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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.

38 **INTRODUCTION**

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

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

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

60 The technique of timber densification was already presented more than a century
61 ago and in the last decades it is evolved to hydro-thermo-mechanical treatment (HTM)

62 consisting of multi-stage process of wet/moist cycles at various temperatures and
63 pressure (Sears 1900, Welzbacher *et al.* 2008, Navi and Pizzi 2015).

64 The major drawback of the resulting compressed wood is the springback effect
65 which occurs when the material is exposed to high moisture environment or in direct
66 contact with water. Several studies involving resin impregnation, thermal and oil based
67 post-treatment were already studied to minimize this effect (Gabrielli and Kamke 2010,
68 Gong *et al.* 2010, Fang *et al.* 2012) and the oil heat-treatment (OHT) have the additional
69 advantage of increasing the biological resistance of the treated samples (Dubey *et al.*
70 2012, Pelit *et al.* 2015).

71 Other studies have shown that the densification at >180 °C carries to a
72 significant reduction of the mechanical properties (Navi and Pizzi 2015, Pelit *et al.*
73 2015, Pertuzzatti *et al.* 2018), whereas when too low temperature are applied, the
74 springback effect is remarkable and a stabilizing post-treatment is required (Dubey *et al.*
75 2012). For this reason, in this study, we investigate the technological properties of the
76 wood treated at 140 °C and 160 °C and applying the OHT to find the most suitable
77 treatment for enhancing the properties of the four *Eucalyptus* species considered (*E.*
78 *grandis*, *E. dunnii*, *E. cloeziana* and *E. tereticornis*).

79 MATERIAL AND METHODS

80 Materials

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

87 60 samples of 40 cm x 15 cm and different thickness according to density, were
88 prepared and stabilized until equilibrium moisture content (30 days under conditions –
89 20 °C and 65 % of relative humidity) in order to obtain samples having thickness of 2
90 cm after densification.

91 **Sample preparation**

92 **Hydro-thermo-mechanical treatment (HTM) - densification**

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

98 **Oil heat treatment (OHT)**

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

104 **Table 1:** Hydro-thermo-mechanical (HTM) treatments on *Eucalyptus grandis*,
105 *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

106

107

108

109 **Sample characterization**

110 **Physical analysis**

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

114

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

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

117

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

$$MV = (M_{Ad} - M_{Bd} / M_{Bd}) \times 100 (\%) \quad (2)$$

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

124

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

$$\rho_b = M / V \text{ (kg/m}^3\text{)} \quad (3)$$

127 where M is the mass of the samples in the condition of 12 % relative humidity (20 °C
128 and 65 % RH) (kg), and V is the sample volume in the condition of 12 % relative
129 humidity (20 °C and 65 % RH) (m^3).

130

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

$$SR=(T_a - T_f/T_i - T_f) \times 100_p) \times 100 (\%) \quad (4)$$

135

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

138 **Mechanical analysis**

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

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

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

151

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

154 **Wettability**

155 Contact angle (CA) measurements were done with a DataPhysics OCA (DSA
156 25, Krüss, Hamburg, Germany) instrument at 20 °C ± 1 °C laying 5 µL drop of
157 deionized water on the compressed face of the stabilized specimens. The contact angle
158 was registered after 10 s, 30 s, 50 s, 70 s and 90 s.

159

160 **Data analysis**

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

165

166 **RESULTS AND DISCUSSION**

167 **Intrinsic properties**

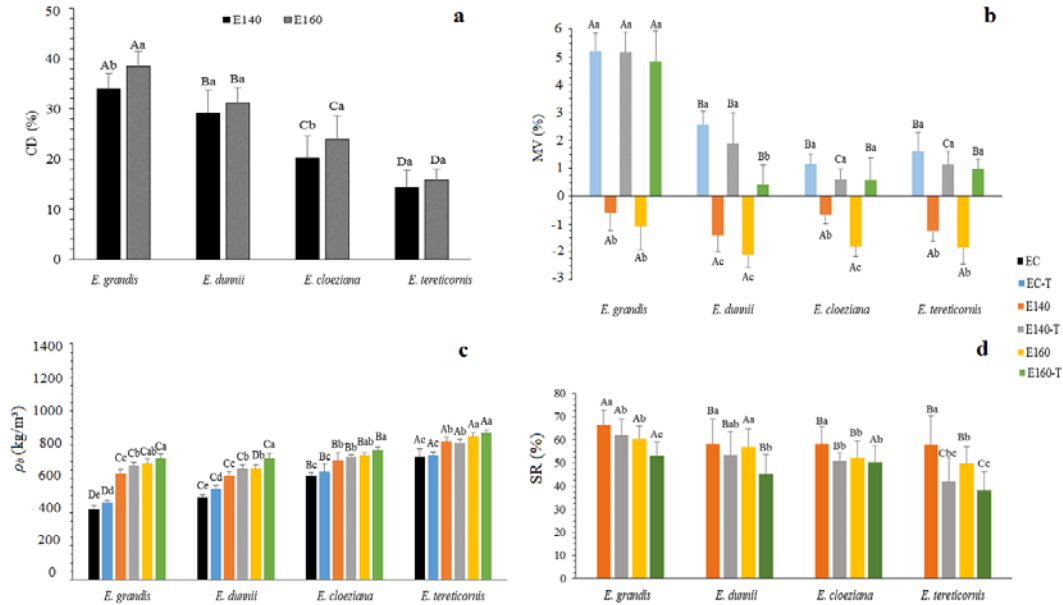
168 During the HTM densification process, volume, mass and hence density of the
169 treated samples modified. In Figure 1 compression degree (a), mass variation (b),
170 density (c) and set-recovery after water cycle (d) are presented.

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

181 Mass loss (Figure 1b) also occurred when wood was subjected to HTM
182 densification processes. On the one hand the volatile substances (e.g. VOC, water) and
183 the small moieties obtained by thermal degradation of hemicelluloses evaporated during
184 the process and on the other hand the equilibrium moisture content after treatment was
185 lower because of the less hydrophilic surface which equilibrated with less water (Alén

186 *et al.* 2002; Pertuzzatti *et al.* 2018). Consistently, higher mass losses were observed
 187 when higher densification temperature were applied.

188



189 **Figure 1:** Intrinsic properties of densified and oil post-treated *Eucalyptus*. a)
 190 Compression degree; b) Mass variation; c) Density and d) Set recovery (%). The
 191 standard deviation is reported on top of each bar. The capital letters describe the comparison
 192 between species while the lowercase compare between treatments according to Tukey test 5 %
 193 significance.

194

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

199 As a consequence of the previous two greatness, density increases were
 200 registered for every densification and oil treatment (Figure 3c) meaning that the mass
 201 loss is less important than the volume decrease. Volume reductions from 13,3 % to 38,5
 202 % and the weight reduction of 0,6 % to 2,2 % resulting in a density increase of 10,9 %
 203 to 61,9 % were observed.

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

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

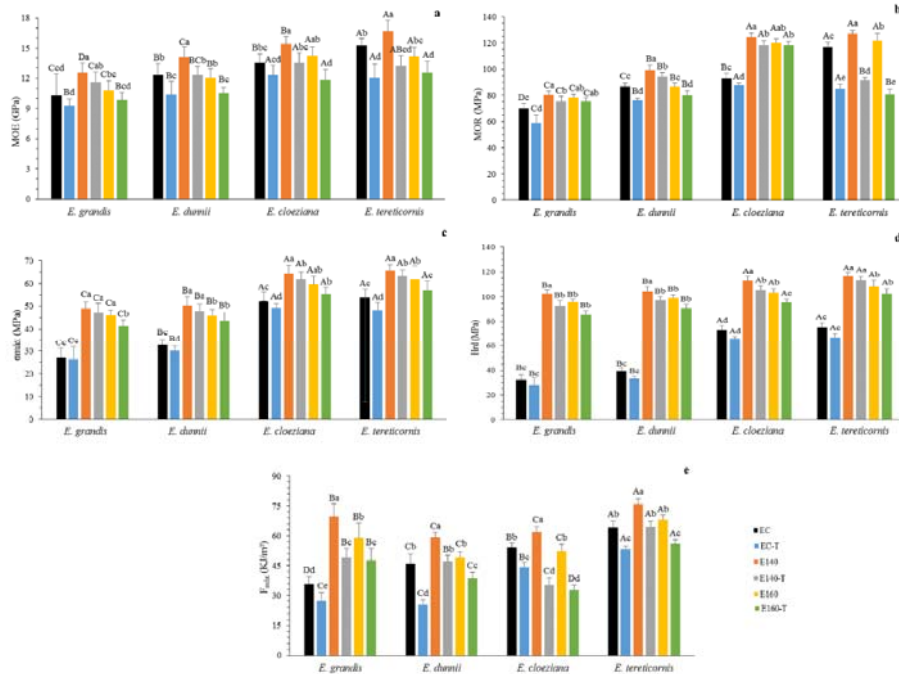
220 According to Navi and Pizzi 2015, there are two mechanisms acting on set-
221 recovery. After densification, the wood is cooled below the glass transition temperature
222 of lignin, that is, from rubbery to the glassy state. In this way, cellulose will be confined
223 in this rigid matrix. During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

229 allow to dissipate the deformation energy. These changes in hemicelluloses result in
230 decreases in mechanical properties (similar to thermal treatments), which would make
231 the wood modification method unfeasible. Thus, there must be a balance between
232 depolymerization of hemicelluloses, changes from Tg of lignin and densification rates,
233 aiming at the minimum loss of mechanical resistance.

234 **Mechanical properties**

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

243



244

245 **Figure 2:** Mechanical properties of densified and oil- post-treated Eucalyptus. a)
 246 Modulus of elasticity (MOE); b) Modulus of rupture (MOR); c) Compression resistance
 247 (σ_{max}) d) Janka hardness (Hrd) and e) Impact resistance (F_{max}). The standard deviation is
 248 reported on top of each bar. The capital letters describe the comparison between species while
 249 the lowercase compare between treatments according to Tukey test 5 % significance.

250

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

258 The densification process significantly enhanced the surface hardness. This was
 259 due to the nature of the process which compress to a higher extent the surface layers that
 260 results denser than the inner core. The surface hardness after compression was similar

261 for all the considered genres of *Eucalyptus* and therefore when hardness had a major
262 impact (e.g. flooring purposes), the variability of *Eucalyptus* can be reduced with the
263 densification process. The surface hardness had also particularly high impact in the
264 surface wettability.

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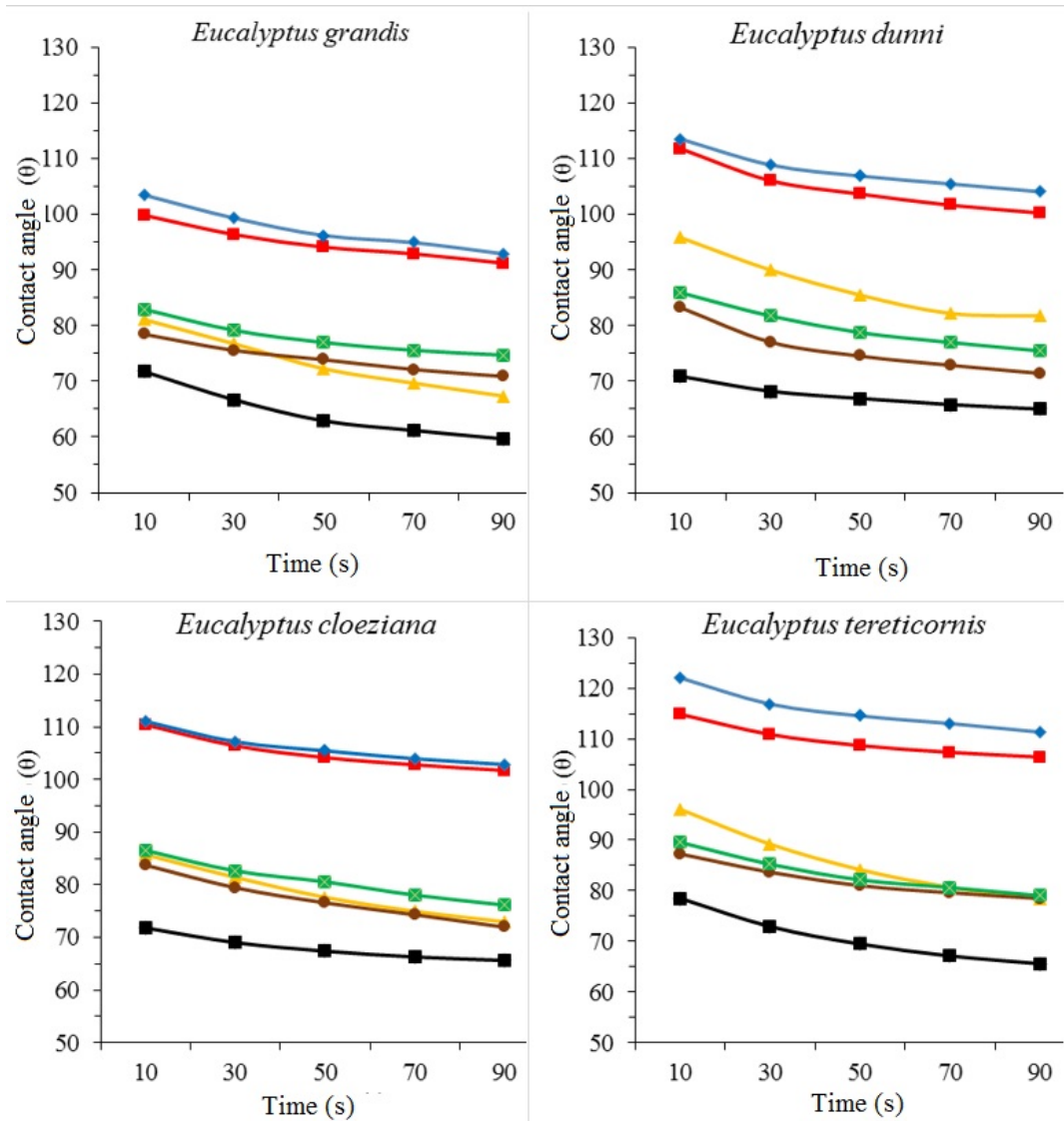
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274 **Wettability**

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

286 possibly due to the smoother surface of the densified samples (Christiansen 1991;
287 Pertuzzatti *et al.* 2016).
288



289
290 **Figure 3:** Contact angle trend over time for the four *Eucalyptus* species undergoing
291 densification and oil post-treatment.

292
293 According to Wålinder and Gardnerb (1999), when the water drop lays with
294 contact angle $< 90^\circ$ a faster penetration, typical for hydrophilic surfaces, occurs. This
295 means that without oil post-treatment the densification process alone does not guarantee

296 to significantly modify the wettability to hydrophobic and the surface can still be easily
297 treated with coatings and adhesives. Conversely, when the HTM densification and the
298 OHT oil post-treatment are combined, the contact angle increases of around 50 % and
299 wood surface remains hydrophobic for longer than 90 seconds.

300 Considering the average price of *Eucalyptus* ($d = 550 \text{ kg/m}^3$) of 190 €/m^3 we can
301 estimate the cost of the densified wood after HTM treatment to around 335 €/m^3 ($d =$
302 700 kg/m^3). The OHT post-treatment can be estimated with 115 €/m^3 and therefore the
303 cost of the HTM and OHT treated wood will be around 450 €/m^3 . This price is highly
304 competitive because with the mechanical and surface properties observed promote the
305 densified *Eucalyptus* can be considered also for flooring and cladding applications for
306 which tropical species like Mogno (*Swietenia macrophylla*), Ipê (*Tabebuia* sp.) and
307 Cedrus (*Cedrela fissilis*) are used and their costs is, at present, around 700 €/m^3 .

308 CONCLUSIONS

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

312 It was observed that limited loss of mass and consistent loss of volume with
313 resulting increase of density were registered for every *Eucalyptus* species. In particular,
314 the ones having lower density reached higher compression degrees and the ones
315 originally heavier reached density over 800 kg/m^3 after HTM process and presented
316 reduced set-recovery after water immersion (down to $< 40 \%$). The study of the
317 mechanical properties highlighted that milder HTM treatment produces more
318 performing densified wood for bending, compression, impact and mostly hardness
319 resistance. Both, densification and oil post-treatment increased significantly the
320 hydrophobicity of the wood surface, but only the joint application of the two treatments

321 allowed to get surfaces with contact angle $> 90^\circ$. The densification process enhanced
322 the mechanical properties and homogenized the properties of the four *Eucalyptus*
323 species studied, while the hot-oil post-treatment was required to obtaining a more
324 hydrophobic surface and for containing the springback effect. The combination of the
325 two treatments carried to an interesting, cheap biomaterial that can be suitable for
326 indoor sight application such as flooring, cladding and ceiling.

327

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