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Effect of the post-heat treatment on the properties of medium density particleboard of *Eucalyptus sp.*

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Abstract

Heat treatment aims to reduce the compression stresses generated during the panel production, improving its dimensional stability and providing greater resistance to attack by xylophagous organisms, despite decreasing the mechanical properties. This work aimed to evaluate the physical-mechanical properties of medium density particleboard (MDP) of *Eucalyptus sp*. subjected to post-heat treatment with two temperature levels (200 and 260ºC) and two periods (5 or 10 minutes). The apparent density, water absorption and thickness swelling (TS) after 2 and 24h, internal bonding, MOE and MOR in static bending were evaluated. The post-heat treatment at 200 °C for 5 minutes was the most efficient, improving the TS, with smaller reductions in the mechanical properties. The temperature had a greater influence in the first hours of immersion in water (TS2h), while for a longer period (TS24h) the heat treatment time was more effective. The temperature influenced the mechanical properties more negatively than the heat treatment time.

Keywords: Composites, Dimensional stability, Physical-mechanical properties, Thermal modification.

1. INTRODUCTION

Particleboards are one of commonly produced wood materials in the world with application in many branches of industry, in particular in furniture, construction or packaging industries (Borysiuk et al., 2019). World production of wood panels (particleboard) was 97.5 million m^3 in 2019 (FAO, 2020), with Brazil accounting for 3.2 million m^3, with 3.2% increase in the production of medium density particleboard (MDP) in relation to the previous year (IBÁ, 2019).

The particleboard provides the advantages of using lower quality raw materials and adequate mechanical properties and surface finish (Mendes et al., 2018; Narciso et al., 2020), but the dimensional instability due its hygroscopic behavior is one of the shortcomings (Salca & Hiziroglu, 2014; Majka et al., 2016). Many studies have been carried out using different modification processes to decrease such disadvantage (Lee et al., 2017; Özgenç et al., 2017). Heat treatment is reported to be an effective method since it significantly improves the hydrophobic properties and dimensional stability of wood and wood products (Poncsák et al., 2007; Tarmian & Mastouri, 2019; Soratto et al., 2020).

Heat treatment also changes the wood structure and degrades partially cell wall compounds and wood extractives (Nuopponen et al., 2003; Silva et al., 2017; Melo et al., 2018). Although there is also a mass loss in lignin and cellulose, the thermal degradation of the hemicelluloses during heat treatment is much more intense and expressive (Van Der Stelt et al., 2011; Silva et al., 2017), due to its heterogeneous structure, amorphous structure and low molecular weight relative to the other polymers in the wood (Figueroa & Morais, 2009). In wood panels, this treatment also aims to reduce the compression stresses generated during the panel manufacturing process (Del Menezzi et al., 2009; Mendes et al., 2013a).

There are two heat treatment procedures for the wood panels: (i) the pre-treatment performed in the raw material

of panels, i.e., in the particles (Ayrilmis et al., 2011; Mendes et al., 2013a; Mendes et al., 2013b; Ribeiro et al., 2020), and (ii) the post-treatment performed in the panels after being consolidated and stabilized (Mendes et al., 2013b; Carvalho et al., 2015; Oliveira et al., 2017; Hill et al., 2021). Therefore, regardless of how it is applied, the heat treatment consists of the application of temperature between 100 and 250 °C in the wood or wood products (Kocaefe et al., 2010; Silva et al., 2018; Wang et al., 2018).

Thus, some researches have been aiming to evaluate its influence on MDP (Carvalho et al., 2015; Melo et al., 2018), medium density fiberboard - MDF (Lee et al., 2015; Oliveira et al., 2017; Wang et al., 2020), Plywood (Lunguleasa et al., 2018; Bekhta, 2020) and oriented strand board - OSB (Okino et al., 2007; Mendes et al, 2013ab). The results were promising in most cases and the heat treatment in already consolidated panels provided an improvement in material's dimensional stability and resistance to the attack of xylophagous organisms, despite decreasing the mechanical properties.

In order to obtain a treatment that improves the dimensional stability of eucalyptus panels with the least possible effect on the reduction of mechanical properties, the objective of this work was to evaluate the physical and mechanical properties of MDP subjected to post-heat treatment with two temperature levels and two times of duration.

2. MATERIAL AND METHODS

2.1. Obtaining of material

One commercial MDP produced with *Eucalyptus* sp. particles, urea-formaldehyde adhesive and dimensions of 2.750 x 1.830 x 0.015 m (length, width and thickness, respectively) was evaluated. The panel was cut in specimens with dimensions of 50 x 50 cm (length and width) and acclimatized in a room with a temperature of 20 ± 2 °C and relative humidity of 65 ± 5%, up to constant mass.

The choice to use only one commercial panel, and its division in specimens for heat treatment in a laboratory press, aimed at greater homogenization of the initial properties of the panel for the different treatments, allowing the evaluation of the effect of the heat treatment on its physical and mechanical properties.

2.2. Heat treatment

The heat treatment of the samples was performed in a hydraulic press for 5 and 10 minutes at 200 and 260 °C (Table 1), with three replications for each treatment. The pressure used was 0.5 kgf/cm² to promote a good contact between the press plates and the sample and hence facilitate the temperature conduction. The parameters evaluated in this study were based on Oliveira et al. (2017) and Carvalho et al. (2015).

Table 1. Description of heat treatments.

Treatments	Temperature (°C)	Time (min.)
Control	-	-
T1	200	5
T2		10
T3	260	5
T4		10

2.3. Physical and mechanical characterization of the samples

Samples of MDP, treated and untreated, were cut using a circular saw. The apparent density, water absorption (WA) and thickness swelling (TS) after 2h and 24h of immersion, and internal bonding (IB) were performed following the requirements of the ASTM - D1037 standard (ASTM, 2012). The modulus of rupture (MOR) and modulus of elasticity (MOE) in static bending were determined according to the DIN 52362 NHSS standard (DIN, 1982). The EN 312 standard (ECS, 2003) was used as a comparison parameter for the results obtained.

2.4. Statistical analysis

The results of the physical and mechanical properties in relation to the heat treatment of the MDP samples were analyzed in a completely randomized design, in a factorial arrangement with two temperatures (200 and 260 °C) and two times (5 and 10 minutes) and a control (MDP without heat treatment). The means were grouped using the Scott-Knott test ($p \leq 0.05$). The results considered the values of the control and were submitted to the Dunnett test ($p \leq 0.05$).

3. RESULTS AND DISCUSSION

3.1. Physical properties

Table 2 shows the values of apparent density and thickness obtained for samples with and without heat treatment. The apparent density was 9.5% higher in the untreated (control) than in samples treated for 10 minutes at 260 °C (Table 2). The NBR 14810 standard (ABNT, 2018) establishes that medium density panels are those with values between 0.51 and 0.75g/cm³. In view of this classification, only samples treated at 260 °C for 10 min do not fit the category of medium density panels.

The decrease in the apparent density with the increase in the heat treatment time and temperature is due partially to the mass loss of the MDP by the thermochemical degradation of its volatilized constituents; this reduces the mass ratio over the sample volume and, consequently, the apparent density (Mendes et al., 2013a; Ribeiro et al., 2020). In additional, the thickness increased with the increase in the heat treatment temperature and time from 15.58 cm (control) to 16.08 cm in the 260 °C/10 min treatment (Table 2). The reduction of the apparent density of the material is linked to the increase of the sample's final thickness due to the partial release of compression stresses (Mendes et al., 2013b). According to Maloney (1993), an increase or expansion in the thickness occurs for most of the particleboards following the opening of the hot press, at the end of pressing, following the pressing pressure relaxation.

Mendes et al. (2013a) when evaluating post-heat treatment in OSB panels at temperatures of 220 °C for 12 minutes, observed a reduction in the panels density between 4 to 6.7%. Ribeiro et al. (2020) when studying the heat treatment in sugarcane bagasse MDP at temperatures of 170, 200 and 230 °C observed density reductions in the range of 5.5 to 6.3%. Carvalho et al. (2015) applied heat treatment at 200, 230 and 260 °C for 8 and 12 min in MDP produced with sugarcane bagasse and observed a reduction from 0.0% to 8.2% in the final density of panels by increasing the temperature and the duration of treatment. In this study, the reduction of apparent density was about 4.8 to 9.5%.

Table 2. Effect of heat treatment on apparent density and thickness of MDP.

Treatments	Apparent density (g/cm³)	Thickness (cm)
Control	0.63 $^{(0.01)}$ a	15.58 $^{(0.07)}$ a
200 °C/5 min	0.60 $^{(0.01)}$ b	15.77 $^{(0.12)}$ b
200 °C/10 min	0.60 $^{(0.01)}$ b	15.73 $^{(0.10)}$ b
260 °C/5 min	0.60 $^{(0.01)}$ b	15.78 $^{(0.10)}$ b
260 °C/10min	0.57 $^{(0.01)}$ c	16.08 $^{(0.05)}$ c

Means followed by the same letter in column do not statistically differ according to Scott-Knott test at 5% significance. The standard deviation is indicated in parentheses.

Table 3 shows the values of water absorption and thickness swelling obtained for samples with and without heat treatment. All values of WA2h of the heat-treated samples differed statistically from the control, increasing from 15.16 to 32.42%, depending of the time and temperature used. The higher water absorption is due to the compression stresses present in the MDP, which are released after the heat treatment, increasing its thickness (Table 2) and allowing more water entering the interior in a shorter time. After 24h contact with water, this effect of heat treatment is no longer observed, since the time is sufficient for all the hygroscopic sites of the material to have contact with the water.

It was observed that the increase provided by the release of pressing pressures of eucalyptus MDP had a more marked effect than the thermal degradation of hemicelluloses. Thus, the water absorption of the samples was not reduced, as observed by other authors (Tjeerdsma & Militz, 2005; Ayrilmis et al, 2009; Carvalho et al, 2015). This fact may be associated, besides the release of pressing pressures, with the higher thermal depolymerization of the cellulose that occurs between the temperatures 200 and 280 ºC (Figueroa & Moraes, 2009), being more pronounced from 240 °C (Cavdar et al., 2014) what could favor the water absorption of eucalyptus wood.

Table 3. Effect of heat treatment on WA and TS of MDP.

Treatments	WA2h	WA24h	TS2h	TS24h
	%			
Control	26.19$^{(3.43)}$	67.87$^{(2.20)}$	10.16$^{(1.37)}$	19.36$^{(0.99)}$
200°C/5 min	33.26$^{(4.83)}$ *	69.57$^{(5.81)}$ns	8.36$^{(0.66)}$*	16.82$^{(1.23)}$*
200°C/10 min	30.55$^{(6.38)}$ *	69.92$^{(6.92)}$ns	7.53 $^{(1.07)}$ *	18.79$^{(1.63)}$ns
260°C/5 min	30.39$^{(6.69)}$*	64.74$^{(8.99)}$ns	9.03$^{(0.27)}$ns	16.68$^{(0.34)}$*
260°C/10 min	34.68$^{(6.46)}$*	68.56$^{(5.35)}$ns	10.05$^{(0.55)}$ns	17.82$^{(0.84)}$ns

*Differences, at the same column, among treatments and the control by the Dunnett test (α= 0.05). ns does not differ, at column, from the control treatment by the Dunnett's test (α = 0.05). The standard deviation is indicated in parentheses.

Interactions were not observed between both temperatures (200 and 260 °C) and both times (5 and 10 min) of heat treatment when evaluating the properties of WA2h and WA24h, as well as no effect of the level of temperature and time of heat treatment was verified when evaluated separately.

The TS2h values of samples heat-treated at 200 °C differed statistically from the control, showing a reduction of 17.71% and 25.86% for the 5 and 10 minutes periods, respectively (Table 3). For TS24h, the treatments at 200 and 260 °C with duration of 5 minutes showed a significant improvement in relation to samples without heat treatment (control), because the values of this property reduced 13.12% and 13.84%, respectively (Table 3).

Winandy and Krzysik (2007) studied the temperature and time effects of heat treatment on the properties of MDF panels and affirm that the decrease in thickness swelling values is related to the inhibition of moisture absorption of the material due to the reduction of hemicellulose present in wood. While Sivonen et al. (2002) explain that heat treatment increases the degree of entanglement within the lignin matrix and that this fact may be associated with the reduced hygroscopicity of thermally treated wood, thus improving the dimensional stability of the material. In the case of chemical modification, the hemicelluloses are initially broken down into monomeric

structures, which are then dehydrated in aldehydes and form furfural units when derived from the pentoses and hydroxymethylfurfural units when derived from the hexoses, thus rendering a material less hygroscopic (Tjeerdsma & Militz, 2005; Yildiz & Gumuskaya, 2007; Winandy & Krzysik, 2007; Uimonen et al., 2020; Hill et al., 2021).

The reductions in thickness swelling values are also associated with the release of compression stress before the panel is exposed to moisture (Carvalho et al., 2015). Del Menezzi et al. (2008) explain that the stress releases are due to the nature of polymers that compose the wood, mainly lignin, which are viscoelastic materials, i.e., the polymer becomes viscous with increasing temperature, losing its stiffness and hence releasing the compression stresses.

Table 4 shows that there were interactions between the two temperatures (200 and 260 °C) and the two times (5 and 10 min) of the heat treatment in relation to the values of TS2h and TS24h. The increase of temperature and time had a negative effect on the TS2h property with highest value in the treatment of 10 min and 260 °C. In the TS24h property, there was a significant effect of the heat treatment time only at the temperature of 200 °C, and the duration of 10 min showed the highest average value of swelling.

Table 4. Interactions between temperature and heat treatment time on the TS2h and TS24h of MDP.

Temperature/Time	TS2h (%)	
	5 min	10 min
200 °C	8.36 (0.66) Aa	7.53 (1.07) Aa
260 °C	9.03 (0.27) Aa	10.05 (0.55) Bb
	TS24h (%)	
200 °C	16.82 (1.23) Aa	18.79 (1.63) Ab
260 °C	16.68 (0.34) Aa	17.82 (0.84) Aa

Means followed by the same uppercase letter, per column, or lower case, per line, do not differ by Scott-Knott test (p> 0.05). The standard deviation is indicated in parentheses.

Particleboards produced with the *Populus* wood and heat-treated with a temperature of 190 °C in times of 5, 10, 15, 20 and 25 min, and at 220 °C for 5 and 10 min showed reductions between 14.9% and 25.0% for the TS24h property (Xiangquan et al., 1997). Panels produced with sugarcane bagasse and heat-treated at 260 °C for 12 min had reductions for TS2h and TS24h of 26.6% and 47.0%, respectively (Carvalho et al., 2015).

The samples without heat treatment and treated at 200 and 260 °C did not meet the requirements of the EN 312 standard (ECS, 2003) and NBR14810 (ABNT, 2018) which establishes maximum value for TS24h of 14% for MDP of general use under moist conditions. However, all treatments met the NBR14810 (ABNT, 2018) standard for non-structural use in dry conditions, which determines the TS24h value of 22%.

3.2. Mechanical properties

Table 5 shows the values of MOE, MOR and IB obtained for samples with and without heat treatment. The values of MOE differed statistically being between 19.77% and 27.24% lower than control treatment. All values of MOR of the heat-treated samples differed statistically from the control, reducing from 26.69% to 54.11%, depending of the time and temperature used. The reduced density of heat-treated samples (Table 2), due to the release of pressing pressures, affected negatively the mechanical properties, since there is a positive linear relationship between the density of the material and its mechanical behavior (Maloney, 1993; Iwakiri et al., 2008; Ayrilmis et al., 2009).

The IB values of heat-treated samples differed statistically from the control, showing a reduction between 19.15% and 72.34% according to the increase of time and temperature (Table 5). The reduction of the internal bonding property using the heat treatment is related to the adhesive present in the panel that is also volatilized at the temperatures used in this work (Kaboorani & Riedl, 2011; Stefanowski et al., 2016). According to Oliveira et al. (2017), the thermal degradation of the urea-formaldehyde adhesive starts at a temperature of 220 °C, with peak near 250 °C, factor that directly affects the mechanical properties of the panels.

Table 5. Effect of heat treatment on MOE, MOR and IB of MDP.

Treatments	MOE	MOR	IB
	MPa		
Control	2336.47 (50.78)	17.65 (1.08)	0.47 (0.06)
200 °C/5 min	1874.44 (108.87) *	12.94 (0.42) *	0.37 (0.03) *
200 °C/10 min	1883.71 (118.10) *	12.74 (0.52) *	0.38 (0.02) *
260 °C/5 min	1758.20 (126.78) *	9.60 (1.12) *	0.38 (0.05) *
260 °C/10 min	1700.12 (138.63) *	8.10 (0.75) *	0.13 (0.01) *

*Differences, at column, between treatments and the control by the Dunnett test (α= 0.05).

There were interactions between the two temperatures (200 and 260 °C) and the two times (5 and 10 min) used in the heat treatment in relation to MOE, MOR and IB (Table 6). The heat treatment time in association with the temperature of 200 °C did not have effect for any of the evaluated mechanical properties. However, the values of MOR and IB properties reduced with the increase of the heat treatment time in the 260 °C temperature.

Table 6. Interactions between temperature and heat treatment time on the MOE, MOR and IB of MDP.

Temperature/Time	MOE (MPa)	
	5 min	10 min
200 °C	1874.44 (108.34) Aa	1883.71 (122.24) Aa
260 °C	1758.19 (101.63) Aa	1700.12 (143.05) Ba
	MOR (MPa)	
200 °C	12.94 (0.55) Aa	12.74 (0.59) Aa
260 °C	9.59 (0.97) Ba	8.10 (0.66) Bb
	IB (MPa)	
200 °C	0.37 (0.03) Aa	0.38 (0.03) Aa
260 °C	0.38 (0.04) Aa	0.13 (0.01) Bb

Means followed by the same uppercase letter, per column, or lower case, per line, do not differ by Scott-Knott test (p> 0.05). The standard deviation is indicated in parentheses.

Changing from 200 °C to 260 °C temperature with 10 minutes of heat treatment duration had significant effect on all properties (MOE, MOR and IB). There was significant effect for the MOR property at the time of 5 min too. In both cases, the heat-treated panels at 260 °C obtained the lowest average values. The decrease in mechanical properties is associated with loss of panel mass caused by heating during heat treatment, as well as by the release of pressing stresses that promote the increase of empty spaces, acting as stress concentrators and decrease of panels density (Ayrilmis et al., 2009; Mendes et al., 2013a; Surdi et al., 2018).

Only heat-treated samples at a temperature of 260 °C for 10 min did not meet the established minimum value by EN312 standard (ECS, 2003) of 0.30 MPa and by NBR14810 (ABNT, 2018) of 0,35 MPa for the internal bonding. The samples without heat treatment and treated at 200 °C in both times meet the requirements of the EN 312 (ECS, 2003) and NBR14810 (ABNT, 2018) which establishes minimum value for MOR of 11 MPa for MDP of internal use in dry conditions. For the MOE, only the samples without heat treatment (control) had a mean value higher than the minimum established value of 1950 MPa (ECS, 2003; ABNT, 2018) for use in moist conditions. However, all treatments met the NBR14810 (ABNT, 2018) and EN 312 (ECS, 2003) for non-structural use in dry conditions, which determines 1600 MPa for MOE.

4. CONCLUSIONS

The heat treatment at temperature of 200 °C for 5 minutes was the most suitable for eucalyptus MDP, because the properties of TS2h and TS24h were improved and mechanical properties had smaller reductions.

For the dimensional stability of the samples, the temperature level had a greater influence in the first hours of immersion in water (TS2h), while for a longer period (TS24h) the heat treatment time was more effective. The temperature level influenced the mechanical properties more negatively than the heat treatment time.

Only the heat-treated samples at a temperature of 260 °C, in both treatment times, did not meet the requirements determined by the standards NBR14810 (ABNT, 2018) and EN 312 (ECS, 2003) for non-structural use in dry conditions.

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