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EFFECT OF THE BRAZILIAN PROCESS OF THERMAL MODIFICATION ON THE PHYSICAL PROPERTIES OF *Eucalyptus grandis* JUVENILE WOOD

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ABSTRACT

This study is a continuation of a previous study published in this journal, with the aim of evaluating the effect of the Brazilian industrial thermal modification process on some physical properties of *Eucalyptus grandis* juvenile wood. Flatsawn boards of juvenile wood were tested for four treatment levels: untreated and thermally modified wood at final cycle temperatures of 140 °C, 160 °C and 180 °C. Physical properties were assessed according to a standard of the Comisión Panamericana de Normas Técnicas and a method proposed by the specialized literature encompassing equilibrium moisture content (a measure of wood's hygroscopicity), density (oven-dried and air-dried) and radial, tangential and volumetric swelling (from oven-dried to green moisture content). Thermally modified *Eucalyptus grandis* wood became less hygroscopic, more dimensionally stable (tangential and volumetric swelling) and less dense, even at the lowest temperature tested (140 °C), except for radial swelling, which did not differ from untreated wood.

Keywords: Density, equilibrium moisture content, heat treatment, hygroscopicity, swelling.

INTRODUCTION

Thermal modification significantly affects the properties of wood. Considering just physical properties, thermally modified wood is less hygroscopic and consequently more dimensionally stable. On the other hand, density also declines due to cell wall mass loss, leading to a reduction of mechanical resistance (Esteves and Pereira 2009, Hill 2006, Xie *et al.* 2002).

Considering this, thermal modification processes must be developed and assessed to achieve a balance between the pros and cons imparted to wood properties, considering the end use. This makes it possible to reach the best process variables (time, temperature and treatment atmosphere, for example) for a specific raw material (species, mature or juvenile wood, heartwood or sapwood, hardwood or softwood), aiming at a specific end use (indoor or outdoor).

In Brazil, *Eucalyptus grandis* is the most studied species regarding thermal modification because of its large planted area, meaning plentiful availability to supply the wood industry. For this species, some physical properties, such as density, hygroscopicity and dimensional stability are the most studied (Batista and Klitzke 2010, Batista *et al.* 2011, Cademartori *et al.* 2012, Cademartori *et al.* 2015,

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Calonego *et al.* 2012, Calonego *et al.* 2014, Garcia *et al.* 2012, Modes *et al.* 2013).

These physical properties are among the most studied because represent simple and cheap analysis. Density, for example, is a good parameter for classifying wood quality once it is associated with dimensional stability, mechanical resistance and other wood properties (Stamm 1964). But other properties of thermally modified *Eucalyptus grandis* wood have also been studied, such as colorimetric variables (Cademartori *et al.* 2013, Garcia *et al.* 2014, Lazarotto *et al.* 2016, Zanuncio *et al.* 2014a, Zanuncio *et al.* 2014b) and decay resistance (Calonego *et al.* 2013, Calonego *et al.* 2010, Lazarotto *et al.* 2016), for example.

This study is a continuation of Batista *et al.* (2016), with the aim of evaluating the effect of the Brazilian industrial process of thermal modification on some physical properties of *Eucalyptus grandis* juvenile wood.

MATERIAL AND METHODS

Wood collection, sampling and thermal modification

The *Eucalyptus grandis* W. Hill wood used in this study was from an 18-year-old stand (from seeds) planted in the municipality of Telêmaco Borba, Paraná state, southern Brazil. Five trees were felled, but for this study, we sampled only the “A” boards (juvenile flatsawn heartwood), cut from the first three logs (3300 mm length) of each tree (Figure 1). The rest of the wood was also broken down and used to load the thermal modification equipment and for other studies.

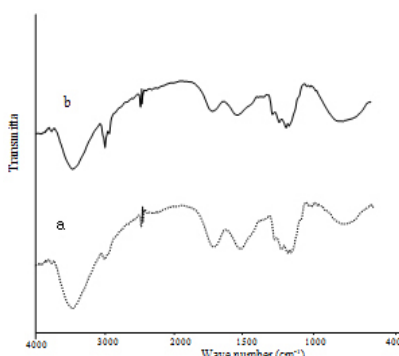


Figure 1. Scheme of the transversal section of a log showing the sampled “A” boards.

The fifteen sampled boards were then trimmed into four parts with equal length (820 mm) and equally distributed among the four treatment levels investigated: untreated wood and thermally modified wood at final cycle temperatures of 140 °C, 160 °C and 180 °C. These temperatures are the ones commonly used by the company where the research was carried out. The products are intended for indoor use.

Prior to thermal modification, the wood was seasoned until 15% final moisture content, after which it was thermally modified according to the Brazilian process of thermal modification, in an industrial scale device (steel cylinder with 6 m³ capacity), where one batch was processed separately for each final cycle temperature. Steam generated in a boiler was injected into the cylinder as the heat-transfer medium, which also limits oxidative processes, classifying the process as hygrothermal (Hill 2006). The process is typically divided into five phases, each lasting approximately eight hours, with eight more hours of cooling. The exact schedule is proprietary information, but a more detailed description of the process can be found in Batista *et al.* (2015).

Physical properties

Dimensional stability was assessed according to the swelling test (from the oven-dried to green moisture content) prescribed by standard 462 of the *Comisión Panamericana de Normas Técnicas* (Copant 1972) adapted by Batista *et al.* (2011). The specimens were taken from the middle position of the width of the trimmed “A” boards (Figure 1), properly oriented and defect free, with the size of 25 x 25 x 100 mm (radial, tangential and longitudinal). We tested 30 specimens for each treatment, two per trimmed “A” board. The same specimens were used in the assessment of the apparent densities (oven-dried and climatic chamber) and equilibrium moisture content (EMC). The latter was used as a measure of hygroscopicity.

The specimens were initially weighed on a digital semi-analytical scale (0,01 g), oven-dried (103 ± 2 °C) to constant mass (around 24 hours) and measured with a digital caliper (0,01 mm) in the longitudinal direction and with a digital micrometer (0,001 mm) in radial and tangential directions. After that, they were conditioned in a climatic chamber (35 ± 2 °C and 65 ± 3%; 11,5% EMC) to constant mass (around 15 days), weighed and measured again. Finally, the specimens were immersed in distilled water until complete saturation (constant mass, around seven days), weighed and measured again.

Swelling (radial, tangential and volumetric), apparent densities (oven-dried and climatic chamber) and EMC were calculated according to Equation 1, Equation 2, Equation 3 and Equation 4.

$$\alpha_{(t,r,v)} = \frac{(D_{green} - D_{0\%})}{D_{0\%}} \times 100 \quad (1)$$

$$\rho_{0\%} = \frac{M_{0\%}}{V_{0\%}} \quad (2)$$

$$\rho_{cc} = \frac{M_{cc}}{V_{cc}} \quad (3)$$

$$EMC = \frac{(M_{cc} - M_{0\%})}{M_{0\%}} \times 100 \quad (4)$$

Where – $\alpha_{(t,r,v)}$: tangential, radial or volumetric swelling (%); D_{green} : corresponding green dimension (mm); $D_{0\%}$: corresponding oven-dried dimension or volume (mm); $\rho_{0\%}$: oven-dried density (g/cm³); $M_{0\%}$: oven-dried mass (g); $V_{0\%}$: oven-dried volume (cm³); $\rho_{cc\%}$: climatic chamber density (g/cm³); M_{cc} : equilibrium mass in the climatic chamber (g); V_{cc} : equilibrium volume in the climatic chamber (cm³); EMC: equilibrium moisture content (%).

Statistical analysis

Statistical analysis was performed according to a completely randomized design, with 95% confidence level. The effect of the treatments was checked by applying the analysis of variance (ANOVA), with Bartlett's test used for validation, which verifies a basic premise for the applying ANOVA, the homogeneity of variances among treatments (Ribeiro-Júnior 2001). In cases of homogeneous variances, ANOVA was applied, while in cases of statistically significant difference between means, Tukey's multiple range test was used to determine which means were different.

If at least one of the variances was not statistically equal, the Kruskal-Wallis H-test was applied, which is a nonparametric method for ANOVA, for classification of an acceptance criteria or experiments with one factor (Spiegel 1994). In this test, the original data of all treatments are increasingly ordered and receive scores, giving a mean score per treatment instead of an overall mean. When at least one mean score was not statistically equal (p-value < 0,05) a box-and-whisker plot was used to identify which were different.

RESULTS AND DISCUSSION

Equilibrium moisture content (EMC)

The means of EMC of the four groups (untreated and thermally modified at 140 °C, 160 °C and 180 °C) were 8,8%; 7,6%; 5,7% and 5,2% respectively. According to Bartlett's test (1,55*) the variances of the treatments were not homogeneous with 95% confidence level and the use of ANOVA was unmanageable. Therefore, we applied the H-test (108,49*), which indicated a significant difference between the mean scores of the treatments with 95% confidence.

According to Figure 2, there was no overlap of the areas of the notches of any box for the EMC, which means that all the mean scores of the treatments were statistically different with 95% confidence.

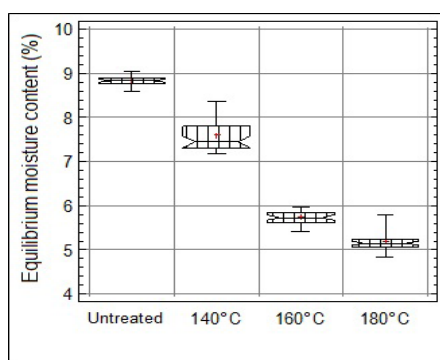


Figure 2. Comparison of mean EMC scores.

EMC of thermally modified wood was significantly lower than untreated wood by 14%, 35% and 41%, respectively, for treatments at 140 °C, 160 °C and 180 °C, so the highest reduction happened between 140 °C and 160 °C (21 percentage points). In this work, EMC was measured as an indicator of hygroscopicity.

Although the Brazilian process is very different from the European ones, mostly because of the use of different species and the lower temperatures, it is worthwhile making some comparisons. The reductions in hygroscopicity achieved by the treatments at 160 °C and 180 °C were higher than those for Plato® (25%), but smaller than those for ThermoWood® (50%) and Perdure® (62,8% to 64,4%) (Finnish Thermowood Association 2003, Perdure 2011, Platowood 2011). These comparisons show the potential of the Brazilian thermal modification process, mostly because the effect was positive for all the assessed temperatures.

Unfortunately, there are no other studies about *Eucalyptus grandis* thermally modified wood in an industrial scale process, but it is still informative to compare the results to other studies carried out in laboratory scale, which used ovens and dry heat for thermal modification. In general, the temperatures applied in these studies are higher, reaching 220 °C and 240 °C (Batista and Klitzke 2010, Batista *et al.* 2011, Cademartori *et al.* 2015, Calonego *et al.* 2012, Garcia *et al.* 2012), because it is easier to control higher temperatures in small devices. Nevertheless, in some of these studies and in the study of Calonego *et al.* (2014), 180 °C was also assessed.

Comparing the treatment at 180 °C, higher reductions were achieved in the industrial process (41%) than in the studies of Calonego *et al.* (2012) – 21%, Calonego *et al.* (2014) – 14% and Garcia *et al.* (2012) – 10%. The reduction was higher than that achieved for treatments carried out at higher temperatures (200 °C), as reported by Batista and Klitzke (2010) – 10%. On the other hand, Cademartori *et al.* (2015) reported 55% reduction in the EMC for 180 °C and Batista and Klitzke (2010) reported similar results (60%) for 230 °C.

Reduction in hygroscopicity of thermally modified wood is mostly related to the loss of

hemicelluloses, as well as the production of a high content of extractives, which also contributes to reduced hygroscopicity (Esteves and Pereira 2009, Hill 2006, Stamm 1964, Xie *et al.* 2002).

Figure 3 depicts the behavior of EMC when compared to the chemical composition, according to data published by Batista *et al.* (2016), who assessed the same material (species, process, and treatments). Batista *et al.* (2016) found that the temperatures used were not high enough to cause mass loss of cellulose, so the holocellulose reductions resulted from thermal degradation of hemicelluloses.

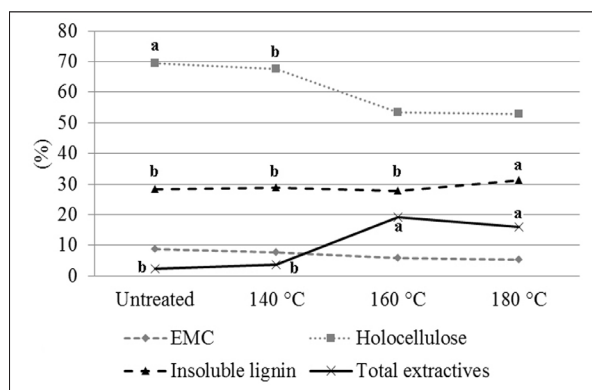


Figure 3. Behavior of EMC and chemical composition of wood (adapted from Batista *et al.* 2016) – the letters in the graph represent the Tukey tests statistics.

EMC of thermally modified wood had the same tendency as holocellulose content, where the smaller the holocellulose content was, the lower the EMC was because carbohydrates are the most hygroscopic cell wall compounds and their mass loss represents a reduction in the sites for water bonding. For extractives content and EMC, the effect was inverse to that for holocellulose and EMC. The extractives content increased significantly, indicating that the mass loss resulted in the production of chemical compounds of low molecular mass, also assessed as extractives content.

Comparing EMC with lignin content, we noted that its effect was not significant for hygroscopicity reduction. According to the literature, lignin is the least hygroscopic compound of the cell wall (Skaar 1972) and is significantly degraded only at temperatures above those tested in this study (Fengel and Wegener 1989). For further discussion of the chemical composition of thermally modified *Eucalyptus grandis* wood, see the study of Batista *et al.* (2016).

Based on the chemical results of the treatments at 160 °C and 180 °C, which had the smallest average EMC values, some considerations can be made: the means of holocellulose did not differ significantly; the average lignin content at 160 °C was smaller; the mean extractives content at 160 °C was higher; and the mean EMC at 180 °C was smaller.

So, isolated analysis of the chemical composition of wood did not permit establishing a clear relationship between cause and effect in EMC as in the overall analysis of the treatments. These results indicate that the real behavior of wood is due to the synergy of its chemical compounds and not to their isolated effects.

It is also necessary to consider that not all the extractives produced by the degradation of holocellulose and lignin have the same nature, so their behavior and role in the cell wall also differ, which can explain the EMC results.

Further, regarding the EMC results, it should be noted that the hysteresis effect is added to the effects previously discussed, according to findings of Batista and Klitzke (2010), who used the same method for EMC determination.

Density

Both apparent densities (oven-dried and climatic chamber) had the same behavior. Thermal modification reduced wood density significantly and the higher the temperature, the higher the reduction (Table 1). It is noteworthy that the means of the treatments at 140 °C and 160 °C did not differ significantly for both apparent densities.

Treatment at 180 °C caused the highest reductions in density, with 13% and 16% for oven-dried and climatic chamber, respectively. Reduction in density has the same origin as hygroscopicity: mass loss of cell wall compounds, mostly hemicelluloses, and it is a positive effect of thermal modification processes. Conversely, reduction in density results in weaker mechanical resistance. That is why it is important to find a balance between the pros and cons of the process according to a particular end use for the wood product, because the mass loss is dependent on the process, mainly time, temperature, atmosphere condition (gas, pressure) and the raw material.

Table 1. Means of wood density.

Treatment	Density (g/cm ³)			
	Oven-dried	Reduction (%)	Climatic chamber	Reduction (%)
Untreated	0,46 a	-	0,49 a	-
140 °C	0,44 b	4	0,46 b	6
160 °C	0,44 b	4	0,46 b	6
180 °C	0,40 c	13	0,41 c	16
Bartlett's test	1,05 ^{ns}	-	1,06 ^{ns}	-
ANOVA -F test	15,24*	-	19,19*	-

Means followed by the same letter in the column do not differ significantly according to Tukey's test (95% confidence level).

ns: not significant (95% confidence level). *: significant (95% confidence level).

The reductions in density caused by temperature increase are in accordance with what was reported in the literature for the European processes, although the kind of density assessed is not mentioned to provide a proper comparison. Density is reduced up to 12.5% by the ThermoWood® process, regarding 230 °C (Finnish Thermowood Association 2003). For Plato®, up to 10% is expected (Platowood 2011), while for Retification®, it varies from 4% to 9% (Retiwood 2011).

Comparing the results to those of a laboratory scale process for the same species and temperatures (140 °C, 160 °C and 180 °C), smaller reductions were reported by Calonego *et al.* (2012) for both oven-dried (2,1% to 6,7%) and air-dried (0,5% to 4,1%) densities. In some cases, thermal modification had no effect on the oven-dried density of wood at 180 °C (Calonego *et al.* 2014) and even 200 °C (Batista *et al.* 2011). Garcia *et al.* (2012) also reported a smaller reduction in air-dried density (10%) at 180 °C. Only Batista *et al.* (2011) reported a higher reduction in oven-dried density (20%) at 230 °C.

These results indicate that the Brazilian industrial process, which uses wet heat, is more aggressive regarding the mass loss of cell wall compounds than the laboratory scale processes, which use dry heat.

Swelling

Thermal modification reduced the samples' hygroscopicity and density, and since both properties are directly related to swelling (Stamm 1964), we expected the swelling to be reduced as well.

The process of thermal modification (Table 2) significantly reduced the swelling of *Eucalyptus grandis* wood with increasing temperature, except the radial swelling did not have the same behavior

because the mean of the treatment at 140 °C (3,85%) did not differ significantly from the untreated (4,18%). Also for radial swelling, we found an unexpected result because the mean of the treatment at 180 °C was higher than that at 160 °C.

Table 2. Averages of swelling (oven-dried to green).

Treatment	Swelling (%)			Volumetric reduction (%)
	Radial	Tangential	Volumetric	
Untreated	4,18 a	8,88 a	13,40 a	-
140 °C	3,85 a	7,06 b	11,13 b	17
160 °C	2,56 c	5,99 c	8,82 c	34
180 °C	3,05 b	5,45 d	8,77 c	35
Bartlett's test	1,06 ^{ns}	1,05 ^{ns}	1,06 ^{ns}	-
ANOVA – F test	58,74*	234,00*	246,57*	-

Means followed by the same letter in the column do not differ significantly according to Tukey's test (95% confidence level).
ns: not significant (95% confidence level). *: significant (95% confidence level).

The average volumetric swelling values of the treatments at 160 °C and 180 °C did not differ significantly and they achieved the highest reductions (34% and 35%). These results are similar to those of Retification® - 25% to 43% (Retiwood 2011) but are smaller than those reported for Perdure®, Plato® and ThermoWood®, with 39% to 64%, 50% to 67% and 68%, respectively (Finnish Thermowood Association 2003, Perdure 2011, Platowood 2011).

Comparing the results to those of a laboratory scale process for the same species and temperatures, the Brazilian industrial process was more effective in reducing the volumetric swelling. Calonego *et al.* (2012) reported reductions of 8,4%; 14,5% and 23,2% respectively at 140 °C, 160 °C and 180 °C. For 180 °C, Calonego *et al.* (2014) reported reductions of 14,7% and Batista *et al.* (2011) reported 20% reduction for treatments at 200 °C.

We calculated the linear correlation coefficient between the data of EMC x volumetric swelling, oven-dried density x volumetric swelling and air-dried density x volumetric swelling. We used Student's t-test to analyze the significance of the correlations. All the correlations were positive and significant at 95% confidence and the best results were achieved for the correlation between EMC and volumetric swelling ($r=0,917^*$), while the correlations between EMC and oven-dried density ($0,595^*$) and air-dried density were weaker ($r=0,620^*$).

Improvements in dimensional stability caused by the reduction in hygroscopicity, imparted by thermal modification processes, is desired for lumber mostly in indoor products with high added value. This is perhaps the most noteworthy advantage of thermally modified *Eucalyptus grandis* juvenile wood, which is otherwise recognized for its poor dimensional stability.

On the other hand, it is necessary to assess the effects of thermal modification on mechanical resistance because of the reductions in density.

CONCLUSIONS

Thermally modified *Eucalyptus grandis* juvenile wood became less hygroscopic, more dimensionally stable and less dense, even at the lowest temperature tested (140 °C), except for radial swelling at 140 °C which did not differ from control.

Equilibrium moisture content and tangential swelling continuously decreased with increasing temperature from 140 °C to 180 °C.

Higher reductions in hygroscopicity and density were achieved at the highest temperature tested (180 °C), but for volumetric swelling, there was no difference between the treatments at 160 °C and 180 °C.

The effect of thermal modification was higher in the reduction (%) of hygroscopicity and dimensional stability than in the reduction (%) of the apparent densities.

Reductions in hygroscopicity and density were caused by the mass loss of hemicelluloses and by the increase in the extractives content, produced mainly, but not exclusively, by carbohydrate degradation.

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