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HYDROTHERMAL TREATMENT OF *EUCALYPTUS* STRAND PARTICLES FOR IMPROVEMENT OF ORIENTED STRAND BOARD (OSB)

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The aim of this study was to evaluate the effect of hydrothermal treatment on the chemical composition of *Eucalyptus* strand particles and on the physical and mechanical properties of oriented strand board (OSB) panels. Strand particles of *Eucalyptus* were pre-hydrolysed at 130, 150 and 170 °C for 7 and 21 minutes. The extractives, lignin, carbohydrates, pH, equilibrium moisture content, mass loss and contact angle were evaluated. The OSB panels were fabricated using a phenol-formaldehyde adhesive (8%), 0.7 g cm^{-3} nominal density and a pressing cycle with 170 °C, 3.14 MPa for 8 minutes. These panels were kept in a climate chamber at 20 ± 2 °C and 65 ± 3% relative humidity to determine their physical and mechanical properties. The hydrothermal treatment, mainly at 170 °C, degraded the hemicelluloses, especially galactans, xylans and arabians. The treatments at this temperature showed the best results for improving the dimensional stability, as well as reducing the moisture equilibrium content, water absorption and thickness swelling after 24 hours of immersion, without losing mechanical resistance. Therefore, hydrothermal treatment can be an important alternative for improving OSB panel quality.

Keywords: Dimensional stability, particleboard, physical properties, pre-hydrolysis, wood chemistry

INTRODUCTION

Oriented strand board (OSB) panels are commercially produced with the genus *Pinus*, with a wood density ranging from 0.3 to 0.5 g cm^{-3} (Mendes et al. 2013a, Sanches et al. 2016, Delucis et al. 2017). Panel production demands timber with up to 0.55 g cm^{-3} density (Moslemi, 1974). Many fast-growing *Eucalyptus* species with high productivity can also be used to increase the raw material supply and substitute *Pinus* spp. (Bufalino et. al. 2015).

The panel's capacity of absorption and desorption of moisture from wood polysaccharides when exposed to relative humidity causes changes in its volume. Cellulose and hemicellulose absorb water through hydrophilic sites which connect with water through hydrogen bonds. The hemicelluloses are more thermally unstable and reducing these polysaccharides in wood may increase their dimensional stability (Kok and Ozgur 2017, Korosec et al. 2017).

The search for alternatives to improve the dimensional stability of reconstituted wood products is mainly based on pre- and post-production heat treatment (Del-Menezzi and Tomaselli 2006, Mendes et al. 2013b, Carvalho et al. 2015, 2018).

Pre-hydrolysis is a hydrothermal process with liquid heated water under pressure, used to hydrolyse hemicelluloses, which are easier to hydrolyse then cellulose, with a selective removal of these structures (Ruiz et. al. 2013). Acetyl groups and uronic acids resulting from hydrolysis, originally present in the hemicelluloses, catalyse the hydrolysis of those hemicelluloses (Yu et al. 2010). Hydrothermal pre-treatment in pulping process has been studied with a variety of raw materials, such as *Pinus*, *Eucalyptus*, sugarcane bagasse and *Leucaena* (Chirat et al. 2012, Feria et al. 2012, Saukkonen et al. 2012, Andrade and Colodette 2014). However, there are few studies using this treatment in the production of wood panels.

The hydrothermal process of pre-hydrolysis has potential for improving OSB dimensional

stability. Therefore, the objective of this study was to evaluate the hydrothermal treatment of *Eucalyptus* strand particles and their effects on OSB´**s** physical and mechanical properties.

MATERIALS AND METHODS

Particle extraction and hydrothermal treatment

Three *Eucalyptus* trees were harvested and their logs were sawn to obtain particles which were subjected to hydrothermal treatment. Logs were sawn into 9 cm lengths with wood grain, and 2.3 cm thickness. These pieces were submerged in water until complete saturation and processed in a wood chipper with blades adjusted to generate thickness of around 0.30 mm to obtain particle strands of 90 mm length × 23 mm width × 0.30 mm to thickness.

The particles were subjected to air-drying until they reached equilibrium moisture content. Then, they were subjected to hydrothermal treatment at 130, 150 and 170 °C with 0.228, 0.448 and 0.862 MPa pressure, respectively, in a Parr reactor with an 18.75 L capacity and a water-wood ratio of 8:1.5. The heating rate was 1.71 °C min^{-1} for seven and twenty-one minutes.

The pre-hydrolysed particles were washed in running water to remove potential extractives on their surface, and then dried at 25 °C until equilibrium of moisture content. The pre-hydrolysis yield was determined by equation 1:

$$ML(\%) = \left(\frac{(Mi-Mf)}{Mi}\right) * 100 \quad (1)$$

where ML (%) is the weight loss, and Mi and Mf are the mass of particles before and after the hydrothermal treatment, respectively.

Wood particles were conditioned in a climatic chamber at 20 ± 2 °C and relative humidity of 65 ± 3% until constant mass to determine the equilibrium moisture content, as shown in equation 2:

$$EMC\ (\%) = \left(\frac{(Mw-Md)}{Md}\right) * 100 \quad (2)$$

where EMC (%) is the equilibrium moisture content of the samples, and Mw and Md are the wet and oven-dried particle mass, respectively.

Wood chemical composition

Wood particles were transformed into sawdust in a laboratory mill. The portion which passed through a 40-mesh sieve but retained by a 60-mesh sieve was used. The total extractives content, carbohydrate content and the soluble and insoluble lignin contents were determined as per Kymalainen et al. (2017), with adaptations.

Contact angle, pH and wood buffer capacity

The contact angle of the phenol-formaldehyde resin with hydrothermally treated particle surface was obtained after adding 5 µL phenol-formaldehyde resin to the particles. The reading was carried out five seconds after mixing the resin and the wood particles, using a goniometer connected to a digital camera and a computer.

The pH was determined using the same sample as in the chemical characterisation. A total of 15 g of dry sawdust from each treatment and 150 mL distilled water at 100 °C were added into a beaker. This mixture was maintained for 30 minutes while stirring. Then the mixture was filtered in a funnel (porosity 2), and a 50 mL aliquot with the resulting liquid was analysed in a digital pH meter.

The buffer capacity was obtained by measuring the pH of the pure extract and the subsequent addition of sodium hydroxide solution (NaOH) at 0.025 molar concentration. This alkaline solution was added until the sawdust solution reached pH 7. The buffer capacity in mmol L^{-1} was calculated according to the alkali quantity used.

Panel production and characterisation

Strand particles were dried in an oven with forced air circulation until the moisture level reached 3% (dry matter basis of the particles). The OSB panels with a nominal density of 0.70 g cm^{-3} were produced for each treatment. Phenol-formaldehyde resin was used with viscosity 943 cP, solids content of 53.4%, gel time at 170 °C of 64.3 s and pH 10.7. A total of 8% resin of particle mass (dry basis) was sprayed using a rotary drum-type glue blender. After resin spraying, particles were placed in a 40 × 40 × 1 cm forming box to perform strand orientation in three layers (25% + 50% + 25% of particles

total mass). Subsequently, the panel was pressed at 170 °C for eight minutes at pressure 3.14 MPa. Then, the OSB panels were conditioned to 65% relative humidity at 23 °C until constant mass. Two centimeters were cut from each side to avoid edge effects.

Density, moisture (dry basis), thickness swelling and internal bond was determined according to the European committee for standardisation (EN 323 1993, EN 322 1993, EN 317 1993 and EN 319 1993) standards respectively, while water absorption, modulus of elasticity and modulus of rupture in static bending were according to EN 310 2006 standard. The results were compared with the requirements of the EN 300 2006 standard.

Data statistical analysis

Data were submitted to the Lilliefors test for normality, and to the Cochran test for homogeneity of variances. The experiment was analysed according to a completely randomised design. Data were subjected to variance analysis by F test, and the means were compared using Scott-Knott test at 5% significance level.

RESULTS

The content of extractives and insoluble, soluble and total lignin, as well as polysaccharides of both the control (untreated wood chips) and the treated particles are presented in Table 1.

The hydrothermal treatment at 130 °C reduced the extractives in wood particles, but increased at 150 °C and reached the highest values at 170 °C. The insoluble lignin content remained constant, while the soluble lignin decreased with the treatment. Total lignin content remained unaltered by hydrothermal treatment.

Among the treatments, the galactan and xylan levels were lowered with hydrothermal treatment above 130 °C. The arabinans, galactans and xylans showed their lowest values at 170 °C, and the arabinans had similar values in treatments at 150 and 170 °C. *Eucalyptus* wood contains mannans (Gírio et al. 2010), but the equipment did not detect this sugar. The 170 °C treatment for 21 minutes caused a decrease of 28.9, 62.1 and 62.2% in arabinan, galactan and xylan contents in the particles, respectively, compared to control.

The average values of equilibrium moisture content (EMC), mass loss, bulk density, pH variation of *Eucalyptus* particles and buffering capacity are shown in Table 2.

Heating the wood particles to 170 °C for seven and twenty-one minutes reduced the EMC in the particles by 25.8 and 26.6%, respectively, while heating to 150 °C reduced it by 10.2 and 18.8%, respectively. The treatment at 130 °C did not affect the particle EMC. The sugar degradation of hemicelluloses (Table 1) reduced the particle hygroscopicity due to the availability of hydroxyl groups to make hydrogen bonds with water molecules.

Table 1 Chemical composition of *Eucalyptus* particles as a function of temperature and exposure time to hydrothermal treatments

%	Control	T1	T2	T3	T4	T5	T6
Total extractives	2.22^{b}	1.61^{a}	1.77^{a}	4.38^{d}	3.13^{c}	8.97^{e}	8.77^{e}
Insoluble lignin	27.73^{a}	27.83^{a}	27.27^{a}	26.93^{a}	26.66^{a}	28.49^{a}	27.90^{a}
Soluble lignin	2.17^{c}	1.97^{c}	1.98^{c}	1.68^{c}	1.90^{c}	1.25^{b}	1.08^{a}
Total lignin	29.9^{a}	29.8^{a}	29.25^{a}	28.61^{a}	28.56^{a}	29.74^{a}	28.98^{a}
Arabinans	0.38^{a}	0.34^{a}	0.34^{a}	0.22^{b}	0.23^{b}	0.24^{b}	0.27^{b}
Galactans	0.66^{a}	0.54^{c}	0.66^{a}	0.60^{b}	0.59^{b}	0.33^{d}	0.25^{e}
Glucans	43.64^{d}	42.45^{e}	42.23^{e}	44.24^{d}	46.10^{c}	52.11^{b}	55.82^{a}
Xylans	14.48^{a}	13.12^{b}	12.70^{b}	11.73^{c}	12.92^{b}	7.01^{d}	5.47^{e}
Mannans	ND	ND	ND	ND	ND	ND	ND

Means followed by the same letters per row do not differ at 95% probability level by Scott Knott test; T1 = 130 °C - 7 min, T2 = 130 °C - 21 min, T3 = 150 °C - 7 min, T4 = 150 °C - 21 min, T5 = 170 °C - 7 min, T6 = 170 °C - 21 min

Table 2 Weight loss, equilibrium moisture content, pH, buffer capacity and contact angle of pine particles as a function of temperature and exposure time to hydrothermal treatment

Treatments	Weight loss (%)	EMC (%)	pH	Buffer capacity (mmol L^{-1})	Contact angle
Control	-	12.8[a]	4.53[a]	0.0680[a]	120.7[a]
130°C - 07 min	3.7[a]	11.9[a]	4.08[b]	0.0693[a]	126.9[b]
130°C - 21 min	4.5[a]	12.0[a]	4.07[b]	0.0717[a]	130.4[c]
150°C - 07 min	6.3[b]	11.5[b]	3.39[c]	0.1361[b]	132.2[c]
150°C - 21 min	7.9[b]	10.4[b]	3.37[c]	0.1434[b]	127.4[b]
170°C - 07 min	13.5[c]	9.5[c]	3.09[d]	0.2503[c]	118.7[a]
170°C - 21 min	16.3[c]	9.2[c]	3.08[d]	0.2420[c]	115.7[a]

Means followed by the same letters do not differ at 95% probability level by Scott Knott test; EMC = equilibrium moisture content

The mass loss was highest at 170 °C, and the degradation of wood constituents, mainly hemicelluloses, was greater at higher temperatures which resulted in greater wood mass losses.

The *Eucalyptus* wood pH was 4.53, which decreased with hydrothermal treatment as the temperature increased. The treatment period per temperature did not affect the pH values.

The hydrothermal treatment at temperatures 130 and 150 °C increased the contact angle between *Eucalyptus* particle surfaces and phenol formaldehyde adhesive, in relation to the control. The wetting properties of the adhesive on particle surface decreased due to lower surface area at the given volume. The treatment at 170 °C did not influence the adhesive wettability of the particles.

Characterisation of oriented stand board (OSB) panels

Physical properties of OSB panels

The average values of density, EMC, water absorption (WA) and thickness swelling (TS), after 24 hours of immersion of the OSB in water, are shown in Figure 1

The panels produced with particles treated at 170 °C were included in the category of high-density panels (over 0.80 g cm^{-3}), while the others were classified as medium-density panels, with values between 0.64 and 0.80 g cm^{-3} (American National Standard 1993).

The 170 °C temperature decreased EMC by 18.9% (seven minutes) and 18.2% (21 minutes) compared to control panel. The water absorption values were lowered for panels produced with particles treated at 170 °C (Figure 1). The TS24h panels, containing particles treated hydrothermally at 170 °C for 21 minutes was lowest, while the 130 °C treatment for 21 minutes, and the 150 °C treatment for seven minutes increased TS24h.

Mechanical properties of oriented strand board (OSB)

Average values of mechanical properties, modules of rupture and bending elasticity of OSB are presented in Figure 2.

The panels treated at 150 and 170 °C for 21 minutes had the highest density and modulus of elasticity in perpendicular directions (MOE//).

The modulus of elasticity in parallel directions (MOE⊥) and modulus of rupture in perpendicular directions (MOR//) were not affected by the treatments, and their values in all the treatments were the same as control (untreated wood chips). The panels produced with wood particles hydrothermally treated at 150 °C for seven minutes showed lower values of modulus of rupture in parallel directions (MOR⊥) than control. The MOE and MOR static bending did not show a tendency to increase or decrease with the treatment intensity.

There was no appreciable effect of hydrothermal treatment on the internal bond of the panels. Control had 0.47 MPa, while the treatments were 0.42 MPa (130 °C/7 min), 0.39 MPa (130 °C/21 min), 0.38 MPa (150 °C/7 min), 0.40 MPa (150 °C/21 min), 0.52 MPa (170 °C/7 min) and 0.43 MPa (170 °C/21 min).

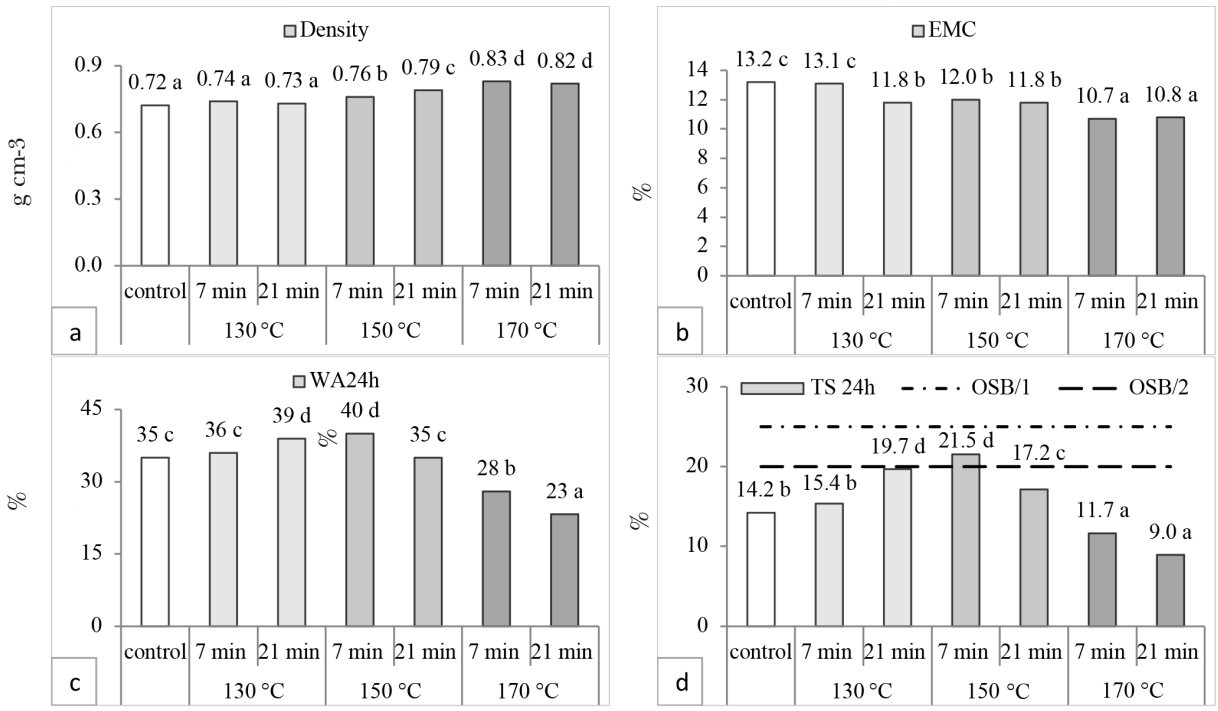

Figure 1 Apparent density (a), equilibrium moisture content (b), water absorption (c), and thickness swelling (d) after 24-hour immersion of the OSB panels following hydrothermal treatment; means followed by the same letters in the column do not differ at 95% probability level by the Scott Knott test; EMC = equilibrium moisture content, WA = water absorption, TS = thickness swelling

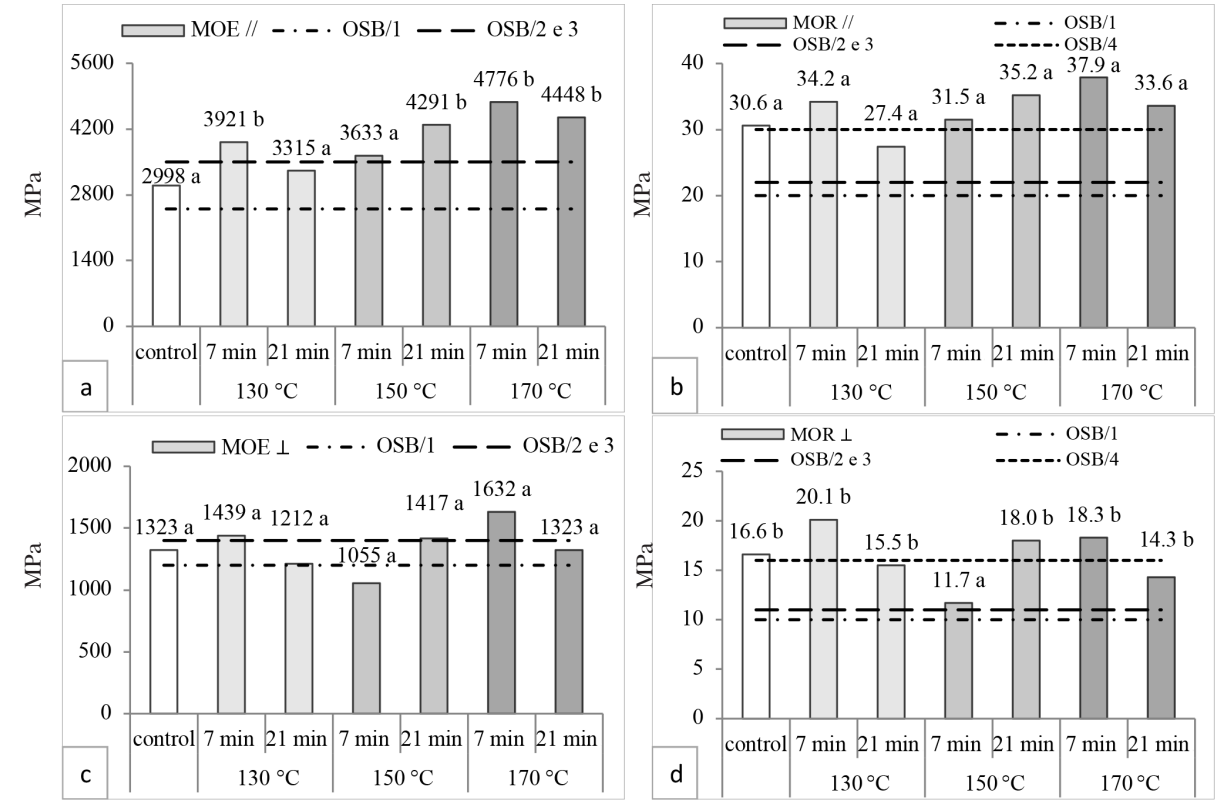

Figure 2 Parallel elasticity modulus (a), parallel rupture modulus (b), perpendicular elasticity modulus (c) and perpendicular rupture modulus (d) as functions of temperature and period of hydrothermal treatment; means followed by the same letters in the column do not differ at 95% probability level by Scott Knott test

DISCUSSION

The increase in extractive content in hydrothermal treatmenst of 150 and 170 °C (Table 1) is due to degradation of hemicelluloses (Brito et al. 2008; Esteves and Pereira 2009). Hemicelluloses are polysaccharides which are thermally unstable, and when submitted to thermal treatment, they are degraded (Esteves et al. 2013). Once submitted to hydrothermal processing, hemicellulose depolymerisation occurs, forming soluble compounds such as oligosaccharides, monosaccharides, sugar-decomposition products and acetic acid (Gullón et al. 2010). Zanuncio et al. (2014) also reported the increase of extractive content in heat-treated *Eucalyptus grandis* wood at 200 and 230 °C.

Reduction in the sugars composing the hemicellulose structure (Table 2) can be explained by the fact that hemicelluloses are less tolerant to heat because their ramifications are easily removed from the main chain, degrading into volatile compounds (Wang et. al. 2017). The glucan content, as a component of cellulose, has greater thermal stability compared to other carbohydrates (He et al. 2019), increasing its percentage when treated at 150 and 170 °C for 21 minutes (Table 1).

Altering the treatment period at the same temperature did not affect mass losses or EMC, but altering the temperature affected mass loss and reduced EMC, with major differences occurring in the particles between treatments at 150 and 170 °C (Table 2).

The pH reduction was attributed to the formation of carbonic acid, acetic acid (mainly because of deacetylation during the hydrolysis of polyose), and acetyl groups (Tjeerdsma and Militz 2005, Gullón et al. 2010).

The wood buffering capacity showed its ability to resist pH changes in the environment. The temperatures of 150 and 170 °C increased the wood chip buffering capacity. Phenol formaldehyde adhesives cure faster in alkaline pH, increasing the buffering capacity of the particles in acidic pH, influencing the adhesive curing speed and decreasing the curing time.

The contact angle did not show a sequential increase as a function of increasing temperature, with higher contact angles at higher temperatures. The contact angle of heat-treated *Eucalyptus cloeziana* wood in distilled water showed an increase, confirming that wood heat treatment reduces its hygroscopicity (Cardematori et al. 2013). The authors also did not observe a sequence of increasing contact angles as a function of increasing temperature, with higher contact angles at temperatures of 180 and 200 °C than at 220 and 240 °C.

Characterisation of panels

Physical properties of oriented stand board (OSB) panels

The density of the panels produced with particles treated at 150 and 170 °C was higher than control and those treated at 130 °C.

Lower thickness leads to lower panel volume, and the panel density is determined by the ratio between mass and volume. Thus, it is possible to infer that the increased density of panels produced with pre-hydrolysed particles was mainly based on their lesser thickness.

In another study, a reduction in the thickness of the panels was observed in OSB heat-treated at 250 °C for 4.7 and 10 minutes, but mass losses of the panel corresponding to increase in density was not observed (Del-Menezzi and Tomaselli 2006). The lack of effect of mass loss on panel density was due to the application of hydrothermal treatment to particles before constructing the panel. This explains the increase in density of the panels after hydrothermal treatment, and a decrease in the panel thickness.

The panel EMC followed the same trend as the strands, with temperature of 170 °C being the most efficient for reducing this parameter. The water absorption values were lower for the panels produced with particles treated at 170 °C (Figure 1).

The hydrothermal treatment at temperature 170 °C was the most efficient for improving dimensional stability of OSB. However, the WA24h and TS24h (water absorption and thickness swelling after 24 h of OSB immersion in water) did not show a tendency to increase with treatment temperature or time. In a similar fashion, thermally treated strand particles of pine at 200 and 240 °C did not display a direct corelation between treatment temperature and OSB dimensional stability, with an increase in thickness swelling values at 200 °C and a reduction at 240 °C (Mendes et al. 2013b).

The results were related because hydrothermal treatment degraded the hemicelluloses (Table 1) and hydrophilic sites of the particles, resulting in lower panel EMC, WA and TS.

The panels containing particles treated at 170 °C for seven minutes reached minimum TS value stipulated for OSB type 4 (TS24h < 12%). The chips treated at 170 °C for both seven and twenty-one minutes, as well as control, met the minimum TS value required for OSB type 3 (TS24h < 15%). However the particles treated at 150 °C for seven minutes did not meet the minimum TS value for OSB type 2 (TS24h < 20%). All panels attended the minimum TS value stipulated by the relevant standard for OSB type 1 (TS24h < 25%) (EN 300 2006).

Mechanical properties of oriented stand board (OSB)

All treatments attended the minimum value stipulated for MOE// by the European norm EN 300 (2006) for OSB type 1, with a minimum of 2500 MPa for this property. The panels produced with particles treated at 130, 150 and 170 °C for seven minutes also met the standard MOE// requirement for OSB type 2 and 3, with a minimum of 3500 MPa.

All panels attendeded the minimum value stipulated for MOR// by EN 300 (2006) for OSB types 1, 2 and 3, where the requirement is 20, 22 and 22 MPa, respectively. The panels containing particles treated at 130 °C for 21 minutes did not meet the standard for OSB type 4, which requires at least 30 MPa.

Panels produced with particles treated at 150 °C for seven minutes also did not meet the requirements for MOE⊥ of the European standard for OSB type 1 (EN300 2006), a minimum of 1200 MPa. The panels containing particles treated at 130 °C for seven minutes, 170 °C for seven minutes and 150 °C for 21 minutes met the minimum MOE⊥ values for OSB panels type 2 and 3 (1400 MPa).

All panels attend the European standard requirements for OSB type 1, 2 and 3 (EN300 2006) for MOR⊥. The control OSB panels and those with particles treated at 130 °C for seven minutes, 170 °C for seven minutes and 150 °C for 21 minutes reached the minimum value stipulated for type 4 OSB panels.

All panels met the minimum value established for internal bonding by the EN 300 (2006) standard for type 1, 2 and 3 OSB panels, with values higher than 0.3, 0.34 and 0.34 MPa, respectively.

CONCLUSIONS

Hydrothermal treatment altered the chemical composition of wood particles. The increase in temperature, mainly at 170 °C, degraded the hemicelluloses, reducing the galactan, xylan and arabinan contents, resulting in a loss of particle density. The degradation of these components reduced the wood´s adsorption capacity and, therefore, the OSB panels submitted to the hydrothermal treatment had lower equilibrium moisture content and swelling. In addition, panels treated at higher temperatures maintained their mechanical resistance, due to their higher densities, which contributed to this resistance.

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