

1
2 **EFFECT OF THE THERMAL TREATMENT ON THE CHEMICAL**
3 **COMPONENTS, SORPTION, AND SHRINKAGE PROPERTIES OF**
4 ***Tectona grandis* JUVENILE WOOD**

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22 **ABSTRACT**

23 The effect of thermal treatment on the chemical components, equilibrium moisture content (EMC),
24 and shrinkage of teak juvenile wood was studied. Heartwood and sapwood samples were thermally-
25 treated at 180 °C and 200 °C. Extractive, Klason lignin, holocellulose, and α -cellulose contents, as
26 well as pH on untreated and thermally-treated woods, were determined. The EMC was reached at
27 five relative humidity (RH) levels using saturated salt solutions: 86 % (KCl), 76 % (NaCl), 58 %
28 (NaBr), 33 % (MgCl₂), and 0 % (P₂O₅). Linear and volumetric shrinkages were calculated for all
29 EMCs. The ratio of sorption (S), coefficient of shrinkage (h), and fiber saturation point (FSP) were
30 also determined. Thermally-treated wood exhibited lower holocellulose and α -cellulose contents
31 than untreated wood and increased acidity due to degradation of the hemicelluloses. The thermal
32 treatment reduced the EMC of heartwood and sapwood. However, sapwood was more sensitive to
33 RH variations than heartwood regardless of the treatment. Thermally-treated woods had higher
34 hygroscopic and dimensional stabilities, and lower FSP than untreated wood. The thermal treatment
35 did not affect radial shrinkage of the heartwood between 33 % and 86 % RH. Heartwood was more
36 sensitive to the effect of the thermal treatment on shrinkage and degradation of cell wall polymers
37 compared to sapwood.

38 **Keywords:** Chemical modification, dimensional stability, equilibrium moisture content, shrinkage,
39 teak, *Tectona grandis*.

40 INTRODUCTION

41 Thermal modification is a technique that has been extensively studied in the last years because it
42 enhances several wood properties such as lower hygroscopicity and higher dimensional stability
43 (Giebeler 1983) and decay resistance (Weiland and Guyonnet 2003). The treatment provokes
44 physical and chemical changes to wood, such as extractive volatilization, formation of new
45 compounds (e.g. anhydrosugars and phenolic compounds), degradation of the hemicelluloses
46 (Esteves *et al.* 2011), changes in the lignin structure (Kim *et al.* 2014), increase of crystalline
47 cellulose (Li *et al.* 2015), and mass losses (Garcia *et al.* 2012), among other modifications. These
48 chemical changes reduce the OH groups available in cell walls, promoting lower hygroscopicity
49 and higher dimensional stability to thermally-treated woods.

50 The teak (*Tectona grandis*) plantations in Brazil have shorter cutting cycles (15 to 25 years)
51 compared to those in its native region and other regions around the world (Tsukamoto Filho *et al.*
52 2003, FAO 2000) due to favorable edaphoclimatic conditions and management techniques.
53 However, teak wood from fast-grown plantations exhibits high proportions of juvenile wood and
54 sapwood, which have different anatomical, physical, chemical, and aesthetic properties than those
55 of mature wood and heartwood (Bhat *et al.* 2001, Lopes 2012, Darmawan *et al.* 2015). Studies have
56 shown that the proportion of juvenile wood measured at breast height (1.3 m) is 80-100 % for 20-
57 year-old trees (Bhat *et al.* 2001) and 100 % for 10-year-old trees (Darmawan *et al.* 2015). Juvenile
58 wood presents, for example, lower density, lower shrinkage, shorter fibers, higher microfibril angle,
59 and lower bending strength than mature wood (Darmawan *et al.* 2015). Teak heartwood presents
60 good dimensional stability and high biological durability due to some substances such as
61 caoutchouc (Yamamoto *et al.* 1998), tectoquinone (β -methyl anthraquinone), 2-(hydroxymethyl)
62 anthraquinone, and lapachol (Niamké *et al.* 2011). On the other hand, teak sapwood presents a
63 lighter color, lower dimensional stability and low resistance to the attack of xylophagous organisms

64 than heartwood (Lopes 2012, Motta *et al.* 2013). Therefore, treatments to improve the properties of
65 sapwood are required to increase the yield of this raw material. Previous studies have shown that
66 thermal treatments provide color uniformity to pieces of teak wood containing heartwood and
67 sapwood (Lopes *et al.* 2014). In addition, thermally-treated teak wood can show higher color
68 stability after exposure to ultraviolet radiation depending on the treatment conditions (Garcia *et al.*
69 2014).

70 Studies about the dimensional stability of wood are essential for its use because it depends on the
71 relationships between specific gravity and the relative humidity (RH) of the air (Tsoumis 1991).
72 Particularly for teak species, studies have reported the volumetric swelling (Kartikawati *et al.* 2020)
73 and anti-swelling efficiency of heat-treated and untreated 15-year-old teak wood from Indonesia
74 (Priadi *et al.* 2019), the maximum shrinkage of teak wood from the West Africa region (tree age
75 was not specified) (Govorčín *et al.* 2010) among others. However, there are no studies on the
76 dimensional stability of thermally-treated teak juvenile wood under different RH conditions, in
77 particular teak wood from fast-growing plantations in Brazil, which highlights the originality of our
78 study. In general, teak wood is popular in outdoor applications (Weaver 1993), which require
79 materials with attractive color, high dimensional stability under different climatic conditions, and
80 high resistance to pathogens and weathering (Kokutse *et al.* 2006).

81 With the ultimate goal of contributing to a better understanding of the properties of teak wood after
82 heat treatment and to enhance the use of teak wood from young plantations in Brazil, this study
83 aimed, in the first part, to assess the effect thermal treatments on the chemical composition of
84 juvenile teak wood (heartwood and sapwood), while in the second part, we studied the EMC and the
85 variation of shrinkage under different RH of thermally-treated wood.

86 MATERIAL AND METHODS

87 Material and thermal treatment

88 Twelve-year-old teak (*Tectona grandis* Linn. f.) trees were harvested from a plantation located in
89 Caceres city, Mato Grosso State, Brazil (Latitude: 16° 04' 14" S, Longitude: 57° 40' 44" W). Based
90 on previous studies (Bhat *et al.* 2001; Flórez *et al.* 2014; Darmawan *et al.* 2015), we have assumed
91 that the samples contained only juvenile wood as they were obtained from fast-growing young
92 trees. In addition, samples were taken from the second log cut from the diameter at breast height,
93 where the proportion of juvenile wood is greater. Samples of 150 mm (L) x 75 mm (T) x 20 mm (R)
94 were prepared from heartwood or sapwood.

95 Wood samples were heat-treated in a muffle furnace of 600 mm x 600 mm x 700 mm equipped with
96 a temperature and time controller. The treatment was performed at four steps: (1) heating up to 100
97 °C for 2 h, (2) heat increasing from 100 °C to final temperature (180 °C or 200 °C) for 30 min, (3)
98 thermal exposure at the final temperature for 2,5 h, and (4) cooling for approximately 24 h. Before
99 and after thermal treatment, samples were conditioned at 20 °C and 65 % RH until mass equilibrium
100 was reached.

101 **Chemical analysis**

102 Chemical analyses were done according to ASTM-D1105-96 (ASTM 2001) and ASTM-D1106-96
103 (ASTM 2001) to determine extractive, Klason lignin, holocellulose, and α -cellulose contents. The
104 extractive contents were obtained after extractions in a Soxhlet apparatus for 24 h using three
105 organic solvents of increased polarity: cyclohexane, ethyl acetate, and methanol.

106 The Klason lignin was obtained after treatment of approximately 300 mg extractive-free dry ground
107 wood with 72 % sulfuric acid (H_2SO_4) in a water bath at 30 °C for 1 h, followed by dilution to 84
108 mL distilled water and refluxing for 4 h. The residue was washed with 500 mL hot distilled water
109 and oven-dried at $103\text{ °C} \pm 2\text{ °C}$ for 24 h. The Klason lignin content was estimated by dividing the
110 oven-dry weight of the residue (insoluble lignin) by the weight of the extractive-free wood.

111 The holocellulose fraction was determined by the chlorination method using 2,5 g of extractive-free
112 dry wood added of 80 mL hot distilled water, 0,5 mL acetic acid, and 1 g sodium chlorite ($NaClO_2$).

113 The mixture was heated in a water bath at 70 °C, and another dose of 0,5 mL acetic acid and 1 g
114 NaClO₂ were added every 1 h until the fibers were completely separated. The mixture was left for
115 24 h without reagent addition and was then filtered under vacuum and washed with distilled water
116 until the yellow color and chlorine odor completely disappeared. The residue was oven-dried at 103
117 °C ± 2 °C for 24 h and weighed. The holocellulose content was then estimated by dividing the oven-
118 dry weight of the residue (holocellulose) by the weight of the extractive-free wood.

119 The α-cellulose was obtained after treatment of 2 g dry holocellulose with 10 mL of 17,5 % sodium
120 hydroxide (NaOH) in a water bath at 20 °C. Another 5 mL of 17,5 % NaOH was added to the
121 mixture at 5 min intervals for a total treatment of 45 min. A 33 mL distilled water at 20 °C was
122 added and left for 1 h. The mixture was filtered under vacuum and subsequently washed with 100
123 mL of 8,3 % NaOH and with distilled water. The cellulose residue was oven-dried at 103 °C ± 2 °C
124 for 24 h. The cellulose content was estimated by dividing the oven-dry weight of the cellulose
125 residue by the weight of the dry holocellulose residue.

126 The pH of wood was determined as described by Lelis (1995). A 5 g of ground wood oven-dried at
127 103 °C ± 2 °C for 24 h was mixed with distilled water at room temperature and left for 24 h. The
128 mixture was filtered and the pH was measured with an electronic pHmeter.

129 Three replicates per treatment were used for each chemical component and pH analysis.

130 **Sorption tests**

131 Samples of 20 mm (L) x 15 mm (T) x 13 mm (R) were prepared with the untreated and heat-treated
132 wood pieces of each wood type: heartwood and sapwood. Firstly, the samples were full saturated
133 following a mild procedure in three steps: (1) adsorption in desiccators containing potassium
134 chloride - KCl (86 % RH) at 21 °C with mass measurements until equilibrium was reached; (2)
135 adsorption in desiccators over deionized water until mass equilibrium was reached; and (3)
136 immersion in deionized water for seven days until full saturation was reached. Afterward, samples
137 were separated into five matched groups. One group was immediately put in a desiccator over

138 phosphorus pentoxide - P_2O_5 (0 % RH). The other four groups were placed for desorption in
139 desiccators at four levels of RH: 86 %, 76 %, 58 %, and 33 %, by using saturated salt solutions of
140 potassium chloride (KCl), sodium chloride (NaCl), sodium bromide (NaBr), and magnesium
141 chloride ($MgCl_2$), respectively. A total of 42 samples, 7 replicates for each treatment [wood type
142 (sapwood and heartwood) and temperature (control or untreated, and treated at 180 °C, and 200 °C)]
143 were used for each sorption condition. Samples were placed into two desiccators per RH condition.
144 Desorption tests were conducted simultaneously on all samples using sorption vats as described by
145 Hernández and Pontin (2006). The temperature of the vats was established at 21 °C. These vats
146 provided temperature control of $\pm 0,01$ °C for long periods and thus allowing control of RH in the
147 various desiccators serving as small sorption chambers. The samples were placed in plastic supports
148 with a perforated base to promote the exchange of moisture. The weight of samples was measured
149 periodically without removing them from the desiccators until a constant mass was reached.
150 All mass of samples was taken to the nearest 0,001 g and their dimensions were measured in the
151 three principal directions with a micrometer to the nearest 0,001 mm after full saturation.
152 Dimensions and mass of the four groups of conditioned samples were also measured once their
153 corresponding sorption equilibrium condition was reached and then they were immediately oven-
154 dried at 103 °C for 24 h. Samples dimensions at the oven-dried state were measured after a 20 min
155 period of cooling over P_2O_5 .

156 **Equilibrium moisture content and ratio of sorption**

157 The EMC of untreated and thermally-treated woods was calculated with the mass of the samples at
158 equilibrium and the oven-dried mass, expressed as a percentage of oven-dry mass.

159 The sorption ratio (S) is a parameter of hygroscopicity proposed by Noack *et al.* (1973) to
160 characterize the sensitivity of the EMC variation (ΔEMC) related to the variation in RH (ΔRH)
161 (Equation 1). This parameter assumes that there is a linear relationship between EMC and RH.
162 Therefore, the S-ratio was calculated for desorption between 33 % and 76 % RH.

163
$$S = \Delta EMC / \Delta RH \quad (1)$$

164 **Shrinkage and coefficient of shrinkage**

165 The linear shrinkages in the tangential (β_T), radial (β_R), and longitudinal (β_L) directions were
166 calculated in percentage. Volumetric shrinkage was estimated as the sum of these three directional
167 measurements [β_V involves the product $\beta_T \times \beta_R$ in order to achieve sufficient accuracy within the
168 range of β_T and β_R values normally found for wood, as described by Skaar (2012)] (Equation 2).
169 Shrinkages of untreated and thermally-treated woods were obtained for all EMCs.

170
$$\beta_V(\%) = \beta_T + \beta_R + \beta_L - (\beta_T \times \beta_R) \quad (2)$$

171 The coefficient of shrinkage (h) is a general index of dimensional stability proposed by Noack *et al.*
172 (1973) to determine the swelling/shrinkage of the wood for every 1 % RH. The h -coefficient was
173 calculated with the volumetric shrinkage ($\Delta\beta_V$) for desorption between 33 % and 76 % RH
174 (Equation 3).

175
$$h = \Delta\beta_V / \Delta RH \quad (3)$$

176 **Statistical analysis**

177 Statistical analyses were done with a STATISTIC 10.0 software. Analysis of variance (ANOVA)
178 and Tukey's tests (at 0,01 of the probability level) were used to verify the effects of the wood type
179 (heartwood and sapwood) and temperature (control or untreated and treated at 180 °C, and 200 °C)
180 on the extractives, Klason lignin, hemicellulose, and α -cellulose contents, pH, EMC, and shrinkage
181 components (β_T , β_R , β_L , and β_V). The normality and homogeneity of variance were verified by the
182 Shapiro-Wilk and Levene's tests.

183 **RESULTS AND DISCUSSION**

184 **Chemical changes**

185 A significant interaction was found between wood type (heartwood, sapwood) and temperature for
186 all chemical components (extractive, Klason lignin, holocellulose, and α -cellulose) and pH (Tables
187 1 and 2). The untreated heartwood had a higher extractive content than the untreated sapwood. The

188 extractive content gradually increased as the temperature of treatment increased for both wood
 189 types (Table 3). The total extractive content of treated heartwood and sapwood respectively
 190 increased by 53 % and 64 % at 180 °C and by 122 % and 100 % at 200 °C compared to untreated
 191 woods. Similar behavior for the extractive content was observed by other authors after thermal
 192 treatments (Gašparík *et al.* 2019, Bellon 2013, Lengowski 2011). Gašparík *et al.* (2019) found an
 193 increase of 56 % on teak extractive after the ThermoWood® treatment at 210 °C. Also, the
 194 extractive content increased by 135 % (Bellon 2013) and 158 % (Lengowski 2011) in the 160 °C-
 195 treated teak wood from VAP HolzSysteme® industrial process compared to untreated wood.
 196 However, these studies did not separate heartwood from sapwood material. The increase of the
 197 extractive content is not related to the natural extractives of wood, which are in large part
 198 volatilized during the thermal treatment, but to by-products formed mainly from the degradation of
 199 the hemicelluloses. According to International ThermoWood Association (2003), temperatures
 200 ranging from 120 °C to 180 °C increase the extractive content of wood while the temperature up to
 201 230 °C causes a decrease in extractives.

202 **Table 1:** F-values obtained from the ANOVA of the effects of the wood type (heartwood, sapwood)
 203 and temperature treatment on the extractive content of teak wood.

Source of variation	Extractive content			Total extractive content
	Cyclohexane	Ethyl acetate	Methanol	
Wood type	37,4**	1367,2**	722,5**	34,9**
Temperature	865,1**	443,4**	375,0**	1218,6**
Wood type*Temperature	111,8**	15,4**	23,3**	24,7**

**Significant at the 1 % probability level.

204

205 **Table 2:** F-values obtained from the ANOVA of the effects of the wood type (heartwood, sapwood)
 206 and temperature treatment on the chemical components and pH of teak wood.

Source of variation	Klason lignin	Holocellulose	α -cellulose	pH
Wood type	6,1*	784,0**	1312,7**	2212,1**
Temperature	6,6*	707,2**	33,3**	512,1**
Wood type*temperature	2,0 ^{ns}	109,1**	2,6 ^{ns}	21,6**

*, ** Significant at the 5 % and 1 % probability level respectively; ^{ns} not significant.

207 **Table 3:** Means of the extractive contents of untreated and thermally-treated teak heartwood and
 208 sapwood.

Wood type	Temperature	Extractive content (%)	Total extractive
-----------	-------------	------------------------	------------------

	(°C)	Cyclohexane	Ethyl acetate	Methanol	content (%)
Heartwood	Control	1,60 (0,12) d	2,63 (0,05) e	2,63 (0,05) c	6,86 (0,21) c
	180	2,15 (0,08) b	4,58 (0,05) b	3,73 (0,08) b	10,46 (0,25) b
	200	4,52 (0,05) a	5,13 (0,08) a	5,56 (0,10) a	15,21 (0,40) a
Sapwood	Control	0,51 (0,06) e	2,70 (0,05) e	0,61 (0,08) f	3,82 (0,16) d
	180	1,93 (0,03) c	3,23 (0,08) d	1,12 (0,08) e	6,28 (0,01) c
	200	2,41 (0,05) b	3,71 (0,08) c	1,50 (0,05) d	7,62 (0,03) c

The number in parentheses are standard errors. Means within a column followed by the same letters are not significantly different at the 1 % probability level.

209
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Table 4: Means of the chemical component contents and pH of untreated and thermally-treated teak heartwood and sapwood.

Wood type	Temperature (°C)	Klason Lignin (%)	Holocellulose (%)	α -cellulose (%)	pH
Heartwood	Control	29,3 (1,5) b	63,6 (0,4) b	45,4 (1,1) c	5,55 (0,03) d
	180	31,3 (1,5) ab	57,0 (0,2) d	42,6 (1,4) c	4,86 (0,02) e
	200	35,7 (3,1) a	47,3 (0,8) e	39,6 (1,4) d	4,72 (0,09) f
Sapwood	Control	29,7 (3,2) ab	66,5 (0,6) a	60,8 (1,6) a	6,53 (0,01) a
	180	27,7 (1,5) b	63,6 (0,7) b	58,6 (0,8) ab	5,72 (0,03) c
	200	31,3 (1,5) ab	59,4 (0,1) c	57,5 (0,5) b	5,92 (0,02) b

The number in parentheses are standard errors. Means within a column followed by the same letters are not significantly different at the 1 % probability level.

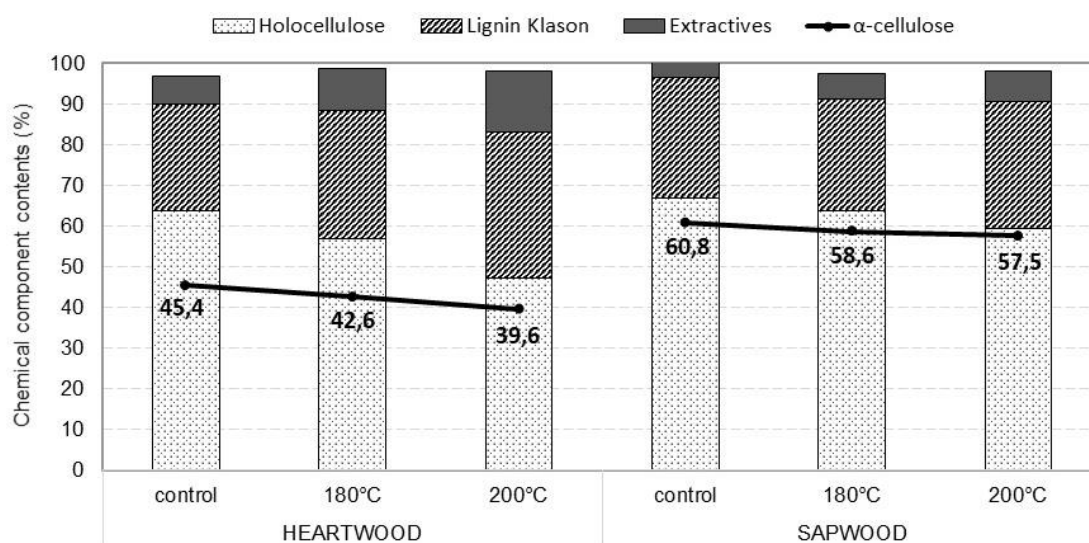
211 The treatment at 200 °C increased the Klason lignin content in heartwood but not in sapwood
 212 (Table 4). The increase in lignin content was also found in thermally-treated teak (Bellon 2013;
 213 Lengowski 2011), Caribbean pine (Poubel *et al.* 2013), and eucalypt (de Moura *et al.* 2012) woods.
 214 Several works have reported chemical changes in lignin structure due to thermal treatments.
 215 Chemical analysis of thermally-treated teak wood assessed by Fourier-transform infrared (FTIR)
 216 spectroscopy showed an increase in band intensity at 1328 cm⁻¹ assigned to C-H vibration in
 217 cellulose and C₁-O vibration in syringyl derivative-condensed structures in lignin, which suggest
 218 the formation of condensed structures (Li *et al.* 2015). Kim *et al.* (2014) also reported
 219 depolymerization (cleavage of β -O-4 linkage) and condensation between lignin fragments during
 220 thermal treatment. Although the thermal treatment affects lignin due to depolymerization,
 221 hemicelluloses reach a more advanced stage of degradation starting at 180 °C compared to lignin,
 222 which results in an apparent lignin increase in relation to the other components of the cell wall
 223 (Sundqvist 2004).

224 The untreated sapwood exhibited the highest holocellulose and α -cellulose contents (Table 4).
225 According to Stamm (1964), hemicellulose and cellulose (amorphous portion and crystallite
226 surfaces) are the main responsible for the hygroscopicity of wood while lignin plays a secondary
227 role.

228 Holocellulose content decreased gradually with the temperature increase for both wood types (Table
229 4). The holocellulose content of treated heartwood and sapwood decreased by 10 % and 4 % at 180
230 °C, and by 26 % and 11 % at 200 °C, respectively. The holocellulose decrease in treated woods was
231 probably due to the degradation of hemicelluloses. The results regarding the heartwood and
232 sapwood behavior confirm those of previous works. Lopes *et al.* (2018) used infrared spectroscopy
233 to assess the chemical modification of the surface of thermally-modified teak wood. A more
234 pronounced decrease in OH⁻ groups in heartwood than in sapwood after heat treatment was
235 observed. The authors provide a good discussion about the different chemical changes in teak
236 heartwood and sapwood after heat treatment and the greater sensitivity of the heartwood to thermal
237 degradation compared to sapwood, but it is not yet clear why this occurs. No significant differences
238 were found between α -cellulose contents of untreated and 180 °C-treated woods. On the other hand,
239 the α -cellulose content decreased in the 200 °C-treated heartwood and sapwood. Among the
240 chemical constituents of the cell wall, hemicelluloses are primarily consumed because they are
241 easily degraded at low temperatures (140 °C) while cellulose is more resistant to thermal
242 degradation (Sundqvist 2004). Other authors reported a holocellulose decrease of 24-25 % in teak
243 (Bellon 2013, Lengowski 2011), 18,6 % in *Eucalyptus grandis*, and 4,6 % in *Pinus taeda* woods
244 after thermal treatment at 160 °C by the VAP HolzSysteme[®] process (Lengowski 2011).

245 In summary, the chemical changes caused by the thermal treatment were more important in
246 heartwood than in sapwood (Figure 1). The apparent total extractive content (or by-products formed
247 mainly from the degradation of hemicelluloses) increased more in the thermally-treated heartwood
248 than in the thermally-treated sapwood due to greater susceptibility to thermal degradation of the

249 former. The relative lignin content increased by 20 % in 200 °C-treated heartwood while no
 250 significant change was found in the sapwood. The holocellulose content decreased more in
 251 heartwood than in sapwood at both thermal treatment degrees. The α -cellulose content decreased by
 252 12,8 % in heartwood and 5,4 % in sapwood after the treatment at 200 °C. Studies realized by Lopes
 253 *et al.* (2018) using FTIR spectroscopy also reported a more important thermal degradation in
 254 heartwood than in sapwood.



255 **Figure 1:** Chemical component contents of untreated and thermally-treated teak wood.
 256

257 The untreated heartwood was more acid (pH = 5,55) than the untreated sapwood (pH = 6,53) (Table
 258 4). The high acidity of heartwood compared to sapwood has often been reported in the literature for
 259 several hardwood species (Kakavas *et al.* 2018; Passialis *et al.* 2008). Untreated woods usually have
 260 a pH near to 5,0-5,5 (Boonstra *et al.* 2007), however, pH values depend on the extraction conditions
 261 (Geffert *et al.* 2019). Lower pH values are found with hot water extraction compared to cold water,
 262 due to the greater release of acetic acid (Geffert *et al.* 2019). Therefore, the extraction method used
 263 in our study can explain the high pH values found in untreated teak woods, principally sapwood.
 264 The thermal treatment significantly decreased the pH values of both types of wood, principally of
 265 the heartwood. The most expressive chemical degradation in the heartwood after thermal treatment
 266 can be also confirmed by its higher acidity. The increased acidity in thermally-treated woods can be
 267 explained by the hemicellulose degradation and formation of acetic acid. This acid also acts as

268 catalysts in the depolymerization process of the cellulose microfibrils breaking them into smaller
269 chains (Boonstra *et al.* 2007), in addition to the condensation and degradation reactions in lignin
270 structure (Li *et al.* 2015) that result in the formation of phenolic groups (Kim *et al.* 2014).

271 **Equilibrium moisture content**

272 The ANOVAs showed respectively a significant effect of the heat treatment and wood type on the
273 EMC for all moisture sorption conditions, except in the anhydrous state (Table 5). As expected, the
274 analysis of untreated samples showed that the EMC was lower in heartwood than sapwood (Table
275 6). This can be attributed to the higher proportion of extractives present in heartwood (Table 3).
276 Previous work about the chemical composition of teak juvenile wood assessed by FTIR
277 spectroscopy also showed a higher extractive content (quinones, oils, and waxes) in heartwood than
278 sapwood (Lopes *et al.* 2018). In fact, several works have shown that teak wood and other tropical
279 woods have a hydrophobic behavior due to the presence of extractives in their tissues (Hernández
280 2007a, Jankowska *et al.* 2017). Furthermore, EMC decreased due to the heat treatments (control to
281 180 °C to 200 °C) for all moisture sorption conditions and wood types (Table 6). For instance, the
282 EMC of heartwood and sapwood treated at 180 °C significantly decreased 3,9 % and 1,1 % MC
283 compared to the corresponding untreated samples in desorption at 86 % RH, respectively. EMC
284 decreased even more for samples treated at 200 °C, namely 4,9 % for heartwood and 4,7 % MC for
285 heartwood and sapwood at the same RH. For all cases, the decrease in EMC due to the heat
286 treatment was higher for heartwood than for sapwood.

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Table 5: F-values obtained from the ANOVAs of the effects of the wood type (heartwood, sapwood) and temperature treatment on the EMC and each shrinkage component.

0% RH					
Source of variation	EMC	β_T	β_R	β_L	β_V
Wood type	4,5 ^{ns}	29,3 **	35,1 **	0,01 ^{ns}	40,7 **
Temperature	1,3 ^{ns}	16,4 **	26,6 **	4,8 ^{ns}	29,5 **
Wood type*temperature	1,9 ^{ns}	4,4 *	3,1 ^{ns}	0,01 ^{ns}	5,2 *
33% RH					
Source of variation	EMC	β_T	β_R	β_L	β_V
Wood type	14,3 **	21,9 **	12,0 **	3,3 ^{ns}	19,3 **
Temperature	67,6 **	11,7 **	2,4 ^{ns}	0,8 ^{ns}	5,8 **
Wood type*temperature	4,7 *	3,4 *	1,6 ^{ns}	0,4 ^{ns}	4,8 *
58% RH					
Source of variation	EMC	β_T	β_R	β_L	β_V
Wood type	40,1 **	80,5 **	25,05 **	0,6 ^{ns}	34,3 **
Temperature	84,9 **	12,8 **	2,3 ^{ns}	1,75 ^{ns}	9,8 **
Wood type*temperature	3,4 *	2,2 ^{ns}	2,6 ^{ns}	0,33 ^{ns}	3,7 *
76% RH					
Source of variation	EMC	β_T	β_R	β_L	β_V
Wood type	49,1 **	112,1 **	15,2 **	0,01 ^{ns}	83,7 **
Temperature	107,5 **	20,8 **	2,8 ^{ns}	1,7 ^{ns}	14,3 **
Wood type*temperature	2,6 ^{ns}	3,7 *	2,2 ^{ns}	2,2 ^{ns}	5,9 **
86% RH					
Source of variation	EMC	β_T	β_R	β_L	β_V
Wood type	126,9 **	34,6 **	2,8 ^{ns}	2,2 ^{ns}	13,2 **
Temperature	101,1 **	6,1 **	1,9 ^{ns}	2,7 ^{ns}	2,2 ^{ns}
Wood type*temperature	12,6 **	2,3 ^{ns}	0,1 ^{ns}	0,1 ^{ns}	1,6 ^{ns}
*,** Significant at the 5 and 1 % probability level respectively; ^{ns} not significant.					

Table 6: Means of the equilibrium moisture content (EMC) of untreated and thermally-treated teak heartwood and sapwood for different sorption conditions at 21 °C.

Temperature (°C)	EMC in desorption (%)									
	0 % RH		33 % RH		58 % RH		76 % RH		86 % RH	
	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood
Control	0,09 Aa	0,04 Aa	7,3 Aa	7,5 Aa	12,2 Aa	12,7 Aa	16,1 Ba	17,1 Aa	19,9 Ba	21,9 Aa

	(0,08) ¹	(0,04)	(0,1)	(0,1)	(0,2)	(0,1)	(0,2)	(0,1)	(0,3)	(0,02)
180	0,07 Aa (0,06)	0,01 Aa (0,04)	5,7 Bb (0,2)	7,0 Aa (0,6)	9,7 Bb (0,3)	11,4 Ab (0,1)	13,0 Bb (0,3)	15,3 Ab (0,1)	16,0 Bb (0,4)	20,8 Ab (0,3)
200	0,05 Aa (0,03)	0,01 Aa (0,04)	4,9 Ac (0,2)	5,3 Ab (0,4)	8,3 Bc (0,3)	10,0 Ac (0,4)	10,5 Bc (0,5)	12,8 Ac (0,4)	15,0 Bb (0,7)	17,2 Ac (0,3)

¹ The standard error is given in parentheses. Means followed by the same letters are not significantly different at the 5 % probability level. Uppercase letters: mean comparisons between wood types, for each temperature and RH separately. Lowercase letters: mean comparisons among thermal treatments within a column.

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304 The difference in EMC between untreated samples and both treated woods (180 °C or 200 °C),
 305 increased as RH increased from 33 % to 86 %, for both heartwood and sapwood (Figure 2). The
 306 most important decrease in EMC occurred at 200 °C. The 200 °C-treated wood had lower
 307 holocellulose and α -cellulose contents (Table 4) and higher total extractive content (by-products
 308 formed during thermal degradation) (Table 3) than other treatments. These chemical modifications
 309 may explain the lowest EMC of 200 °C-treated heartwood and sapwood compared to the other
 310 treatments. Other studies have reported reductions in EMC of teak wood thermally-treated at 160 °C
 311 (Lengowski 2011, Bellon 2013).

312 Generally, the sapwood had higher EMC than the heartwood after the heat treatments (Table 6,
 313 Figure 2). This is due to the higher holocellulose and α -cellulose contents in sapwood (Table 4)
 314 which are the most hygroscopic polymers of cell walls. The higher extractive content of treated
 315 heartwood could also affect the EMC because they decrease the wood hygroscopicity.

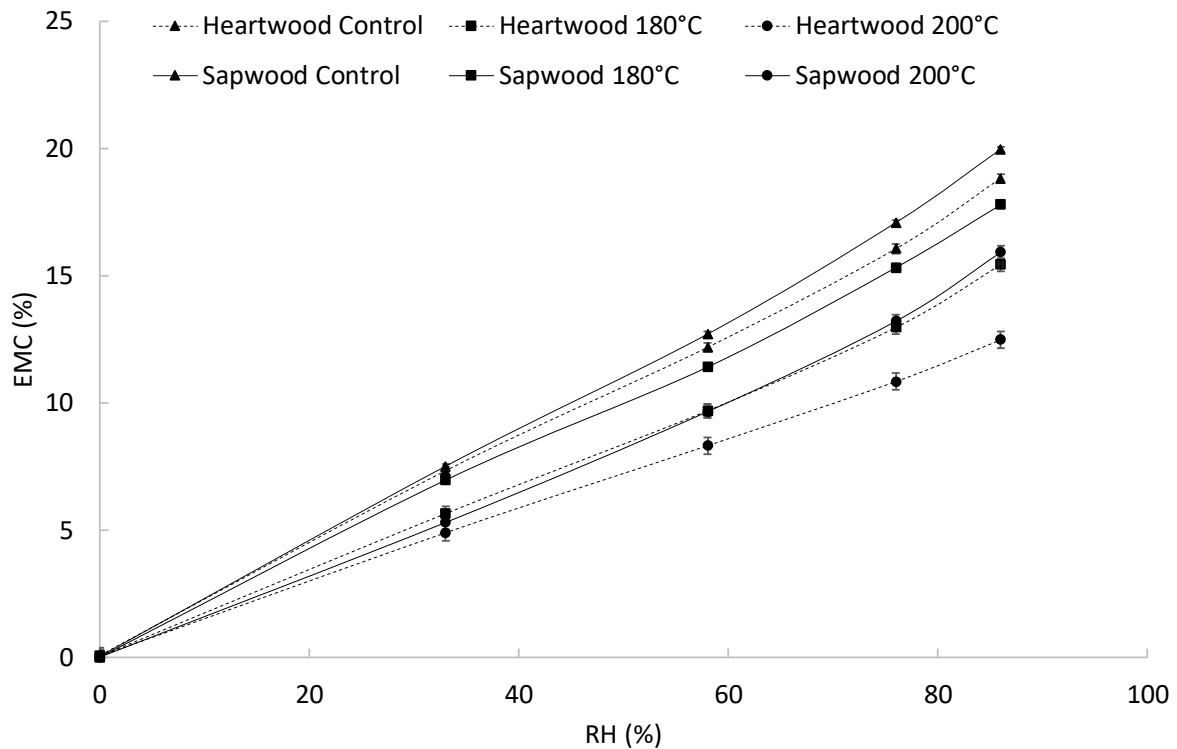


Figure 2: Equilibrium moisture content (EMC) in desorption of untreated and thermally-treated teak wood as a function of relative humidity (RH) at 21 °C.

Shrinkage

The results of untreated samples confirmed that teak wood presents low values of partial and total shrinkages compared to other tropical hardwoods (FPL 2010). As expected, untreated heartwood had lower total and partial linear and volumetric shrinkages than untreated sapwood at all moisture sorption conditions. The total β_T , β_R , β_L , and β_V of heartwood were of 4,6 %, 2,6 %, 0,5 %, and 7,7 % while those of untreated sapwood were of 5,2 %, 3,3 %, 0,5 %, and 8,8 %, respectively (Table 7). However, given the very low values, no significant difference was found between heartwood and sapwood for β_L . These results are similar to those reported by Miranda *et al.* (2011) to 50-70 years-old teak wood, which were 5,2 %, 3,5 %, and 7,6 % (mean from pith to bark) for β_T , β_R , and β_V , respectively. The lower shrinkage in heartwood is attributed to the high extractive content observed in this wood type (Hernández 2007b, Jankowska *et al.* 2017).

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347 **Table 7:** Means of the shrinkage of untreated and thermally-treated teak heartwood and sapwood
 348 for different sorption conditions at 21 °C.

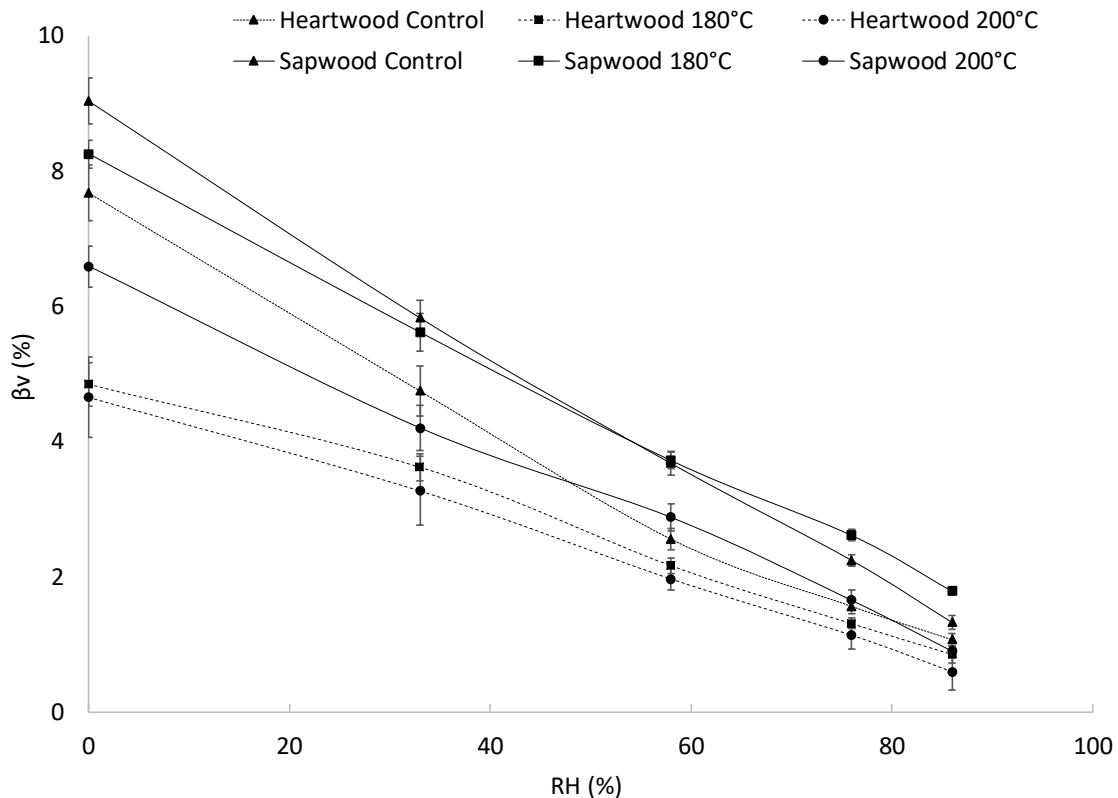
Tangential shrinkage – β_T (%)										
Temperature (°C)	0 % RH		33 % RH		58 % RH		76 % RH		86 % RH	
	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood
Control	4,6 Ab (0,3)	5,2 Aa (0,4)	3,1 Ba (0,3)	3,8 Aa (0,2)	1,7 Ba (0,1)	2,5 Aa (0,1)	0,9 Ba (0,08)	1,5 Ab (0,07)	0,4 Ba (0,1)	0,9 Aa (0,1)
180	2,9 Ba (0,2)	5,2 Aa (0,1)	2,2 Bb (0,2)	3,8 Aa (0,1)	1,3 Bb (0,1)	2,5 Aa (0,06)	0,8 Ba (0,08)	1,8 Aa (0,05)	0,3 Ba (0,04)	0,8Aa (0,06)
200	2,7 Ba (0,3)	3,7 Ab (0,3)	2,1 Ab (0,3)	2,6 Ab (0,3)	1,2 Bb (0,07)	1,9 Ab (0,2)	0,5 Bb (0,1)	1,1 Ac (0,08)	0,3 Ba (0,04)	0,5 Ab (0,04)
Radial shrinkage – β_R (%)										
Temperature (°C)	0 % RH		33 % RH		58 % RH		76 % RH		86 % RH	
	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood
Control	2,6 Ba (0,2)	3,3 Aa (0,1)	1,5 Aa (0,1)	1,9 Aa (0,2)	0,8 Ba (0,07)	1,0 Aa (0,1)	0,5 Aa (0,06)	0,6 Aa (0,04)	0,4 Aa (0,03)	0,9 Aa (0,1)
180	1,5 Bb (0,1)	2,7 Ab (0,1)	1,2 Ba (0,1)	2,0 Aa (0,2)	0,7 Ba (0,04)	1,2 Aa (0,04)	0,5 Ba (0,02)	0,8 Aa (0,06)	0,3 Aa (0,05)	0,4 Aa (0,1)
200	1,7 Ab	2,1 Ac	1,2 Aa	1,5 Aa	0,7 Aa	0,9 A	0,4 Aa	0,5 Aa	0,1 Aa	0,3 Aa

	(0,2)	(0,2)	(0,2)	(0,1)	(0,1)	(0,02)	(0,06)	(0,07)	(0,03)	(0,1)
Longitudinal shrinkage – β_L (%)										
Temperature (°C)	0 % RH		33 % RH		58 % RH		76 % RH		86 % RH	
	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood
Control	0,51 Aa (0,01)	0,50 Aa (0,04)	0,33 Aa (0,06)	0,23 Aa (0,02)	0,14 Aa (0,07)	0,15 Aa (0,07)	0,11 Aa (0,03)	0,12 Aa (0,02)	0,15 Aa (0,05)	0,10 Aa (0,04)
180	0,42 Aa (0,02)	0,43 Aa (0,06)	0,27 Aa (0,03)	0,24 Aa (0,04)	0,16 Aa (0,05)	0,08 Aa (0,04)	0,04 Aa (0,02)	0,09 Aa (0,04)	0,20 Aa (0,04)	0,16 Aa (0,03)
200	0,34 Aa (0,04)	0,34 Aa (0,04)	0,25 Aa (0,04)	0,19 Aa (0,04)	0,07 Aa (0,02)	0,04 Aa (0,05)	0,13 Aa (0,04)	0,07 Aa (0,02)	0,27 Aa (0,04)	0,19 Aa (0,06)
Volumetric shrinkage – β_V (%)										
Temperature (°C)	0 % RH		33 % RH		58 % RH		76 % RH		86 % RH	
	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood
Control	7,7 Ba (0,4)	8,8 Aa (0,4)	4,7 Ba (0,3)	5,8 Aa (0,3)	2,7 Ba (0,2)	3,7 Aa (0,2)	1,5 Ba (0,1)	2,2 Ab (0,08)	0,9 Aa (0,1)	1,4 Aa (0,2)
180	4,9 Bb (0,3)	8,3 Aa (0,2)	3,6 Bb (0,3)	6,1 Aa (0,4)	2,2 Bab (0,1)	3,7 Aa (0,1)	1,3 Bab (0,08)	2,6 Aa (0,08)	0,6 Ba (0,2)	1,4 Aa (0,1)
200	4,7 Bb (0,5)	6,1 Ab (0,4)	3,9 Aab (0,5)	4,2 Ab (0,3)	2,0 Ab (0,2)	2,5 Ab (0,3)	1,1 Bb (0,2)	1,7 Ac (0,1)	0,7 Aa (0,07)	0,9 Aa (0,1)
The standard error is given in parentheses. Means followed by the same letters are not significantly different at the 5 % probability level. Uppercase letters: mean comparisons between wood types within a row, for each RH separately. Lowercase letters: mean comparisons among temperatures within a column, for each shrinkage type separately.										

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350 The ANOVAs showed a significant effect of the wood type and the thermal treatment on the linear
 351 (except for β_L) and volumetric shrinkages for almost all moisture sorption conditions. The wood
 352 type showed a more important effect on partial and total shrinkages compared to the temperature
 353 treatment (Table 5). The thermal treatment decreased partial shrinkage in both heartwood and
 354 sapwood, however, this effect would be more important in sapwood (Table 7, Figure 3). Total linear
 355 and volumetric shrinkages also decreased with the thermal treatments for both wood types but this
 356 effect would be more important in heartwood (Table 7). At 200 °C, the β_T of the heartwood and
 357 sapwood decreased respectively by 41 % and 29 %, β_R decreased by 35 % and 36 % and β_V
 358 decreased by 39 % and 31 %, compared to untreated woods.

359



360
361 **Figure 3:** Volumetric shrinkage (β_v) of teak wood as a function of relative humidity (RH) at 21 °C.
362

363 The effects of the thermal treatment were more important in β_T than in β_R . However, β_L was not
364 affected neither by the wood type nor the thermal treatment (Table 5). The greater dimensional
365 variations of the wood occur in the tangential direction from 2,4 % to 11 % followed by radial
366 direction from 3,5 % to 5 % while those in the longitudinal direction are almost negligible from 0,1
367 % to 0,6 % (Kollmann and Côté Jr. 1968). Several factors can explain the anisotropy of wood such
368 as anatomical and chemical structure, mainly due to restriction of rays in the radial direction and the
369 helical arrangement of the cellulose microfibrils in the cell wall (Kollmann and Côté Jr. 1968).
370 Nevertheless, the relationships between anatomical structure, chemical composition, extractive
371 content, and density cause variations in shrinkage and swelling among wood species.

372 The hygroscopic and dimensional stability of teak wood were improved after thermal treatment.
373 The increase in the temperature treatment resulted in a decrease of the EMC. The S-ratio was
374 calculated from the EMC-RH curve in the highest sorption linear range from 76 % to 33 % RH
375 (Figure 2). The S-ratio was higher in sapwood than in heartwood in all thermal treatments (Table 8).

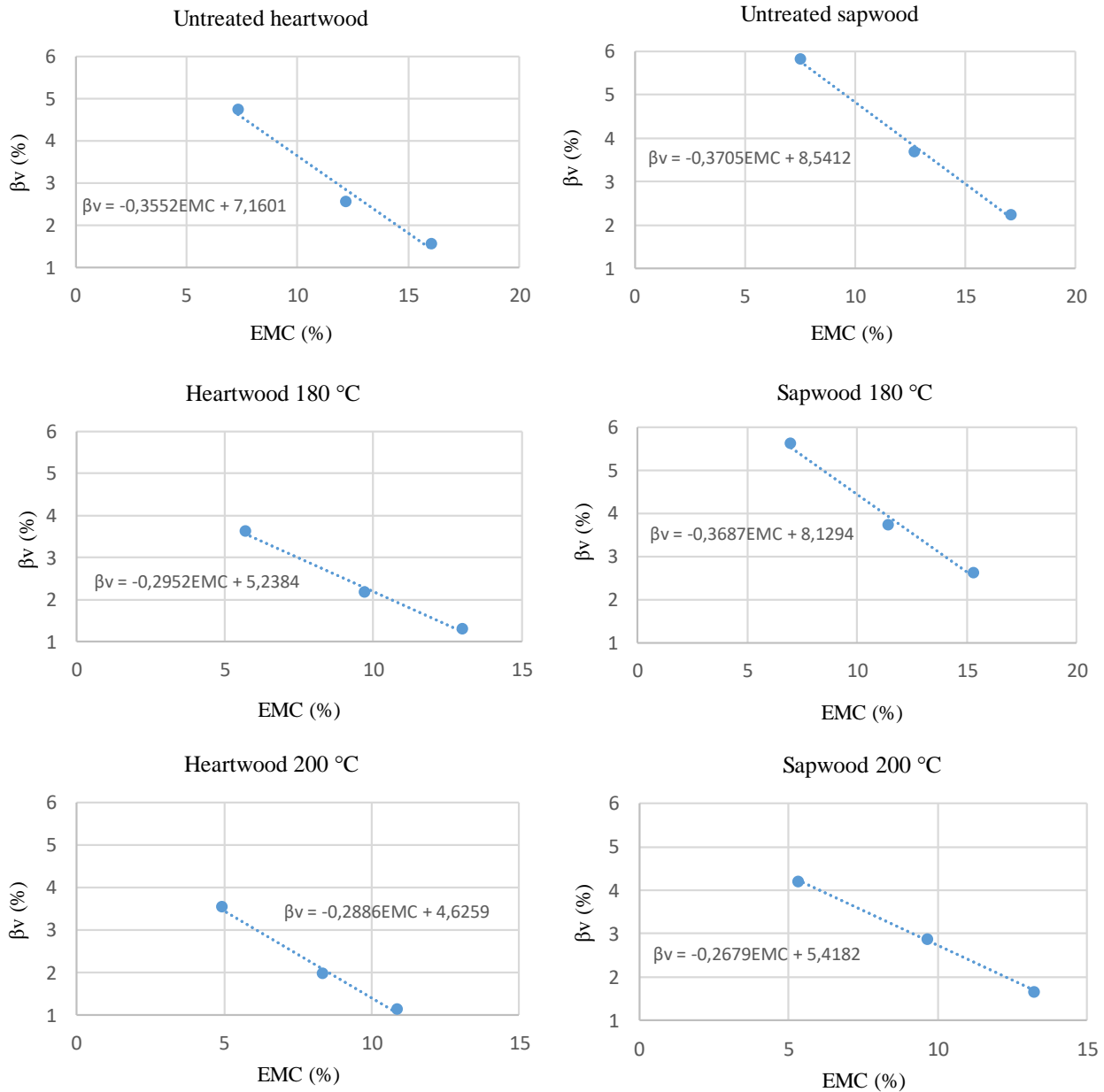
376 It clearly shows that sapwood is more sensitive to RH changes than heartwood. These results can be
 377 explained by the higher holocellulose and α -cellulose contents in sapwood compared to heartwood
 378 (Table 4) and also by the lower natural extractive content in the untreated sapwood (Table 2). The
 379 caoutchouc found in the cell walls and lumens is responsible for the greater hydrophobicity of teak
 380 heartwood (Yamamoto *et al.* 1998) which has higher concentrations than that of the sapwood
 381 (Lopes *et al.* 2018). The highest decrease in the S-ratio was found with the 200 °C treatment in both
 382 wood types (heartwood and sapwood), which could be explained by the greater chemical changes.

383 **Table 8:** Ratio of sorption (S), coefficient of shrinkage (h), and estimated fiber saturation point
 384 (FSP) of untreated and thermally-treated teak woods.

Temperature (°C)	S-ratio Desorption 76 % - 33 % (% EMC / % RH)		h-coefficient Desorption 76 % - 33 % (% β_v / % RH)		FSP	
	Heartwood	Sapwood	Heartwood	Sapwood	Heartwood	Sapwood
Control	0,203	0,223	0,074	0,083	20,2	23,1
180	0,170	0,194	0,054	0,070	17,7	22,1
200	0,138	0,184	0,056	0,059	16,0	20,2

385
 386 Heartwood was more dimensional stable compared to sapwood in untreated and thermally-treated
 387 conditions. Thermally-treated woods showed lower h-coefficient values compared to untreated
 388 woods (Table 8), which means that the temperature treatments improve the dimensional stability for
 389 both wood types. For sapwood, the treatment at 200 °C was more efficient compared to 180 °C
 390 since it provided greater dimensional stability (h-lower coefficient). For heartwood, both
 391 temperature treatments resulted in a similar h-coefficient. The curve of the β_v as a function of EMC
 392 from 33 % to 76 % RH was used to estimate the FSP by the intersection to zero shrinkage method
 393 for all samples (Figure 4). Sapwood exhibited a higher FSP than heartwood for all treatments.
 394 Heartwood FSP was similar to that found by Kokutse *et al.* (2010). The higher h-coefficient and
 395 higher FSP of the sapwood (Table 8) again show that it is less stable than heartwood and more
 396 prone to defects caused by the shrinkage of wood during drying. The greater dimensional stability
 397 of thermally-treated woods is due to lower hygroscopicity caused by chemical changes in the cell
 398 wall polymers which decrease the water absorption sites (mainly OH⁻ groups), and consequently the

399 FSP, and shrinkage (Priadi and Hiziroglu 2013). Overall, the improvement of the dimensional
 400 stability of teak wood with temperature treatments, as a result of the diminution of its
 401 hygroscopicity (EMC, S, and FSP) and changes in its dimensions (h-values) will optimize the
 402 utilization of the wood in several end-uses, such as outdoor furniture, decks, and indoor flooring.



403 **Figure 4:** Volumetric shrinkage (β_v) of teak wood as a function of equilibrium moisture content
 404 (EMC) between 33 % and 76 % RH.
 405
 406

407 CONCLUSIONS

408 The effects of thermal modification of teak heartwood and sapwood on the chemical components,

409 EMC, and shrinkage at various RHs were investigated. The following conclusions can be drawn:

410 1. The untreated heartwood had higher extractive and Klason lignin contents than those of
411 untreated sapwood, which exhibited higher holocellulose and α -cellulose contents.

412 2. The thermal modification decreased the holocellulose and α -cellulose contents in heartwood
413 and sapwood and increased the acidity of wood. The degradation of hemicelluloses started at 180 °C
414 while that of α -cellulose started at 200 °C.

415 3. Chemical changes were more important in the heartwood than in the sapwood.

416 4. The thermal modification reduced the EMC of heartwood and sapwood at sorption
417 conditions from 33 % to 86 % RH. The sapwood was more sensitive to RH variations than
418 heartwood, regardless of the temperature.

419 5. The thermal modification provided higher dimensional and hygroscopic stabilities to wood.
420 Generally, the thermal treatment decreased the shrinkage of heartwood and sapwood, however,
421 shrinkage in heartwood was more sensible to the temperature treatment compared to sapwood.
422 Thermally-treated woods also exhibited lower S and FSP compared to untreated ones.

423 6. Overall, the results of this research lead to a better understanding of dimensional stability of
424 teak wood from young fast-growing plantations after thermal treatment, which may help define,
425 from a practical point of view, the optimal use of juvenile teak wood in different relative humidity
426 conditions.

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