# Effect of Process Parameters in the Thermomechanical Densification of *Pinus elliottii* and *Eucalyptus grandis* Fast-growing Wood

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Densification parameters were investigated for the fast-growing pine and eucalyptus. Both woods showed optimal results in terms of apparent density and mechanical properties when milder treatments of 150 °C were applied. Pine showed mass loss and improved mechanical properties with a longer heating time of 60 min, while eucalypt performed better with shorter treatments of 30 min. Eucalypt has more highly acetylated hemicelluloses, mainly composed of xylose units, which degrade more quickly with consequent decrease in mass and mechanical properties. However, apparent densities close to 1.0 $g \cdot cm^{-3}$ were obtained, and greatly enhanced bending properties, hardness, and impact resistance were observed, especially when the optimal parameters were used. Treatments at 170 °C or greater, while resulting in well-densified specimens, yielded inferior mechanical properties. The densified woods also presented initial apparent contact angles greater than 85°, highlighting a considerable increase of hydrophobicity. The densification process therefore allows these less valuable timber species to be used in applications such as flooring and decking.

*Keywords: Thermal modification; Plastification; Wood resistance; Physical properties; Hydrophobization.*

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## INTRODUCTION

Wood densification has been studied to improve some of the material's properties, including density, mechanical resistance, and wettability. Results of such work have been reported by several researchers (Welzbacher *et al*. 2008; Belt *et al*. 2013). Mainly, two distinct processes can be undertaken for wood densification. One is characterized by the filling of empty spaces in the material with fluids. The other aims for radial compression of wood by applying mechanical force and heat; therefore, it is defined as thermomechanical treatment, with the product sometimes termed "staypak" (Welzbacher *et al*. 2008).

These thermomechanical treatments are possible due to wood's porous structure and the ability of its components, especially lignin and hemicellulose, to soften in the presence of heat and humidity (Schaffer 1973; Rowell 2005). Because humidity and heat act as plasticizing agents, resulting in a more malleable wood, it is possible to apply

compression without any structural breakage (Seborg and Stamm 1941; Kutnar and Kamke 2012). Among several factors, the results of the thermomechanical treatments depend principally on temperature and pressure. The use of elevated temperatures tends to increase the resulting dimensional stability and reduce mechanical properties, while high pressure loads tend to crush the samples and also can reduce their mechanical properties (Kamke and Sizemore 2008). Besides, wood densification causes an increase in density, so stiffness and bending strength for example are improved compare to non-densified wood (Gaff *et al*. 2017a; Gaff *et al*. 2017b).

Heat treatment generally also provides a darkening of the wood surface, mainly due to chemical modifications of wood's constituents and migration of the extractives (Bekhta and Niemz 2005; Cademartori *et al*. 2014). Many studies have reported the decomposition of a large part of hemicelluloses after applied heat treatment on wood. Heat-treated wood generally swells less and presents improvements in dimensional stability; however strength might decrease (Rowell *et al*. 2009).

It has been more than a century (Sears 1900; Olesheimer 1929; Brossman 1931) since wood densification was first reported, demonstrating long-standing awareness of the great value of the technique. Starting from those studies, it is known that densification leads to an increase in the wood's mechanical resistance, especially in hardness, which can be enhanced by over 100%; consequently, this treatment can be beneficial for wood applications in coatings and decks (Fang *et al*. 2012; Fu *et al*. 2016; Laine *et al*. 2016).

It is well known that the mechanical properties of wood are strongly dependent on its density, and therefore fast-growing woods are usually not suitable for structural purposes (Popescu *et al*. 2014; Pelit *et al*. 2017). However, the increase in industrial demand for high quality materials justifies investment into wood densification. Improving the performances of pines and eucalypt wood, indeed, will be a winning point for the Brazilian forestry sector because in 2015 the area panted with these species reached 7.2 million hectares (IBÁ 2016).

North America and Europe are already considering wood densification processes as a good method for enhancing the properties for their lower density timber species. Conversely, more limited interest in the subject was shown in South America, where the process was less studied for the local species and also was not industrially exploited. Therefore, the focus of the present study was to evaluate the effect of the thermomechanical treatments on fast-growing timbers such as *Eucalyptus grandis* and *Pinus elliottii* by analyzing the variation of physical, mechanical and hygroscopic properties resulting from differences in time and temperature applied for the densification.

## EXPERIMENTAL

### Materials

*Selection and sample fabrication*

The materials originated from homogeneous populations of *Pinus elliottii* and *Eucalyptus grandis*, which were 22- and 21-years-old, respectively and located in the municipality of Santa Maria – RS, Brazil (29° 43' 1.95" S, 53° 43' 33.7" W, 110 m above sea level). Five trees were selected in accordance with the American Society for Testing and Materials standard ASTM D5536-94 (2004). All of the samples were prepared from the same log (3 m in length from the tree base), with the cutting carried out in such a way as to avoid sapwood and defects in the samples (Missio *et al*. 2016).

The boards were conditioned in a climate chamber (20°C, 65% RH) for one week, and then samples of 40 cm × 7.0 cm × 3.5 cm (longitudinal, tangential, and radial) were cut to undergo the thermomechanical treatments. The initial densities of the two wood species were 0.53 g·cm$^{-3}$ ± 0.03 g·cm$^{-3}$ for pine and 0.70 g·cm$^{-3}$ ± 0.06 g·cm$^{-3}$ for eucalypt.

*Thermomechanical treatments*

The eucalyptus and pine samples were submerged in boiling water at 100 °C for 20 min (Fig. 1). This pretreatment was used to soften the samples, similar to vaporization for drying and reduction of defects caused by the pressing process. Afterwards, the samples were briefly blotted with absorbing paper and then exposed in the radial direction to the thermomechanical process in an hydraulic press (Omeco, Brazilian Industry, Curitiba, Brazil), with a capacity of 100 tons and an electric heating system.

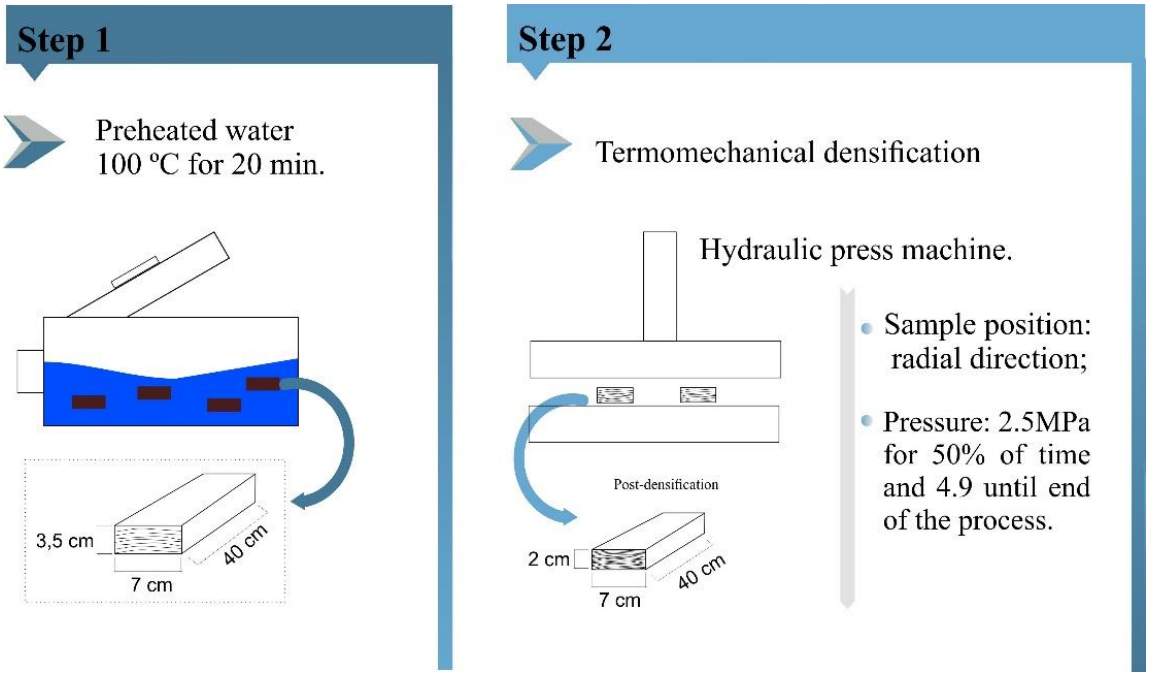



**Fig. 1.** Thermomechanical densification scheme for wood samples of *Eucalyptus grandis* and *Pinus elliottii*

The specific temperatures for each treatment are presented in Table 1. The applied pressure schedule was 2.5 MPa for 50% of the time and then 4.9 MPa until the end of the process according to findings from the authors' previous study (Arruda and Del Menezzi 2016).

The process was concluded by reducing the pressure to minimal values, just to restrain the wood samples to hydraulic press plates, and the heating system cooled at rates of 3.24, 3.35, and 3.53 ºC·min$^{-1}$ for each treatment 150 ºC, 170 ºC, and 190 ºC, respectively. The densified samples were kept inside the press until a temperature of 100 °C was reached and were then conditioned at 20 °C and 65% relative humidity. The thickness after densification process was 2 cm (Fig. 1). The samples then were cut according to Fig. 2.

**Table 1.** Parameters of Thermomechanical Treatments on *Eucalyptus grandis* and *Pinus elliottii* Wood

| Species | Treatment | Temperature (°C) | Time (min) |
|---|---|---|---|
| *Eucalyptus grandis* | Control | - | - |
| | E150-1 | 150 | 30 |
| | E150-2 | | 60 |
| | E170-1 | 170 | 30 |
| | E170-2 | | 60 |
| | E190-1 | 190 | 30 |
| | E190-2 | | 60 |
| *Pinus elliottii* | Control | - | - |
| | P150-1 | 150 | 30 |
| | P150-2 | | 60 |
| | P170-1 | 170 | 30 |
| | P170-2 | | 60 |
| | P190-1 | 190 | 30 |
| | P190-2 | | 60 |

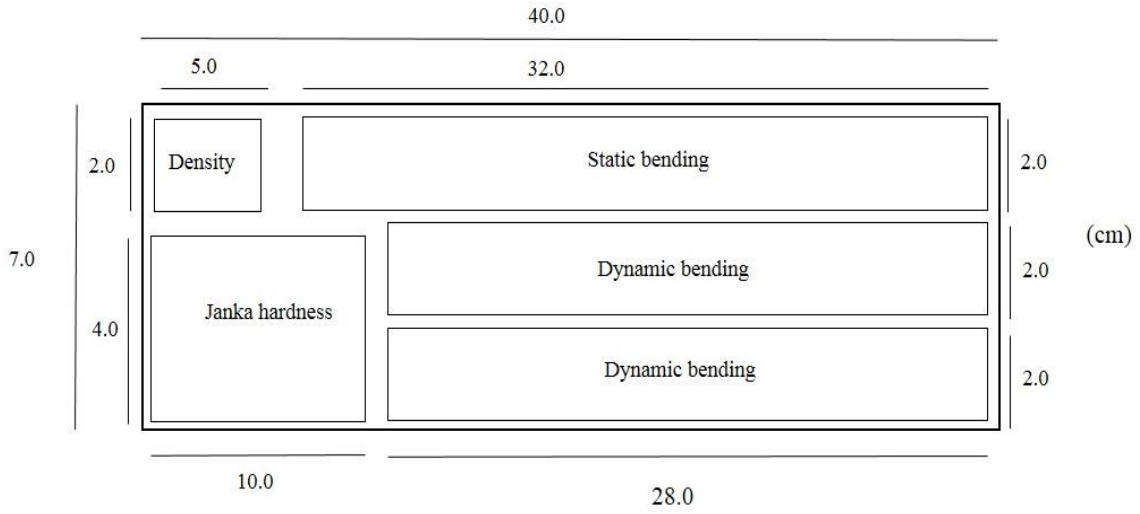



**Fig. 2.** Sample dimension scheme (in cm) for physical and mechanical properties analysis

## Methods

*Physical analysis*

The compression degree ($C_d$) was calculated starting from the relation between the initial and final thicknesses of the wooden samples (Eq. 1). The mass loss (*ML*) was calculated using the samples' mass variation from before to after densification (Eq. 2). The apparent density ($\rho_{ap}$) was determined after the conditioning of the samples (Eq. 3),

$$C_d = \frac{T_i - T_f}{T_f} \cdot 100 \quad (1)$$

$$ML = \frac{M_{BH} - M_{AH}}{M_{BH}} \cdot 100 \quad (2)$$

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

where $C_d$ is the compression degree (%), $T_i$ is the initial thickness (mm), $T_f$ is the final thickness (mm), $ML$ is the mass loss (%), $M_{BH}$ is the sample dry mass before densification (g), $M_{AH}$ is the sample dry mass after densification (g), $\rho_{ap}$ is the apparent density of the samples (g/cm$^3$), $M_{clim}$ is the conditioned sample mass (g), and $V_{clim}$ is the conditioned sample volume (cm$^3$).

*Mechanical analysis*

Static bending tests were performed following the ASTM D143-94 (2000) standard on 12 wood samples for each treatment, measuring 32 cm × 2 cm × 2 cm (longitudinal, radial, and tangential). A universal testing machine (EMIC®, DL2000/1000, Equipamentos e Sistemas de Ensaio Ltda, São José dos Pinhais, Brazil) with a capacity of 30 kN and a loading cell of 5000 N was employed at a rate of 1.04 mm/min to obtain the modulus of elasticity (MOE) and the modulus of rupture (MOR).

The impact resistance test was conducted respecting the NBR 7190 (1997) standard, by means of a Charpy pendulum (PW 15/10, Wolpert®, Ludwigshafen am Rhein, Germany). The test consists of the pendulum falling from a height of 1 m directly onto the sample, with dimensions 28 cm × 2 cm × 2 cm (longitudinal, radial, and tangential), placed into an opening of 24 cm (Pertuzzatti *et al.* 2017). The absorbed work ($W$) and the maximum resistance to impact ($F_{max}$) were obtained through Eq. 4,

$$F_{max} = \frac{1000 \cdot W}{b \cdot h} \quad (4)$$

where $F_{max}$ is the maximum resistance to impact (kJ·m$^{-2}$), $W$ is the absorbed work (J), and $b$ and $h$ are the transverse sample dimensions (mm).

The Janka hardness test was performed with the aid of a universal testing machine (EMIC®, DL2000/1000, Equipamentos e Sistemas de Ensaio Ltda, São José dos Pinhais, Brazil). Two measurements on the compression direction (radial) were recorded. The test comprised the insertion of a sphere with 1 cm$^2$ of cross-sectional area into the samples at a 6 mm·min$^{-1}$ rate.

*Wettability*

To evaluate the wood surface wettability, the apparent contact angle (ACA) was recorded using a DataPhysics OCA goniometer (DSA 25, Krüss, Hamburg, Germany) at 20 ºC ± 1 °C. The solvent used was deionized water (surface tension of 72 mN·m$^{-1}$), and the test was performed by the sessile drop method.

Before the analysis, the samples were placed in a climate chamber (20 °C ± 1 °C and 65% ± 5% relative humidity) until hygroscopic equilibrium was reached. Afterwards, the samples were removed and stored in a desiccator until testing.

The measurements were taken on the sample surface by placing 5 µL drops of deionized water on the tangential surface of control and densified samples. Six samples were tested for each treatment, and the apparent contact angles were recorded 2 s, 6 s, and 10 s after drop deposition.

*Data analysis*

The normality and homogeneity of the data's variance were verified by the White and the Shapiro-Wilk tests, respectively. The results were satisfactory for the application of parametric tests. In the case of null hypothesis rejection, a comparison between the averages was performed by the Tukey test with a significance level of 5%.

The statistical analysis was performed with Statgraphics Centurion (Statgraphics Technologies, Inc., Version 15.2.05, Warrenton, VA, USA) and Assistat (Federal Univerrsity of Campina Grande, Version 7.7, Campina Grande, Brazil) proceeded with a completely randomized design, with six combinations of temperature and compression time, as well as the control treatment. The data were interpreted through analysis of variance (ANOVA) with a 5% significance level.

## RESULTS AND DISCUSSION

Figure 3 summarizes the physical properties related to the compression process. All of the densification tests showed that compression degrees of around 40% are possible for both wood species. The pine samples presented similar compression degrees for all treatments except the one at 150 °C for 30 min, which resulted in less compression. However, the eucalypt samples presented a continuous increase in compression degree, which is sensitive to temperature and time. This observation is in line with the results of Gong and Lamason (2007), where more difficulties were observed in the compression of softwoods than in the compression of hardwoods. This result suggested that the eucalypt can ideally be further compressed, while pine appeared to reach a compression plateau after 60 min at 150 °C. This phenomenon might be due to the partial collapsing of the tylose-obstructed cell structure (tylose) of eucalypt when water vapor is freed during heat exposure.

The mass losses during the process for the two wood species were significantly different. Indeed, pine showed relatively similar mass loss for every treatment, from 2.0% to 3.3%, while eucalypt lost only 1.1% for light treatments but 4.8% for exposure of 60 min at 190 °C. This result means that pine quickly loses some volatiles, and its mass then remains relatively constant. Meanwhile, eucalypt loses material constantly, meaning that the thermal treatment induces continuous changes. This observation was in line with the compression degree observation, where pine reaches its plateau while eucalypt continuously rearranges. This result meant that the continuous increase of compression degree was accompanied by a loss of wood constituents, with collapsing due to water vaporization greatly contributing to the volume reduction.

The stronger mass loss observed for eucalypt than for pine during heating can be attributed to its hemicelluloses (Esteves and Pereira 2009; Missio *et al*. 2015), for two reasons. Firstly, they are easier to degrade, for they are mainly composed of xylans (Alén *et al*. 2002). Secondly, they are more acetylated, leading to release of acetic acid *in situ*, which catalyzes further hydrolysis (Wang *et al*. 2017). Nevertheless, hemicelluloses degradation for both species was low, mainly because a significant its degradation occurs in a range between 220 °C and 315 °C and until 190 °C there is a loss of ~10% of its mass (Yang *et al*. 2007). Some authors also have verified a strong influence of temperature and time of exposition of heat treatments on wood, causing initially a degradation of hemicellulose at low temperatures, for example 180 °C (Missio *et al*. 2016), and consequently increasing its mass loss (Gaitán-Alvarez *et al*. 2017).

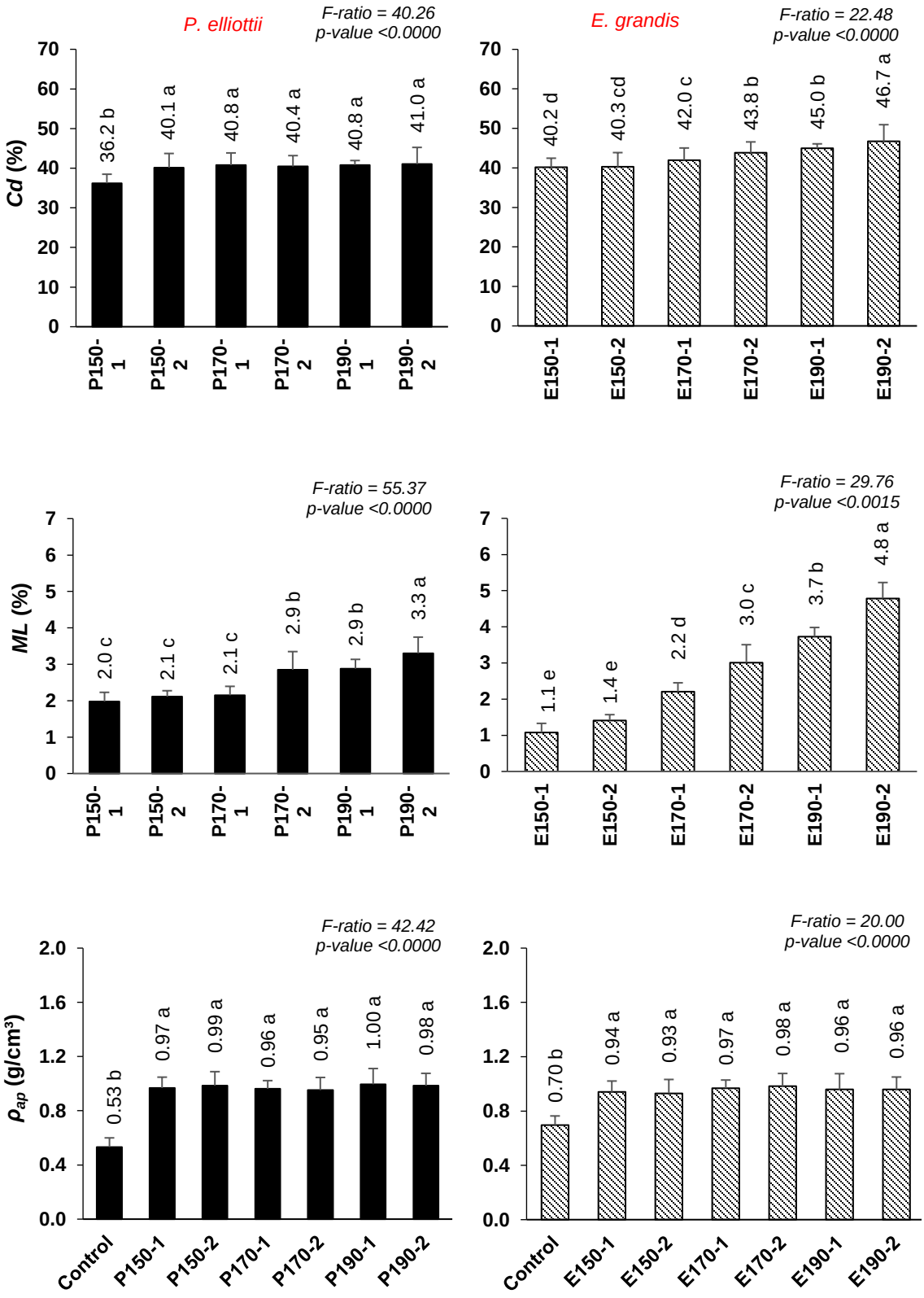



**Fig. 3.** Compression degree (*Cd*), mass loss (*ML*), and apparent density ($\rho_{ap}$) for *P. elliottii* and *E. grandis* woods after different thermomechanical treatments; average values followed by the same letter do not present a significant difference at 5% of significance level for the Tukey test; the error bars at the top of each column indicate the standard deviation

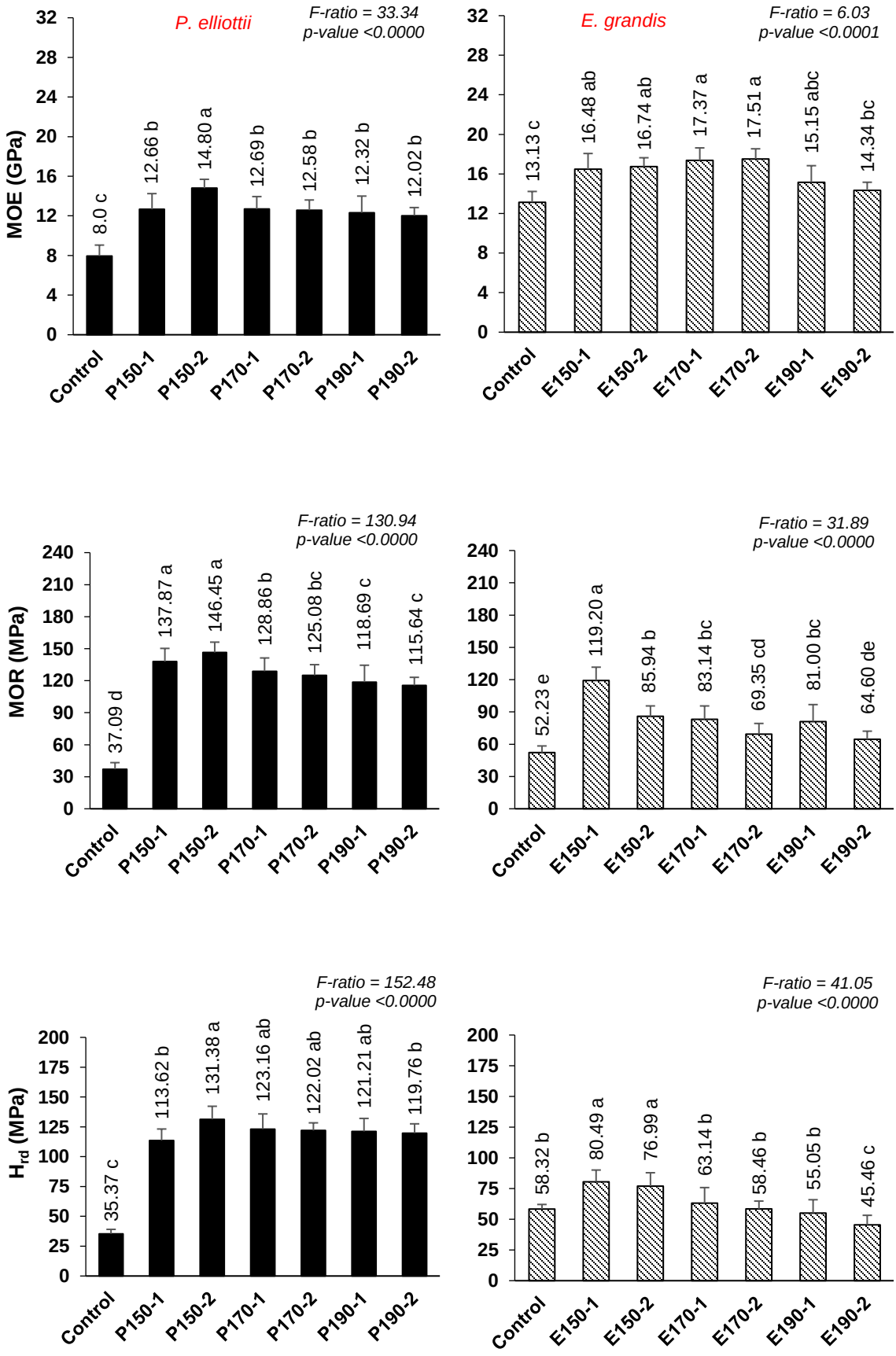
The other components of the cell wall remained unchanged and therefore had no influence on the mass loss. Cellulose has a lower degradation percentage than hemicellulose, due to the lower accessibility of acetic acid and water to the β-D glyosidic bonds (Boonstra and Tjeerdsma 2006). Besides, cellulose exhibits weight loss in the range of 315 to 400 °C, while lignin exhibits degradation at a broader range temperatures, between 160 °C and 900 °C, because its chemical bonds presents different degradation points. However, lignin degrades slowly and has lower weight loss until relatively high temperature values are reached, especially in comparison to hemicellulose losses (Yang *et al*. 2007).

The combination of the volume reduction and the mass loss resulted in a density change for both species. Even if the original densities of the two wood species are different, the products obtained after the same modification process had similar densities of approximately 0.95 $g \cdot cm^{-3}$. It appeared that pine rearranged rapidly, losing its mass and reaching a densification plateau after treatment at 150 °C for 30 min. The eucalypt increased its density but lost mass and volume continuously, which yielded products with similar densities independent of the process applied but with increased chemical and structural degradation as the process was prolonged. From these observations, it is reasonable to infer that the densification processes of lower temperature and duration should produce mechanically superior products with less degradation to hemicelluloses.

## Mechanical Properties

The mechanical analysis of the samples is summarized in Fig. 4. The modulus of elasticity, the Janka hardness, and the impact resistance presented similar trends for pine. The treatment particularly improved the mechanical properties of the material when applied at 150 °C for the longer duration of 60 min. When the densification temperature increased, the performance fell. These observations can be easily described: The densification process reached a plateau at 150 °C; then, albeit relatively slowly, the mechanical properties decreased. Conversely, for eucalypt, the modulus of rupture and the hardness presented a different trend from the modulus of elasticity for the mildly treated samples. Like pine, eucalypt showed peak performances in MOR and Janka hardness when the milder treatment of 150 °C for 30 min was applied. Then, the mechanical weakening began with the treatment at 150 °C for 60 min, meaning that an increase in the energy applied increased the wood's degradation and diminished its mechanical properties. The MOE of eucalypt was relatively constant among mild treatments (150 °C and 170 °C), decreasing only at 190 °C. This region of similar performance for MOE could be due to competing factors: an increase of rigidity due to the initiation of furanization of hemicelluloses and a softening of the lignin due to the breaking of secondary bonds and amorphization.

Another difference in behavior between the wood species was visible when their impact resistances were analyzed. Densified pine reacted very well to mild treatment and performed best with low temperature and long exposure (60 min). This result meant that the wood components did not lose their elasticity during the densification process. Conversely, the eucalypt suffered degradation, with the elasticity against impact decreasing and with the treated samples registering lower performances than the untreated samples. The densification process by itself has little influence on these results, since densification has more effect on a layer close to surface than in all wood sample (Gašparík *et al.* 2016).


P. elliottii
F-ratio = 33.34
p-value <0.0000
MOE (GPa)
8.0 c
12.66 b
14.80 a
12.69 b
12.58 b
12.32 b
12.02 b
Control
P150-1
P150-2
P170-1
P170-2
P190-1
P190-2
E. grandis
F-ratio = 6.03
p-value <0.0001
13.13 c
16.48 ab
16.74 ab
17.37 a
17.51 a
15.15 abc
14.34 bc
Control
E150-1
E150-2
E170-1
E170-2
E190-1
E190-2
F-ratio = 130.94
p-value <0.0000
MOR (MPa)
37.09 d
137.87 a
146.45 a
128.86 b
125.08 bc
118.69 c
115.64 c
Control
P150-1
P150-2
P170-1
P170-2
P190-1
P190-2
F-ratio = 31.89
p-value <0.0000
52.23 e
119.20 a
85.94 b
83.14 bc
69.35 cd
81.00 bc
64.60 de
Control
E150-1
E150-2
E170-1
E170-2
E190-1
E190-2
F-ratio = 152.48
p-value <0.0000
Hrd (MPa)
35.37 c
113.62 b
131.38 a
123.16 ab
122.02 ab
121.21 ab
119.76 b
Control
P150-1
P150-2
P170-1
P170-2
P190-1
P190-2
F-ratio = 41.05
p-value <0.0000
58.32 b
80.49 a
76.99 a
63.14 b
58.46 b
55.05 b
45.46 c
Control
E150-1
E150-2
E170-1
E170-2
E190-1
E190-2

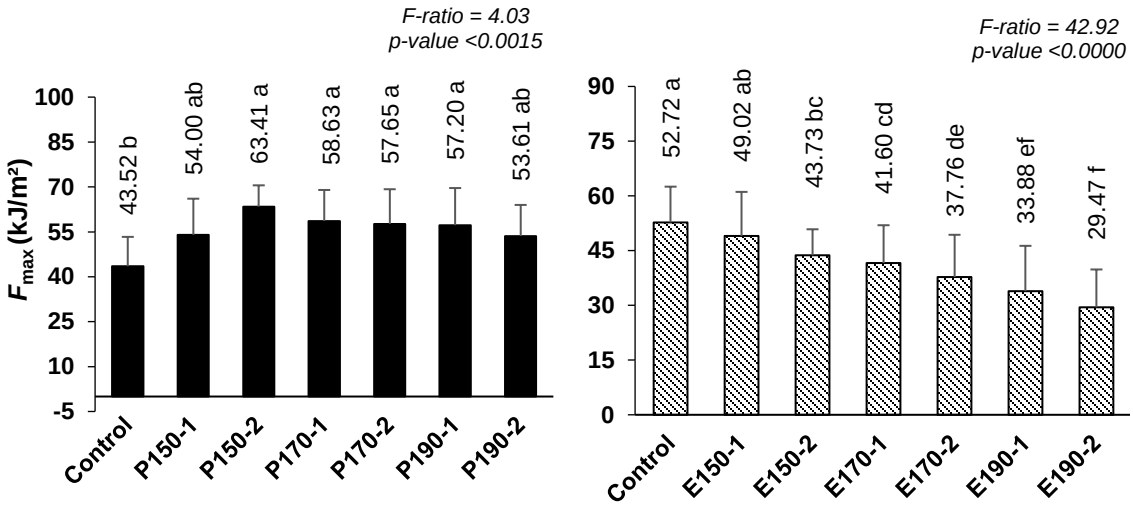



**Fig. 4.** Modulus of elasticity (MOE), modulus of rupture (MOR), Janka hardness in the radial direction ($H_{rd}$), and impact resistance ($F_{max}$) for *P. elliottii* and *E. grandis* wood after different thermomechanical treatments; average values followed by the same letter do not present a significant difference at 5% significance level for the Tukey test; the error bars at the top of each column indicate the standard deviation

However, according to the literature, hemicelluloses are the first structural component to be thermally affected (Missio *et al.* 2015), beginning their degradation process by debranching and deacetylation. The released acid acts as a catalyst for the further depolymerization of this polysaccharide (Awoyemi *et al.* 2009). Also, hardwoods typically present a higher content of pentosans (hemicellulose) than softwoods (Hill 2006). This explains, with through the loss weight results as well, why eucalypt presented a decrease in strength properties.

It is well known that the stiffness increase obtained through densification can be negated by the thermal degradation of the wood's chemical constituents (Esteves and Pereira 2009; Ulker *et al*. 2012; Cademartori *et al*. 2014; Missio *et al*. 2015). Accordingly, wood densification at elevated temperatures might cause an increase in fragility and a consequent decrease in load resistance.

## Wettability

In Fig. 5 the ACA after 2 s, 6 s, and 10 s are reported for both wood species. For all the samples, the apparent contact angle decreased with time, but for the densified surfaces the behavior of ACA at these times was different (increase of the ACA) due to four phenomena: decreased dimensions of pores and micropores, increased hydrophobicity due to thermal rearrangement, migration of extractives and resins to the surface, and molecular reorientation (Christiansen 1991; Awoyemi *et al.* 2010; Pertuzzatti *et al*. 2016). Besides, there is no significance statistical difference among the treatments, except between them and the control samples.

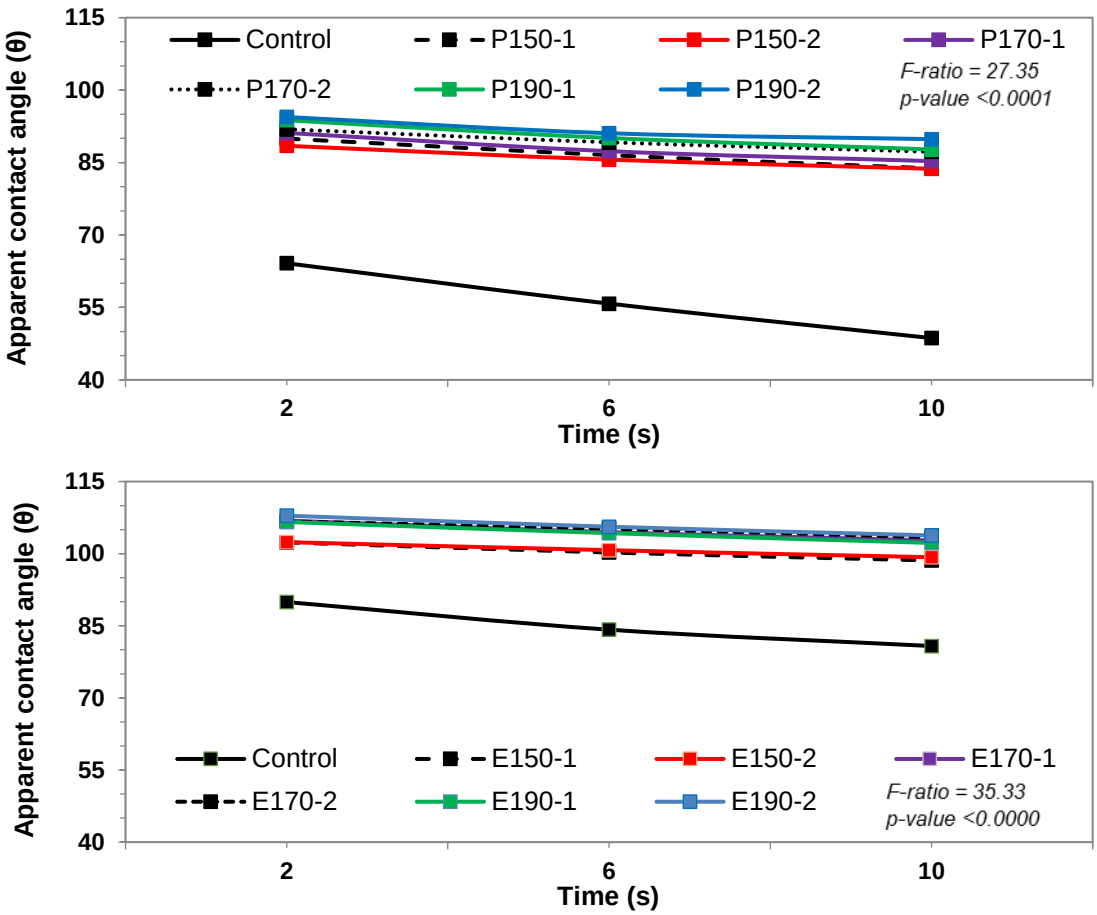



**Fig. 5**. Behavior of the apparent contact angle (θ), in degrees (°), in *P. elliottii* and *E. grandis* wood subject to densification.

According to Wålinder and Gardner (1999), when liquid deposition on a porous material yields an angle less than 90°, a fast penetration is occurring, typical of hydrophilic materials. For pine, the results were similar between 2 s and 10 s of analysis, ranging from 87° to 94º. For the eucalypt, only the control recorded an angle of $\theta < 90°$ (hydrophilic character) indicated that the densification treatments at all the assessed temperatures were efficient in increasing surface water repellency. As Wålinder and Johansson (2001) reported, the wettability is influenced by several factors, including porosity, surface roughness, surface polarity, pH, fiber direction, and extractive content.

Conforming to Cademartori *et al*. (2015), wettability variations after application of elevated temperature are related to material surface inactivation. This phenomenon is influenced by the reduction of attractive forces on the wood surface, *via* pore closure, molecular reorientation, rearrangement of hydroxyl groups in the surface, surface oxidation, and migration of extractive products to the surface (Christiansen 1991). Moreover, densification has an impact on repellency of polar solvents, an interesting peculiarity for materials that naturally have an elevated degree of hydrophilicity and notable interactions with polar solvents.

## CONCLUSIONS

1. Wood densification for fast-growing wood species of *Eucalyptus grandis* and *Pinus elliottii* led to an increase in apparent density up to 0.95 $g \cdot cm^{-3}$ to 1.00 $g \cdot cm^{-3}$ through strong volume reduction and contained mass loss.
2. During the heat treatment, hemicelluloses are the first component that degrades. *Eucalyptus grandis* was consequently more sensitive than *Pinus elliottii* because the former contains more xylose and acetylated groups in its hemicelluloses.
3. The improvement in the mechanical properties was generally superior when treatments of milder temperature (150 °C) were performed. Longer exposures (60 min) were preferred for *P. elliottii*, while *E. grandis* behaved better with shorter exposures (30 min). This result was because pine reached a degradation plateau, while eucalypt lost mass continuously, and the consequent chemical degradation was progressive.
4. The best resulting process found for the densification of *P. elliottii* was 150 °C for 60 min, while for *E. grandis* it was 150 °C for 30 min. All the processes involving higher temperatures or longer times resulted in similar densifications but inferior mechanical properties due to increased chemical and structural degradation.
5. Mechanical properties, in general, were improved for both densified wood species, except the impact resistance of the treated eucalypt.
6. The densification process significantly decreased the hydrophilicity of the wood surfaces.

## ACKNOWLEDGMENTS

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