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Effect of thermal treatment on the physical properties of GG100 clone *Eucalyptus* wood

Felipe Gomes Batista[1], Dayane Targino de Medeiro[1], Adriano Reis Prazeres Mascarenhas[1], Lourival Marin Mendes[1], Danilo Wisky Silva[1], Daniel Tavares de Farias[2], Alexandre Santos Pimenta[3], Francisco Rodolfo Junior[4], Edgley Alves de Oliveira Paula[5], Rafael Rodolfo de Melo[5]

[1]Universidade Federal de Lavras. Aquenta Sol, Caixa Postal 3037, 37200-900, Lavras, MG, Brasil.

[2]Universidade Federal de Santa Maria, Centro de Ciências Rurais, Departamento de Engenharia Florestal. Prédio 42, Av. Roraima, nº 1000, Bairro Camobi, 97.105-900, Santa Maria, RS, Brasil.

[3]Universidade Federal do Rio Grande do Norte, Escola Agrícola de Jundiaí. RN 160, Km 03, 59.280-000, Macaíba, RN, Brasil.

[4]Universidade Federal do Piauí. Campus Professora Cinobelina Elvas, Rodovia Bom Jesus, Viana, Km 01, Planalto Horizonte, 64.900-000, Bom Jesus, PI, Brasil.

[5]Universidade Federal do Semiárido, Departamento de Ciências Agronômicas e Florestais. Av. Francisco Mota, 572, Costa e Silva, 59.625-900, Mossoró, RN, Brasil.

e-mail: felipejp.gomes@gmail.com, dayanemedeirost@gmail.com, adriano.mascarenhas@unir.br, rafael.melo@ufersa.edu.br, lourival@ufla.br, danilowisky@gmail.com, daniel.bky@gmail.com, alexandre_spimenta@hotmail.com, rodolfo@ufpi.br, edgley.22@gmail.com, rafael.melo@ufersa.edu.br

ABSTRACT

Thermal treatment can modify undesirable characteristics of some woods, dimensional instability, and excessive water adsorption. The present research work aimed to assess the effect of thermal treatment on the physical properties of clone GG100 *Eucalyptus* wood. Wood samples were obtained from 9 year-old trees. Samples were subjected to three types of thermal treatment (T1, T2, and T3). In T1, the wood was kept in a laboratory kiln at 100 °C for 180 min. In T2, hydrothermal treatment was performed in an autoclave at 120 °C for 120 min. In T3, the combined autoclave and laboratory kiln treatments were employed. The properties evaluated were apparent density (ρa); mass loss (ML); longitudinal (βl), radial (βr), tangential (βt), and volumetric shrinkage (βv); anisotropic factor; and equilibrium moisture content. The treatments caused a decrease of ρa and increase of ML. The Equilibrium Moisture Content (EMC) was lowest for T3, and had a negative correlation with ML, βt, and Δv. The Pearson correlation analysis indicated the results of the thermal treatments were similar, but T1 presented the lowest variation of the physical properties compared to the other treatments, giving it the best predictability for practical uses.

Keywords: Dimensional stability; Thermal rectification; Wood moisture.

1. INTRODUCTION

In the timber market, it is increasingly common to replace tropical wood from native species with wood from fast-growing forest plantations, mainly of the genus Eucalyptus. Whose planted area has increased greatly due to its adaptability, high productivity, and the possibility of multiple uses, such as pulp, charcoal, panels, among others [1–3]. However, fast-growing species usually produce wood with high quantities of juvenile wood, elevated microfibrillar angle, high variability of density, great dimensional instability, low mechanical strength, and susceptibility to warping and cracks, which can make use for furniture and civil construction difficult, among other issues [4–7].

The employment of processes able to modify the wood structure can be successful to improve wood properties. Among the possibilities, thermal treatments can be highlighted. During them, the wood is subjected to high temperatures for given periods [8, 9]. Thermal treatment is also known as thermal rectification and as the potential to improve the performance of wood-based products from several standpoints [10].

Thermal treatments decrease wood's hygroscopicity due to the degradation of the hemicelluloses that occurs more intensely than to other wood components due to their structural heterogeneity, presence of

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amorphous regions, and low molecular mass [11]. When properly carried out, the treatments also cause higher dimensional stability and resistance to decay [12, 13]. The wood color is also altered, leaving it with darker and more uniform tones [14]. Finally, thermal treatments are environmentally friendly, since they do not require the use of chemical products, and the energy demand in the process is low [15].

To make the thermal treatments of wood and its products effective, it is necessary to find the proper combination of temperature and exposure time, as a function of the wood species and type of final use, whether structural or merely aesthetic [16, 17]. Other variables important in the process are the atmosphere of treatment, type of system (open or closed), and wood dimensions [18]. Considering those aspects, we aimed to assess the effects of different thermal treatments on the physical properties of the wood of the GG100 clone (*Eucalyptus urophylla x Eucalyptus grandis* hybrid), mainly the changes in apparent density and dimensional stability after the treatments.

2. MATERIAL AND METHODS

2.1. Sample collection and thermal treatments

For the experimental procedures, the GG100 clone (Eucalyptus urophylla x Eucalyptus grandis hybrid) was employed. Ten trees were harvested from an experimental plot (9 years old) located at the municipality of Macaíba, state of Rio Grande do Norte, Brazil (5° 51' 36" S, 35° 20' 59" W, and altitude of 15 m). Trees were grown with a spacing of 3.0 m × 2.0 m. Four short logs were sawn from each harvested trunk and 10 test specimens (3 cm × 2 cm × 5 cm) were collected from each one of them obtaining always wood samples free of knots, cracks, grain slope and other defects, according to the procedures recommended by the standard NBR 7190 [19].

The test specimens were conditioned in a climatic chamber kept at 65% relative humidity and 25 °C, until they reached the equilibrium moisture content of 12%. Then the test specimens' weight and dimensions (radial, tangential and longitudinal directions) were measured.

The experiment was conducted according to an entirely randomized design, considering three thermal treatments compared to a control (untreated wood at 12% moisture content). Test specimens were subjected to three types of thermal treatment (T1, T2, and T3). In T1, the wood was kept in a laboratory kiln at 100 °C for 180 min; in T2, the wood was subjected to hydrothermal treatment in an autoclave at 120 °C for 120 min; and in T3, the wood was subjected to both autoclave and laboratory kiln treatment, with 120 °C for 120 min followed by 100 °C for 180 min. For each experimental condition and the control treatment, 40 test specimens were employed. Figure 1 displays the experimental conditions employed in the present study.

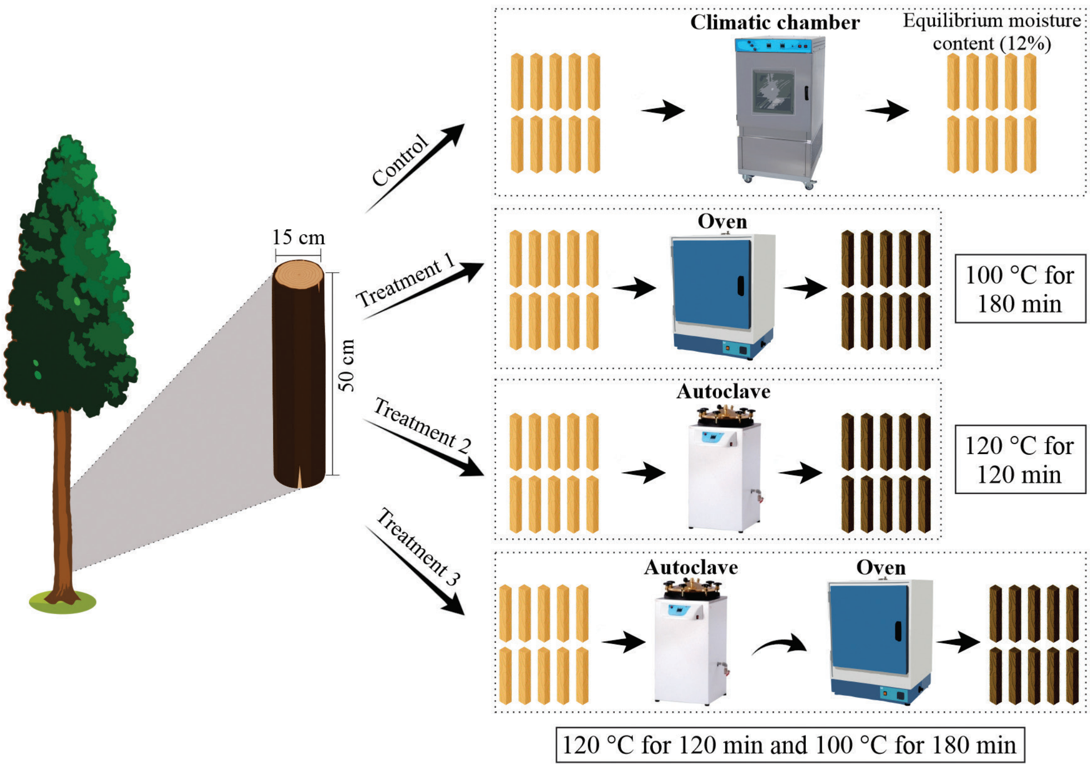

Figure 1: General scheme of thermal treatments applied to the GG100 clone.

2.2. Sample collection and thermal treatments

All the physical properties were determined by following the procedures described in the standard NBR 7190 [19]. Except for the control, the apparent density was calculated after the thermal treatment, according to Equation 1.

$$\rho a = \frac{m_{eq}}{V_{eq}} \quad (1)$$

Where: ρa = apparent density (g cm^{-3}); m_{eq} = test specimen weight at the equilibrium moisture content (g); v_{eq} = test specimen volume at the equilibrium moisture content (cm³).

Also, the mass loss was determined as the ratio between the difference of weights before and after the thermal treatment. To characterize the dimensional stability, the linear (longitudinal, radial and tangential) and volumetric shrinkage were determined through Equations 2 and 3, respectively. The anisotropy factor (fa) was obtained as the ratio between tangential and radial shrinkage.

$$\beta l,r,t = \left(\frac{d_{l,r,t_{(28)\%}} - d_{l,r,t_{(0\%)}}}{d_{l,r,t_{(28)\%}}} \right) \times 100 \quad (2)$$

$$\Delta v = \left(\frac{v_{28\%} - v_{0\%}}{v_{28\%}} \right) \times 100 \quad (3)$$

Where: $\beta l,r,t$ = longitudinal, radial or tangential shrinkage (%); $d_{l,t,r(28\%)}$ = longitudinal, radial or tangential dimension after water saturation (cm); $d_{l,r,t(0\%)}$ = longitudinal, radial or tangential dimension in dry condition (cm); **Δv** = volumetric shrinkage (%); $v_{28\%}$ = volume after water saturation (cm³); $v_{0\%}$ = volume in dry condition (cm³).

The equilibrium moisture content of each experimental treatment was determined according to the procedures described by standard NBR 11941 [20]. For this, the test specimens were kept in a climatic chamber at 65% relative humidity and 25 °C until reaching constant weight, defined when the difference between two successive weightings did not exceed 0.5 g.

2.3. Statistical analyses

Experimental data were subjected to the Fisher test at 95% probability, and when statistical differences between means were observed, the Scott-Knott test at 95% probability was applied. Besides this, principal component analysis (PCA) was employed to group the experimental treatments in clusters according to the variation of their main characteristics. This analysis was employed with the maximum number of 8 principal components. To verify the existence of correlations between the physical properties of the thermally treated wood, applying Pearson correlation at 95 and 99% probability, which was analyzed by plotting a correlogram.

3. RESULTS

For apparent density (ρa), significant statistical differences were detected between the thermal treatments and the control (Figure 2). Regardless of the thermal treatment applied, a decrease of 2.6% of this property was

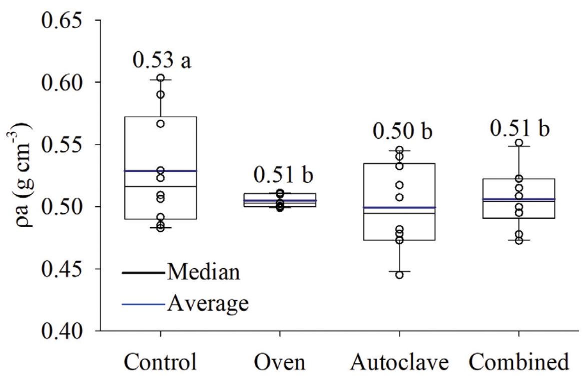

Figure 2: Values of apparent density (ρa) for the GG100 clone wood before (control) and after different thermal treatments (T1, T2, and T3).

*Values above the box-plots indicate that means followed by the same letters do not differ from each other by the Scott-Knott test at 95% probability.

detected compared to the control. Although not significantly differing from the other experimental treatments, T1 resulted in the lowest heterogeneity of the apparent density values. For T2 and T3, high variability above and below the mean, both with positive asymmetry (mean > median), was observed.

The lowest ML values were observed for the wood subjected to T3, which combined thermal treatments in an autoclave and a laboratory kiln (Figure 3). However, the treatments T1 and T2, in which the thermal treatments with laboratory kiln and autoclave were applied alone, did not differ from each other and resulted in the highest values of ML.

Concerning the dimensional stability (Table 1), the standard deviations were low for all experimental treatments. The lowest values of βr and Δv were achieved with T3 (hydrothermal followed by thermal treatment). Regarding βt, all experimental treatments brought a decrease in values in comparison with the control, while for βl and fa, no significant effects were observed.

The lowest value of EMC was observed for the wood subjected to T3 compared to the control (Figure 4). However, T1 did not differ statistically from T2, although both resulted in a decrease of 20% of that property compared to the control treatment. With respect to the variability of the experimental data, the same trend was observed for ρa, i.e., T1 presented the lowest dispersion of values. In the dataset obtained for wood subjected to the hydrothermal treatment, negative asymmetry of that property was observed (mean < median), while for T3, the asymmetry was positive.

According to the perceptual map displayed in Figure 5, the first principal component (PC1) explained 50.9% of the variability of the experimental data, while 20.4% was explained by the second component (PC2). Thus, the dimensional perceptual map was adequate to assess the relationship between the studied variables, considering that both components together explained 71.3% of the variance.

The eigenvalues of the control samples are grouped on the right of the perceptual map. The eigenvalues obtained for T1, T2, and T3 are grouped to the left with overlaps between groups. The eigenvalues determined for T1 and T2 presented less dispersion compared to the control and T3. Based on the directions of the eigenvectors, the studied parameters varied for all treatments in the multivariate space. All physical properties tended to have lower values regardless of the thermal treatment, except for ML. In Figure 6, these results can be observed more clearly. A highlight needs to be made for the characteristics ML and Δv, βt, EMC, where a strong negative

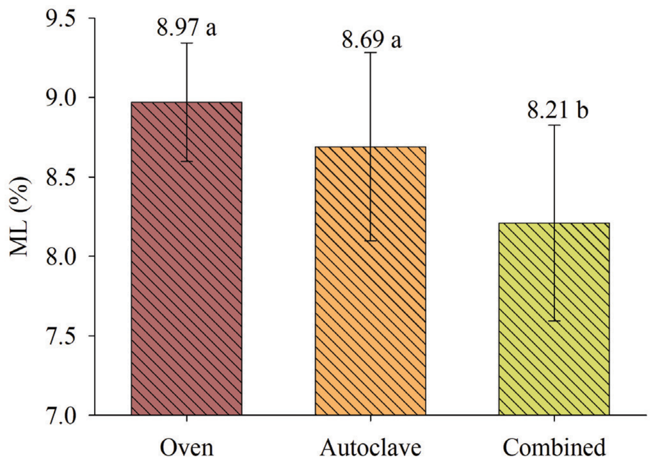

Figure 3: Mass loss (ML) of the GG100 clone wood before (control) and after different thermal treatments (T1, T2, and T3). *Values above the bars indicate that means followed by the same letters do not differ from each other by the Scott-Knott test at 95% probability.

Table 1: Values of longitudinal (βl), radial (βr), tangential (βt), and volumetric (Δv) shrinkages; and anisotropy factor (fa) determined for the GG100 clone wood before (control) and after different thermal treatments T1, T2, and T3.

TREATMENT	βl (%)	βr (%)	βt (%)	Δv (%)	fa
Control	0.29 ± 0.11a	5.16 ± 0.79a	7.90 ± 1.05a	12.90 ± 1.23a	1.57 ± 0.34a
T1	0.24 ± 0.15a	4.47 ± 0.81b	5.88 ± 0.97b	10.31 ± 0.94b	1.32 ± 0.35a
T2	0.23 ± 0.15a	3.84 ± 0.60c	5.28 ± 1.23b	9.12 ± 1.49c	1.39 ± 0.33a
T3	0.24 ± 0.19a	3.62 ± 0.73c	5.46 ± 0.76b	9.10 ± 1.27c	1.58 ± 0.17a

Means followed by the same letters in the columns do not differ from each other by the Scott-Knott test at 95% probability.

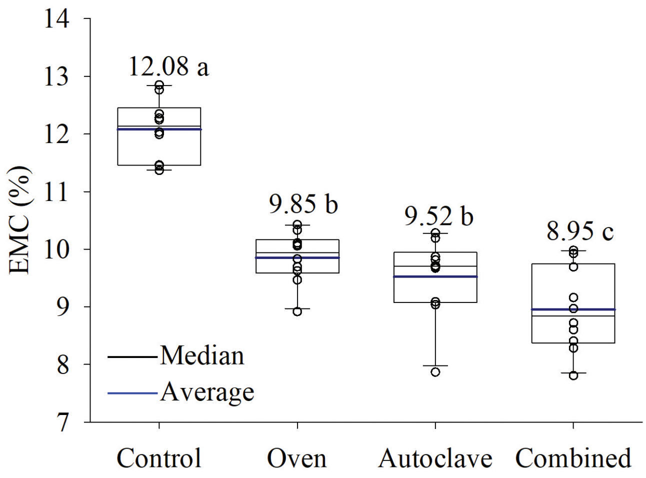

Figure 4: Equilibrium moisture values (EMC) for the GG100 clone wood before (control) and after different heat treatments T1, T2, and T3.
*Values above the box-plots indicate that means followed by the same letter do not differ from each other by the Scott-Knott test at 95% probability.

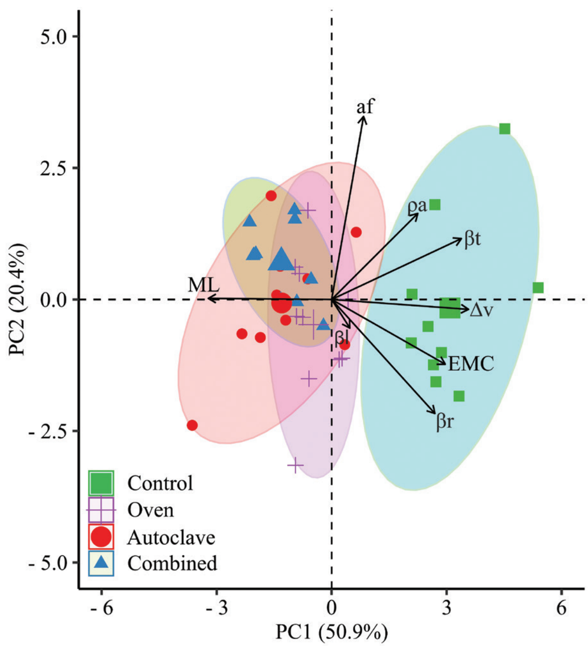

Figure 5: Perceptual map with the results of the principal component analysis (PCA) for apparent density (ρa); mass loss (ML); longitudinal (βl), radial (βr), tangential (βt), and volumetric (Δv) shrinkages; anisotropy (fa); and equilibrium moisture content (EMC); and grouping of physical properties before (control) and after treatments (T1, T2, and T3) applied to GG100 clone wood.

correlation could be observed. Similarly, Δv, βt, and βr were negatively correlated with EMC. When PCA was associated with Pearson correlation analysis, greater intensity of ML caused by thermal treatments is associated with lower values of EMC and higher dimensional stability, mainly concerning βt and Δv.

4. DISCUSSION

As observed in the present study, the thermal treatments applied to the GG100 clone wood caused significant changes. As a result of T1, the most intense and predictable change was related to ρa. Furthermore, according to the classification proposed by RUFFINATTO *et al.* [21], the wood after T1 remained in the intermediate density class (0.40 to 0.75 g cm^{-3}). This result can be considered positive for thermal treatment, since several studies have indicated broad variation in the apparent density of the wood as a function of the high heterogeneity of this

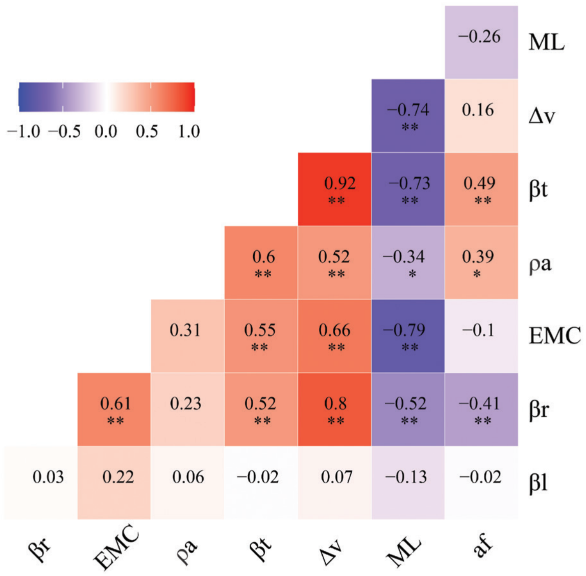

Figure 6: Correlogram from the Pearson correlation analysis of the physical properties, as follows: apparent density (ρa); mass loss (ML); longitudinal (βl), radial (βr), tangential (βt), and volumetric (Δv) shrinkages; anisotropy factor (fa); and equilibrium moisture content (EMC) obtained for the GG100 clone wood after different heat treatments.

*Values in decimals indicate Pearson's correlation coefficient; * = significant at 95% probability; ** = significant at 99% probability.

aspect. It is well known that density is affected by growth conditions, age and cell wall thickness [22, 23], so the possibility of negative effects caused by thermal treatment sometimes tends to be unpredictable.

The aspects discussed above can be related to the high dispersion of the values of ρa observed in treatments T1 and T2. Since the test specimens were collected in different axial and radial positions along the tree trunks, the occurrence is natural of variations in the proportion of juvenile and mature wood, and thus in the chemical composition of the samples, as observed by BILLARD *et al.* [24] and MASCARENHAS *et al.* [25]. In other words, more aggressive thermal treatments can intensify undesirable changes in apparent density as a function of the position from which the samples are collected, which did not occur in the present work. To cite an example to support this statement, ARANTES *et al.* [26] reported that the lignin and extractives contents of *Eucalyptus urophylla* x *Eucalyptus grandis* decreased in the pith-bark and base-top directions. So, the bonds between hemicelluloses and lignin and cellulose and lignin can be broken more easily in positions with low lignin contents and vice versa, which can negatively affect the density when the wood is thermally treated [27]. Another issue to be considered is that hemicelluloses and extractives undergo degradation at high temperatures, which can result in a greater mass loss [28]. Also, MELO *et al.* [29] reported that the mass loss that occurs during thermal treatments is a characteristic effect that can be around 20%, depending on the temperature, duration of the process and the forest species.

Other researchers have presented results similar to those in the present work. For instance, CADEMARTORI *et al.* [30], when assessing the effect of thermal treatment on two species of eucalyptus, found that hydrothermal treatment in an autoclave induced ML and decreased the EMC of the studied species, while in the laboratory kiln treatment, the changes in the dimensional stability values were higher. When studying the influence of thermal treatments on the wood of different clones of *Eucalyptus urophylla* x *Eucalyptus grandis*, JUIZO *et al.* [31] obtained ML varying from 17 to 22% employing a temperature of 200 °C for 4 h in a laboratory kiln. According to CZAJKOWSKI *et al.* [32], ML is due to the thermal degradation of cellulose, hemicelluloses and lignin, as well as the volatilization of some extractives.

In the present study, the benefits of thermal treatments for the reduction of wood hygroscopicity were clear. Similarly, FERREIRA *et al.* [33] also found the same effect on *Hymenolobium petraeum* ('angelim-pedra') wood, where the EMC decreased by 23% after thermal treatment with a temperature of 180 °C for 2 h. In the same context, ESTEVES and PEREIRA [10] reported that thermal treatments with milder temperatures (around 100 °C, as in this work), can permanently decrease the amount of water absorbed by the wood. To explain this observation, TJEERDSMA *et al.* [34] and HILL *et al.* [35] indicated that reduction in the content of hydroxyls (–OH) is responsible. However, it depends on the sorption ability, and the decrease in the hygroscopicity can be recovered by humidification.

Regarding the dimensional stability, the treatment T3, which combined hydrothermal with thermal treatment, was more effective since it promoted a higher decrease in the values of βr and Δv, compared to the other conditions of wood treatment. For this property, the GG100 clone wood assessed in this work can be reclassified from medium to low. Furthermore, MODES *et al.* [36] studied the treatment of *Eucalyptus grandis* and *Pinus taeda* wood samples submitted to autoclave and laboratory kiln treatment and achieved better dimensional stability compared to other treatments. The results of these other studies indicate that the employment of combined hydrothermal and thermal treatment can be successfully extended to other types of wood. The phenomenon of reduced dimensional stability was also reported by BATISTA *et al.* [8], and GASSON *et al.* [37], explained because higher temperature is associated with more intense decrease of the transversal section of the wood due to the tangential reduction of vessels, consequently decreasing the intensity of shrinkage.

As a general rule, the linear and volumetric shrinkages are considered very important for practical applications, such as the structural use of the wood. High values of these parameters can result in cracks and warping of wood products that demand high dimensional stability, as is the case of furniture and door/window frames [38–40]. Similar results to ours, regarding the effect of thermal treatment on the dimensional stability of wood, have been reported for other wood species. For instance, FERREIRA *et al.* [33], cited above, found a decrease in the radial, tangential and volumetric shrinkages after exposing the wood of *Hymenolobium petraeum* to temperatures ranging from 180 to 200 °C at times between 120 and 240 min. Studying the effects of thermal treatment on the *Pinus nigra* wood, GULLER [40] verified that when the temperature increased from 190 to 225 °C and the exposure time from 60 to 180 min, the volumetric shrinkage decreased by 72% compared to the control samples. Also, LIU *et al.* [41] determined that thermal treatments with temperatures of 140, 160, and 180 °C, with exposure times of 2 and 4 h, resulted in a decrease in the hygroscopicity and 66% of the volumetric shrinkage of *Ailanthus desf* wood.

Our results are also in line with those of BORŮVKA *et al.* [18] regarding the effects of thermal treatment on density and ML. They authors mentioned that wood subjected to thermal rectification can be employed for residential indoor uses as panels, furniture and flooring, and also for some musical instruments. They also emphasized that thermally treated wood can be employed to produce doors, door/window frames, fences, items for garden decoration, and playground equipment. Based on these observations, it is important to highlight that PCA showed that the thermal treatments employed here brought similar results. T1 promoted the lowest variability in density and EMC, while T2 caused the lowest ML. Furthermore, the thermal treatments applied in this work were effective to improve the dimensional stability of the GG100 clone wood. The Pearson correlation analysis indicated that higher ML was associated with lower EMC values, so the wood subjected to thermal treatment will absorb less moisture from the surrounding environment, resulting in greater dimensional stability.

5. CONCLUSIONS

Thermal treatments applied to the GG100 clone promoted a decrease in the apparent density and higher values of ML, as temperatures and exposure time increased. The EMC was the lowest when the combination of hydrothermal (autoclave) and thermal (laboratory kiln) treatment was applied, correlating negatively with ML, βt, and Δv.

The principal component analysis revealed that the thermal treatments had similar results regarding physical properties. The thermal treatment conducted in the laboratory kiln resulted in the lowest variation in the physical properties, giving higher predictability to the results than the other treatments.

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