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2 **THERMOCHEMICAL BEHAVIOR OF *EUCALYPTUS GRANDIS* WOOD**
3 **EXPOSED TO TERMITE ATTACK**

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16 **ABSTRACT**

17
18 This study aimed to evaluate the variations in thermal and chemical characteristics of
19 juvenile *Eucalyptus grandis* wood submitted to a deterioration test by *Nasutitermes*
20 termites. For this purpose, a biodeterioration test with termites was conducted according to
21 ASTM D 3345 (2008), in which, after the end of the period corresponding to the test (40
22 days), we evaluated the mass loss, chemical composition and thermal stability of the main
23 components of the deteriorated wood samples and those belonging to the control group. We
24 found that deterioration due to exposure of the samples to *Nasutitermes* sp. termites caused
25 a mass loss of 66.88% for wood with a density at 12% moisture content of 0.412 g.cm⁻³. The
26 quantitative chemical composition showed a reduction in the contents of cellulose,
27 hemicellulose and lignin. Analysis of the variations of the organic functional groups related
28 to the chemical composition of the wood by Fourier Transform Infrared Spectroscopy and
29 relative intensity of the spectral bands also showed reductions, demonstrating homogeneous
30 deterioration of the main components of the deteriorated woods. The thermal stability
31 showed an increase in deteriorated wood for most of the temperature ranges, mainly for
32 those that corresponded to losses in moisture and volatiles (25 °C - 100 °C), hemicelluloses
33 (240 °C - 300 °C), celluloses, and together with initial lignin degradation (310 °C to 400
34 °C), possibly due to the removal of cellulose and hemicellulose, as well as the deposition of
35 substances expelled by the termites in the cell wall. From the results, we conclude that the
36 termites do not have specificity regarding the chemical component and that the deterioration
37 caused variations in the chemical composition of the wood, whereas the opposite was
38 observed for thermal stability, which presented an increase in most of the temperatures
39 ranges for the deteriorated woods compared to the control group.

40 **Keywords:** Chemical composition; *Nasutitermes*; thermal stability; wood deterioration; wood protection.

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45 **1 INTRODUCTION**

46 In 2016, Brazil had a total of 7.84 million hectares of planted forests, of which 5.67
47 million were registered as *Eucalyptus*, 1.58 million *Pinus* and 0.59 million of other genres.
48 From 2015 to 2016, only *Eucalyptus* presented growth of 0.5% (BTI 2017). These figures
49 indicate that Eucalyptus species have a great potential, mainly due to their high productivity
50 and rapid growth (HUBBARD et al. 2010), thus presenting as an alternative to the use of
51 native woods in the timber productive chain.

52 Due to their interesting and varied properties, this genus, mainly *Eucalyptus grandis*,
53 is widely used in the paper and cellulose industries, as well as for manufacturing of
54 furniture, railway sleepers, fence posts and construction. However, as it is a material of
55 organic origin, like any other wood type, it is vulnerable to the attack of several biotic
56 agents, such as termites, which are a big threat to the wood's useful lifespan. This attack is
57 due to the ease that these xylophagous organisms have in digesting the lignocellulosic
58 biomass that constitutes the cell wall of the wood in order to meet their energy demands
59 (COUTURIER et al. 2015).

60 Due to the large variety of wood species, it should be noted that the durability of this
61 material depends on several factors: the proportion of heartwood and sapwood, the chemical
62 composition (mainly content and composition of extractives and lignin), density, moisture
63 content, hardness, and use conditions (HAUPT et al. 2003, BRISCHKE et al. 2014,
64 DELUCIS et al. 2016).

65 Termite attacks on wood cause significant damages, sometimes requiring
66 replacement, depending on the degree of deterioration. Besides the social and economic
67 impacts, this deterioration changes the technological properties of the wood, such as
68 chemical, physical and mechanical properties (MALAKANI et al. 2014), which
69 compromises its use and durability. According to Constantino (2002), there are many
70 species of termites in South America, several of them considered structural and agricultural

71 pests, because of their highly destructive potential in using vegetable and wood components
72 as food resources. The subfamily Nasutitermitinae is among those that cause most damage
73 to wood in tropical regions (BOULOGNE et al. 2017). Among the known genres, the
74 *Nasutitermes* includes approximately 54% of all species present in this region. They are
75 considered different structural pests that are able to attack woods with different density
76 classes (CONSTANTINO 2002, STALLBAUN et al. 2017).

77 Thus, aiming to obtain information about wood durability of one of the most used
78 and planted species in the country, the present work aimed to analyze the variations in
79 chemical characteristics and thermal stability in spawood of *Eucalyptus grandis* wood
80 resultant of the deterioration caused by termites *Nasutitermes* sp.

81

82 **2 MATERIAL AND METHODS**

83 **2.1 Material used**

84 For this study, we used specimens measuring 2.0 x 2.0 x 15cm (tangential plane x
85 radial x longitudinal, respectively) from a 12-year-old *Eucalyptus grandis*, collected in a
86 forest garden near Guaíba city, Rio Grande do Sul, Brazil. Subsequently, the 30 specimens
87 were kept in a room with controlled conditions (20 ° C temperature and 65% air humidity)
88 until mass stabilization occurred (hygroscopic equilibrium of 12%).

89

90 **2.2 Experimental Procedure**

91 After the stabilization period, the density at moisture content ($\rho_{12\%}$) was determined
92 using Equation 1. For that, the mass ($M_{12\%}$) and volume ($V_{12\%}$) of the test specimens were
93 obtained using an analytical balance and a digital pachymeter (accuracy of $\pm 0.001\text{mm}$),
94 respectively, obtaining the volume by stereometric method.

95

$$\rho_{12\%} (\text{g/cm}^3) = \frac{M_{12\%} (\text{g})}{V_{12\%} (\text{cm}^3)} \quad (1)$$

96

97 In order to perform the biodeterioration test, 15 specimens were placed inside a
98 microfiber box (2,000 liters capacity) containing a 10 cm layer of sand (periodically
99 moistened) as a substrate, under which they were partially buried (approximately 2/3 of
100 their length). The *Nasutitermes* termite test was conducted adapting the standard of the
101 American Society for Testing and Materials (ASTM D 3345, 2008), for a 40-day period.

102 Once the biodeterioration test was finalized, the specimens were removed from the
103 exposition to the termites and carefully cleaned (substrate and termites were removed)
104 through the use of a spatula and a brush. They were then placed back in the room under the
105 conditions of temperature and humidity mentioned previously.

106 **2.3 Analysis of wood deteriorated by *Nasutitermes***

107 The deterioration caused by termites *Nasutitermes* sp. in the *Eucalyptus grandis*
108 wood was verified by analysis of mass loss, changes in chemical properties and thermal
109 stability. The adaptation of standard ASTM D 2017 (2005) made it possible to determine the
110 mass loss (Equation 2), where ML is the mass loss (percentage) and, M_B and M_A are the
111 masses (grams) of the samples before and after the biodeterioration test by termites,
112 respectively.

113

$$114 \quad ML = \left(\frac{M_B - M_A}{M_B} \right) \times 100 \quad (2)$$

114

115 Aiming to verify variances in homogeneity, the mass loss data was converted into
116 Arcsen ($\sqrt{ML / 100}$), as suggested by Steell and Torrie (1980). Subsequently, the test
117 specimens of the control and deteriorated groups were milled in a Willey-type knife mill,
118 passing through a set of sieves (40 and 60 meshes, respectively). The powder retained in the
119 60 mesh was used for the analysis of the deterioration caused by *Nasutitermes* sp. in the
120 other properties of interest to the present study.

121 The thermal behavior of the wood was evaluated by thermogravimetric analysis
122 (TGA). For this, a Netzsch TG 209F1 equipment was used with the following parameters:
123 initial temperature of 30 °C, heating rate of 10 °C.min⁻¹, final temperature of 700 °C, under
124 inert atmosphere, with nitrogen gas flow.

125 Regarding the chemical analysis, quantitative and qualitative chemical analyses were
126 carried out. The quantification of the main chemical components of wood from the control
127 group and the sample deteriorated by termites was carried out in triplicate, according to an
128 adaptation of the methodology described by TAPPI (2007) standard and Rowell et al.
129 (1983), presented in Table 1.

130

131 **Table 1:** List of methodologies applied for the determination of the wood's chemical
132 components.

Chemical Compound	Methodology used
Cellulose	Rowell (1983)
Hemicellulose	
Extractives	TAPPI T-204 cm-97
Lignin	TAPPI T-222 om-98

133

134 The Fourier transform Infrared Spectroscopy (FT-IR) was used for the qualitative
135 study of the recurrent variations in the chemical composition, as a function of the termite
136 attack. The parameter used was absorbance, regarding a total spectrum resulting from 32
137 readings in the spectrum range between 1800 and 600 cm⁻¹. The spectrum was normalized
138 using the wavelength of 1030 cm⁻¹, as it does not subject to change when exposed to
139 different conditions used in the material (CHEN et al., 2012; MISSIO et al., 2015).

140 Aiming to facilitate an understanding of the recurrent variations in the chemical
141 components, a complementary analysis was developed by obtaining the ratio between the
142 relative intensities of spectra related to the lignin band (1508 cm⁻¹) and carbohydrates (890
143 cm⁻¹, 1370 cm⁻¹, 1420 cm⁻¹, 1740 cm⁻¹). The 1508 cm⁻¹ band was used as reference, since it

144 is considered pure, without influence of other chemical components (PANDEY and
145 PITMAN, 2003).

146

147 **3 RESULTS AND DISCUSSION**

148 The mass loss due to deterioration caused by termites of *Nasutitermes* genus in
149 *Eucalyptus grandis* species was of 66.88% for the wood used, with density at 12% moisture
150 content ($\rho_{12\%}$) of 0.412 g/cm³. Table 2 shows that the deterioration caused by termites
151 resulted in reductions in contents of the cellulose, hemicellulose and lignin (main
152 components) of the deteriorated woods compared to the control group, whereas the the
153 inverse occurred with the extractives. The increase in the percentage of secondary
154 metabolites can be justified by the deterioration of the other components of the cell wall of
155 the wood.

156

157 **Table 2:** Average values of chemical composition of woods of the control group and
158 deteriorated by termites of *Nasutitermes* genus.

Treatment	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Extractives (%)
Control	53.24	17.58	22.11	1.11
Deteriorated	50.53	16.31	20.33	1.66

159

160 According to Watanabe and Tokuda (2010), these insects degrade the cellulose and
161 hemicellulose present in the cell wall of the wood by means of the symbiotic association
162 with bacteria and or protozoa residing in their digestive tracts, which release enzymes that
163 act to break and convert main components into smaller molecules, making them available as
164 a nutrient source for the termites. Differently, the lignin suffers with degradation mainly due
165 to scarification process.

166 The relation between the wavelengths and the functional groups, the types of
 167 vibrations and the respective chemical components analyzed in this study are presented in
 168 Table 3.

169

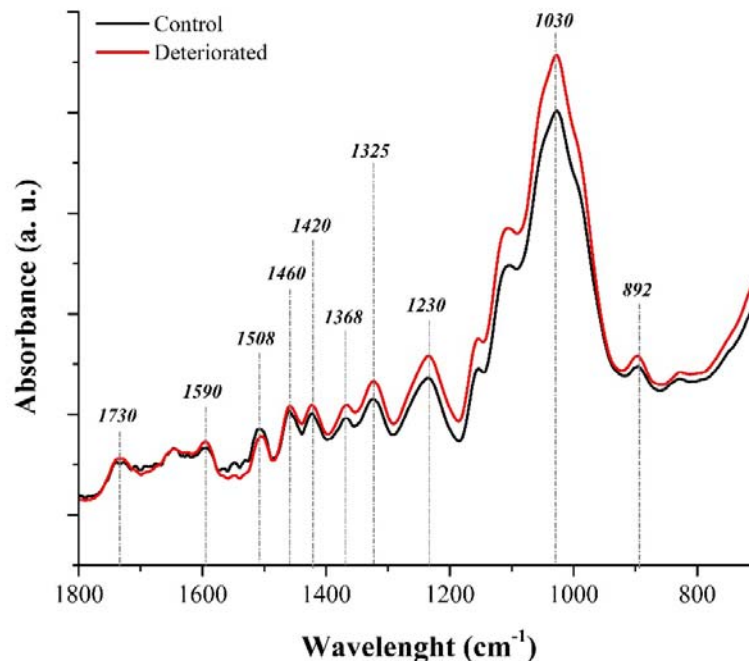
170 **Table 3:** Relation between the main chemical components of wood (cellulose = 1;
 171 hemicellulose = 2; lignin = 3) with the respective bands of the infrared spectrum.

Chemical compound	Wavelength (cm ⁻¹)	Functional group / Vibration type	Reference
1	890	C–OH stretching vibration;	Zhang et al. (2015)
1 e 2 e 3	1030	C–O stretching and C–H deformation;	Darwish et al. (2013)
2	1230	Acetyl and carboxyl vibration in xylan;	Yilgor et al. (2013)
1 e 3	1325	C–H vibration in cellulose and C–O vibration in syringyl derivatives	Pandey e Pitman (2003)
1 e 2	1368	C–H deformation;	Tomak et al. (2013)
1	1420	C–H ₂ scissor vibration;	Tomak et al. (2013)
3	1460	Aromatic C–H deformation;	Fackler et al. (2007)
3	1508	C=C stretching vibration in aromatic ring	Darwish et al. (2013)
3	1590	Aromatic skeletal vibrations;	Pozo et al. (2006)
2	1730	C=O stretching vibration;	Zhang et al. (2015)

172

173 Figure 1 shows an increase in the intensities of the spectral of wood deteriorated by
 174 termites in comparison with the control group, mainly in the bands of 1368 cm⁻¹, 1325 cm⁻¹,
 175 1230 cm⁻¹, 1030 cm⁻¹ and 890 cm⁻¹. This is not indicating an increase in the quantity of the
 176 components, but rather the occurrence of a change in the chemical composition of the wood,
 177 which is associated with the degradation of cellulose, hemicellulose and lignin due to the
 178 termite attack.

179



180

181 **Figure 1:** FTIR spectra of the woods of the control group and deteriorated by termites of
182 *Nasutitermes* genus.

183

184 Zhang et al. (2015) and Yilgor et al. (2013) have argued that changes in peak
185 intensities of spectra are caused by changes in the amounts of the chemical compounds
186 present in the wood. Besides the deterioration of cellulose, hemicellulose and lignin,
187 variations in intensities are associated with the appearance of new functional groups
188 (PANDEY and PITMAN, 2003). The increase in peaks intensities of 1368 cm^{-1} , 1325 cm^{-1} ,
189 1230 cm^{-1} , 1030 cm^{-1} and 892 cm^{-1} are associated with the removal of carbohydrates and
190 increased concentration of different chemical structures of lignin (for example, guaiacil and
191 syringyl).

192 Aiming to prove the variation of functional groups related to the wood's chemical
193 composition, Table 4 presents the relative intensity values of the lignin / carbohydrate ratio.

194

195

196

197 **Table 4:** Relative values of the bands relating lignin (I1508) with carbohydrates (I890;
 198 I1370; I1420; I1740) from the woods of the control group and deteriorated by termites of
 199 *Nasutitermes* genus.

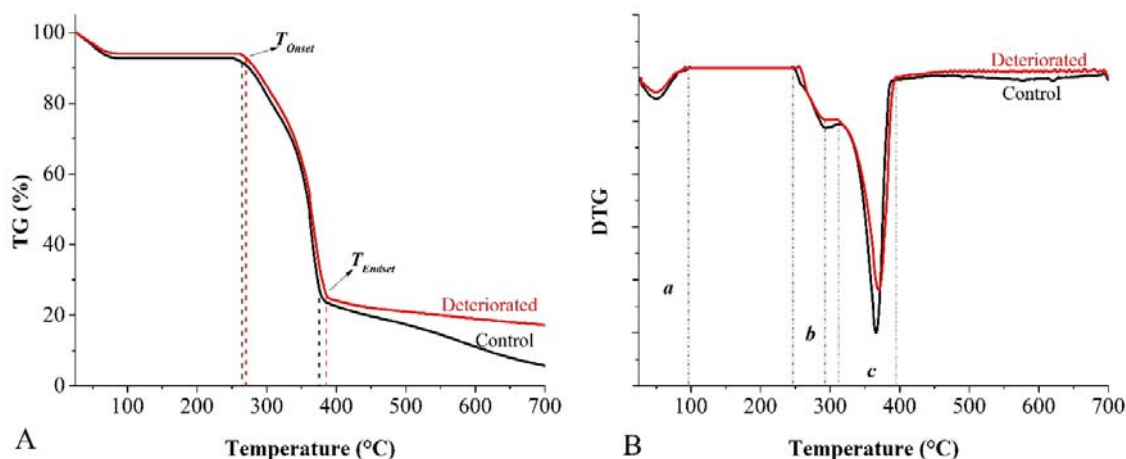
Treatment	I_{1508} / I_{890}	I_{1508} / I_{1370}	I_{1508} / I_{1420}	I_{1508} / I_{1740}
Control	0.627	0.951	0.859	0.531
Deteriorated	0.586	0.840	0.803	0.592

200

201 The decrease in the lignin / carbohydrate ratio is possibly associated to the decrease
 202 in the amount of holocellulose (cellulose and hemicellulose) in relation to lignin with a
 203 more accentuated rate of deterioration, which presented the oposite behavior in relation to
 204 the relative intensity of carbonyl (I_{1508} / I_{1740}). This proves that the termites deteriorate the
 205 main components of wood in a similar way, and have no specificity in relation to a
 206 particular chemical component.

207 Graphs A and B of Figure 2 show that the thermo-degradation for both treatments
 208 occurred in three main zones (a, b and c). The first one shows the loss mass as a function of
 209 humidity (25 °C to 100 °C) present in the wood, while the second and third zones are
 210 mainly related to the hemicelluloses and the volatiles (240 °C to 300 °C) and the cellulose
 211 (310 °C to 400 °C), respectively, with lignin slowly degrading from the beginning of the
 212 thermal process.

213



214

215 **Figure 2:** TGA (A) and DTG (B) curves of the woods of the control group and deteriorated
 216 by termites of *Nasutitermes* genus.

217 Extrapolating the degradation curves of the hemicelluloses and cellulose, represented
218 by the points at the TOnset and TEndset temperatures (Figure 2 - A), there is a slight
219 displacement of the deteriorated wood to a higher temperature when compared to the
220 control.

221 Observing the derivative (Figure 2-B), the presence of a small shoulder at
222 approximately 290 °C is noted, characterizing the hemicelluloses. These were noticeably
223 degraded due to the termite attack, because the shoulder of the deteriorated wood presents a
224 reduction when compared to the control treatment.

225 Cellulose is characterized by having the highest peak at about 370 °C. Like the
226 hemicelluloses, there was a decrease in the intensity of the peak (mass loss) of the
227 deteriorated wood, indicating a decrease in the amount of cellulose, as a result of termite
228 degradation. As for lignin, the presence of a bending in the control treatment after 450 °C
229 was observed, while the deteriorated wood remains practically constant, indicating again
230 that the termites also degrade the lignin.

231 Table 5 presented the decreases of mass for the deteriorated wood in comparison to
232 the control group in most of the temperature ranges, with the exception of the temperature
233 range of 350 - 400 °C, which mainly covers the cellulose and lignin. As shown,
234 approximately 60% of the mass loss occurred in the temperature range between 300 and 400
235 °C, as well as in the study carried out by Crespo et al. (2015) with young wood of *Acacia*
236 *mangium* (80% of mass loss).

237

238

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242

243 **Table 5:** Percentage values of mass loss (%) as a function of different temperature ranges
 244 for woods of the control group and deteriorated by termites of *Nasutitermes* genus.

Temperature ranges (°C)	Control	Deteriorated
30 – 100	7.25	5.84
100 – 150	0.00	0.00
150 – 200	0.00	0.00
200 – 250	0.66	0.54
250 – 300	10.44	9.97
300 – 350	21.11	20.73
350 – 400	38.77	40.05
400 – 450	2.81	1.99
450 – 500	2.31	1.11
500 – 550	2.88	1.00
550 – 600	3.43	1.01
600 – 650	3.11	0.93
650 – 700	2.23	0.86
Residual mass	4.99	15.97

245

246 Due to the removal of cellulose and hemicellulose from the cell wall, which have a
 247 large amount of hydroxyl groups, the wood tends to decrease its moisture exchange
 248 capacity. The hemicelluloses have lower thermal stability in relation to the other
 249 components of the wood (ALFREDSSEN et al. 2012), so the decrease in quantity of these
 250 components implies a lower mass loss as a function of the temperature. The chemical
 251 component with the highest thermal stability is cellulose (SEBIO-PUÑAL et al. 2012).
 252 which presented similar degradation profiles for the two treatments.

253 In this context. Gašparovič et al. (2012) observed that hemicelluloses and cellulose
 254 presented the highest degradation rates in temperatures of 290 °C and 350 °C, respectively,
 255 similar to the degradation profiles observed in this study.

256 With regards to the lignin, the removal of this component as a function mainly of the
 257 scarification process changes the original characteristics of this component, causing
 258 variations in resistance to thermo-degradation. Therefore, the smaller mass loss
 259 corresponding to the lignin region is associated to the degradation mechanism used by the

260 termites and the removal of this component from the interior of the wood by these
261 xylophagus.

262 Regarding the residual mass at the end of the test, it was evident that the deteriorated
263 samples had a higher percentage value when compared to the control. Aydemir et al. (2011)
264 also observed that the degradation of the main chemical components causes an increase in
265 the residual mass of degraded woods (*Carpinus betulus* and *Abies bornmulleriana*)
266 subjected to thermal treatment, when compared to the control treatment.

267 This is also explained by the removal of the major chemical components from the
268 wood, especially the hemicelluloses. Removal of these components from the interior of the
269 wood may have led to a reduction in the mass loss because they are thermally unstable.

270 Also, considering that the amounts of these components were reduced as a function
271 of the degradation caused by the termites, the material that was previously degraded as a
272 function of heat (of the samples from the control group) was not present in the analysis and,
273 for this reason, was not degraded as a function of temperature.

274

275 **4 CONCLUSIONS**

276 The deterioration caused by termites of the genus *Nasutitermes* sp. changed the
277 chemical characteristics and thermal stability of *Eucalyptus grandis* wood. Based on the
278 quantitative chemical analysis, the occurrence of a non-preferential degradation of the main
279 chemical components of the wood (cellulose, hemicellulose) was observed, proving the low
280 specificity of this xylophagus for the component and the type of wood to be deteriorated.

281 The infrared spectroscopy technique, together with the analysis of the relative
282 intensity of the bands of spectra and modifications of the functional groups, contributed
283 positively to the study, since they enhanced the understanding of the recurrent variations in
284 the chemical structure of the wood deteriorated by the termites in relation to the control
285 group.

286 The application of the thermogravimetric analysis allowed to correlate the mass loss
287 with the attack of the termites, demonstrating that there was a reduction in the mass loss of
288 the deteriorated wood due to the termite attack, which is related to the degradation of the
289 primary components (cellulose, hemicellulose and lignin) of the wood.

290

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294

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