

EFFECT OF HYDROTHERMAL AND FREEZING TREATMENT ON THE PHYSICAL AND MECHANICAL PROPERTIES OF EUCALYPTUS WOOD¹

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ABSTRACT - Wood, in general, is a material with excellent properties; however, some features may limit its use. In this sense, various thermal treatments are emerging as alternatives to improve the technological properties of wood. In this context, this study aims at analyzing the effects of time and temperature (hydrothermal treatment and freezing) on the physical and mechanical properties of *Eucalyptus grandis* W. Hill ex Maiden. Three trees were selected, and only the heartwood was used to obtain the test body dimensions of 30 × 8 × 3 cm (length × width × thickness). The treatments were carried out at three temperatures of -20, 60, and 100°C, each at three exposure times (5, 10, and 15 h). Temperature treatment at -20°C was conducted in a domestic freezer, while the treatments at 60 and 100°C were administered in a Parr reactor, where the timber was submerged in water. Regardless of the exposure time, the timbers heat-treated at 100°C showed more efficiency, since this treatment unclogged the pores, reduced hygroscopicity, and promoted further darkening and uniformity of color, without changing the density and mechanical strength of the wood.

Keywords: Heartwood. Parr reactor. Heat treatment.

EFEITO DO TRATAMENTO HIDROTÉRMICO E CONGELAMENTO NAS PROPRIEDADES FÍSICAS E MECÂNICAS DA MADEIRA DE EUCALIPTO

RESUMO - A madeira, de modo geral, é um material com excelentes propriedades, mas ressalta-se que algumas características podem limitar o seu uso. Neste sentido, alguns métodos de tratamentos térmicos vêm surgindo como alternativa para melhorar algumas propriedades tecnológicas da madeira. Neste contexto, o objetivo do trabalho foi avaliar o efeito do tempo e da temperatura do tratamento hidrotérmico e do congelamento nas propriedades físicas e mecânicas da madeira de *Eucalyptus grandis* W. Hill ex Maiden. Foram utilizadas três árvores, selecionando-se apenas a madeira do cerne para obtenção dos corpos de prova de dimensões 30 x 8 x 3 cm (comprimento x largura x espessura). Os tratamentos foram realizados nas temperaturas de -20, 60 e 100 °C combinados a três tempos de exposição (5, 10 e 15 horas). O tratamento na temperatura de -20 °C foi realizado em um freezer doméstico, e os tratamentos a 60 e 100 °C em um Reator Parr, onde as madeiras ficaram submersas em água. Independente do tempo de exposição, os tratamentos realizados a 100 °C mostraram-se mais eficientes, pois desobstruíram os poros, reduziram a higroscopicidade e promoveram maior escurecimento e homogeneização da cor das madeiras, sem alterar a densidade e a resistência mecânica das mesmas.

Palavras-chave: Madeira de cerne. Reator parr. Tratamento térmico.

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INTRODUCTION

Total cultivated forests in Brazil cover an area of 7.74 million ha, the majority of which are *Eucalyptus* sp. plantations with more than 5.1 million ha (IBÁ, 2015). These plantations make Brazil one of the largest producers of eucalyptus in the world. Eucalyptus use in the solid wood industry is emerging, yet its potential is limited by the effects of rapid growth: dimensional instability, anisotropy, heterogenic coloring, cracking, and collapsing from the unfolding and drying processes.

In this context, thermal treatment appears to be an alternative for improving some technologic proprieties of the wood. This method requires exposure of the wood to adverse treatment conditions involving temperature, time, pressure, and work atmosphere, searching the improvement of its quality for different uses. It is worth mentioning that thermal treatment can cause alterations in the physical, chemical and mechanical proprieties of eucalyptus by degrading and/or altering the structure of the main chemical components of the wood (cellulose, hemicellulose, and lignin) (RODRIGUES, 2009).

Until recently, chemical treatment methods have been developed mainly in the European continent: like the *Finnish ThermoWood®* method (Finland - VTT), in which the basic principle is the exposure of wood to steam; the *French Rectification* and *Bois Perdure* methods (France), that consists of exposing wood to nitrogen and hot air; and the *Oil Heat Treatment* method (Germany – MENZ HOLZ), in which the wood is immersed in hot oil, mainly of vegetable origin (MILITZ, 2002; RODRIGUES, 2009).

Another treatment method is the pre-freezing of wood, which has been tested in its industrial process (GIOVANELLA; MUNIZ, 2010). According to Illic (1999), pre-freezing may reduce defects during drying, increase the drying rate, and also

reduce retractability due to the tensions generated in the cell walls owing to the freezing and expansion of water.

It is noted that hydrothermal treatment does not require the use of acids; it is thus unnecessary to work with highly corrosion-resistant reactors, reducing the costs of the process (RUIZ et al., 2013). Once thermally treated, the wood may be used for coatings, floors, decks, indoor and outdoor furniture, fences, musical instruments, doors, and windows.

Therefore, independent of the thermal treatment type, a temperature-based treatment seems to be an alternative method for increasing value and improving some of the wood's intrinsic characteristics and appearance, in a species whose use is limited owing to its unwanted coloration.

The objective of this study is to evaluate the effects of time and temperature of hydrothermal treatment and freezing on the physical and mechanical proprieties of *Eucalyptus grandis* W. Hill ex Maiden wood.

MATERIAL AND METHODS

Wood precedence, processing and treatment

Three trees of a *Eucalyptus grandis* clone were utilized, approximately 15 years old and with a median diameter at breast height (DBH) of approximately 30 cm. The subject trees were taken from a population located at the Zootechnics Department of Universidade Federal de Viçosa- MG. A five m section of the lower trunk was used. From each log, three planks, three centimeters thick and embracing only of heartwood, eliminating the pith and the sapwood, were taken. From the planks specimen with dimensions of 30 ´ 8 ´ 3 cm (length ´ width ´ thickness, respectively) were obtained (Figure 1).

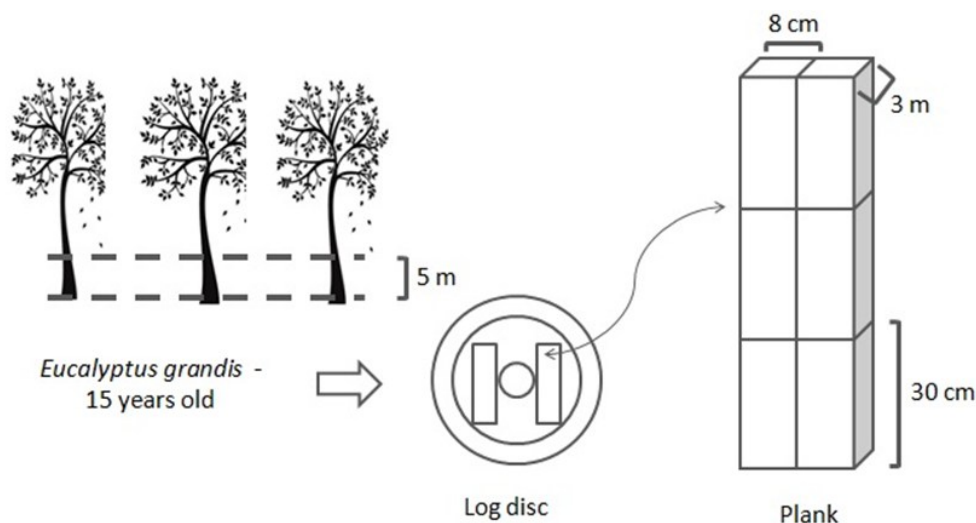


Figure 1. Schematics of the material obtainment: selection of three trees, demarcation of the heart region, plank extraction and test specimen production with dimensions of 30 × 8 × 3 cm (length × width × thickness, respectively).

The wood was exposed to two treatment conditions: freezing and wet, or hydrothermal, treatment. For freezing, the wood was exposed to -20°C in a domestic freezer for 5, 10, and 15 h. For the hydrothermal treatment, the wood was submerged in water in a digester (Parr Reactor, equipped with a heat exchanger - Parr 4848M). In this reactor, the wood was exposed to 60 and 100°C temperatures, also for 5, 10, and 15 h.

In each treatment, six specimens were used, all with an initial humidity of approximately 74%. After the treatment period, the specimens were removed from the freezer, and the resulting water from the hydrothermal process was drained for the material removal.

The specimens were next maintained at room temperature for 7 d. After this period, they were put in a climatic chamber at $23 \pm 0.5^{\circ}\text{C}$ and $65 \pm 1\%$ relative humidity, until constant mass was reached, for later characterization (CARVALHO, 2015).

Scanning electron microscope and colorimetric properties

Only the treatments with longer exposure time (15 hours), and the control were selected to illustrate the effects of treatment temperatures. For the scanning electron microscopy (SEM), cuts in the transversal planes of the specimen were made. Each wood treatment block was cut into 30 samples, $2.5 \times 2.5 \times 1.0$ cm, then fixed in aluminum stubs with carbon glue. They were next metalized with gold for two minutes on a vacuum sputter with argon. For the visualization, a vacuum scanning electron microscope with nitrogen was used.

For the colorimetric analysis the CIE-L* a*b* system (Commission Internationale de L'Éclairage) (CARVALHO et al., 2014), was employed. This is an efficient system for wood color determination using colorimeters or spectrophotometers. Three readings along the surface of each wood piece were taken using a colorimeter.

Physical and chemical properties

The basic density of the wood and the equilibrium humidity were obtained according to the Brazilian Regulation Standard – NBR 11941, Brazilian Association of Technical Standards - ABNT (2003) and NBR 7190, ABNT (1997).

To test compression parallel to the fibers, static flexion, and retractability (tangential, radial, longitudinal), the procedures established by the Technological Research Center – IPT (1956) were

used. This procedure was selected because the limitations of wood thickness prevented specimen preparation in the dimensions specified in the NBR 7190, ABNT (1997) standard.

Statistical analysis

The experiment was prepared according to a completely randomized design (CRD) in a factorial scheme comprised of three temperatures (-20 , 60, and 100°C) at three exposure times (5, 10, and 15 h) with six repetitions, for a total of nine treatments, in addition to the control, for a total of 60 sample units.

The data were submitted to variance analysis (ANOVA). When a significant effect was observed, the average results were compared using the Tukey test. Each treatment was compared to the control using the Dunnett test. For each case, 95% probability was required.

RESULTS AND DISCUSSION

Scanning electron microscopy and colorimetric properties

It was observed that in the longer exposure time (15 h), the hydrothermal treatments (C and D) promoted partial clearances in the vessel elements, probably due to the solubilization of tyloses in hot water, independent of temperature (Figure 2). The water-soluble wood constituents are mainly salts or inorganic minerals, sugars, and polysaccharides of low molar mass (MORAIS; NASCIMENTO; MELO, 2005). The wood pore clearance is important because it can increase permeability, and also positively influence the drying process, therefore decreasing defects and raising the drying tax, making it more economic.

For the wood treated in freezing conditions (Figure 2. (B)) there was no observable clearance of the vessels. When the wood is exposed to negative temperatures, it is disposed to internal collapse with water expansion, which may cause an increase in permeability (TAIZ; ZEIGER, 2006). In addition, depending on the treatment conditions, this may cause losses in mechanical resistance.

Variance analyses indicate that all the colorimetric parameters were influenced by the treatment, and only for the L parameter (luminosity) was there significant correlation between time and temperature. For all other values (a-tom and b-saturation) there were only isolated effects due to temperature (Tables 2, 3 and 4).

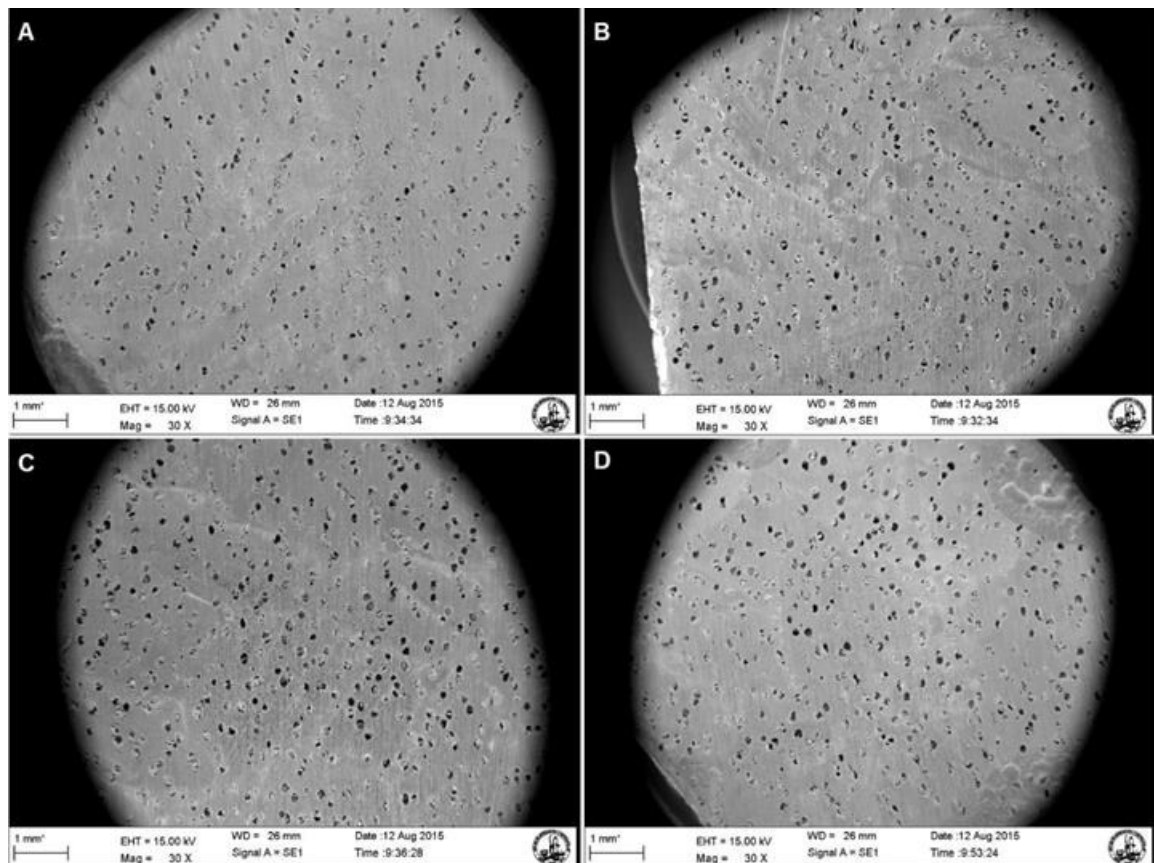


Figure 2. *Eucalyptus grandis* W. Hill ex Maiden control surface area (A), treatment at -20°C (B), treatment at 60°C (C), and treatment at 100°C (D), after 15 exposure hours.

Table 1. Average values of the L parameter (Luminosity) of *Eucalyptus grandis* for temperature (-20 , 60 and 100°C) and wood exposure time (5, 10 and 15 h).

Temperature ($^{\circ}\text{C}$)	Time (hours)			Average
	5	10	15	
-20	67.64 Aa ^{n.s}	68.08 Aa ^{n.s}	69.41 Aa ^{n.s}	68.38
60	69.70 Aa ^{n.s}	67.67 Aa ^{n.s}	69.34 Aa ^{n.s}	68.90
100	59.52 Ba [*]	53.87 Bb [*]	52.47 Bb [*]	55.29
Average	65.62	63.20	63.74	
Control	67.80			

Average values followed by the same superscript letters among columns (exposure time) and capital letters among rows (temperature) do not differ. Non-significant differences are indicated by “^{n.s},” and “^{*}” indicates significant differences between the average values and the control.

Independent of treatment time, it was observed that the higher the temperature, the lower the L value, which indicates lower wood luminosity and thus increased darkening (Table 1). It is notable that only the wood treated at 100°C significantly affected the exposure time, with the L value considerably greater for the 5 h exposure. The average value of L observed on wood treated at 100°C , independent of exposure time, was significantly lower than values observed for the

control, due to greater darkening of the wood and. The other treatments did not differ significantly from the control.

The chromatic coordinate “a” represents the red matrix. The greater its value, the closer to red on the color spectrum; lower values indicate color closer to green. According to Table 1, only the treatments at 60°C differed from the other, presenting a lower average value of the chromatic coordinate “a”. Those also differed from the control.

Table 2. Average values of the chromatic coordinate “a” for *Eucalyptus grandis* for temperatures (-20, 60, and 100°C) and exposure time (5, 10, and 15 h).

Temperature (°C)	Time (hours)			Average
	5	10	15	
-20	19.98 ^{n.s}	19.22 ^{n.s}	18.29 ^{n.s}	19.16 A
60	14.77 [*]	13.83 [*]	14.66 [*]	14.42 B
100	16.29 ^{n.s}	19.36 ^{n.s}	18.99 ^{n.s}	18.21 A
Average	17.01	17.47	17.31	
Control	20.06			

Average values followed by the same superscript letters among columns (exposure time) and capital letters among rows (temperature) do not differ. Non-significant differences are indicated by “^{n.s},” and “^{*}” indicates significant differences between the average values and the control.

Table 3 indicates a negative correlation between thermal treatment temperature and the values for “b”. This coordinate represent the yellow matrix. The greater the value, the closer to yellow on

the color spectrum; lower values indicate closer to blue. Only the treatments at 60°C for 15 h and at 100°C for 5 h were statistically significant when compared to the control.

Table 3. Average values of “b” (chromatic coordinate) for *Eucalyptus grandis* at temperatures (-20, 60, and 100°C) and exposure time (5, 10, and 15 h).

Temperature (°C)	Time (hours)			Average
	5	10	15	
-20	21.73 ^{n.s}	22.79 ^{n.s}	21.43 ^{n.s}	21.99 A
60	18.99 ^{n.s}	18.86 ^{n.s}	17.81 [*]	18.55 B
100	17.83 [*]	19.01 ^{n.s}	19.17 ^{n.s}	18.67 B
Average	19.52	20.22	19.47	
Control	21.57			

Average values followed by the same superscript letters among columns (exposure time) and capital letters among rows (temperature) do not differ. Non-significant differences are indicated by “^{n.s},” and “^{*}” indicates significant differences between the average values and the control.

In general, the treatments with greater exposure times and temperatures were the most notable with regard to the darkening of the wood, because the real color tone is determined by the combination of three parameters.

Figure 3 demonstrates the colors of the *Eucalyptus grandis* wood, in natural condition and after the thermal treatments in which the L, a, and b values are transformed in RGB (abbreviations of the additive color system formed by red, green and blue). The treatment at 100°C for 10 and 15 h promoted a greater darkening of the wood.

The chromophore composition of extractives such as tannic acids, resins and water-soluble pigments, may cause darkening of the wood that undergoes the hydrothermal process, due to

oxidation of these compounds (SUNDQVIST; MORÉN, 2002).

Treatment at -20°C did not alter the wood color because the experiment did not involve the removal of extractives, only the freezing of free and adhesion water present in the cells. Freezing does not allow water movement inside the cells’ lumen, hampering the oxidation of extractives (LIU; GAO; CHEN, 2015).

Color change is frequently considered a positive effect, valorizing lower value species that can replace the more expensive or exotic noble woods. Color is an important consideration in wood species used for furniture and decoration (KORKUT, 2012).

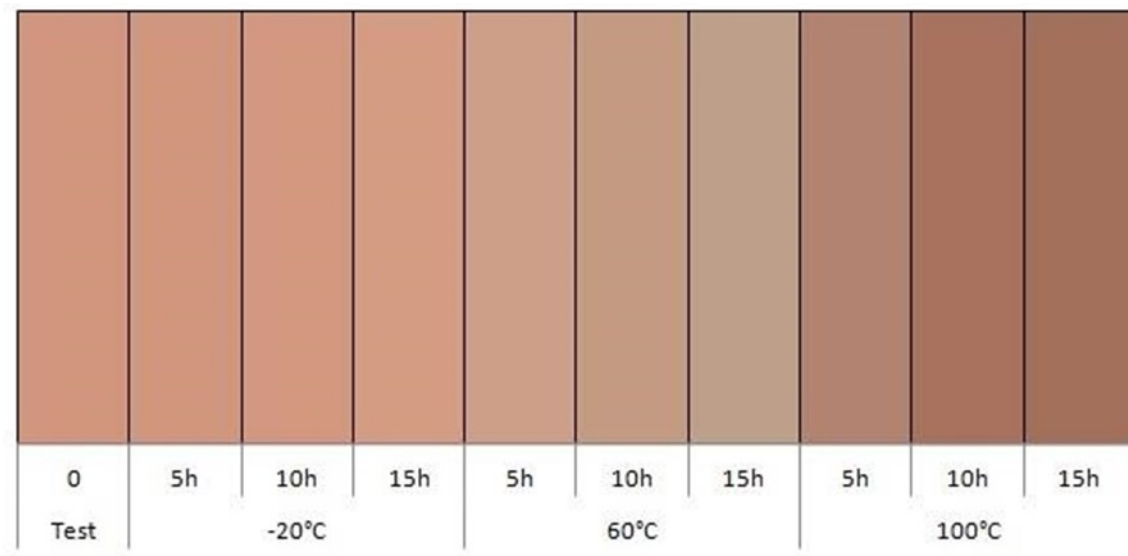


Figure 3. Representation of *Eucalyptus grandis* wood submitted to thermal treatment (temperatures: -20, 60, and 100°C and exposure time: 5, 10, and 15 h), and also the *in natura* wood (control).

Physical and mechanical properties

Variance analysis indicates that there is no significant effect of thermal treatment on basic wood density. The average density value of the thermal treatments was 0.526 g cm⁻³ and 0.525 g cm⁻³ in the control.

The adopted treatment temperatures in the present study were not high enough to degrade the structural components of the wood. Consequently, there neither significant mass loss that could cause a reduction in density nor an alteration in the proportion of mass to volume. The biomass is altered drastically when exposed to temperatures over

160°C, with the greatest loss of mass occurring at more elevated temperatures (LE VAN, 1992). Density may be altered if the mass or volume is altered, but if both remain constant, density is maintained. Density is an important property that relates dry mass and volume, and is directly related to several other important properties, such as mechanical resistance. Therefore, it is interesting to note that the thermal treatments reduced neither the density nor the mechanical resistance.

Variance analysis indicates that the hygroscopic balance moisture was affected by the treatments, based on the interaction between time and temperature of treatment (Table 4).

Table 4. Average values of hygroscopic balance moisture (%) for *Eucalyptus grandis* samples treated at varying temperatures (-20, 60, and 100°C) and exposure times (5, 10, and 15 h).

Temperature (°C)	Time (hours)			Average
	5	10	15	
-20	14.1 Aa ^{n.s}	14.0 Aab ^{n.s}	13.7 Ab ^{n.s}	13.9
60	13.4 Ba [*]	13.7 Aa ^{n.s}	13.5 Aa [*]	13.5
100	12.8 Cb [*]	13.7 Aa ^{n.s}	13.7 Aa ^{n.s}	13.4
Average	13.4	13.8	13.6	
Control	14.1			

Average values followed by the same superscript letters among columns (exposure time) and capital letters among rows (temperature) do not differ. Non-significant differences are indicated by “^{n.s}” and “^{*}” indicates significant differences between the average values and the control.

The moisture balance of wood treated for 5 h differs significantly from the others, indicated by a reduction of this variable with an increase in temperature. The treatments at 60°C for 5 and 15 h, and 100°C for 5 h had significant changes when compared to the control.

The wood exhibits high hygroscopicity due mainly to the presence of hemicellulose, which is a branched chain polymer that is found only in amorphous regions, free hydroxyl groups and therefore, have great capacity for adsorbing humidity from the environment (BRITO, 2006).

The thermal treatments did not induce significant degradation of these polymers, because polymer degradation occurs at 140°C (GIRARD; SHAH, 1991). Nevertheless, the results may be due to the thermal treatment acting on the polymer regions of the wood. Polymer alterations may be caused by phenomena like recombination, substitution, chain breaking, or partial elimination of the OH groups present in the hemicellulose, resulting in a restriction of the capacity of the wood to exchange water with the surrounding environment (HOMAN et al., 2000). Another possibility is the removal of hydrophilic extractives from the water as a result of the hydrothermal treatments, which may

contribute to the capacity of the wood to adsorb humidity.

Variance analysis indicates that the retraction of the longitudinal direction was not affected by the treatments. The average values determined for the thermal treatments and the control were 0.326% and 0.346%, respectively.

Variance analysis indicates that the retractions of the tangential and radial planes were affected by thermal treatment. In the tangential plane, there was significant correlation between time and temperature; there was only an isolated effect of these variables in the radial plane (Tables 5 and 6).

Table 5. Average values of the tangential plane retraction (%) for *Eucalyptus grandis* exposed to temperatures (-20, 60, and 100°C) at varying exposure times (5, 10, and 15 h).

Temperature(°C)	Time (hours)			Average
	5	10	15	
-20	7.94 Aa ^{n.s}	7.98 Aa ^{n.s}	7.93 Ba ^{n.s}	7.95
60	7.77 Aa ^{n.s}	8.39 Aa ^{n.s}	7.86 Ba ^{n.s}	8.01
100	7.99 Ab ^{n.s}	7.73 Ab ^{n.s}	9.19 Aa [*]	8.28
Average	7.90	8.03	8.31	
Control	7.90			

Average values followed by the same superscript letters among columns (exposure time) and capital letters among rows (temperature). Non-significant differences are indicated by “^{n.s}” and “*” indicates significant differences between the average values and the control.

Table 5 indicates a significant difference between the temperatures only when the wood was exposed to the treatment for a period of 15 h, or only for samples treated at 100°C. The average value of the treatment at 100°C for 15 h differ from the other, being the only one to present significant difference when compared to the control, having higher retraction.

There was notable variation in results between the temperatures and the exposure times (Table 6). Among the test temperatures, the treatments at 60°C presented lower retraction in the radial plane. Among the exposure times, the wood exposed for 5 and 10 h presented lower retractions as well. The treatment results did not differ from the control, being the lowest retraction in the radial plane the treatments at -20°C exposed for 5 and 10 h when compared to the control. It is likely that water present in the wood freezes, causing an expansion of the cells' lumen that can generate stress in the cellular walls, thus making them more rigid and reducing contraction (LIU; GAO; CHEN, 2015).

The greater retractions in the tangential and radial planes occurring with treatments of higher temperature and more time can be explained by the

structural reassignment of linkages, making them tighter, and consequently reducing the wood volume. This alteration in the final volume was due to the increase in retractability. An error in sampling and the difficulty in representing heartwood can contribute to the alteration of wood retractability. In general, the wood retraction in the tangential and radial planes does not show great variation.

Kollmann and Coté Junior (1968) claimed that the difference between radial and tangential retractability, rather than explained only by the restrictive influence of the radius in the radial direction, may also be explained by the helical arrangement of the microfibrils in the tangential and radial walls. Varying behaviors and movements caused by the different kinds of links present in each wood plane. The longitudinal plane is the most stable, followed by the radial and then the tangential. The longitudinal plane shows the lowest retraction percentage when compared to the others.

Variance analysis indicates that the compression resistance parallel to the fibers was not influenced by the treatments. The average values for the thermal treatments were 53.13 and 53.00 MPa for the control, respectively.

Table 6. Average values of the radial plane retraction (%) for *Eucalyptus grandis* exposed to temperature (-20, 60, and 100°C) and exposure times (5, 10, and 15 h).

Temperature(°C)	Time (hours)			Average
	5	10	15	
-20	6.68 ^{n.s.}	7.10 ^{n.s.}	7.39 ^{n.s.}	7.06 A
60	6.30 ^{n.s.}	6.42 ^{n.s.}	6.33 ^{n.s.}	6.35 B
100	6.87 ^{n.s.}	6.99 ^{n.s.}	8.08 ^{n.s.}	7.29 A
Average	6.62 b	6.84 ab	7.24 a	
Witness	7.22			

Average values followed by the same superscript letters among columns (exposure time) and capital letters among rows (temperature) do not differ. Non-significant differences between the average values and the control are indicated by “n.s.”

Variance analysis also indicates that the resistance to static flexion was not affected by the treatments. The average values of modulus of elasticity (MOE) and modulus of rupture (MOR) for the treatments were 7243.00 and 82.00 MPa, respectively. For the control, the average values of MOE and MOR were 6988.00 and 89.00 MPa, respectively.

The treatments at 60 and 100°C did not degrade the basic compounds of wood, such as cellulose, hemicellulose, and lignin, and thus did not alter the density and mechanical resistance of wood. The treatment at -20°C caused only internal freezing of the free and adhesive water present in the cells.

Other analyses, such as tomography, will allow for the visualization of additional effects of thermal treatments, for example, internal collapse, fissures, and cracks, which are visually undetectable.

CONCLUSION

Independent of the exposure time, the treatments at 100°C were optimal for the homogenization and darkening of the wood surface, in addition to reducing the hygroscopicity without altering the density and mechanical resistance of the wood.

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