík F, Gaff M, Vondrová V, Bubeníková

T, Kubovský I. Impact of thermal modification on color and chemical changes of spruce and oak wood. Journal of Wood Science. 2018;64(4):406–416. doi:10.1007/s10086-018-1721-0

Silva MR da, Machado GDO, Brito JO, Junior CC. Strength and stiffness of thermally rectified eucalyptus wood under compression. Materials Research 2013;16(5):1077–83. doi:10.1590/S1516-14392013005000086

Soltani A, Hosseinpourpia R, Adamopoulos S, Taghiyari HR, Ghaffari E. Effects of heat-treatment and nano-wollastonite impregnation on fire properties of solid wood. BioResources. 2016;11(4):8953–67. doi:10.15376/biores.11.4.8953-8967

TAPPI Standards - TAPPI. Test methods: T 264 cm- 97: Preparation of wood for chemical analysis. Atlanta, USA: 1997.

TAPPI Standards - TAPPI. Test methods: T 257 cm-85: Sampling and preparing wood for analysis. Atlanta, USA: 1985.

Tjeerdsma BF, Militz H. Chemical changes in hydrothermal treated wood: FTIR analysis of combined hydrothermal and dry heat-treated wood. Holz Als Roh - Und Werkstoff. 2005;63(2):102–111. doi:10.1007/s00107-004-0532-8

Wikberg H, Maunu SL. Characterisation of thermally modified hard- and softwoods by13C CPMAS NMR. Carbohydrate Polymers. 2004;58(4):461–466. doi:10.1016/j.carbpol.2004.08.008

Yildiz S, Gümüşkaya E. The effects of thermal modification on crystalline structure of cellulose in soft and hardwood. Building and Environment. 2007;42(1):62–67. doi:10.1016/j.buildenv.2005.07.009

Zanuncio AJV, Nobre JRC, Motta JP, Trugilho PF. Química e colorimetria da madeira de *Eucalyptus grandis* Hill ex Maiden termorretificada. Revista Árvore. 2014;38(4):765–770. doi:10.1590/S0100-67622014000400020