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EFFECT OF HEAT TREATMENT ON PHYSICAL, MECHANICAL AND CHEMICAL PROPERTIES OF ANGELIM WOOD

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ABSTRACT

This study had the objective to heat treated angelim vermelho (red) wood and then to analyze its effects on the physical, mechanical and chemical properties of the wood. The wood was treated at 180 ºC and 215 °C for 20 min and 40 min in a muffle furnace. The basic density, shrinkage, anisotropy, the modulus of rupture and modulus of elasticity, as well as the holocellulose, lignin, extractives and ash content values were obtained for the treated and untreated (control) wood. The results indicated that the basic density was not changed and there was a decrease in volumetric shrinkage in the most severe treatment. The modulus of rupture did not change and the chemical analysis indicated a decrease in the holocellulose and extractives content resulting in lignin content percentage increase, mainly in the most severe test.

Keywords: Chemical analysis, dimensional stability, *Dinizia excelsa*, heat treatment, technological properties

INTRODUCTION

Wood is one of the most used materials by man since our beginnings, having an important role in the development of civilization, both for its versatility and for its high performance. It is an important resource and present in the daily lives in several sectors such as energy, civil construction, cellulose and paper production, and manufacturing of furniture and panels, among others.

In order to use a resource efficiently, it is necessary to understand its characteristics and the best way to manage it to support the final product. In this sense, wood is increasingly studied, and one of the major concerns related to its use is to reduce its dimensional instability and increase the natural durability associated with this material due to its anisotropic and biological nature. It is possible to increase the added value of wood and the competitiveness of sectors which use its products by improving its multiple characteristics.

Intuitively, the technique of treating wood with heat was developed by the Vikings, who heated the ends of the logs as a way to increase the natural durability of the wood. Heat treatment has an effect on the chemical composition of wood by altering its properties. It increases dimensional stability and darkens the color of the wood, making it closer to widely used tropical species (Stamm *et al*. 1946, Larenstein 2009, Kocaefe *et al*. 2015).

Heat treatment reduces the hygroscopicity of wood by degrading its most hydrophilic constituent, hemicellulose. Decreasing the wood's ability to exchange water with the medium minimizes contraction and swelling problems (Aytin *et al*. 2015). When a piece of wood swells or retracts it undergoes deformations due to the dimensional variation provided. Studies involving reduced defects caused by dimensional variation are frequent, since they may enable using wood which has previously been discarded by

the industry, in addition to obtaining a product with higher quality (Borges and Quirino 2004).

Heat treatment is generally applied to low-value species to enable their utilization in harsher environments. Although thermally modified wood tends to be more dimensionally stable than unmodified wood of the same species, mechanical properties generally have a negative effect (Esteves *et al*. 2021). In view of the above, studies that look for the physical, mechanical and chemical effects of treatments are essential and justify this study.

The angelim vermelho (red) wood (*Dinizia excelsa*) has been standing out in recent years as one of the forest species most used by the timber segment in the states of Pará and Amazonas. Wood is used indoors, in the manufacture of wooden frames, stair steps and widely in civil construction (Baraúna 2011). The basic density for red angelim according to the IPT - Institute of Technological Research (2013) is 830 kg/m³, tangential contraction of 6,6 % and radial contraction of 4,2 %, modulus of elasticity in the green condition of 14,073 MPa. Thus, improving dimensional stability conditions can expand the uses of this species in the country.

In an attempt to study the effect of heat treatment on hygroscopicity and durability of wood, this work aimed to apply heat treatments of different temperatures and times to the angelim vermelho (red) wood, and then to evaluate the effects on the wood's physical, mechanical and chemical properties, thereby seeking to reduce dimensional instability problems with minimal compromise to other properties, in turn valuing and expanding the use of wood of this species.

MATERIALS AND METHODS

Origin and manufacture of the specimens

The angelim *vermelho* wood (*Dinizia excelsa* Ducke) used in the study came from an industry in the Federal District of Brasília, Brazil. Three initial heartwood pieces of 30 cm x 6 cm x 200 cm (width, thickness and length) were obtained from three different trees who had their identification confirmed by wood anatomists of the Wood Anatomy Sector of the Forest Products Laboratory of the Brazilian Forest Service (LPF-SFB). The pieces were sawn into specimens in the Carpentry Sector of the LPF into 35 samples of 2 cm x 2 cm x 30 cm (width, thickness and length) and 35 samples of 2cm x 2cm x 10cm, totaling 70 samples.

The specimens were placed in an air-conditioned room (21 ºC and 65 % relative humidity) where they remained for 50 days to reach an average moisture content of 12 % and then proceeded to receive heat treatments.

Heat treatments

The heat treatment tests were carried out at the Forest Products Laboratory (LPF) in the Energy Sector, using a Quimis muffle furnace.

Preliminary tests were carried out to verify the best conditions for the treatments and the heating rate adopted from these tests was 2 °C/min. The heat treatments were divided into 3 phases: Phase 1 was heating, defined from t0 to t1. The time that the samples remained in the muffle furnace after reaching the final temperature (T*) is called the final threshold, defined from t1 to t2, and constitutes Phase 2 of the process. The heat treatment ends at the end of the final stage and the muffle furnace moves to the cooling stage, defined as Phase 3 (t2 to t3). The initial moisture content of the heat-treated samples was 12 %. Figure 1 shows the program adopted for the heat treatments.

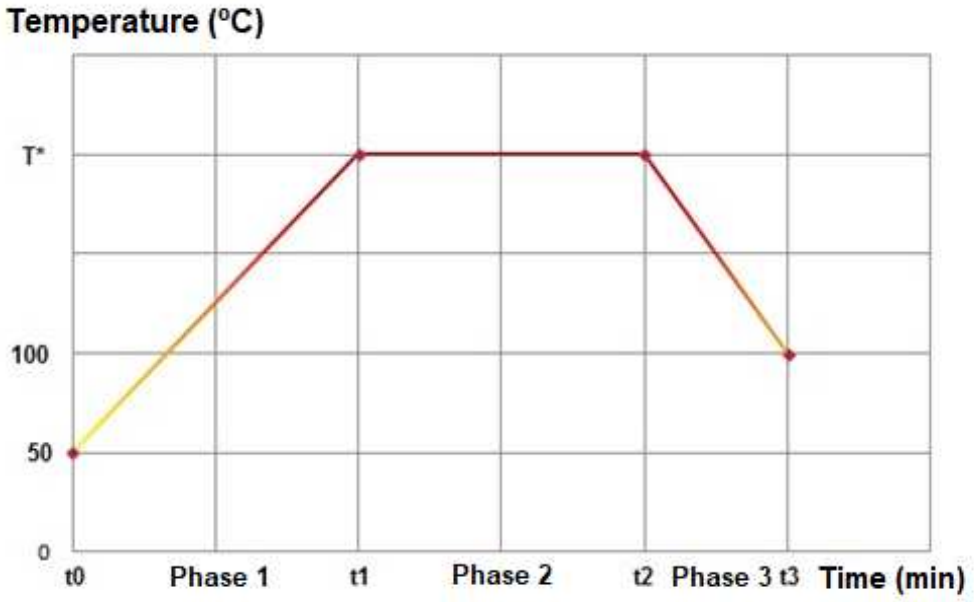

Figure 1: Temperature program and time applied for the heat treatments of the angelim wood samples.

As can be seen in Figure 1, the performed heat treatments started at a temperature of 50 ° C. The samples were then removed from the muffle furnace in the cooling step when it reached a temperature of around 100 ° C. These temperatures were chosen based on the results observed in the preliminary tests. Specimens with dimensions of 2 cm x 2 cm x 10 cm and 2 cm x 2 cm x 30 cm were used for the tests, being arranged in the furnace so as not to touch the internal walls by means of wooden separators.

The samples were divided into five treatments (Control, T1, T2, T3 and T4) in which 30 samples were used for each treatment. The heat treatments (T1, T2, T3 and T4) were separated according to the time and exposure temperature. The temperatures and times tested were 180 ° C and 215 ° C and 20 min and 40 min, respectively, as described in Table 1.

Table 1: Treatments used for wood samples as a function of temperature and time.

Treatment	Final temperature (°C)	Furnace time – Phase 2 (min)
Control	0	0
T1	180	20
T2	180	40
T3	215	20
T4	215	40

The specimens with dimensions of 2 cm x 2 cm x 10 cm were subsequently used in the density, mass loss and shrinkage tests, while those of 2 cm x 2 cm x 30 cm were used in the static bending tests and in the chemical analysis.

Physical and mechanical properties

Density, mass loss, retractability and anisotropy coefficient

The basic density was determined according to the COPANT 461 (1972a). The moisture content of the wooden pieces before the heat treatment was 12 %. It was initially necessary to calculate the dry mass of the wood based on its moisture to evaluate the mass loss, according to Equation 1.

$$dm = \frac{100\ x\ im}{M+100} \quad (1)$$

In which: dm = dry mass of wood before heat treatment (g); im = initial mass of the wood (g); M = initial moisture content of the wood (%);

The mass loss was calculated using the dry mass result based on Equation 2.

$$ML = \frac{dm-fm}{dm}\ x\ 100 \quad (2)$$

In which: ML = mass loss (%); dm = dry mass of the wood before heat treatment (g); fm = final mass of wood after heat treatment (g).

143 COPANT 462 (1972b) was used to determine the sample retractabilities and the
144 calculated anisotropy coefficients were equal to the ratio between tangential and radial
145 retractabilities. The specimens had their measurements of radial, tangential and
146 longitudinal dimensions obtained after the treatments in oven dry condition at 103 ºC ± 2
147 ºC and in condition of complete fiber saturation, after submerged in water for 40 days,
148 using a digital caliper. Equations 3 and 4 were used to calculate linear (radial and
149 tangential) shrinkage and volumetric shrinkage, respectively. Based on the values
150 obtained for radial and tangential shrinkage, the anisotropy coefficient was obtained by
151 Equation 3.

152 $$Rt\ or\ Rr = \frac{Du-Ds}{Du}\ x\ 100 \quad (3)$$

153 In which: Rt = tangencial retractability (%); Rr = radial retractability (%); Du = linear
154 dimension (tangential or radial face) of the sample in saturated condition (cm); Ds = linear
155 dimension (tangential or radial face) of the sample in dry condition (cm);
156

157 $$Rv = \frac{Vu-Vs}{Vu}\ x\ 100 \quad (4)$$

158 In which: Vr = volumetric retractability (%); Vu = sample volume in saturated condition
159 (cm³); Vs = sample volume in dry condition (cm³);
160

161 $$AC = \frac{Rt}{Rr} \quad (5)$$

162 In which: AC = anisotropy coefficient; Rt = tangencial retractability (%); Rr = radial
163 retractability (%).
164

165 **Modulus of elasticity and modulus of rupture**

166 Equations 6 and 7 were used to determine the elastic modulus (MOE) and modulus
167 of rupture (MOR), respectively, and the results were transformed to MPa. The test was
168 carried out at the Wood Technology Laboratory in the Forest Engineering Department

(UnB) using an EMIC DL model universal testing machine with a load capacity of 30 kN, following the COPANT 555 (1972c) standard.

$$\mathrm{MOE} = \frac{PL^3}{4bdh^3} \quad (6)$$

In which: MOE = modulus of elasticity to static bending (kgf/cm²); P = load at the proportional limit (kg); d = deformation corresponding to the load at the proportional limit (cm); L = distance between supports, free span (cm).

$$\mathrm{MOR} = \frac{3PmL}{2bh^2} \quad (7)$$

In which: MOR = modulus of rupture with static bending (kgf/cm²); Pm = maximum applied load (kg); L = distance between supports, free span (cm); b = sample base (cm); h = height of the sample (cm).

The values were later transformed from kgf/cm² to MPa.

Chemical analysis

The chemical analysis was conducted at the Chemistry Sector of LPF. The wood was prepared for the analysis following the procedures of TAPPI 257 cm-85 (1996), the moisture content was determined according to TAPPI 264 om-88 (1996), and the extractives content according to TAPPI 204 om-88 (1996). Total lignin was obtained by adding the soluble and insoluble lignin obtained using LAP 003 and 004 laboratory procedures. The ash content of the wood was measured according to the TAPPI 211 om-93 standard (1996); and finally, holocellulose, which is the sum of cellulose plus hemicellulose, was obtained through the difference between the mass of the extractive-free material, the total lignin and the ash (Severo *et al*. 2012)

194 **Statistical analyses**

195 The values of the results obtained in the tests were submitted to analysis of
196 variance (ANOVA) using the the ASSISTAT 7.7 program in order to verify if there was
197 a statistical difference between the treatments. The Tukey means test at 5 % significance
198 was applied for data which differed statistically, meaning when the F value was
199 significant ($\alpha = 0,05$).

200 The normality of the distributions between the means was verified by the Shapiro-
201 Wilk test at 95 % probability before the analysis of variance.

202

203 **RESULTS AND DISCUSSION**

204 **Physical and mechanical properties**

205 **Density, mass loss, retractabilities and anisotropy coefficient**

206 Table 2 contains the mean values of the physical properties of the angelim
207 *vermelho* wood.

208 **Table 2:** Values of the physical properties of angelim wood.

Treatment	Bd (kg/m³)	Vr (%)	Rt (%)	Rr (%)	AC	ML (%)
Control	853a (80)	10,85a (1,29)	6,46a (0,68)	4,39a (0,74)	1,49a (0,17)	- -
T1	872a (30)	9,64ab (1,22)	5,65a (1,00)	3,98ab (0,48)	1,43a (0,24)	7,49a (0,02)
T2	853a (70)	9,05ab (1,09)	5,48a (1,02)	3,57b (0,20)	1,53a (0,28)	7,73a (0,02)
T3	835a (50)	9,17ab (1,67)	5,64a (1,41)	3,53b (0,35)	1,59a (0,32)	8,09a (0,02)
T4	850a (20)	8,44b (1,37)	4,99a (1,19)	3,45b (0,24)	1,44a (0,28)	8,86a (0,02)

Bd = basic density; Vr = volumetric retractability; Rt = tangencial retractability; Rr = radial retractability; AC = anisotropy coefficient; ML = mass loss; The values in parentheses refer to the standard deviation. Values marked with different letters for the means, within the same column, differ from each other at the level of 5 % significance by the Tukey test.

209

210 The average basic density found for untreated wood was 0,85 g/cm³, close to that
211 found by Nascimento *et al*. (1997) of 0,88 and by the IBDF (1983) of 0,83 g/cm³. The

212 tangential and radial retractability values of untreated wood were equal to 6,46 % and
213 4,39 %, respectively, being lower than those found by the IBDF (1983) of 9,5 % and 5,7
214 %, and by Chichignoud *et al*. (1990) of 8,3 % and 5,7 %, and very close to those found
215 by the IPT (2013) of 6,6 % for tangential contraction and 4,2 % for the radial contraction.
216 The heat treatments did not statistically change the basic density, the tangential
217 retractability or the anisotropy coefficient. On the other hand, volumetric and radial
218 retractability obtained a significant reduction in their values from the first heat treatment
219 (T1). They had the greatest reduction for the T4 treatment at 215 ºC for 40 min, being
220 22,21 % for volumetric retractability and 21,41 % for radial retractability in relation to
221 the control. There was no statistical difference for the mass loss of angelim wood between
222 the studied heat treatments; however, it is possible to notice that there was a slight
223 increase in mass loss with the increase in temperature and time. The same was reported
224 by Juizo *et al*. (2018), who subjected eucalyptus wood to heat treatment in a closed system
225 oven at temperatures and exposure times ranging from 180 ºC to 240 ºC and 2 h and 4 h.
226 These authors found a reduction in mass loss and a consequent reduction in apparent
227 density, with a tendency to further decrease with increasing temperature, reaching a
228 reduction of around 16,43 % in relation to untreated wood for the highest exposure
229 temperature of 240 ºC.
230 Ferreita *et al*. (2019) heat treated angelim pedra (stone) wood using temperatures
231 of 180 ºC and 200 ºC with exposure times varying from 2 h to 4 h in a forced air circulation
232 oven; similarly to this study, they did not obtain a significant reduction in the density of
233 the treated wood. Menezes *et al*. (2014) evaluated the effect of thermal modification on
234 total swelling and anisotropy coefficient in *Corymbia citriodora* and *Eucalyptus saligna*
235 woods treated at temperatures of 140 ºC, 160 ºC and 180 ºC for 2,5 h using a laboratory
236 greenhouse. They observed that the thermal modification was generally efficient in

reducing the total swelling and the anisotropy coefficient of both species, with the most expressive values being observed in the treatments with higher temperatures, just as observed in this study.

Takeshita and Jankowsky (2015) heat treated jatobá and muiracatiara woods at temperatures below those normally used from 60 ºC to 90 ºC. They concluded that the heat treatment with a temperature of 90 ºC was more effective, reducing the hygroscopicity of the wood and consequently its dimensional movement. Batista *et al*. (2011), Borges and Quirino (2004) and Pertuzzatti *et al*. (2016) analyzed the effect of heat treatment on *Eucalyptus grandis*, *Pinus caribaea* and *Pinus elliottii*, respectively, and also concluded that heat treatments were efficient in reducing the dimensional instability of species by reducing their retractability.

Additionally, it is observed that the different temperatures and times studied, as well as the type of wood (species) and the equipment used in each study (laboratory oven, muffle oven and autoclave) showed different behaviors. As in this study, it is generally possible to observe that heat treatment contributes to improving wood stability.

The basic density did not significantly change with the application of heat treatments. Hill (2006) observed similar results, and concluded that the density does not change in cases where the variation in mass and volume happens in a similar way. Just as in this study, Brito *et al*. (2006) did not detect significant changes in the basic density of heat-treated eucalyptus wood. Other authors describe a growth in the mass loss and density trends with increasing temperatures (Borrega and Karenlampi 2008, Surini *et al*. 2012, Moura *et al*. 2012, Bal 2013, Poubel *et al*. 2013, Conte *et al*. 2014, Nabil *et al*. 2018). According to Esteves and Pereira (2009), the degradation of hemicelluloses in volatile products and the evaporation of extracts are the main reasons for the wood density reduction.

Modulus of elasticity and modulus of rupture

Table 3 contains the mean values of the mechanical properties (static bending) of the angelim wood.

Table 3: Mechanical property values of angelim wood.

Treatment	Static bending	
	MOR (MPa)	**MOE (MPa)**
Control	121,38a (23,8)	12600b (876,8)
T1	133,85a (35,4)	15156a (1,408)
T2	89,53a (30,1)	12592b (2,418)
T3	98,01a (30,4)	11344c (1,842)
T4	107,66a (35,0)	13129b (2,220)

MOR = modulus of rupture with static bending; MOE = modulus of elasticity; The values in parentheses refer to the standard deviation. Values marked with different letters for the means within the same column differ from each other at the 5% significance level by the Tukey test.

The values found for the untreated wood (control) were 121,38 MPa for the modulus of rupture and 12600 MPa for the modulus of elasticity to static bending. These values are close to those found in the literature for the species (IBDF – Brazilian Forest Defense Institute 1983, Chichignoud *et al*. 1990, Nascimento *et al*. 1997).

The values related to the modulus of rupture to static bending are statistically equal between the control and the heat-treated samples, showing that the studied heat treatments did not significantly affect this property. The heat treatments did not differ in relation to the control for the modulus of elasticity to static bending, except for the T1 and T3 treatments. The T1 heat treatment favored this property, with a significant increase in the modulus of elasticity to static bending, while the T3 treatment led to a decrease in the value of this property. Despite inconsistent results from more severe treatments with

278 smaller reductions in mechanical properties, the absence of significant statistical change
279 shows that this inconsistency is not relevant.

280 Wood can lose mechanical resistance during heat treatment due to the decrease in
281 components such as xylose, galactose and arabinose. However, each wood can react
282 differently due to the variation between the types of heat treatments and the intrinsic
283 characteristics of each species (Winandy and Rowell 2005). When studying the thermal
284 modification of wood in France, Vernois (2000) points out that depending on the species,
285 the mechanical properties at temperatures up to 210 °C maintain values close to the
286 original values.

287 Motta *et al*. (2013) did not find any influence of heat treatments on MOR and
288 MOE to dynamics bending when heat treating Tectona grandis wood. Zhang *et al.* (2013)
289 observed a slight increase in the modulus of elasticity to static bending, followed by a
290 decrease in this property with the increase in the temperature of the tests, as reported in
291 the study.

292 Likewise, Ferreita *et al.* (2019) thermally modifying the wood of angelim pedra
293 using temperatures of 180 ºC and 200 ºC with exposure times ranging from 2 h to 4 h did
294 not verify changes in the mechanical properties.

295 **Chemical analysis**

296 Table 4 shows the mean values of the chemical analyzes of heat treated and
297 untreated wood (control).

298

299

300

301

302 **Table 4:** Chemical analysis values of angelim wood.

Treatment	EC (%)	LInsC (%)	LSolC (%)	TLC (%)	AC (%)	HC (%)
Control	16,95a (0,36)	35,71a (0,28)	1,70a (0,05)	37,41a (0,33)	0,43a (0,01)	62,17a (0,33)
T1	11,83bc (0,37)	38,06b (0,24)	1,60ab (0,05)	39,66b (0,28)	0,41a (0,06)	59,93b (0,28)
T2	12,68b (0,18)	38,21b (0,85)	1,55bc (0,03)	39,76b (0,84)	0,26a (0,03)	59,99b (0,84)
T3	10,95cd (0,77)	39,11b (0,76)	1,48cd (0,05)	40,58b (0,74)	0,34a (0,08)	59,08b (0,74)
T4	9,83d (0,74)	42,32c (0,33)	1,42d (0,05)	43,74c (0,30)	0,36a (0,01)	55,90c (0,30)

EC = extractives content; LInsC = acid-insoluble lignin content; LSolC. = acid-soluble lignin content; TLC = total lignin content; AC = ash content; HC = holocellulose content. The values in parentheses refer to the standard deviation. Values marked with different letters for the means within the same column differ from each other at the 5% significance level by the Tukey test. Note: The insoluble and soluble lignin, ash and holocellulose contents refer to extractive-free wood, which means removing the extractive content percentage.

303
304 The extractives content decreased with increasing temperatures, while the total
305 lignin behaves in an inverse way so that the increase in temperature and time leads to an
306 increase in the total lignin content due to the percentage decrease of the other components.
307 Brito *et al*. (2008) and Severo *et al*. (2012) also observed a decrease in the holocellulose
308 content and a consequent increase in the total lignin content.

309 Calonego (2017) in a thesis on the thermal modification of *Schizolobium*
310 *parahyba* wood, treated the species in an electric furnace at 180 ºC, 200 ºC and 220 ºC at
311 1,34 ºC/min for 2,5 h. The author reports similar results to the study, in which the
312 treatments caused significant reductions in holocellulose contents and a proportional
313 increase in the respective lignin contents and total wood extractives.

314 The reduction of hemicelluloses and part of the cellulose amorphous region
315 decreases the water absorption sites and hydroxyl groups, thus decreasing the dimensional
316 instability of the wood (Hillis and Rozsa 1985). The most significant reductions in

317 holocellulose occurred in the T4 treatment. Chemical modifications did not significantly
318 alter the mechanical properties of the wood.

319 Menezes (2017) heat treated *Tectona grandis* wood according to the VAP
320 HolzSysteme® industrial process using a final temperature of 160 ºC, and reported that
321 the basic density decreased due to the mass loss of the holocellulose and alteration of the
322 total extracts. Furthermore, the total swelling and anisotropy of wood swelling decreased
323 due to the degradation of holocellulose, as occurred in this study, improving the
324 dimensional stability of the wood.

325

326 **CONCLUSIONS**

327 The heat treatments did not significantly change the basic density and the mass
328 loss was similar between the treatments. The temperature of 215 ºC promoted greater
329 mass loss.

330 Volumetric shrinkage reduced when the wood was treated at 215 ºC for 40 min.
331 There was a significant reduction in the holocellulose content for this same test condition,
332 making the T4 treatment the most recommended to make the wood more dimensionally
333 stable.

334 The chemical analyzes detected a reduction in the extractives content and in the
335 holocellulose content, as well as an increase in the lignin content in relation to the
336 controls. For the lignin and holocellulose content, the most expressive changes occurred
337 in the most severe tests, at 215 ºC for 40 min.

338 The modulus of rupture to bending had no significant change in any of the tested
339 heat treatments, thus the heat treatments did not compromise the analyzed mechanical
340 property.

It is recommended, in future studies, to test other temperatures and different periods of time. The improvement of the equipment, such as the use of other atmospheres, such as nitrogen and vacuum, is also recommended. The use of infrared spectroscopy should be tested as a tool for monitoring the thermal treatments of wood. Chemical determination of cellulose and hemicellulose separately is also recommended.

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