

ENHANCING MECHANICAL AND SURFACE PROPERTIES OF *Eucalyptus* WOOD

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ABSTRACT

Eucalyptus is one of the most fast-growing trees. Therefore, in the last decades it has been extensively planted and harvested so that nowadays *Eucalyptus* is one of the most popular trees of the planet. There are many genres of this plant and they are often treated as a large bunch of the same timber characterized by moderate mechanical and surface properties which hinder their usage for any sight application (e.g. flooring, cladding, ceiling). In this study four species of *Eucalyptus*: *E. grandis*, *E. dunnii*, *E. cloeziana* and *E. tereticornis* were undergone to densification through hydro-thermo-mechanical treatment (HTM) first and then to oil heat-treatment (OHT) in order to improve their mechanical properties and hydrophobicity. It was observed that low density species (*E. grandis*) reaches higher compression degrees while heavier species (*E. tereticornis*) reach densities over 800 kg/m³; however, HTM decrease the variability of the properties. Treatments at higher temperature (160 °C) involves higher compression degree, lower set-recovery and higher surface hydrophobization, but also weaker mechanical properties. The hot oil post-treatment helps to contain the springback effect and to reduce the wettability of each specimen. Densified samples present similar surface hardness. The tailored application of the two treatments improves the properties of every *Eucalyptus* which can gain market also for nobler end-usages.

Keywords: Density enhancement, mechanical resistance, post-treatment, springback effect, surface properties, synergic treatment, wettability.

INTRODUCTION

Until the middle of the last century there was a wide variety of forest species available in Brazil. But the massive harvesting of tropical lumbers has dramatically reduced the stock available and wood with excellent mechanical and durability properties became scarce and expensive. The increasing need for wood has favoured the plantation of fast-growing trees and since then the *Eucalyptus* species are the most abundant in Brazil. Several *Eucalyptus* species were introduced, but due to the limited experience with this specie, they were often harvested jointly and underwent the same processing line, even though their properties are sensibly different.

Due to its features, *Eucalyptus* is particularly interesting for pulping purposes, but it cannot be used for flooring, cladding, ceiling or any other application where mechanical and durability resistance are required.

In this context, a wood modification process that enhances mechanical and surface properties and also

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homogenises the different species is necessary. These modification methods, whether of thermal treatments or also using densification, are currently used by researchers in different species, such as *Pinus caribaea* and *Eucalyptus saligna* (Brito *et al.* 2019), *Eucalyptus nitens* (Wentzel *et al.* 2019), *Eucalyptus grandis* and *Eucalyptus cloeziana* (Dalla Costa *et al.* 2020), *Fagus sylvatica* and *Quercus robur* (Laskowska 2020), *Populus usbekistanica* (Sözbir *et al.* 2019), aiming at increases in the technological properties of wood. The densification process suites well to this purpose because it is proven to significantly increase the mechanical and surface properties of wood (Welzbacher *et al.* 2008, Pertuzzatti *et al.* 2018).

The technique of timber densification was already presented more than a century ago and in the last decades it is evolved to hydro-thermo-mechanical treatment (HTM) consisting of multi-stage process of wet/moist cycles at various temperatures and pressure (Sears 1900, Welzbacher *et al.* 2008, Navi and Pizzi 2015).

The major drawback of the resulting compressed wood is the springback effect which occurs when the material is exposed to high moisture environment or in direct contact with water. Several studies involving resin impregnation, thermal and oil based post-treatment were already studied to minimize this effect (Gabrielli and Kamke 2010, Gong *et al.* 2010, Fang *et al.* 2012) and the oil heat-treatment (OHT) have the additional advantage of increasing the biological resistance of the treated samples (Dubey *et al.* 2012, Pelit *et al.* 2015).

Other studies have shown that the densification at >180 °C carries to a significant reduction of the mechanical properties (Navi and Pizzi 2015, Pelit *et al.* 2015, Pertuzzatti *et al.* 2018), whereas when too low temperature are applied, the springback effect is remarkable and a stabilizing post-treatment is required (Dubey *et al.* 2012). For this reason, in this study, we investigate the technological properties of the wood treated at 140 °C and 160 °C and applying the OHT to find the most suitable treatment for enhancing the properties of the four *Eucalyptus* species considered (*E. grandis*, *E. dunnii*, *E. cloeziana* and *E. tereticornis*).

MATERIAL AND METHODS

Materials

Eucalyptus grandis, *Eucalyptus dunnii*, *Eucalyptus cloeziana* and *Eucalyptus tereticornis* trees of 21, 18, 20 and 22 years respectively were abated in the region of Santa Maria - RS, Brasil (29° 43' 1,95" S, 53° 43' 33,7" W). 5 trees for each species were selected according to the ASTM D5536-94 (2004). Each sample was extracted from the region at around 3 m from the tree base, by selecting knots-free heartwood (Missio *et al.* 2016).

60 samples of 40 cm x 15 cm and different thickness according to density, were prepared and stabilized until equilibrium moisture content (30 days under conditions - 20 °C and 65 % of relative humidity) in order to obtain samples having thickness of 2 cm after densification.

Sample preparation

Hydro-thermo-mechanical treatment (HTM) - densification

Eucalyptus samples were initially pre-heated by dipping in 100 °C water for 20 min. After short blotting, the samples were compressed in radial direction with 6 MPa pressure at 140 °C or 160 °C for 40 min (Arruda and Del Menezzi 2016). The samples were measured and stabilized in a climatic chamber (20 °C and 65 % RH) until equilibrium was reached.

Oil heat treatment (OHT)

The stabilized samples were dipped into an oil bath, filled with refined soybean oil (Type 1, density ~ 922 kg/m³ at 20 °C), at 180 °C for 60 min and then dried in a ventilated oven at 103 °C for a standard time of one hour. Finally, the samples were stabilized again (20 °C and 65 % RH). The treatments applied are summarized in Table 1.

Table 1: Hydro-thermo-mechanical (HTM) treatments on *Eucalyptus grandis*, *Eucalyptus dunnii*, *Eucalyptus cloeziana* and *Eucalyptus tereticornis*.

Wood species	Treatment	HTM, Temperature (°C)	OHT at 180 °C
<i>E. grandis</i>	EC	No treatment	No
	EC-T	No treatment	Yes
<i>E. dunnii</i>	E140	140	No
<i>E. cloeziana</i>	E140-T	140	Yes
<i>E. tereticornis</i>	E160	160	No
	E160-T	160	Yes

Sample characterization

Physical analysis

The Compression degree (Cd) was calculated from the relationship between final and initial thickness of the wooden pieces (Equation 1), measured with a digital caliper (0,01mm) immediately after the hydro-thermo-mechanical treatment.

$$C_d = (T_i - T_f / T_f) \times 100 \text{ (\%)} \quad (1)$$

Where T_i is the initial thickness (mm), T_f is the final thickness (mm), T_a is the thickness of the dry sample after 24 h water immersion (mm).

The mass variation (MV) was performed using the relation between the mass of the samples before and after densification (Equation 2). This step was important to determine the influence on the mass of the samples after each step of modification treatments.

$$MV = \left(\frac{M_{Ad} - M_{Bd}}{M_{Bd}} \right) \times 100 \text{ (\%)} \quad (2)$$

where M_{Bd} is the sample dry mass before densification (kg), M_{Ad} is the sample dry mass after densification (kg).

The density of the samples (ρ_b) was performed under the condition of 12 % relative humidity (20 °C and 65 % RH) using Equation 3.

$$\rho_b = M/V \quad (3)$$

Where M (kg) is the mass of the samples in the condition of 12 % relative humidity (20 °C and 65 % RH), and V (m³).is the sample volume in the condition of 12 % relative humidity (20 °C and 65 % RH)

Set recovery (SR) was measured by dipping the samples in a 20 °C water bath for 24 hours and successively drying them at 103 °C until constant mass. The final thickness T_a was registered and SR was calculated according to Equation 4. This calculation is essential for calculating the springback effect.

$$SR = \left(\left(\frac{T_a - T_f}{T_i - T_f} \right) \times 100_f \right) \times 100 \text{ (\%)} \quad (4)$$

where T_i is the initial thickness (mm), T_f is the final thickness (mm), T_a is the thickness of the dry sample after 24 h water immersion (mm).

Mechanical analysis

Bending, compression and Janka hardness were performed on 12 samples per treatment with a universal testing machine EMIC® DL2000/1000. The bending tests were performed according to the ASTM D143-94 (2000) with a rate of 1,04 mm/min on samples of 32 cm x 2 cm x 2 cm and elastic modulus (MOE) and rupture modulus (MOR) were registered. The compression tests were done following ASTM D143-94 (2000) on samples of 10 cm x 2 cm x 2 cm. The Janka tests were run on the compressed face by using a spherical spot with 1 cm² of surface with a penetration rate of 6 mm/min.

Impact resistance was measured according to ABNT NBR 7190 (1997), with a Charpy pendulum (PW 15/10, Wolpert®, Ludwigshafen am Rhein, Alemanha) on 28 cm × 2 cm × 2 cm (Pertuzzatti *et al.* 2018). Absorbed work (W) and maximal resistance (F_{max}) were calculated Equation 5.

$$F_{max} = 100 \times \frac{W}{b} \times h \quad (5)$$

where F_{max} is the maximum resistance to impact (kJ/m²), W is the absorbed work (J), and b and h are the transverse sample dimensions (mm).

Wettability

Contact angle (CA) measurements were done with a DataPhysics OCA (DSA 25, Krüss, Hamburg, Germany) instrument at 20 °C ± 1 °C laying 5 µL drop of deionized water on the compressed face of the stabilized specimens. The contact angle was registered after 10 s, 30 s, 50 s, 70 s and 90 s.

Data analysis

Normality and homogeneity tests of variance were verified with White and Shapiro-Wilk algorithm, respectively. Variance analysis (ANOVA) was performed and the comparison between averages was done with the Tukey test (< 5 %) with the SISVAR program (Ferreira 2011).

RESULTS AND DISCUSSION

Intrinsic properties

During the HTM densification process, volume, mass and hence density of the treated samples modified. In Figure 1 compression degree (a), mass variation (b), density (c) and set-recovery after water cycle (d) are presented.

Applying the same pressure to different *Eucalyptus* species carries to different thicknesses and the compression degree is summarized in Figure 1a. It can be noticed that the original density as a major impact on the compression degree: the lower was the original density, the higher was the compression degree. This phenomenon is well-known in the literature and it is due to the fact that a lighter timber, has also more voids (Unsal *et al.* 2011). It is interesting to observe that the use of higher temperature involved higher compression degrees for every species. This was also a logical consequence because the thermoplastic lignin become softer at higher temperature and hence the specimens result more compressed (Bekhta *et al.* 2012, Wolcott *et al.* 1990, Welzbacher *et al.* 2008).

Mass loss (Figure 1b) also occurred when wood was subjected to HTM densification processes. On the one hand the volatile substances (e.g. VOC, water) and the small moieties obtained by thermal degradation of hemicelluloses evaporated during the process and on the other hand the equilibrium moisture content after treatment was lower because of the less hydrophilic surface which equilibrated with less water (Alén *et al.* 2002, Pertuzzatti *et al.* 2018). Consistently, higher mass losses were observed when higher densification temperature were applied.

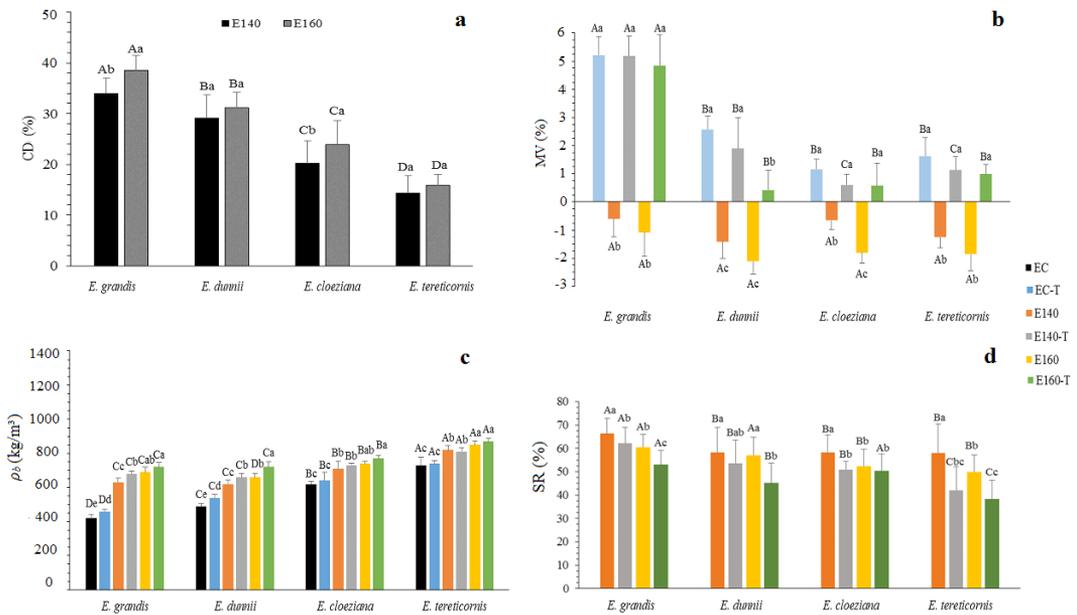


Figure 1: Intrinsic properties of densified and oil post-treated *Eucalyptus*. (a) Compression degree; (b) Mass variation; (c) Density and (d) Set recovery (%).

The standard deviation is reported on top of each bar. The capital letters describe the comparison between species while the lowercase compare between treatments according to Tukey test 5 % significance.

When the samples underwent post-treatment with oil bath OHT, the mass variation was positive because of the penetrated oil, but also in these cases lower absorptions and higher chemical degradations were observed for the more compressed samples.

As a consequence of the previous two greatness, density increases were registered for every densification and oil treatment (Figure 3c) meaning that the mass loss is less important than the volume decrease. Volume reductions from 13,3 % to 38,5 % and the weight reduction of 0,6 % to 2,2 % resulting in a density increase of 10,9 % to 61,9 % were observed.

The density increase registered during densification was more important for the species with originally lower density (420 kg/m^3) which achieved min. 630 kg/m^3 while the heavier species (730 kg/m^3) reached max. density of 870 kg/m^3 . The effect of the oil post-treatment showed density gain of around 30 kg/m^3 , but also in this case the lighter wood *E. grandis* and *E. dunnii* absorbed more oil (approximately 45 kg/m^3) while the heavier *E. cloeziana* and *E. tereticornis* adsorbed less (approximately 15 kg/m^3). Overall the density between the different *Eucalyptus* homogenized, but still significant differences were observed: The natural densities of *E. grandis* (420 kg/m^3) and *E. tereticornis* (730 kg/m^3) was very different ($\Delta = 310 \text{ kg/m}^3$) while the samples compressed at $180 \text{ }^\circ\text{C}$ had density of 720 kg/m^3 and 870 kg/m^3 respectively ($\Delta = 150 \text{ kg/m}^3$).

The set-recovery highlighted that significant springback effect occurred for every treatment. The densification temperature had a lower impact in reducing the set recovery compared to the oil post-treatment, while the original wood density had also a considerable importance, since the *E. tereticornis* presented significantly lower set-recoveries (down to 35 %).

According to Navi and Pizzi 2015, there are two mechanisms acting on set-recovery. After densification, the wood is cooled below the glass transition temperature of lignin, that is, from rubbery to the glassy state. In this way, cellulose will be confined in this rigid matrix. During drying, the formation of hydrogen bonds between the cell wall polymers also contributes to the fixation of the deformed state. However, if the wood comes into contact with water or variations in relative humidity, the hydrogen bonds disrupt, and lignin may become rubbery again. On the one hand, the hydrolysis of hemicelluloses during densification plays an active

role in the dissipation of the tensions stored. During the hemicelluloses hydrolysis, more porous surfaces are formed which allow to dissipate the deformation energy. These changes in hemicelluloses result in decreases in mechanical properties (similar to thermal treatments), which would make the wood modification method unfeasible. Thus, there must be a balance between depolymerization of hemicelluloses, changes from Tg of lignin and densification rates, aiming at the minimum loss of mechanical resistance.

Mechanical properties

Bending, compression and the impact resistances as well as the surface hardness were measured to evaluate the effects of the treatments on the *Eucalyptus* species. These tests are summarized in Figure 2 and show that generally the densified wood increase all mechanical properties: up to 22 % in MOE, up to 34 % in MOR, up to 80 % in σ_{\max} , up to 218 % in hardness and up to 94 % in impact resistance. In particular, the wood densified at milder temperature and without post-treatment were better performing against all mechanical solicitations. This result was confirmed for all species of *Eucalyptus*.

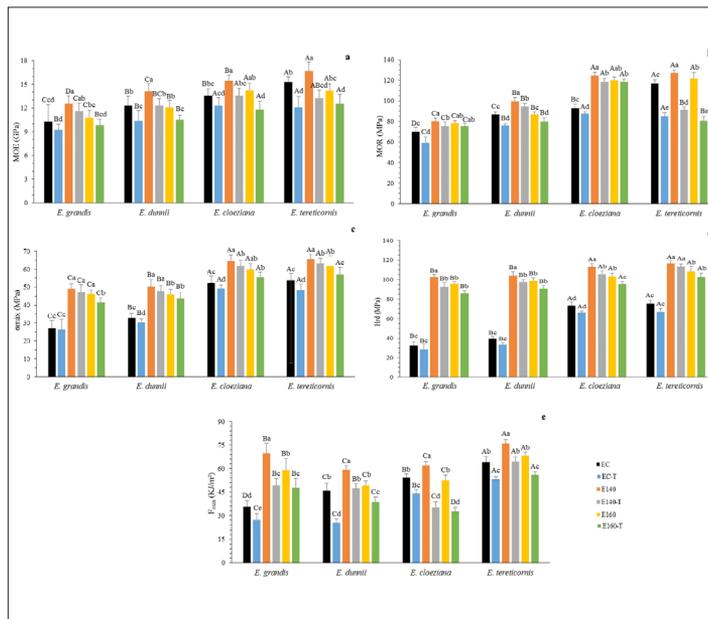


Figure 2: Mechanical properties of densified and oil- post-treated Eucalyptus. (a) Modulus of elasticity (MOE); (b) Modulus of rupture (MOR); (c) Compression resistance (σ_{\max}) (d) Janka hardness (Hrd) and (e) Impact resistance (F_{\max}).

The standard deviation is reported on top of each bar. The capital letters describe the comparison between species while the lowercase compare between treatments according to Tukey test 5 % significance.

The mechanical properties analysed worsen by applying higher temperature and/or oil treatment. These results were due to the degradation of the structure when more aggressive thermal treatment is applied. Thermal treatments caused the increase of micro-fractures, (Ulker *et al.* 2012, Navi and Pizzi 2015, Gašparik *et al.* 2016, Gaff *et al.* 2017, Pelit *et al.* 2018, Pertuzzatti *et al.* 2018) and the hot oil at 180 °C further weakened the structure because the it penetrated the surface and facilitated the heat transfer with consequent increase of degradation inside of the wood structure.

The densification process significantly enhanced the surface hardness. This was due to the nature of the process which compress to a higher extent the surface layers that results denser than the inner core. The surface hardness after compression was similar for all the considered genres of *Eucalyptus* and therefore when hardness had a major impact (e.g. flooring purposes), the variability of *Eucalyptus* can be reduced with the densification process. The surface hardness had also particularly high impact in the surface wettability.

Wettability

In Figure 3 the contact angle measurements at different time were registered for every treatment and species. The oil treated specimens showed the more hydrophobic behavior with similar contact angles (between 90° and 110°) for *E. grandis*, *E. cloeziana* and *E. dunnii*, while the specimens of *E. tereticornis* showed slightly higher values (over 120° for the treatment at 160°C) and this was due to their higher density (Amorim *et al.* 2013). Specimens densified at higher temperatures, resulted more hydrophobic than the homologues treated at milder temperatures while the samples undergoing only oil treatment presented similar trend than the sample densified but not oil-treated, meaning that the two processes allow similar enhancements. The specimens treated only with oil showed steeper slopes than the HTM densified samples which suggest that the OHT modification involves prolonged hydrophobic behavior. This was possibly due to the smoother surface of the densified samples (Christiansen 1991, Pertuzzatti *et al.* 2016).

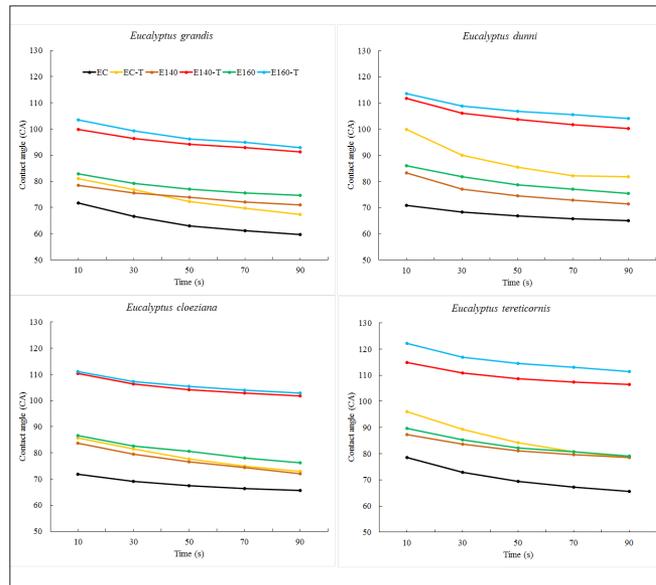


Figure 3: Contact angle trend over time for the four *Eucalyptus* species undergoing densification and oil post-treatment.

According to Wålinder and Gardnerb (1999), when the water drop lays with contact angle $< 90^\circ$ a faster penetration, typical for hydrophilic surfaces, occurs. This means that without oil post-treatment the densification process alone does not guarantee to significantly modify the wettability to hydrophobic and the surface can still be easily treated with coatings and adhesives. Conversely, when the HTM densification and the OHT oil post-treatment are combined, the contact angle increases of around 50 % and wood surface remains hydrophobic for longer than 90 seconds.

Considering the average price of *Eucalyptus* ($d = 550 \text{ kg/m}^3$) of $215 \text{ \$/m}^3$ we can estimate the cost of the densified wood after HTM treatment to around $378 \text{ \$/m}^3$ ($d = 700 \text{ kg/m}^3$). The OHT post-treatment can be estimated with $130 \text{ \$/m}^3$ and therefore the cost of the HTM and OHT treated wood will be around $509 \text{ \$/m}^3$. This price is highly competitive because with the mechanical and surface properties observed promote the densified *Eucalyptus* can be considered also for flooring and cladding applications for which tropical species like Mogno (*Swietenia macrophylla*), Ipê (*Tabebuia* sp.) and Cedrus (*Cedrela fissilis*) are used and their costs is, at present, around $790 \text{ \$/m}^3$.

CONCLUSIONS

In the present study we have analysed intrinsic, mechanical and surface properties of four *Eucalyptus* species undergoing HTM treatment at 140°C and 160°C with an OHT hot-oil post-treatment.

It was observed that limited loss of mass and consistent loss of volume with resulting increase of density were registered for every *Eucalyptus* species. In particular, the ones having lower density reached higher compression degrees and the ones originally heavier reached density over 800 kg/m³ after HTM process and presented reduced set-recovery after water immersion (down to < 40 %). The study of the mechanical properties highlighted that milder HTM treatment produces more performing densified wood for bending, compression, impact and mostly hardness resistance. Both, densification and oil post-treatment increased significantly the hydrophobicity of the wood surface, but only the joint application of the two treatments allowed to get surfaces with contact angle > 90°. The densification process enhanced the mechanical properties and homogenized the properties of the four *Eucalyptus* species studied, while the hot-oil post-treatment was required to obtaining a more hydrophobic surface and for containing the springback effect. The combination of the two treatments carried to an interesting, cheap biomaterial that can be suitable for indoor sight application such as flooring, cladding and ceiling.

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