

HYDROTHERMAL TREATMENT OF *EUCALYPTUS GRANDIS* WOOD

Fabiana Paiva de Freitas^{1*}, Ana Márcia M. L. Carvalho², Angélica de Cássia Oliveira Carneiro², Benedito Rocha Vital², Mateus Alves de Magalhães¹, Mariana Fonseca Xisto³

¹ Universidade Federal de Viçosa, Ciência Florestal, Viçosa-Mg, Brasil (fabianapf@hotmail.com*, mateusmagalhaes91@gmail.com)

² Universidade Federal de Viçosa, Departamento de Engenharia Florestal, Viçosa-Mg, Brasil (ana.marcia@ufv.br, cassiacarneiro1@gmail.com, benedito.vital@gmail.com)

³ Universidade Federal de Viçosa, Biologia Celular e Estrutural, Viçosa – Mg, Brasil (marianaxisto@hotmail.com)

Recebido para publicação: 19/12/2017 – Aceito para publicação: 24/07/2018

Abstract

Among the technological alternatives to improve the quality and use of eucalyptus wood is heat treatment, as the wood has some features that may limit its use, such as dimensional instability, high anisotropy, and heterogeneous coloring. This study is aimed at evaluating the effect of time of hydrothermal treatment on physical, chemical, and mechanical properties of solid wood of *Eucalyptus grandis*. We used three trees of *E. grandis*, selecting only the heartwood, and prepared test specimens with dimensions of 30cmx8cmx3cm (length, width, and thickness). The hydrothermal treatment was performed in a Parr reactor using three reaction times (5, 15, and 25min) at 140°C. Partial removal of extractives occurred, especially in the outer layer of wood. There was an increase of up to 58% of its permeability because of the partial clearing of the vessel elements. There was no degradation of the main constituents of the wood, so loss of mechanical strength was observed. The hydrothermal treatment promoted the partial removal of the hydroxyl groups and/or a structural rearrangement of the hemicelluloses and cellulose, causing a reduction of the hygroscopicity of *E. grandis*.

Key words: Hydrolysis; Pressure; Parr reactor

Resumo

Tratamento hidrotérmico da madeira de Eucalyptus grandis. Entre as alternativas tecnológicas para melhorar a qualidade e o uso da madeira de eucalipto há o tratamento térmico, pois a madeira possui algumas características que podem limitar seu uso, como instabilidade dimensional, anisotropia e coloração heterogênea. Este estudo tem como objetivo avaliar o efeito do tempo de tratamento hidrotérmico nas propriedades físicas, químicas e mecânicas da madeira sólida de *Eucalyptus grandis*. Utilizamos três árvores de *E. grandis*, selecionando apenas o cerne, e foram preparados corpos de prova com dimensões de 30cmx8cmx3cm (comprimento, largura e espessura). O tratamento hidrotérmico foi realizado em um reator Parr usando três tempos de reação (5, 15 e 25min) a 140°C. A remoção parcial de extrativos ocorreu especialmente na camada externa de madeira. Houve um aumento de até 58% de sua permeabilidade devido ao desobstrução parcial dos elementos de vaso. Não houve degradação dos principais constituintes da madeira, como foi observado no teste de resistência mecânica. O tratamento hidrotérmico promoveu a remoção parcial dos grupos hidroxilas e/ou um rearranjo estrutural das hemiceluloses e celulose, causando uma redução da higroscopicidade de *E. grandis*.

Palavras-chave: Hidrolise; Pressão; Reator Parr

INTRODUCTION

Among the technologies studied to improve the quality and use of eucalyptus wood is heat treatment, also known as thermo rectification or thermotreated wood. Heat treatment consists of the application of heat to wood, involving the partial thermo-decomposition of their constituents, especially hemicelluloses, cellulose, and lignin, in the absence or deficiency of oxygen. The temperature ranges normally used in heat treatment can improve some properties of wood products, such as the hygroscopic balance moisture (ZANUNCIO *et al.* 2014).

There are several ways to conduct this treatment in wood, and the main differences between them are related to the stages of the process, the use of oxygen or nitrogen, the presence or absence of steam, the presence or absence of oils, and the type of process (dry or humid) (MENEZES *et al.*, 2014).

The process of humid thermal treatment, the pre-hydrolysis or hydrothermal process, is a type of treatment that has been used by the cellulose industry to degrade the hemicelluloses present in wood. In the solid wood and panels industries, these hydrothermal treatments are used to degrade hemicellulose and to increase the dimensional stability of the same (ZANUNCIO *et al.* 2014). The humid treatment can be less aggressive when compared with the thermal treatment without the immersion of the wood in water (dry), as this can present considerable loss of mass and lead to a reduction of the mechanical resistance.

Besides, treatment using pre-steaming (humid) can cause increased permeability of wood through the clearing of scores and pores, optimizing the drying process (VIVIAN *et al.*, 2011). Consequently, the application

of hydrothermal treatment can obtain products with lower percentages of defects, increased permeability, and better use of the wood.

Thermal treatments generally use higher temperatures to improve wood properties, how to reduce hygroscopicity, but may cause loss of mechanical resistance due to degradation of structural components (cellulose and hemicelluloses). In this sense, it would be interesting to test new methods, which improve the properties of wood, without causing loss of mass and mechanical resistance, as for example hydrothermal treatments. They are treatments that use water, a natural and low-cost resource, and do not use chemicals that cause environmental problems.

This study aims to evaluate the effect of time of hydrothermal treatment on the physical, chemical, mechanical properties and decay resistance of solid wood of *Eucalyptus grandis*.

EXPERIMENTAL

Methodology

For this study, three trees of *E. grandis* from a population located at the Department of Animal Science, Federal University of Viçosa were used. Of the selected trees, five-meter logs from the base of each tree were used. From each log, large planks of three centimeters in thickness, covering only the core, were removed, eliminating the marrow and the sapwood. The samples were short with dimensions of 30cmx8cmx3cm (length, width, and thickness).

The hydrothermal treatment was performed in a digester (Parr Reactor model 4555 – Parr Instrument Company, Moline, Illinois, EUA) equipped with a heat exchange (Parr 4848M - Parr Instrument Company, Moline, Illinois, EUA), according figure 1. In each treatment, six test specimens were used, with an initial moisture content of approximately 74%. These were submerged in water at a temperature of 140°C and a pressure of 3.8kgf/cm². After the reactor reached the desired temperature, the specimens were kept in the reactor for residence times of 5, 15, and 25mi (treatment 1, 2 and 3, respectively), and the control, totaling 4 treatments.



Figure 1. Parr Reactor, model 4555

Figure 1. Reator Parr, modelo 4555

After the treatments, the specimens were packed in a climatic chamber at a temperature of $23 \pm 0.5^{\circ}\text{C}$ and $65 \pm 1\%$ relative humidity to constant weight, for further characterization.

Scanning Electron Microscopy and Colorimetric Properties

Only images of the transversal plane, by means of scanning electron microscopy (MES) were obtained. For the scanning electron microscopy, cuts were made in the transversal planes of the specimens. Thirty specimens with dimensions of 2.5cmx2.5cmx1.0cm (length, width, and thickness) were used for each treatment. The specimens were fixed on aluminum stubs with carbon glue. After that, they were coated with gold for 2min in a metalizer model 500X (Electron Microscopy Sciences - EMS, Hatfield – PA), in a vacuum with argon. For visualization, an electronic sweeping microscope was used, brand LEO *Elektronen Mikroskopie*, model 1430 VP (ZEISS, Oberkochen, Germany), in a vacuum with nitrogen.

For the colorimetric analysis, the CIE-L* a*b* (Commission Internationale de L'Eclairage) was used, which is an efficient system for determination of the color of wood with colorimeters or spectrophotometers. To carry out the analysis, a Konica Minolta (Chiyoda, Tokyo, Japão) model CR - 10 was used (DELUCIS *et al.*, 2015).

Structural Chemical Composition

The structural chemical composition of the wood, both the surface of the sample and its interior, was determined. Once the specimens of each treatment were obtained, they were prepared in accordance with ASTM - ASTM D 1037-28, 1982. The determination of the absolutely dry content of the wood was carried out according to TAPPI 264 on-88 (1996). The extractives contents of the wood were determined in duplicate, in accordance with TAPPI 204 on-88 (2001). The contents of insoluble lignin were determined in duplicate by the Klason method, modified in accordance with the procedure considered by Gomide and Demuner (1986). The soluble lignin was determined by spectrometry, from the dilution of the filtrate, from the procedure for attainment of the insoluble lignin (GOLDSCHIMID, 1971). The content of total lignin was obtained by means of the addition of the values of soluble and insoluble lignin, and the content of holocelluloses was obtained by the sum of extractive contents and total lignin, deducted from 100.

Resistance to Fungus *Trametes versicolor*

The accelerated test for determination of decay resistance, based on standard ASTM D2017 (2005), was carried out at the Laboratory of Fungus of the Area of Biodegradation and Preservation of the Wood, in the Laboratory of Forestry Products (LPF) at the Brazilian Forest Service, in Brasilia, DF. On the basis of the loss of mass of wood, the decay resistance was classified according to criteria established for standard ASTM D2017 (2005), as shown in table 1.

Table 1. Rot Resistance Class as a Function of Mass Loss according to ASTM D2017 (2005)

Tabela 1. Classe de resistência à podridão em função da perda de massa, de acordo com ASTM D2017 (2005).

Resistance Class	Loss of Mass (%)	Residual Mass (%)
Highly Resistant (AR)	0 – 10	90 – 100
Resistant (R)	11 – 24	76 – 89
Moderately Resistant (MR)	25 – 44	56 – 75
Non Resistant (NR)	> 45	> 55

Physical and Mechanical Properties

The basic density of the wood and the equilibrium moisture content were determined in accordance with standard ABNT NBR 11941 (2003) and ABNT NBR 7190 (1997), respectively.

For the analysis of the permeability of the wood, Darcy's law was used for the determination of the gaseous permeability:

$$Kg = \frac{Q.L.Pi}{A.\Delta P.Pm} \quad (1)$$

Where:

Kg : permeability to gas ($\text{cm}^3/\text{cm}.\text{atm}.\text{s}$);

Q : volume of the gas flow that covers the specimen (cm^3/s);

L : length of the sample (cm);

P_i : inlet pressure, of the environment (atm);

A : area of the transversal section (cm^2);

ΔP : pressure difference (atm);

P_m : mean of the pressure in the specimen (atm).

For the assays of parallel compression on fibers, static bending and shrinkage (tangential, radial, and longitudinal) were performed in accordance with the procedures established by Technological Research Center – IPT (1956) were used.

Statistical Analysis

The experiment was conducted entirely at random with four treatments (5, 15, and 25min, and control), in six repetitions, for a total of 24 sample units. The data were submitted to analysis of variance (ANOVA), and when the significant effect of the treatments were observed, the means were compared using Tukey's test at 95% of probability, and each treatment was compared with the control by Dunnet's test at 95% of probability. Only the analysis of the structural chemical composition was carried out in the factorial project, consisting also of four treatments (5, 15, and 25min, and a control) and two positions of analysis of the chemical composition of the wood (external and internal layer), with six repetitions, for a total of 48 sample units.

RESULTS

Scanning Electron Microscopy and Colorimetric Properties

In figure 2, the images obtained from the scanning electron microscopy of the transversal plane of *E. grandis* wood are presented.

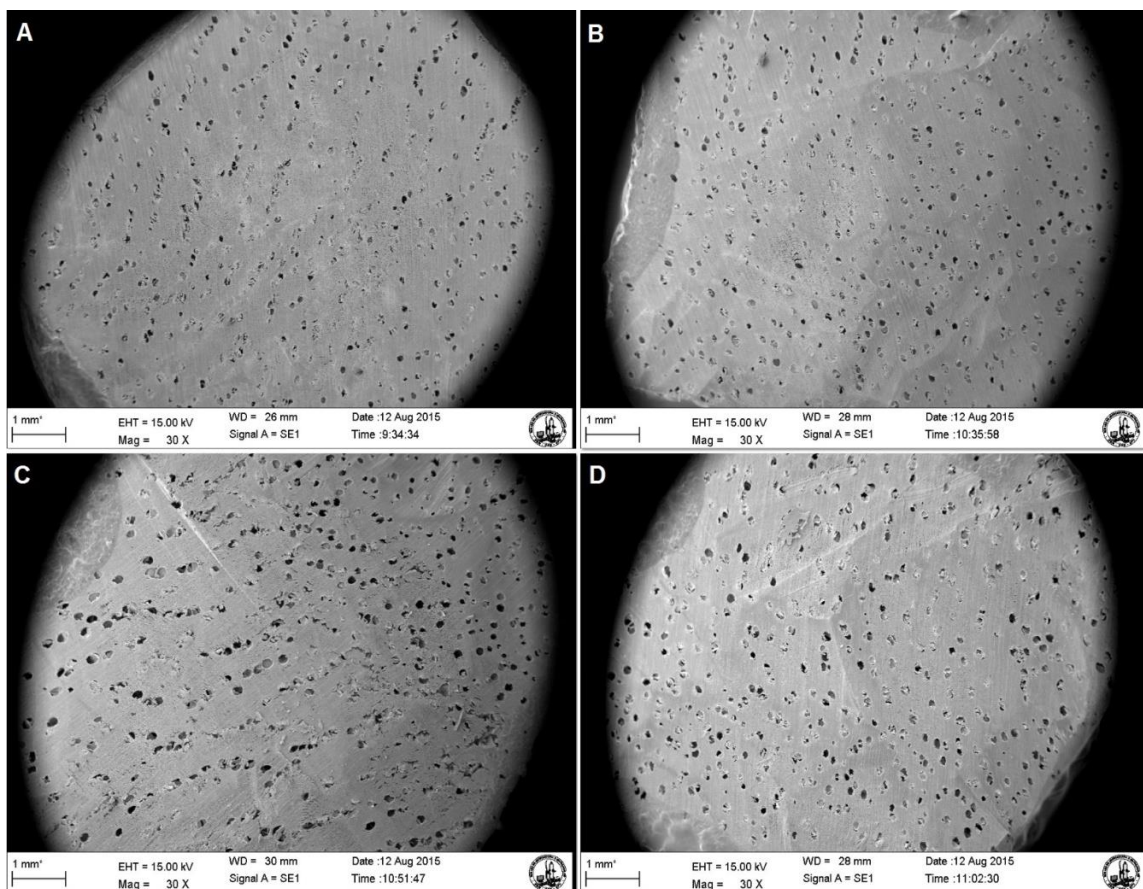


Figure 2. Scanning electron microscopy images of (A) the control wooden surface; and wood treated at (B) 140°C for 5min, (C) 140°C for 15min, and (D) and 140°C for 25min

Figura 2. Imagens de microscopia eletrônica de varredura de (A) a superfície de madeira da testemunha; madeira tratada a 140°C durante 5 minutos (B), 140°C durante 15min (C), e 140°C durante 25min (D).

Figure 2 show that the hydrothermal treatment promoted a partial clearing of the elements of vessels (pores), probably because of the internal pressure generated in the reactor and the solubilization of tyloses in hot water.

In Table 2, the mean colorimetric parameters of the wood specimens of *E. grandis* are presented in natural condition and after the hydrothermal treatments. The variance analysis indicated that the colorimetric properties were affected by the thermal treatment.

Table 2. Mean Values of Colorimetric Parameters of *Eucalyptus grandis* After Treatment

Tabela 2. Valores médios dos parâmetros colorimétricos de *Eucalyptus grandis* após o tratamento

Treatment	<i>L</i> (Luminosity)	<i>a</i> (Chromatic coordinate)	<i>b</i> (Chromatic coordinate)
1	62.5 a ^{n.s.}	18.0 a ^{n.s.}	16.9 a [*]
2	57.0 ab [*]	20.7 a ^{n.s.}	18.3 a [*]
3	51.3 b [*]	18.4 a ^{n.s.}	16.5 a [*]
4-control	67.8	20.1	21.6

Means followed by the same letters between lines do not differ according to Tukey's test. "n.s." indicates differences between the means and controls that are not significant according to Dunnett's test (all at the level of 95% probability).

In figure 3, the colors of the wood parts are presented in natural conditions and after the hydrothermal treatments, where the values of *L*, *a*, *e*, and *b* were transformed into RGB [abbreviation of the system of additives colors formed by Red (R), Green (G), and Blue (B)]. The wood treated for 25min at 140°C became darkest.

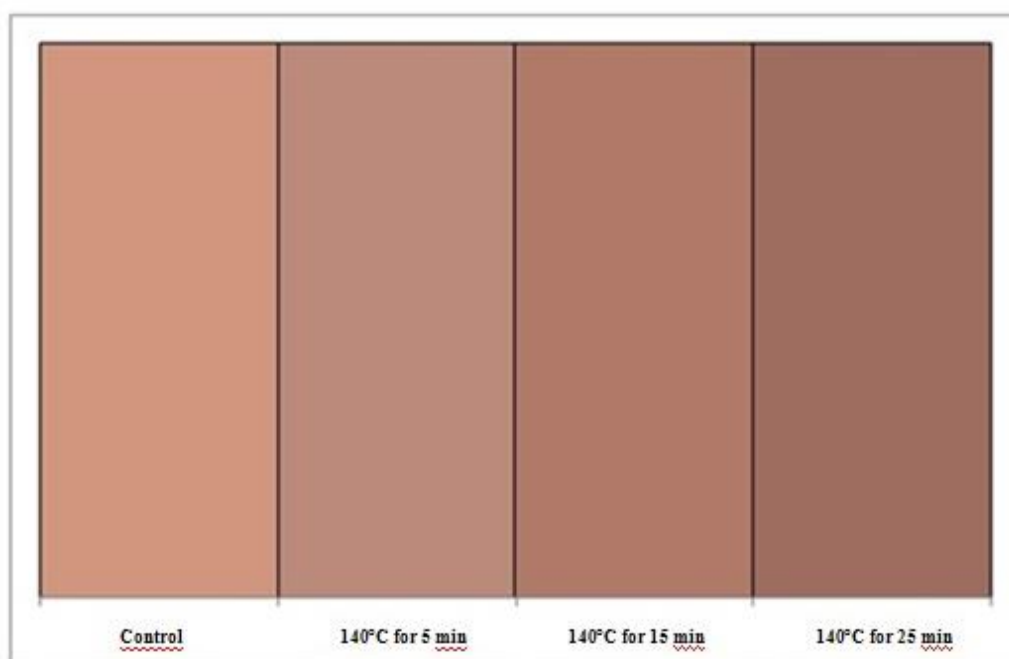


Figure 3. Color representation of the wood of *E. grandis* natural (Control), and of those that were submitted to the hydrothermal process.

Figura 3. Representação de cor da madeira de *E. grandis* natural, e das que foram submetidas ao processo hidrotérmico.

The figure 4 shows the actual color of the wood before and after the hydrothermal treatments.



Figure 4. Actual color of *E. grandis* wood subjected to a hydrothermal process and the wood in nature (the control)
 Figura 4. Cor atual da madeira *E. grandis* submetida ao processo hidrotérmico e da madeira natural (testemunha)

Structural Chemical Composition

In Table 3, the mean values of the content of extractives from the wood for each treatment are presented. The ANOVA indicated only an isolated effect of the position of withdrawal of the wood in the specimen.

Table 3. Mean Values of Extractives (%) of *Eucalyptus grandis* wood after Treatment
 Tabela 3. Valores médios do teor de extrativos (%) da madeira de *Eucalyptus grandis* após o tratamento

Treatment	Position of the Wood		Mean
	External Layer	Internal Layer	
1	3.68 ^{n.s.}	5.94 ^{n.s.}	5.19
2	4.25 ^{n.s.}	4.70 ^{n.s.}	4.55
3	3.91 ^{n.s.}	5.31 ^{n.s.}	4.84
Mean	3.95 b	5.32 a	
4 -Control	5.18		

^{n.s.} indicate differences between the means and control that are not significant according to Dunnett's test (all at the level of 95% probability).

There was no significant difference between the treatment times; the only difference was between the mean values from the external and internal layer from the parts, with nearly equal values at 3.95% and 5.32%, respectively. There was no significant difference for the treatments when compared with the control by Dunnett's test at 95% probability.

The ANOVA indicated that the contents of total lignin and holocelulloses were not affected by the hydrothermal treatments. The treatments did not differ either from the control by Dunnett's test at 95% probability. The mean value of the content of total lignin and holocelulloses of wood treated hydrothermally on the external and internal layer at 32.03% and 32.14%, and 64.02% and 62.54%, respectively. The control values for total lignin and holocelulloses were 30.88% and 63.94%, respectively.

Resistance to Fungus *Trametes versicolor*

In Table 4, the mean values of the mass loss of the treated *E. grandis* wood are presented. The ANOVA indicated that the resistance of the wood to biodegradation was influenced by the hydrothermal treatments.

Table 4. Mean Values of Mass Loss of *Eucalyptus grandis* wood After Treatment
 Tabela 4. Valores médios da perda em massa da madeira de *Eucalyptus grandis* após o tratamento

Treatments	Mass Loss (%)
1	19.04 a *
2	18.86 a *
3	21.23 a *
4 - Control	12.57

Means followed by the same letters between lines do not differ among themselves according to Tukey's test. ^{n.s.} indicate non-significant differences between the means and the control according to Dunnett's test (all at the level of 95% probability).

There was no significant difference in mass loss caused by the fungus *T. versicolor* at different hydrothermal treatment times, but all treatments differed from the control by Dunnett's test.

Table 5 shows that although the hydrothermal treatment of wood promoted weight loss between 19.04% and 21.23% when subjected to the biodegradation resistance test according to the standard, these are still classified as Resistant (R), as shown in table 1.

Physical and Mechanical Properties

The analysis of variance indicated that the mechanical strength of the heat-treated wood was not affected by hydrothermal treatment. The mean values of resistance to compression parallel to the fibers and static bending between the hydrothermal treatments using the modulus of elasticity (MOE) and modulus of rupture (MOR) were 54.00, 6830.00, and 76.00MPa, respectively, and for the control the values found were 53.00, 6988.00, and 89.00MPa, respectively.

Table 5 shows the mean values of physical properties of *E. grandis* wood as a result of the treatments. The analysis of variance indicated that only the moisture of hygroscopic balance and tangential shrinkage were influenced by heat treatment.

Table 5. Mean Values of the basic density of *Eucalyptus grandis* Wood After Treatment
Tabela 5. Valores médios das propriedades físicas da madeira de *Eucalyptus grandis* após o tratamento

Treatments	Basic Density (g/cm ³)	UEH (%)	Shrinkage L (%)	Shrinkage R (%)	Shrinkage T (%)	Permeability (cm ³ /cm.atm.s)
1	0.55 a ^{n.s.}	13.43 a*	0.34 a ^{n.s.}	6.95 a ^{n.s.}	8.03 a ^{n.s.}	9.56 b*
2	0.54 a ^{n.s.}	13.38 a*	0.32 a ^{n.s.}	7.02 a ^{n.s.}	8.90 a ^{ns}	11.59 a*
3	0.55 a ^{n.s.}	12.99 b*	0.33 a ^{n.s.}	8.54 a ^{n.s.}	9.81 a ^{n.s.}	11.67 a*
4 –Control	0.52	14.05	0.395	7.26	7.90	7.38

Means followed by the same letters between lines do not differ among themselves according to Tukey's test. "n.s." indicates non-significant differences between the means and the control according to Dunnett's test (all at the level of 95% probability).

The hydrothermal treatments did not affect the density of wood, and there was no significant difference in treatment compared with the control (0.52g/cm³) according to Dunnett's test at 95% probability

Table 6 shows that with longer duration of treatment, the moisture of hygroscopic equilibrium content of the wood decreased. It also shows that all the treatments differed from the control according to Dunnett's test at 95% of probability.

The shrinkage of the wood did not change with the treatments, and it did not differ either from the control according to Dunnett's test.

According to Table 5, longer treatment time led to higher permeability of the wood. All treatments differed from the control (7.38cm³/cm. atm.s) according to Dunnett's test.

DISCUSSION

Scanning Electron Microscopy and Colorimetric Properties

Figure 1 shows the constituents of wood soluble in water are mostly salts or inorganic minerals, sugars, and polysaccharides of low molar mass (SARTO E SANSIGOLO, 2010). The tylosis represents the growth of parenchyme cells (material of reserve) for the interior of the vessels (pores) through the scoring (FENG *et al.*, 2013). The clearance of the pores was important because it increased the permeability of the wood, thus positively influencing the drying process, minimizing the defects, and reducing the rate of drying.

According to table 2, analyzing parameter *L*, which represents the luminosity, one observes that there were significant differences with respect to the treatment time: longer treatment time led to lower values of *L*, that is, the specimens were darker. Treatments 2 and 3 were significantly different from the control, as determined by Dunnett's test

The parameter a , which represents the red-green chromatic coordinate, was neither affected by the treatment time nor did it present any significant difference in relation to the control (20.1). This shows that the hydrothermal treatment did not darken the wood too much. The higher the value of a is, the closer to red the color becomes; lower values denote more green color.

For parameter b , which represents the yellow-blue chromatic coordinate, there was no difference between the treatment times, but these differed from the control (21.6). The lower the value of b is, the closer to blue the color becomes; higher values denote more yellow color.

The combination of these three parameters forms the real tone of the color. The results show that the treatments modified the color of the wood because of the alterations in its chemical composition, more specifically because of partial degradation of hemicelluloses and removal of extractives. The possible formation and oxidation of composites such as quinones are also causes of the change in color of the wood (KORKUT, 2012).

According to figure 2 and 3, the change of wood color is often considered a positive effect, increasing the potential of other specimens that can substitute noble wood. The color is generally important for lumber species that are used for furniture and decoration (KORKUT, 2012).

Structural Chemical Composition

According to table 3, the outer layer presented lower value of extractives, and this occurs due to the greater contact of the layer exerts with the water during the treatment. According to Paes *et al.* (2013), the majority of the extractives present in the wood are easily soluble in neutral organic solvents or water. The removal of the extractives can present advantages and disadvantages, depending on the purpose of the material. Therefore, their removal can open the vessels impregnated by tyloses, increasing the permeability of wood; however, it can be harmful because some extractives provide wood with natural resistance to biodegradation.

The thermal treatment is dependent on the type of material, the temperature, the time, and the atmospheric conditions. Very high temperatures normally used in processes of dry thermal treatment can promote the degradation of the structural constituent of the wood and reduce its mechanical resistance. The hydrothermal treatment does not allow very high temperatures because of the generation of pressure in the system; however, it can be an alternative to also improve the undesirable characteristics of the wood without loss of mass occurring, which leads to a reduction of its mechanical resistance (PARSHETTI *et al.*, 2013).

Resistance to Fungus *Trametes versicolor*

According to Table 4, the treatments showed greater loss of mass because of the removal of extractives (Table 3).

The natural durability of wood is provided by its secondary component, as the extractives, which may inhibit the growth of fungi, particularly terpenes and polyphenols. The strength of wood against deterioration is the inherent ability of the specimen to resist the action of deteriorating agents, including biological, chemical, and physical agents.

This resistance is attributed to the presence of substances in wood (extractives), which can be toxic to xylophagous or act as physical barriers, such as the tyloses and/or inorganic substances (ashes) (PAES *et al.* 2013).

Physical and Mechanical Properties

According to the chemical structure results, the treatments did not degrade the structural components of the wood's cellulose, hemicellulose, and lignin, so there was no significant weight loss that might contribute to the reduction in density or change the mass ratio/volume.

Heat treatment acts on the regions of the wood polymers, causing changes either by phenomena such as recombination, substitution, and breaking chains, or elimination of the OH groups, resulting in capacity restriction of the wood water exchange with the surroundings (HOMAN *et al.*, 2000),.

When submitted to heating, wood changes its chemical composition through a thermal degradation that depends on temperature and time of exposure. For example, although wood presents a good thermal stability at 100°C if the treatment time is long enough some chemical bonds begin to break, even for lower temperature. The temperature at which the degradation starts depends on the molecular mass and crystallinity of the wood components (ESTEVEZ *et al.*, 2008).

The greatest dimensional change of wood manifests itself in the tangential direction to the growth rings, followed by the radial dimension, and it is practically negligible in the longitudinal direction. The longitudinal and transverse contraction vary essentially according to the interaction of the amount of substance of the wood, with the mean microfibrillar angle on the cell walls in relation to the longitudinal axis of the cell, and the extent of cell wall lignification. Thus, the longitudinal plane has lower shrinkage, followed by radial and finally tangential. In this study, the shrinkage of the wood did not change with the hydrothermal treatment. Even though the treated wood had less hygroscopicity, the shrinkage was not altered because the treatment caused significant degradation of hemicellulose, and the structural changes caused were not enough to reduce the volumetric shrinkage (VALENTE *et al.*, 2013).

An increased permeability by approximately 58% with increasing treatment time was observed, which can be explained by the removal of extractives and clearing of vessel elements, solubilizing tyloses present in the wood. The translocation of liquids in hardwood is greatly affected by the effect of the pores, which may have tyloses, which are natural barriers (KLITZKE, 2003).

The information on the permeability of the wood contributes to the achievement of quality products, because the conditions and the time processing will be influenced. Thus, high permeability values indicate the ease with which these woods can be processed and handled.

Heating wood modifies the cell wall components in all mass of the wood sample. This chemical modification is accompanied by an increase of dimensional stability, at the expenses of some chemical degradation of wood. Higher levels of stability and liquid water repellence are obtained, but at same time some strength properties became altered, namely hardness and abrasion resistance that are reduced. Wood colour becomes darker in all mass, which can be useful for some species (LI *et al.* 2010).

In general, the temperature and hydrothermal treatment times used in this experiment did not significantly degrade the main constituents of wood, such as cellulose, hemicelluloses and lignin; therefore, there was no loss of mass and the mechanical strength was not reduced.

It is worth pointing out that thermal treatments, primarily those that are carried out in dry conditions (in greenhouses, without the presence of liquid water or vapor) are consequently capable of reducing the mechanical properties and together with the loss of mass, limiting the structural use of wooden parts for civil construction (BORREGA AND KÄRENLAMPI, 2010).

CONCLUSIONS

- Analyzing the physical, chemical, and mechanical properties, in relation to the heat treatment in wood *Eucalyptus grandis*, it can be concluded that hydrothermal treatment promoted changes in some properties, such as the reduction of the hygroscopicity up to 7.5%, increase of up to 58% of the permeability of the wood, and homogenization of the superficial color, adding value to the final product.
- The hydrothermal treatment at 140°C for 25min (T3) was presented as the best alternative for improving the properties of wood of *E. grandis* without loss of mechanical resistance.

ACKNOWLEDGEMENTS

The authors thank the Coordination of Higher Education Personnel (CAPES), the Fungi Biodegradation Area Laboratory and Preservation of Wood, the Forest Products Laboratory (LPF) of the Brazilian Forest Service in Brasilia, DF, the Microscopy Center and Microanalysis at the Federal University of Viçosa (NMM/UFV), the Panels and Wood Energy Laboratory (LAPEM-UFV), the Wood Properties Laboratory (LPM-UFV), and the Pulp and Paper Laboratory (UFV).

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