

# WETTABILITY AND DECAY OF PARTICLEBOARDS MANUFACTURED WITH THERMALLY TREATED SUGARCANE RESIDUE AND BAMBOO (*Dendrocalamus asper*) PARTICLES

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## ABSTRACT

This study aimed to evaluate the chemical composition of wood particles (control and treated), and the effects of thermal modification and adhesive levels on the wettability and biological resistance of particleboards made of sugarcane residue and bamboo (*Dendrocalamus asper*). Therefore, 75% bamboo particles and 25% sugarcane residue (bagasse) were used for producing the particleboards. The particles were treated at 220 °C for 3h35min. Urea formaldehyde (UF) adhesive was used in three solid contents (10%, 12% and 14%) based on the dry mass of the particles. The mat was cold pre-consolidated (pressure of 0,5 MPa for 5 min) and after hot consolidated (3,45 MPa, 180 °C, 10 min). Water and ethylene glycol and two measurement times were used to measure the contact angle. *Gloeophyllum trabeum* and *Rhodonía placenta* (brown rot) and *Trametes versicolor* (white rot) fungi were used for the biological resistance test. There was a change in the chemical composition of the treated particles such as a reduction in the levels of lignin (bagasse and bamboo), total extracts and holocellulose (bagasse). The thermal treatment increased the final contact angles obtained with water. The particleboard surfaces were classified as non-wettable and partially wettable to the tested solvents. The thermal treatment provided biological resistance improvements in the particleboards to the tested fungi, being classified as very resistant to *Rhodonía placenta*, resistant to very resistant to *Gloeophyllum trabeum*, and moderate to resistant to *Trametes versicolor*.

**Keywords:** Chemical composition, contact angle, medium density particleboards, thermal modification, wood decay fungi.

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## INTRODUCTION

Industries use wood from reforestation such as pine and eucalypts to produce particleboards, but these can be produced from any lignocellulosic material, given they provide adequate physical, mechanical and biological properties. The quality of the final product is directly related to the choice of raw material (Melo *et al.* 2015). The need to use of alternative materials for particleboard production is due to the high market demand and the constant lack of traditional raw materials in the industries. An option for these problems would be to use agro-industrial residues such as sugarcane bagasse and fast-growing materials such as bamboo, which promote sustainability and ecological construction, in addition to adding substantial value to lignocellulosic materials.

Bamboo can mitigate impacts and adapt to climate change (Wu *et al.* 2014), being an excellent carbon sink. Song *et al.* (2011) claim that the ability of bamboo to sequester atmospheric CO<sub>2</sub> is due to its rapid growth and potential to store carbon in its biomass. Sugarcane bagasse, in addition to producing heat and energy (Hiloidhari *et al.* 2018, Carvalho *et al.* 2020), can be used as a raw material in the paper industry, particleboard, animal feed and in the production of microbial biomass.

The sugarcane production in Brazil in the 2019/20 harvest was 642,7 million tons (*Companhia Nacional de Abastecimento* - CONAB, 2020). In addition to the use of sugarcane for producing ethanol and sugar, bagasse produced on a large scale can be used for cogenerating clean energy through combustion and gasification processes. However, sugarcane bagasse can also be reused for producing particulate panels (Brito and Bortoletto Junior 2020, Brito *et al.* 2020a).

Regarding the production of particleboards, studies have already been conducted in Brazil and in other countries using sugarcane residue as a raw material associated or not with other materials pointing to its technical viability; for example, studies by Soares *et al.* (2017), that recommended a percentage of 26% of sugarcane bagasse, in association with eucalypts wood, in the production of low density particleboard. Atoyebi *et al.* (2019) concluded that panels produced with 50% sugarcane bagasse and 50% corn cob have good physical and mechanical performance. Brito *et al.* (2020b) noted that the mixture of 75% bamboo and 25% sugar cane provided good performance for the panels. Sugahara *et al.* (2019) demonstrated the potential for using bagasse in the production of high-density particleboard. Yano *et al.* (2020) verified that panels with 50% sawdust and 50% bagasse performed well, indicating the possibility of using panels produced with these residues.

Bamboo can be mentioned in addition to sugarcane bagasse, possessing characteristics such as lightness, flexibility, good resistance properties and rapid growth. On the other hand, it has disadvantages such as being hydrophilic, having dimensional instability and low resistance to deterioration in the inner layer, which shortens its useful service life (Schmidt *et al.* 2011, Lee *et al.* 2018).

Although there is only one industry which uses bamboo for producing cellulose in Brazil, a high local potential in relation to planting and marketing bamboo makes it reasonable to carry out scientific research related to developing high value-added products with bamboo (Gauss *et al.* 2019).

References of Brazilian and international research using bamboo as a raw material associated or not with other lignocellulosic material include those carried out by Dinhan *et al.* (2015) who concluded that panels produced with coconut and bamboo fibers is an innovative proposal for the sustainable production of particleboards, for use indoors and dry places. Zaia *et al.* (2015) stated that bamboo particleboards are an economically viable and sustainable alternative for the use of waste generated during bamboo processing. Almeida *et al.* (2017) found that the addition of 25% and 50% bamboo promoted values higher than those produced with 100% wood. Brito *et al.* (2020a) concluded that bamboo can be used as an alternative material for the manufacture of particleboard for indoor uses. Nasser *et al.* (2020) verified panels made of bamboo and peanut shells met the parameters established by Brazilian Standard - NBR 14.810-2 (2013), Brazilian Association of Technical Standards - ABNT (2013) and American National Standards Institute - ANSI A208-1 (1999).

An interesting option for producing particleboards is the combination of two or more materials. Since little is known about combining particles of alternative raw materials in manufacturing particleboards, this could become a large raw material source for supplying industries. In addition, different physical and chemical characteristics can be combined through mixing the particles to make the gluing and particleboard formation process more feasible (Iwakiri *et al.* 2010). For particleboards produced with bamboo particles and sugarcane bagasse, in proportions (0, 25, 50, 75 and 100%), Brito *et al.* (2020b) verified that the compaction rate increased with

the addition of sugarcane bagasse. However, there was a decrease in the dimensional stability of the panels. On the other hand, there was an increase in the modulus of rupture, elasticity and resistance to surface screw pullout, but reduced top screw pullout and internal adhesion. The best panels were those produced with 75% bamboo particles and 25% sugarcane bagasse.

In addition, there is a growing concern regarding biological resistance, which is an essential test to define the use and application of the final product (Brito *et al.* 2020a, Brito *et al.* 2020b), in addition to saving unnecessary expenses with the replacement of parts and reducing impacts on the environment (Paes *et al.* 2015). Like wood, the chemical constitution from agro-industrial wastes (sugarcane bagasse) and other lignocellulosic materials (bamboo) can favor degradation as they contain high levels of starch (Brito *et al.* 2020a) and function as sources of nutrition for xylophagous organisms. Thus, some treatments have emerged which aim to improve the resistance to moisture, dimensional stability and biological durability. Among these, heat treatments which in addition to promoting the necessary improvements, do not use chemicals which harm the environment (Jirouš-Rajković and Josip Miklečić 2019). Thus, improvements in the properties of the final product (such as reconstituted particleboards) can be obtained.

Heat treatment causes changes in the material structure, which reduces the equilibrium moisture content due to the degradation of chemical components and forms lignin cross-linking, which affects water adsorption (Surini *et al.* 2012). After being treated, the wood becomes hydrophobic, influencing wettability (a term related to the spreading of liquids on a solid surface) due to the plasticization of lignin, which leads to reorganized cellular polymeric components (Hakkou *et al.* 2015). When the surface wettability of a material is changed, its use can be influenced by the adhesion of paints and coatings (Zhang and Yu 2015), and the spreading and coating of particles by adhesives. This can add value to the final product and/or enable the union of its components.

Surface wettability is usually measured by the contact angle. The lower its value, the better the surface wettability (Fang *et al.* 2016). Some studies have evaluated the wettability of particleboards made of heat-treated particles (Unsal *et al.* 2010, Unsal *et al.* 2011, Candan *et al.* 2012) in which there was a tendency to increase hydrophobicity in the heat-treated material. This is caused by the decrease of free OH-groups, mainly in the hemicellulose chains (Kubovský *et al.* 2020). Since between 180 °C and 190 °C, we have a moderate modification with carbohydrate degradation and deacetylation reactions of these components (Bachle *et al.* 2010).

Thus, the chemical composition of the material is also modified during heat treatment by the degradation of compounds and the cell wall extracts (Esteves and Pereira 2009), which in turn can influence the natural durability of material. The importance of this property is related to the behavior and uses of particleboards made with alternative materials. Some research on biological resistance of particleboards manufactured with heat-treated particles has already been conducted (Del Menezzi *et al.* 2008, Mendes *et al.* 2013). These authors indicated that the heat treatment improved the resistance against wood decay fungi.

Wood or other lignocellulosic materials (sugarcane bagasse and bamboo) can be used for producing particleboards when they are transformed into particles. However, few experiments have been carried out with particleboards made from thermally modified sugarcane bagasse and bamboo particles; rare examples are those mentioned by Brito and Bortoletto Júnior (2019) and Ribeiro *et al.* (2020), mainly in relation to technological properties, biological resistance and wettability. Thus, this study had the objective to evaluate the effect of thermal modification on the chemical composition of the particles, wettability and biological resistance of particleboards made from sugarcane bagasse and bamboo (*Dendrocalamus asper*).

## MATERIALS AND METHODS

### Origin and collection of raw materials used

Sugarcane bagasse was collected from a sugar mill located in Santa Bárbara D'Oeste, state of São Paulo, Brazil. The collected residues had good phytosanitary characteristics (no decomposition). After collection, the material was exposed outdoors on a plastic canvas until it reached  $\approx 18\%$  moisture. Drying was subsequently completed in a forced circulation oven ( $70 \pm 2$  °C) until it reached  $\approx 10\%$  moisture. After this process, the

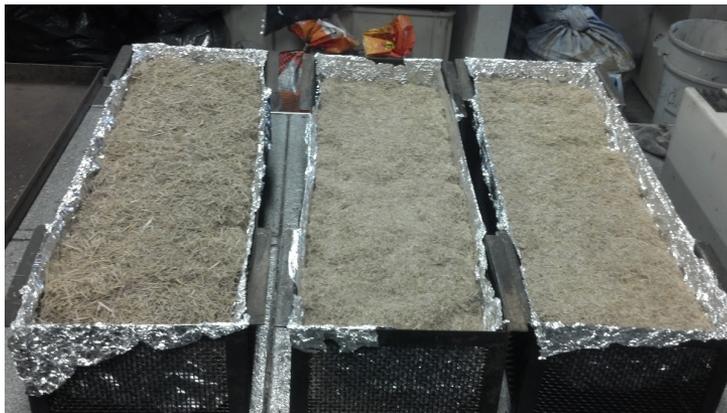
material was classified and the particles with granulometry between 0,50 mm and 0,85 mm were selected by the most appropriate morphology for producing the particleboards. Both were mixed in a 1:1 ratio based on the dry mass of the particles for further heat treatment in an oven.

The bamboo (*Dendrocalamus asper* Schult f. Backer ex Heyne) was aged over three years, and was collected at the Campinas Agronomic Institute located in Tatuí, state of São Paulo, Brazil. The procedures for cutting the culms and transforming them into splinter were described by Brito *et al.* (2018). The selected culm had a height of 15 m and were harvested in the field with a machete and chainsaw. The culms were sectioned in 2 m sections to facilitate transportation to the Lamination and Wood Panels Laboratory, Luiz de Queiroz College of Agriculture, University of São Paulo, Piracicaba Campus, Brazil.

The culms were cut longitudinally in a circular saw to obtain the splinter. The inner and outer layers were removed from the splits, as described by Brito *et al.* (2018), and transformed into chips in a band saw. The chips were dried in a similar way to sugarcane bagasse and transformed into particles in a Thomas Wiley mill (Arthur H. Thomas Company, Philadelphia, Pennsylvania, United States), using the same granulometries and proportions adopted for the sugarcane residue.

### Thermal modification of the particles

The heat treatment process was similar to described by Brito and Bortoletto Júnior (2019). The particles were dried in an oven ( $\approx 3\%$  moisture) and placed into containers made of wire mesh with dimensions of 13 cm x 18 cm x 58 cm (height x width x length), which were previously lined with aluminum foil (Figure 1).



**Figure 1:** Bamboo and sugarcane bagasse particles, packed into containers with dimensions of 13 cm x 18 cm x 58 cm (height x width x length), internally lined with aluminum foil, for insertion in an oven and application of the heat treatment.

The containers were subsequently placed in metal boxes (with capacity for five containers) and placed in an oven equipped with nitrogen injection to avoid the risk of igniting the particles.

The thermal modification of the particles was started at room temperature ( $\approx 28\text{ }^{\circ}\text{C}$ ). The initial heating rate was  $3,33\text{ }^{\circ}\text{C min}^{-1}$  and maintained up to  $100\text{ }^{\circ}\text{C}$ , for 21 min (Mendes *et al.* 2013). Then the heating rate was reduced by  $1\text{ }^{\circ}\text{C min}^{-1}$ , which was maintained until reaching  $220\text{ }^{\circ}\text{C}$  (2h 35 min), and remained in these conditions for another 3h 35 min to perform the heat treatment of the particles. The particles remained inside the oven at the end of the process until they reached room temperature. The particles were turned and removed from the containers and packed in plastic bags.

## Chemical analyzes of the particles

To enable a chemical analysis, the heat treated and untreated particles (control) passed through the wiley mill screen were transformed into sawdust using a material with a 60 mesh size. The analyzes were performed in quadruplicate and the results were expressed on a dry basis. The designations of the Technical Association of the Pulp and Paper Industry - TAPPI 222-02 (2002) were followed for the Klason lignin content (insoluble). Soluble lignin was made according to Novo (2012). The total lignin was the sum of the obtained contents.

TAPPI 207-99 (1999) was followed to determine the extracts in hot water. The levels of total extracts were carried out according to TAPPI 204-97 (1997) using a cyclohexane and ethanol mixture (2:1, volume: volume), followed by extraction in hot water. Next, TAPPI 211-02 (2002) was adopted for ash content. The holocellulose percentage was obtained by the difference, according to the Equation 1.

$$\text{Holocellulose (\%)} = 100 - [\text{extractives (\%)} + \text{total lignin (\%)} + \text{ashes (\%)}] \quad (1)$$

## Production of sugarcane bagasse and bamboo particleboards

A mix made up of 75% bamboo particles and 25% sugarcane bagasse particles were used to produce the particleboards, based on the dry mass of the particles. This proportion was adopted based on previous experiments carried out by the authors. The granulometries used were 0,50 mm and 0,85 mm due to providing better morphology for manufacturing the particleboards. The production parameters were similar to those adopted by Brito and Bortoletto Júnior (2020). The pre-established nominal density was 0,65 g/cm<sup>3</sup> with a nominal thickness of 1,57 cm. Urea-formaldehyde (UF) adhesive was used in three solid contents (10%, 12% and 14%).

The adhesive used had a solids content of 64,16%, a density of 1,27 g/cm<sup>3</sup> and a pH of 7,88. A solution of ammonium sulfate (catalyst) in the proportion of 5% solids was incorporated into the adhesive. The mixture was homogenized and sprayed on the particles in a rotating drum (12 revolutions per minute - rpm) for 5 min, and paraffin emulsion (1,0% solids) was applied to the mixture (5 min - 12 rpm).

The particles were weighed and deposited in a hollow wooden mold with dimensions of 40 cm x 40 cm, placed on an aluminum plate (50 cm x 50 cm). The mat was cold pre-consolidated (pressure of 0,5 MPa, 5 min) and hot consolidated (3,45 MPa, 180 °C, 10 min). The obtained particleboards were air-conditioned (22 ± 2 °C and 65 ± 5 % relative humidity - RH) before the samples were removed for the wetting and biological resistance tests.

## Particleboard surface wettability

For the wettability tests, the goniometer KSV CAM 200 (Bionavis, Tampere, Pirkanmaa, Finland) was used. Samples with dimensions of 2,50 cm x 2,50 cm x 1,57 cm (length x width x thickness) were sanded (sandpaper # 200) to improve the contact angle measurements (θ°). Three replicates per treatment (particleboards) were used to test the wettability, for which a sample was taken from each particleboard and two measurements per sample were performed on opposite sides of the particleboards, totaling six measurements per treatment.

The solvents used (water and ethylene glycol) have a polar character, and provide good solvent-wood interactions. Water can act as a Lewis acid and ethylene glycol as a base (Walinder and Johansson 2001). A 10 µl syringe graduated in 1 µl positioned 8 mm in relation to the sample surface and a drop of 4 µl of the solvent was used for the application.

The KSV Contact Angle Measurement System software program was used to determine the θ°. As indicated by César (2011), two measurement times for the angles (initial and final) were considered. The initial time was determined right after depositing the solvent drop on the sample surface, and the final time after stabilization of the θ°.

## Biological resistance of the particleboards

The resistance of the particleboards to wood-decay fungi was verified and classified according to the

American Wood Protection Association - AWP A E-30 (2016). *Rhodonia placenta* (Fr.) Niemelä, K.H. Larss. & Schigel (Mad 698-R) and *Gloeophyllum trabeum* (Pers: Fr.) Murr. ((Mad 617) which cause brown rot, and *Trametes versicolor* (L.) Lloyd (Mad 697) which causes white rot were used, being obtained from pure cultures. Samples with dimensions of 25,0 mm x 25,0 mm x 15,7 mm (length x width x thickness) were dried in an oven maintained at  $103 \pm 2$  °C to obtain the initial dry mass.

The glass flasks (600 mL) used in the experiment were filled with 300 g of soil (red latosol) from horizon B with a low amount of organic matter. The soil pH was 6,5 and the water retention capacity was 25%. Next, 67 ml of distilled water was added to the flasks and two feeder strips with dimensions of 0,3 cm x 2,8 cm x 3,5 cm (thickness x width x length) made of *Pinus elliottii* wood and sterilized ( $121 \pm 2$  °C, 103 kPa, 30 min). After cooling, the flasks were placed in an incubation room ( $27 \pm 2$  °C and  $65 \pm 5\%$  RH).

The fungi inoculation ( $\approx 0,5$  cm x 0,5 cm inoculum) on the feeder strips was carried out in a laminar flow hood. Two particleboard samples were then added per flask after the inoculum growth and the beginning of soil colonization (30 days). The experiment was kept in the incubation room for 12 weeks. They were removed from the flasks and carefully cleaned with a brush to remove the mycelium and soil granules which were adhered to the material (Figure 2).



**Figure 2:** (a) Incubation room experiment; (b) Samples colonized by fungi; (c) Clean samples for oven drying.

The samples were dried ( $103 \pm 2$  °C) until they reached constant and heavy masses (final dry mass). Mass loss was calculated (AWPA E-30 2016) and the strength classes of the particleboards were determined. Six samples were used for each treatment for operational mass loss, which were kept under the same conditions but without contact with the fungi in order to determine the mass loss caused by handling the samples (Mendes *et al.* 2013).

### Experimental design and data analysis

Descriptive statistics (mean and standard deviation) were used for the chemical composition of the particles (treated and control). Three adhesive levels were adopted (10%, 12% and 14%) for producing the particleboards, having those produced with non-heat-treated particles and glued with 10% adhesive as control.

Analyzes of variance and F tests ( $p < 0,05$ ) were performed for the wettability and biological resistance tests, and the Tukey test ( $p < 0,05$ ) was used to discriminate the means. The data normality was verified by the Lilliefors test and the homogeneity of the variances by the Cochran test.

## RESULTS

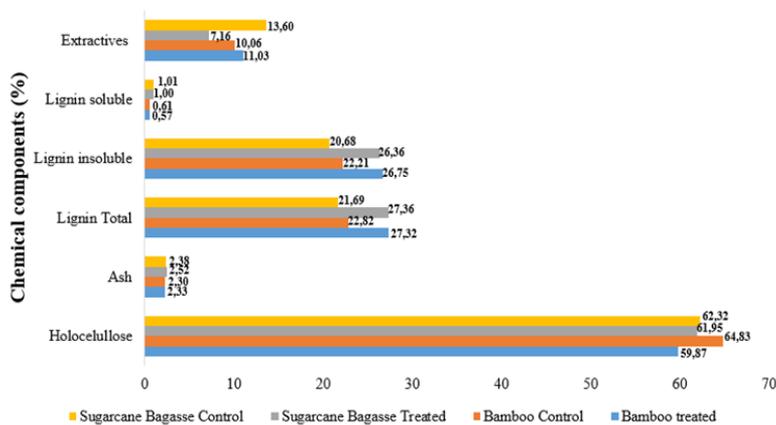
### Particle color change and chemical analysis

After the heat treatment it was found that the particles acquired a darker color (Figure 3).



**Figure 3:** (a) Control particles; (b) Treated particles (Brito 2018).

The chemical components of the bamboo particles and sugarcane bagasse are represented in the Figure 4.



**Figure 4:** Chemical components of bamboo particles and sugarcane bagasse.

### Particleboard surface wettability

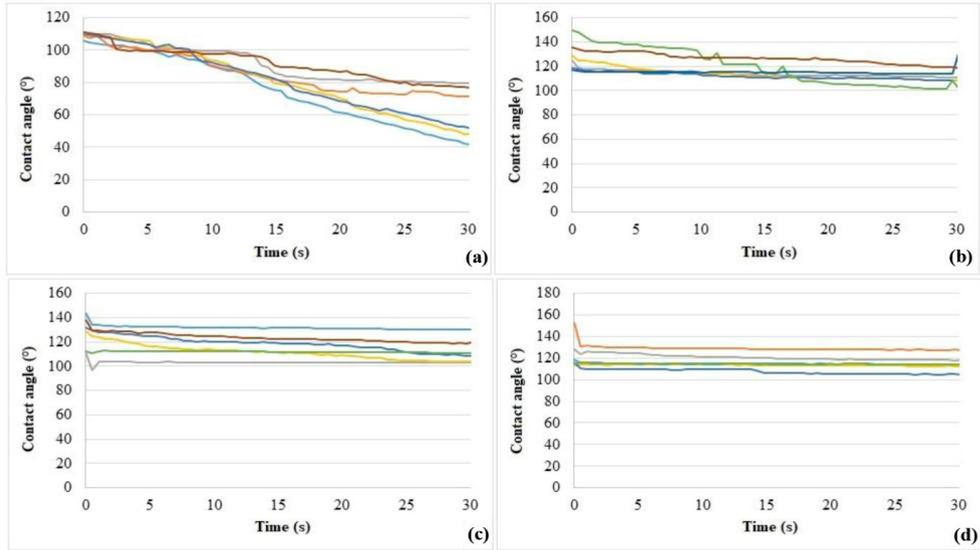
Table 1 describes the values obtained for the wetting angles of the particleboards. Figure 5 and Figure 6 represent the behavior of the contact angles measured in two stages (initial and final).

**Table 1:** Means values of contact angles obtained with water and ethyleneglycol. measured in two periods of particleboards for tested treatments.

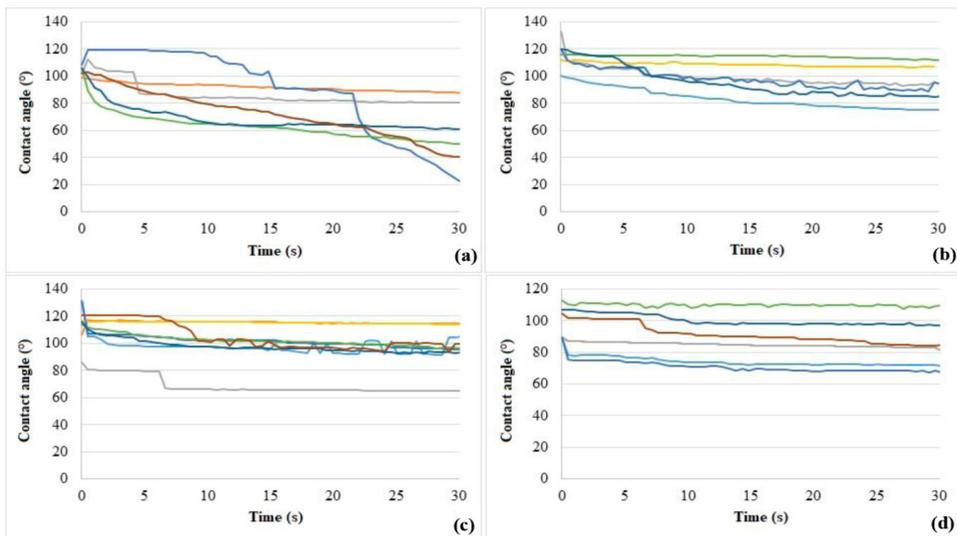
Treatments	Contact Angle ( $\theta^\circ$ )			
	Water		Ethylene glicol	
	Initial**	Last	Initial**	Last*
*T1	108,80 a (0,58)	83,59 b (4,32)	105,42 a (4,09)	81,69 a (16,60)
T2	123,42 a (10,94)	106,20 ab (16,09)	111,44 a (16,80)	93,51 a (12,49)
T3	132,07 a (18,54)	116,67 a (10,14)	116,04 a (7,92)	100,28 a (8,43)
T4	123,99 a (10,32)	116,28 a (5,40)	97,53 a (9,60)	85,70 a (11,94)
Overall mean	122,07	105,68	107,61	90,29
CV (%)	9,78	9,57	9,90	14,06

\* Data contained in Brito *et al.* (2020b), used as comparison in this article; T1 - Control/10% urea formaldehyde (UF); T2 - Thermal treated/ 10% UF; T3 - Thermal treated/ 12% UF; T4 - Thermal treated/14%UF. \*\*Do not differ (F test;  $p > 0,05$ ).

Means followed by the same letter in the column do not differ (Tukey test;  $p > 0,05$ ); Values in parentheses are the standard deviation; CV: Coefficient of variation.



**Figure 5:** Evolution of contact angles to water in function of time: (a) T1 - Control/10% urea formaldehyde - UF (Brito *et al.* 2020b); (b) T2 - Thermal treated/10 % UF; (c) T3 - Thermal treated/12% UF; (d) T4 - Thermal treated/14% UF.



**Figure 6:** Evolution of contact angles to ethylene glycol in function of time: (a) T1 - Control/10% urea formaldehyde - UF (Brito *et al.* 2020b); (b) T2 - Thermal treated/10% UF; (c) T3 - Thermal treated/12% UF; (d) T4 - Thermal treated/14% UF.

## Biological resistance of the particleboards

Table 2 shows the values obtained to resistance of particleboards to wood decay fungi.

**Table 2:** Mean values of mass loss of particleboards to wood decay fungi, and respective resistance classes of panels for tested treatments.

Treatments	Mass loss - ML (%) / Wood decay fungi					
	Brown rot				White rot	
	<i>Rhodonía placenta</i>		<i>Gloeophyllum trabeum</i>		<i>Trametes versicolor</i>	
	<sup>1</sup> ML (%)	<sup>2</sup> CR	ML (%)	CR	ML (%)	CR
* T1	4,43 a (1,05)	<sup>3</sup> HR	22,34 a (3,27)	<sup>4</sup> R	31,33 a (1,11)	<sup>5</sup> SR
T2	1,25 b (1,61)	HR	16,32 b (1,61)	R	24,57 b (0,43)	R
T3	1,28 b (0,69)	HR	8,34 c (2,42)	HR	21,11 c (0,51)	R
T4	1,38 b (0,70)	HR	8,73 c (0,14)	HR	20,69 c (0,18)	R
Overall mean	2,08	-	13,93	-	24,42	-
CV (%)	14,39	-	8,28	-	8,31	-

\* Data contained in Brito *et al.* (2020b), used as comparison in this article. T1 - Control/10% urea formaldehyde (UF); T2 - Thermal treated/10% UF; T3 - Thermal treated/12% UF; T4 - Thermal treated/14% UF. <sup>1</sup>ML: Mass loss; <sup>2</sup>CR: Class of Resistance (AWPA E-30, 2016); <sup>3</sup>HR: Highly resistant; <sup>4</sup>R: Resistant; <sup>5</sup>SR: Slightly resistant. CV: Coefficient of variation. Means followed by the same letter in the column do not differ (Tukey test;  $p > 0.05$ ); Values in parentheses are the standard deviation; CV: Coefficient of variation.

## DISCUSSION

### Particle color change and chemical analysis

The darkening acquired by the material after heat treatment can often be explained due to the formation of oxidation products and degradation of lignocellulosic compounds, especially those of lower molecular weight (Chen *et al.* 2012).

Some researchers have already worked with thermally modified particles from alternative materials such as sugarcane bagasse and bamboo, and noticed the color change after heat treatment; for example; Zhang *et al.* 2013, Zhang *et al.* 2017, Lee *et al.* (2018), Brito and Bortoletto Júnior (2019), and Brito *et al.* (2020a).

Regarding the chemical analysis, it is observed that the particleboard treatment 2 (T2) (Figure 4) obtained a lower average in relation to the total extractives content, with a reduction of 47,35 % in relation to the initial control content (T1).

For sugarcane bagasse particles treated with three temperatures (170 °C, 200 °C, and 230 °C), Ribeiro *et al.* (2020) found mean values of 24,18%, 22,20%, and 14,35 %, respectively, while they have obtained a value of 17,03 % for the control samples. The authors noted an increase in the amount of extractives up to 170 °C, but then the values were reduced. This fact corroborates with the present study, in which a temperature higher than 170 °C (220 °C) was used and the extractives content was reduced. All values were higher than those obtained in the present study.

For particleboards produced with sugarcane bagasse and *Pinus* spp. particles, Protásio *et al.* (2015) obtained 12,46 % of extractives for sugarcane bagasse, which is lower than that obtained for treatment 1 (Figure 4). Based on the Finnish Thermowood® Handbook (2003), there is an increase in the extractive levels when the heat treatment is carried out with temperatures < 180 °C, and can be reduced when they are close to 230 °C. This occurs due to the degradation of chemical components, mainly hemicelluloses, the modification of starch and the volatilization of volatile extracts.

However, for bamboo there was a tendency to increase the content of total extracts with the thermal treatment (T4). This observation corroborates the study by Brito *et al.* (2020a) who worked with *Dendrocalamus giganteus*, and observed an increase in the total extractive levels of bamboo with an increase in

temperature from 6,30% (100 °C) to 7,16% (140 °C), 7,84% (160 °C), 8,42% (180 °C), and 8,83% (200 °C). Výbohá *et al.* (2018) observed that 200 °C caused an increase in the extractives content regarding the effect of three temperatures (160 °C, 180 °C, and 200 °C) on the chemical composition of the wood. The researchers attributed this result to the release of lignin and saccharide degradation products in the extraction mixture or condensation reactions with the extracts originally present in the untreated wood.

Regarding the total lignin content, there was a tendency to increase the values for both materials (Figure 4) after the heat treatments (T2 and T4). The value obtained for the sugarcane bagasse control samples was higher than those reported by Ribeiro *et al.* (2020), who obtained control samples of 20,30%; and by Protásio *et al.* (2015) of 15,72%. Ribeiro *et al.* (2020) obtained average values of 27,25% (170 °C), 24,55% (200 °C), and 30,50% (230 °C) for heat treatments.

Regarding bamboo, Brito *et al.* (2020a) obtained 25,59% for the control samples, constituting a higher value than that obtained in the present study. The result obtained after the heat treatment (T4) was lower than that obtained by Brito *et al.* (2020a), who reported 28,01% (140 °C), 27,91% (160 °C), 31,45% (180 °C), and 28,59% (200 °C). This occurs because some by-products can be formed after the heat treatment resulting from the dehydration of polymers or the formation of new components (Inari *et al.* 2006). This is caused by the polycondensation reactions of the cell wall components, which results in the crosslinking process contributing to an apparent increase in the lignin content (Esteves and Pereira 2009, Ferreira 2014), which probably occurred with the particles subjected to heat treatment in the present research.

It is noted that the ash contents showed similar averages for all treatments (Figure 4). The values obtained in the present study for sugarcane bagasse were higher than those reported by Ribeiro *et al.* (2020) who obtained 0,59% for the control samples, and Protásio *et al.* (2015) who found 0,71%. Ribeiro *et al.* (2020) obtained 0,25% (170 °C), 0,38% (200 °C), and 0,59% (230 °C) after the heat treatment.

There was a tendency to reduce the holocellulose fraction content in the modified particles for both material, with the bamboo particles having a greater reduction (Figure 4). The value obtained for sugarcane bagasse (T1) was lower than those reported by Ribeiro *et al.* (2020) who obtained 63,12% for the control samples, and Protásio *et al.* (2015) with 71,11%. The value obtained after the heat treatment was higher than those found by Ribeiro *et al.* (2020) of 49,45% (170 °C), 52,45% (200 °C), and 55,60% (230 °C). Protásio *et al.* (2015) and Ribeiro *et al.* (2020) attributed this to a significant reduction in hemicelluloses, which is the fraction most sensitive to heat treatment.

Brito *et al.* (2020a) obtained an average value of 68,11% for bamboo, which is higher than the value obtained in the present study (T3). The average value obtained after the heat treatment was 59,87%, which is lower than the average values obtained by Brito *et al.* (2020a), who also observed a decrease in values of 68,83% (140 °C), 64,25% (160 °C), 60,23% (180 °C), and 62,88% (200 °C). For oil-treated bamboo at three temperatures (140 °C, 180 °C, and 220 °C), Salim *et al.* (2008) observed that the higher the temperature, the greater the mass loss of the holocellulose fraction.

According to Brito *et al.* (2006), hemicelluloses are the first affected in a heat treatment due to the reduction of xylose, arabinose, galactose, and mannose contents by the hydrolysis of acids. Degradation begins with deacetylation of hemicelluloses, followed by depolymerization of polysaccharides, catalyzed by the release of acetic acid. This statement corroborates the study by Ferreira (2014), who states that there is a reduction in the carbohydrates' mass during the heat treatment due to their greater sensitivity to thermal degradation, resulting in mass loss of the materials.

### Particleboard surface wettability

Based on the classification by Myers (1999) and according to the values of the initial angles obtained with water and ethylene glycol (Table 1), the surface of all particleboards was classified as "non-wettable" ( $\theta > 89^\circ$ ). The final angles allow classifying T1 (water and ethylene glycol - EG) and T4 (EG) as "partially wettable" ( $\theta < 89^\circ$ ), and treatments T2 and T3 as "non-wettable". It was found that there was only a significant difference for the final angle obtained with water.

There was a tendency to increase the contact angles (initial and final) obtained with water and EG (Table 1), indicating that the particleboards constituted with thermally modified particles and encased with 12% UF (T3) showed a reduction in the wettability of the material in relation to T1 control particleboards. However, an

improvement of 15,95° (initial) to 14,98° (final) in T4 was observed for EG in relation to T3. This may have a positive influence on the quality of protection products and finishing of the particleboards (paints), on the gluing of decorative sheets, on their union with pegs and on the absorption of protection products.

Zhang and Yu (2015) tested the contact angle for water, formamide, and diiodomethane for samples treated with bamboo with different heat treatment temperatures (100 °C, 140 °C, and 180 °C) for 4 h, in addition to a control sample, observing that those obtained with distilled water were higher after heat treatment. They noticed an increase from 49,74° (control) to 104,01° (treated at 180 °C). Also according to Zhang and Yu (2015), the degradation of chemical components may have influenced the availability of OH-free groups in bamboo and the crystallinity of microfibril amorphous regions. The cellulose crystallinity increased, resulting in the formation of internal hydrogen bonds and ethers in the cellulose, making the bamboo repellent to water.

Other researchers who worked with the wettability of particleboards made of treated wood elements also noticed an increase in surface hydrophobicity (Unsal *et al.* 2010, Unsal *et al.* 2011, Candan *et al.* 2012). According to Hakkou *et al.* (2005), the heat treatment reduces the wettability because it causes a reduction of the hydroxy groups (-OH) in the treated particles, which results in partial surface inactivation. This occurs because the temperature can modify the structure of the cell wall components, mainly of the hemicelluloses, causing a reduction in the wettability of the particleboards, in addition to causing component migration generated by the temperature increase to the surface of the particles.

According to Figure 5 and Figure 6, it appears that there was a reduction in the contact angle values for the T1 treatment, which indicates a reduction in the surface wettability of the particleboards, meaning that the solvent absorption rate in the T1 particleboards was faster and formed smaller angles than the T2, T3, and T4 treatments, which showed low variation of the angle values over time. This fact is explained by the thermal treatment of the particles used in manufacturing the particleboards which reduced the permeability of the material and conferred less surface wettability.

It is worth noting that a rapid absorption of solvents by particleboards (treatment 1 - T1) occurred during the experiment, and are characterized in Figure 5 and Figure 6 by a sharp drop in the lines (Figure 5a and Figure 6a). The particleboards manufactured with “*in natura*” material (T1) had greater porosity, being made up of particles which were not thermally modified, so they remained with the same anatomical arrangement, including the empty intercellular spaces and were manufactured with 10% adhesive, and therefore would have fewer particles covered by adhesive. It is believed that porosity substantially contributed to the decreases observed in the lines for T1.

Nurhazwani *et al.* (2016) performed a study with a hybrid particleboard made with bamboo (B) (*Dendrocalamus asper*) and rubber wood (R) (*Hevea brasilienses*) with a thickness of 12 mm and nominal density of 0,70 g/cm<sup>3</sup>, constituted with proportions of 100B:0R; 70B:30R; 50B:50R; 30B:70R and 0B:100R (control), glued with 12% UF-based adhesive with 65% solid content and 1% ammonium chloride as a catalyst and without paraffin, along with cold pressing of 3,5 MPa for ≈ 30 s, and hot pressing (pressure of 11,7 MPa, 160 °C for 6 min). These authors noted that porosity influenced wettability, in which bamboo particleboards had the smallest contact angles on the outer surface (greater porosity) with values from 0° to close to 35° when compared to the inner surface (lower porosity), where the angles varied between 0° and close to 60°.

Thus, in addition to the chemical composition of the surface and porosity, other factors influence wettability such as density and surface tension of the liquid (Rolleri and Roffael 2008).

### Biological resistance of the particleboards

According to AWWA E-30 (2016), the particleboards produced with 10% UF were highly resistant (HR) to *R. placenta*, as they showed mass losses below 10% and resistant (R) to *G. trabeum* (T1 and T2), as they showed mass loss between 11 and 24%, T3 and T4 treatments (12 and 14% UF) were HR (Table 2). All the particleboards composed of thermally modified particles were resistant (R) to *T. versicolor* (white rot).

For *R. placenta*, it is observed that the particleboards constituted with thermally modified particles and encased with 10% UF (T2) had a decrease in mass loss equivalent to 71,78% when compared to the control particleboards (T1), and all particleboards were classified as HR (Table 2). However, the heat treatment of the particles improved the biological resistance of the particleboards to *G. trabeum*. Those coming from T2

had a reduction of 26,95% in relation to the control particleboards (T1), which probably happened due to the decrease in the holocellulose content (Figure 4) and changes in other nutrients used for developing this type of fungus (brown rot).

According to Weiland and Guyonnet (2003), the increase in biological resistance may have been caused by modifying the starch and other simple sugars, the formation of new organic compounds (furfural), and cross-linking these with the remaining lignin chains. These factors make it difficult for the enzymatic system of fungi to recognize chemical components which made up nutrition sources, in addition to forming some types of toxic products which can function as fungicides. In addition, the reduction in hemicellulose content has many hygroscopic sites and reduces free hydroxyl groups (-OH), reducing the moisture acquired from the environment, resulting in increased resistance to the deterioration of heat-modified wood (Dubey *et al.* 2011, Li *et al.* 2016).

It was observed that there was an increase in the biological resistance of  $\pm 50\%$  of the particleboards constituted with 12% (T3) and 14% (T4) of UF in relation to the T2 particleboards. This caused a change in the resistance class of the particleboards, going from resistant to very resistant in relation to *G. trabeum*. Despite the degrading capacity of the strain used for *R. placenta*, which was confirmed by the colonization and consumption of *Pinus elliottii* wood used as feeder strips, it did not consume the tested particleboards.

Regarding *T. versicolor* (Table 2), it was observed that the T2 treatment particleboards had an increase in biological resistance of 21,58 % compared to the control particleboards (T1), resulting in a change from moderate to resistant in the resistance class. The T3 and T4 treatment particleboards differed from the T1 and T2 particleboards (10% UF), indicating that the increase in adhesive content provided an improvement in durability. The evaluated particleboards had less resistance to *T. versicolor* attack, which is capable of degrading all the macromolecular components of the lignocellulosic material.

The mass loss values obtained for *T. versicolor* (T1) were similar to those obtained by Belini *et al.* (2014) for particleboards manufactured with different percentages of sugarcane bagasse particles and *Eucalyptus grandis* fibers, and two urea formaldehyde resin percentages (13 and 16%) for the *Pleurotus ostreatus* white rot fungus. Furthermore, a mass loss of 32,2% (13% of adhesive content) and 36,4% (16%) was obtained for particleboards made up of 25% sugarcane bagasse particles and 75% wood particles. Adhesive contents have no influence on the durability of the particleboards.

Okino *et al.* (2007) conducted a study on the biological resistance of oriented strand board (OSB) glued with urea-formaldehyde (UF) and phenol-formaldehyde (FF), along with 5 and 8% of solids and wood content of *Pinus taeda*, *Eucalyptus grandis*, and *Cupressus glauca*, submitted to *G. trabeum* and *Lentinus* ( $\approx$  *Neolentinus*) *lepidus* (brown rot fungi) and *T. versicolor* and *Ganoderma applanatum* (white rot fungi). They observed that all OSBs had greater mass loss when exposed to *G. trabeum* with the exception for OSBs made from eucalypts wood. The increase in the resinous solids content generally provided greater biological resistance for the particleboards.

In a study by Souza *et al.* (2018), particleboards manufactured with rice husk (fresh or crushed in a hammer mill), glued with tannin-formaldehyde, compacted with different densities (0,65 g/cm<sup>3</sup>, 0,95 g/cm<sup>3</sup>, and 1,15 g/cm<sup>3</sup>) and adhesive contents (7%, 10%, and 13%) were classified as resistant to *T. versicolor* (white rot fungus) and moderately resistant to *G. trabeum* (brown rot fungus). The authors noted that the increase in the adhesive content (particleboards with crushed particles) resulted in increased resistance to *G. trabeum* attack. However, there was no effect of the adhesive content for those produced with natural bark. The increase in compaction and adhesive content generally provided gains in resistance to the deterioration of the particleboards by *G. trabeum*.

According to Souza *et al.* (2018), the worst situation should be considered in indicating the use of particleboards for safety reasons. Since it is not possible to predict which type of fungus will attack the parts in service, data regarding the one with the highest mass loss should be considered as an indicator of the durability of the evaluated material. Thus, the damage caused by *T. versicolor* must be considered in this study for indicating the use of the produced particleboards.

## CONCLUSIONS

The thermal treatment caused a reduction in the levels of total sugarcane bagasse extracts. increased the lignin levels in both materials and reduced the holocellulose fraction, mainly of bamboo.

The manufacturing condition of the particleboards only affected the final contact angle obtained with water. The thermal treatment contributed to increase the contact angles, interfering in the wettability; however, the amount of the adhesive content did not influence this property. The particleboards were classified as “non-wettable” and “partially wettable” in relation to the tested solvents.

The heat treatment of the particles and the increase in the adhesive content promoted improvements in the biological resistance of the particleboards, especially in relation to the fungi which caused greater consumption of materials (*G. trabeum* and *T. versicolor*). The particleboards were generally classified as “resistant” and “very resistant” in relation to these fungi.

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