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Additional Information

1 Acoustic properties of agroforestry waste orange
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18 **Abstract**

19 The present paper investigates the acoustic properties of natural fiber reinforced
20 composites. Fibers from orange tree pruning were obtained and subject to different
21 treatments in order to obtain mechanical, thermomechanical and chemi-

22 thermomechanical pulps. These pulps were used as reinforcement for a
23 polypropylene matrix. The obtained composite materials were submitted to
24 acoustical tests in an impedance tubes device. The transmission losses obtained
25 against the fiber content were obtained and discussed. Latter it was researched the
26 influence of the fiber treatments on the soundproof characteristics. A numerical
27 method was used to preview the acoustic insulation of the materials against the
28 sound frequency. Finally the results were compared with that of the most usual
29 lightweight soundproof solutions.

30 *Keywords:* Soundproofing, Natural fiber composites, Agroforestry recycling

31 **1 Introduction**

32 Noise is considered one of the worldwide biggest polluters [1, 2]. In the 90's, the
33 World Health Organization (WHO) provided worrying data about that kind of pollution
34 in the United States: about 40% of the population were exposed to road traffic noise
35 with an equivalent sound pressure level exceeding 55 dB(A) daytime and 20% were
36 exposed to levels exceeding 65 dB(A). More than 30% were exposed at night to
37 equivalent sound pressure levels exceeding 55 dB(A) which is disturbing to sleep. In
38 the recent decades, the society progressed significantly, from industrial,
39 technological and social points of view. Most of the actions made by the human
40 beings in relation with the industry, the use of new technologies, or in their day to
41 day interactions with the environment are a cause of noise. Therefore, noise
42 pollution remains a matter to be resolved. The effects on the health due to noise
43 exposure are well known [3]. Most health effects are of a sensorial kind as stress,
44 leading to high blood pressure, coronary heart disease, stroke, and other. In most

45 cases the diagnosis is not immediate, aggravating the situation further. Sound
46 insulation is one of the techniques used to reduce the effects of noise in the cited
47 cases. However, the problem is not completely solved and needs further research to
48 find new materials capable of improving the performance of the conventional
49 solutions [4-6] In the case of buildings, the most used solutions are light materials as
50 the laminated gypsum boards. Construction materials as lightweight aggregate
51 concrete have been researched and its acoustic properties have been characterized
52 [7], but, to the best knowledge of the authors, the proposed agroforestry waste
53 reinforced composites have been not studied.

54 On the other hand, there is a need for new and innovative materials capable to
55 satisfy new requirements as lightness, sustainability and cost efficiency [8-11]...
56 Composite materials are a very active research field, and the source of many
57 engineering solutions. As an example, natural fiber reinforced composites provide a
58 way to recover and add value to agro-forestry wastes, avoiding its incineration and
59 the resulting generation of CO₂. For this research, the use of orange tree pruning as
60 reinforcement for composite materials can reduce the need for burning, provide low
61 cost alternatives to wood fibers, and extend the value chain for the agricultural
62 industry [12].

63 The main advantages of using lignocellulosic fibers, instead of mineral fibers, as
64 reinforcement of polyolefin matrices are; their high specific mechanical properties,
65 good aspect ratios, low equipment abrasion during preparation and manufacturing,
66 high availability, low density, and comparatively low cost per volume basis [13]. The
67 last of the potential advantages is clear, as orange tree pruning are agro-forestry

68 wastes with any value. Moreover, the composite materials reinforced with natural
69 fibers, as orange tree pruning, could be considered almost 100% recyclable, as
70 recovering energy by incineration is possible.

71 In many of the States of the European Union the basic quality soundproofing levels
72 that all the buildings and installations must achieve are regulated. Hence, all the
73 proposed solutions must be adapted to these regulations. The norms include
74 materials and procedures to correctly obtain the targets, as well as the tests to be
75 performed, to prove the quality of the results. While some of the regulations are
76 informative, some others are mandatory, setting bounds for the insulation, limit
77 values for the reverberation time and installation vibrations. The energy savings must
78 be also noted, having in account the guidelines to obtain buildings with an envelope
79 that limits the energy demands to obtain a correct thermal comfort. Moreover,
80 Directive 89/106/CEE-construction products, and Regulation UE n. 305/2011, that is
81 applicable from July 2013 and must be developed by the member countries, adds in
82 one of the annexes the regard to the use of sustainable and natural resources. The
83 annex establishes that new building works must be designed, build and demolished
84 in a way that the use of natural resources is sustainable and ensure: a) the reuse
85 and recycling of all the materials after the building is demolished, b) the durability of
86 the building and c) the use of raw materials and secondary materials must be
87 compatible from an environmental point of view. In that context it is possible to
88 propose real sound-proofing solutions, based on the studied composite materials,
89 with application to new building works and to building rehabilitations.

90 In this work, mechanical pulp (MP), thermomechanical pulp (TMP), and chemi-
91 thermomechanical pulp (CTMP) from orange tree pruning were used to prepare
92 composite materials, formulated with a 20 to a 50 wt% of MP, TMP, and CTMP as
93 reinforcement of a polypropylene (PP) matrix. The acoustic properties, against aerial
94 noise, of single layer soundproof elements made with the formulated composites are
95 tested and discussed. The objective of the research is finding the influence of the
96 percentage of reinforcement on the acoustic properties of the composite materials. It
97 was also researched the influence of the chemical treatments on such properties.
98 The researched materials showed themselves light and feasible solutions for
99 soundproofing against aerial noise, and a clear alternative to the conventionally used
100 laminated gypsum boards.

101 **2 Materials and methods**

102 *2.1 Materials*

103 The composites were prepared using polypropylene (PP) homopolymer (Isplen
104 PP099 G2M) with an average melt flow rate (230 °C; 2.16kg) of 55 g per 10 min and
105 a density of 0.905 g/cm³, kindly provided by Repsol-YPF (Tarragona, Spain).
106 Polypropylene functionalized with maleic anhydride (MAH-PP) (Epolene G3015) with
107 an acid number of 15 mg KOH/g and Mn of 24800 Da was acquired from Eastman
108 Chemical Products (San Roque, Spain). Biomass from orange tree pruning fibers
109 (OPF) obtained from seasonal tree pruning was supplied by Mas Clara de Domeny
110 (Girona, catalonia, Spain). Other reactants were used: Diethyleneglycol dimethyl
111 ether (diglyme) was supplied by Clariant and was used as dispersing agent.
112 Decahydronaphthalene (decalin) (190 °C boiling point, 97% purity) supplied by

113 Fisher Scientific was used to dissolve the PP matrix in the fiber extraction from
114 composites process. The reactants that were used for fiber treatment are
115 summarized as follows: sodium hydroxide (Merck KGaA, Darmstadt, Germany),
116 antraquinone (Badische Anilin & Soda Fabric AG, Germany) used without any further
117 purification. Pes-Na (polyethene sodium sulfonate) is an anionic polyelectrolyte.
118 Poly-DADMAC (polydimethyl diallyl ammonium chloride) is a cationic polyelectrolyte.
119 Pes-Na 0.001N and Poly-Dadmac 0.001N were supplied by BTG Instruments GmbH
120 (Germany).

121 *2.2 Preparation of orange tree pruning derivatives*

122 All the biomass from orange tree pruning was submitted to a crushing and
123 classification process. Some OPF samples were submitted to a defibering process
124 under cold aqueous conditions in a Sprout-Waldron equipment to obtain mechanical
125 pulp (MP) with a higher aspect ratio. This process gave almost 100% yield with
126 respect to the starting material [14, 15]. Another OPF sample was submitted to a
127 thermo-mechanical process (vaporization followed by defibering). The OPF were
128 heated to 160 °C for 30 min, and the obtained pulp was rinsed with water and then
129 passed through Sprout-Waldron equipment, resulting in thermo-mechanical pulp
130 (TMP) with an increased reactant surface, and around 95% yield. For OPF chemi-
131 thermomechanical fibers, the OPF were submitted to a sodium/
132 hydroxide/antraquinone (AQ) cooking process (5% NaOH: 0.1% AQ) in a liquid to
133 fiber ratio of 4:1, working at 160 °C for 20 min. Afterwards, the slurry was washed
134 and shredded in Sprout-Waldron equipment, giving around 90% yield.

135 2.3 *Compounding*

136 Composite materials comprising 30 to 50 wt% PP/OPF with and without
137 coupling agent were obtained. The materials were prepared in a Brabender®
138 plastograph internal mixing machine. The working parameters were 80 rpm for OPF
139 during 10 min at a temperature of 180 °C. In the case of the coupled composites, the
140 MAH-PP was added to the plastograph with the PP pellets. The resulting blends
141 were ground with a knives mill, dried, and stored at 80 °C for at least 24 h before
142 processing. Materials with 30 to 50 wt% MP from OPF where prepared and will be
143 referred in the text as MP a%, where a% is the OPF content. Similarly composite
144 materials with a 30 wt% of TMP and CTMP from OPF were obtained.

145 2.4 *Composite processing*

146 The samples for the tensile test were produced with a steel mould in an injection-
147 molding machine (Meteor 40, Mateu & Solé). Ten test specimens from each
148 obtained composite blend were used for the experiment. The processing
149 temperatures were 175, 175, and 190 °C (the machine has three heating areas), the
150 last corresponding to the injection nozzle. First and second pressures were 120 and
151 37.5 kgf/cm², respectively. Standard composite specimen samples (approx. 160 x
152 13.3 x 3.2 mm) were obtained and used to measure the tensile properties in
153 agreement with ASTM D638.

154 2.5 *Mechanical characterization*

155 Prior to the mechanical testing, the specimens were stored in a Dycometal
156 conditioning chamber at 23 °C and 50% relative humidity for 48 h, in agreement with
157 the ASTM D638 standard. Afterwards, composites were assayed in a Universal

158 testing machine (InstronTM 1122), fitted with a 5 kN load cell and operating at a rate
159 of 2 mm/min. Tensile properties were analyzed by means of dog-bone specimens,
160 according to the ASTM D638 standard. The Young's modulus was obtained with an
161 extensometer. Results were obtained from the average of at least 5 samples.

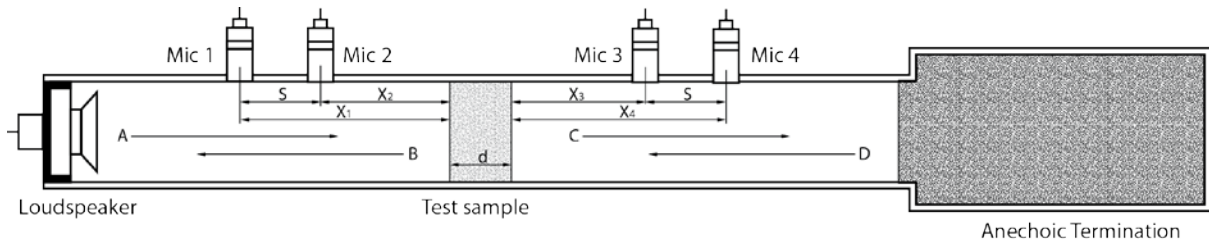
162 2.6 *Acoustic characterization*

163 The acoustic characterization of the materials is a fast growing research line, mainly
164 due to the expanding services that many companies are required to provide. Some
165 examples are the textile, paper and composite materials industries.

166 One of the main acoustic parameters to characterize is the specific flow resistance of
167 the composite materials. There are international norms that provide guidance to
168 measure that parameter (ISO 9053:1991), and as an alternative there are also
169 widely accepted experimental procedures [16]. The procedure is based on
170 measurements in an impedance tube, as one of the methods to measure the
171 absorption coefficient at normal incidence, described by the cited norm [17], a
172 parameter of high interest to acoustically characterize the materials as absorbent.
173 However there are no standard procedures to estimate the transmission losses in
174 materials or panels from the impedance tubes. Nonetheless there are some authors
175 that based their measurements of the transmission losses, and their acoustic
176 characterization of the materials, from impedance tubes tests [18-22].

177 In the Polytechnic School of Gandia, a new method to measure the transmission
178 losses (TL), based on impedance tubes, was developed and used for that research.
179 The design and development was based on the available literature.

180 The device is based in two impedance tubes used to measure the transmission
 181 losses (Fig.1).



182

183 **Figure 1: Diagram of the device used to measure the transmission losses**

184 The loudspeaker, placed at the end of the tube, generates plane waves. The path
 185 followed by these plane waves inside the tube are referenced in figure 1 as A, B, C
 186 and D the microphones, two placed in the tube between the loudspeaker and the
 187 sample, and two placed at the rear end, between the sample and an anechoic
 188 termination. The device represents the description of the transference matrix that
 189 represents the incident and reflected waves from the upper and lower parts of the
 190 sample. If the matrix coefficients are known it is possible to obtain the TL from eq.1:

$$191 \quad TL = 20 \log_{10} \left| \frac{e^{jks} - H_{12}}{e^{jks} - H_{34}} \right| - 20 \log_{10} |H_t| \quad (1)$$

192 Where S is the distance between the microphones, H_{12} and H_{34} represent the
 193 transference function between the microphones 1 and 2 (preceding the sample), and
 194 3 and 4 (subsequent to the sample) respectively, defined by eq.2:

$$195 \quad H_{i,i+1} = P_{i+1} / P_i \quad (2)$$

196 Where P_i is the complex acoustic pressure at point i , and is measured by the
 197 microphones.

198 The relation between the auto spectrums, H_t is defined by the eq.3:

$$199 \quad H_t = \sqrt{|S_d/S_u|} \quad (3)$$

200 Where S_u is the auto spectrum preceding the sample and S_d is the auto spectrum
201 subsequent to the sample, that are obtained by applying Eq 4 and 5.

$$202 \quad S_d = P_3 \cdot P_4^* \quad (4)$$

$$203 \quad S_u = P_1 \cdot P_2^* \quad (5)$$

204 Where P_2^* and P_4^* are the complex conjugates of the complex acoustic pressure at
205 points 2 and 4.

206 There are some particularities that define the test facility designed at the Polytechnic
207 School of Gandia. The tube preceding the sample measures 1315mm, and the
208 section subsequent to the sample measures 1233mm. Both tubes have a 40mm
209 interior diameter. The distances X_1 and X_2 were 120 and 80 mm, respectively, and
210 X_3 and X_4 120 and 80 mm subtracting the sample thickness. The prototype allows
211 for tree different distances between the microphones, while the standard
212 mechanisms allow only two. The distance between the microphones determines the
213 spectrum of frequencies to measure, as it must be ensured a plane propagation
214 wave in the tube (ISO, 1998). In that work, a 32mm distance was used to perform all
215 the measures.

216 *2.7 Prediction of the acoustic insulation*

217 There are a lot of models to predict the acoustic insulation, both for aerial and
218 impact sounds [23-27]. Many models used to describe the airborne insulation are

219 based on the coupling effect between the acoustic impedances of the layers of a
 220 composite to obtain the global isolation of all the layers. The result is the index of
 221 sound reduction (R) or the Transmission Loss (TL) (ISO 10140-2, 2010) (ASTM E90-
 222 09), that could be expressed as a function of the frequency or as a global value.

223 3 Results and discussion

224 Table 1 shows the main mechanical properties of the composites, used to perform
 225 the acoustic insulation calculations. A 15mm gypsum board has a 10 to 12kg/m²
 226 mass, being slightly lighter than the proposed composite materials.

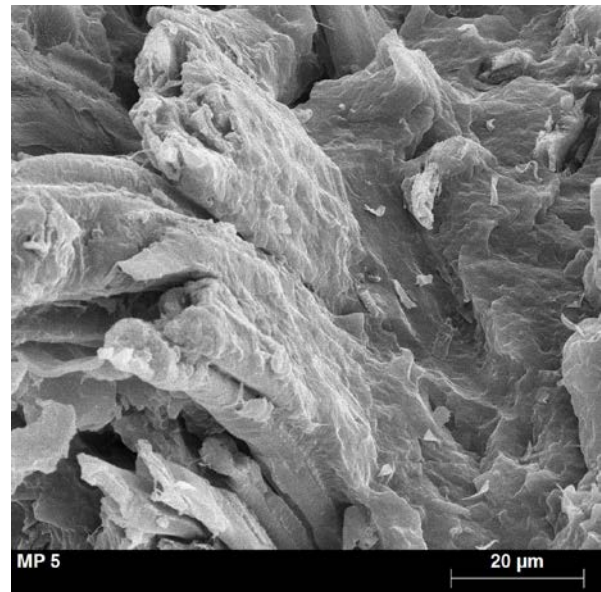
227 All the tested composite materials showed airflow resistance values higher than
 228 1000 kpas/m². The airflow resistance is the resistance experienced by air as it
 229 passes through a material. This property is directly related to the capacity of the
 230 material to absorb o reflect sound energy. The high values for the airflow resistance
 231 shown by the researched composites imply that such materials act as a sound
 232 impervious layer.

Material	Young's Modulus E (GPa)	Thickness (mm)	Mass (kg/m ²)	Critical Frequency (Hz)
MP 20%	2.8	15	14.8	2372
MP 30%	3.7	15	14.8	-
MP 40%	4.3	15	14.8	-
MP 50%	5.1	15	14.8	-
TMP 30%	3.1	15	14.8	2600
CTMP 30%	2.9	15	14.8	2687

233 **Table 1: Main characteristics of the tested specimens**

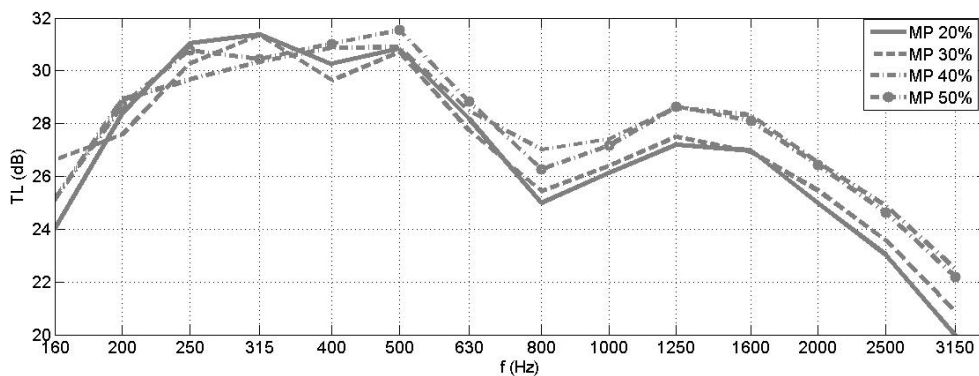
234 The materials showed a slightly toasted color as the amount of reinforcement was
 235 increased (Figure 2). Likewise, the microphotography, of the fracture zone shows the

236 good interface between the fibers and the matrix. The material is compact and not
237 porous, as the matrix totally wets the fibers.



238 **Figure 2: Visual appearance of the composites and microphotography of the fracture zone in a tensile**
239 **strength specimen.**

240 Figure 3 shows the Transmission Loss measured in the tested 20 to 50 wt% MP
241 composite materials.

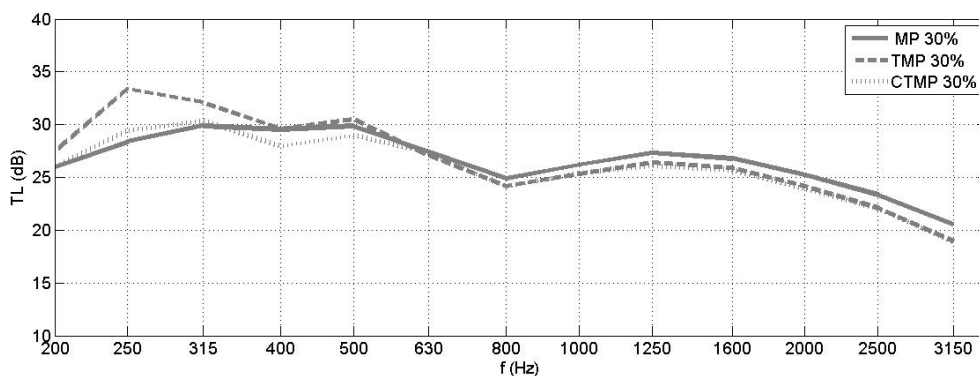


242

243 **Figure 3: Transmission loss against the frequency for the 20 to 50% mechanical pulp polypropylene**
244 **reinforced composite materials**

245 Figure 4 shows the transmission loss or insulation versus the frequency. All the
246 values were obtained under incidence normal conditions. The test were performed in
247 the impedance tube defined in the methods section

248 The results are similar for the 20 to 50 wt% MP from OPF composite materials.
249 Nevertheless, in the case of higher sound frequencies, the composite materials with
250 40 to 50 wt% of MP from OPF showed higher values of sound insulation. In the case
251 of mid to low frequencies, the values are similar for all the tested composite
252 materials. That range of mid to low frequencies are the most interesting to have in
253 account as are the most difficult to attenuate. Having that in account it was decided
254 to continue the test with the composite materials with 30wt% OPF contents.
255 Consequently, MP 30%, TMP 30% and CTMP 30% composite materials were
256 prepared ant tested. Figure 3 compares the Transmission Loss measured for the 30
257 wt% MP, TMP and CTMP composite materials.

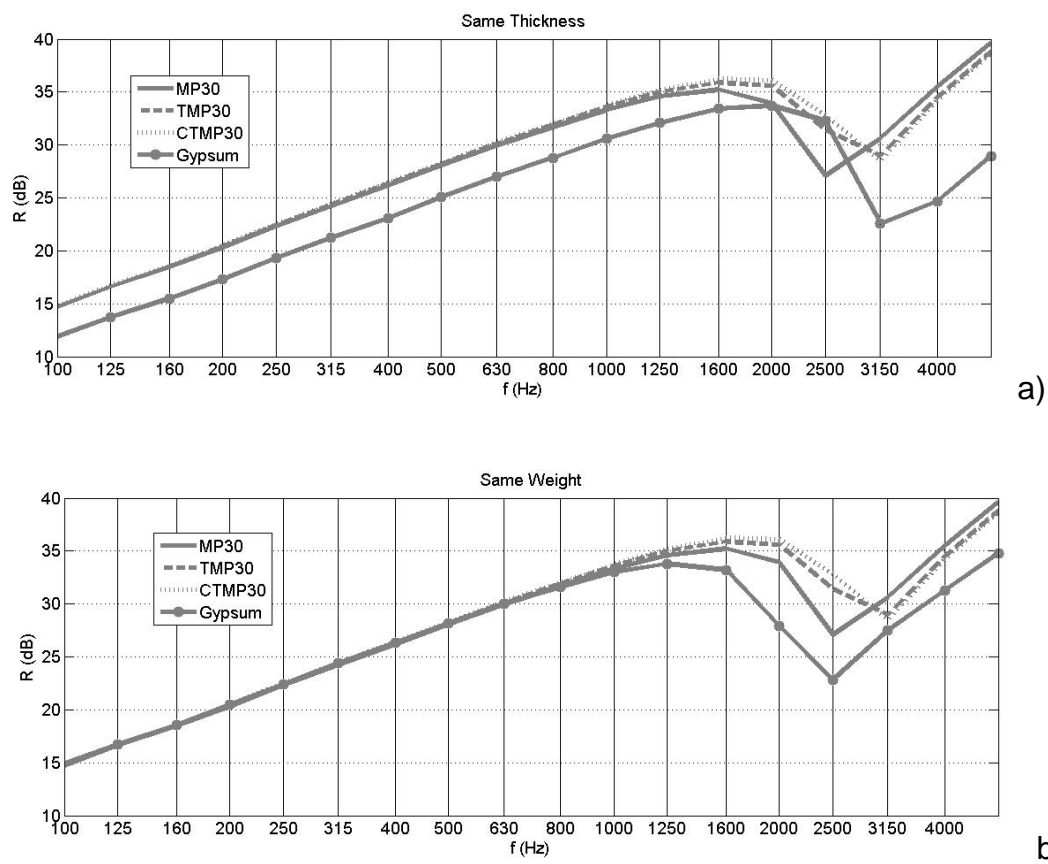


258

259 **Figure 4: Transmission loss against the frequency for the 30% mechanical, thermomechanical and**
260 **chemi-thermomechanical pulp polypropylene reinforced composite materials**

261 In the case of the tested materials, the value of the transmission loses (TL) are very
262 similar, regardless of the fiber treatment. Only the TMP 30% showed higher values
263 for some frequencies.

264 Figures 5a and 5b show the value of the index of sound reduction that are the result
 265 of the numeric simulation. The calculations were made for the 30 wt% MP, TMP and
 266 CTMP composite materials, and were compared with that of a lightweight common
 267 insulation material (laminated gypsum boards). Two different simulations were made,
 268 one supposing a constant thickness for all the samples (fig. 3b), and the other
 269 presuming that the mass of all the samples was the same (fig. 4b).



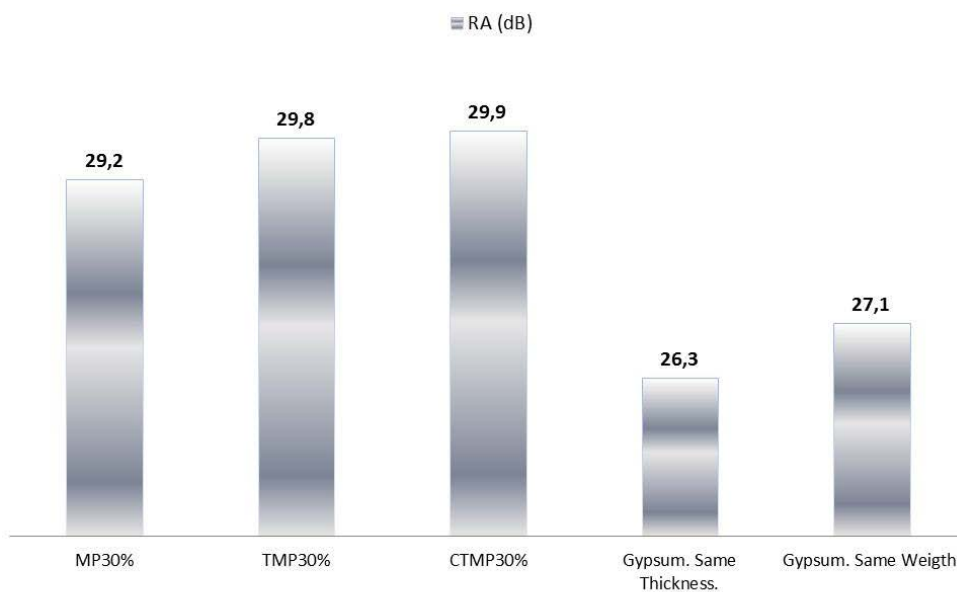
270 **Figure 5: Numeric simulations of the index of sound reduction for the 30% mechanical,**
 271 **thermomechanical and chemi-thermomechanical pulp polypropylene reinforced composite materials,**
 272 **compared with laminated gypsum boards.**

273 The thickness was presumed to be of 13 mm and the mass of 13.1 kg/m².

274 The values for the Transmission Loss or insulation shown in figure 4 were obtained
 275 under diffuse field conditions. The numerical methods that allow obtaining the values

276 require an integration limit angle [28]. The influence of the limit angle on the
277 precision of the computed values is an issue of current interest. The limit angle that
278 was used to obtain the values shown in figure 4a and 4b was 78°. It was found that
279 all the tested materials showed a sharp decrease of the insulation property between
280 2000Hz and 3000Hz. These values match the critical frequency, obtained from the
281 mechanical characterization (Table 1).

282 The weighted airborne sound insulation values are compared in figure 6.



283

284 **Figure 6: Acoustic insulation, global values comparison**

285 The global values of the evolution show that all the tested composite materials had
286 higher sound attenuation index than that of the commonly used insulation materials.

287 The values are 3dB higher in the case of the same thickness specimens, and 2dB

288 higher for the case of same mass specimens.

289 4 Conclusions

290 In this work, orange tree pruning fibers reinforced polypropylene composites are
291 presented as airborne insulation solutions. The study presents the behavior of the
292 acoustic properties of such composites against aerial sound. Their mechanical
293 properties, together with their relative lightness and their soundproofing properties,
294 show similarities to that of the impermeable layers, like the laminated gypsum
295 boards, commonly used by builders.

296 The influence of the reinforcement contents against the acoustic properties was
297 investigated. Reinforcement contents between 20 to 50% were tested. Likewise, it
298 was investigated the incidence of the fiber treatments in the acoustic properties.
299 While such treatments showed some impact on the mechanical properties, only
300 showed a slight incidence on the acoustic ones.

301 Moreover, the obtained insulation properties of the composites are compared with
302 that of the laminated gypsum boards, commonly used as lightweight solution for
303 building. The values were compared by frequencies and by weighted. To obtain this
304 values, a mathematical prediction model was used. The model allowed obtaining the
305 sound reduction index (R) in the diffuse field, understood as random incidence.

306 The tests developed by using an impedance tube, to investigate the impact of the
307 reinforcement content, showed that by increasing the percentage of reinforcing
308 fibers, the insulation could increase by 2dB. The Transmission Loss increased
309 significantly with reinforcement percentages higher than the 20%, mostly for the
310 medium and high frequencies. It was found that for reinforcement contents higher
311 than the 40 to 50% the soundproofing remained saturated

312 Composites reinforced with a 30% of MP, TMP and CTMP fibers were submitted to
313 test in the impedance tube. The results showed that the fibers treatment had little
314 influence in the sound insulation properties.

315 Predictive models were used to compare the acoustic properties of the composites
316 with that of the laminated gypsum boards. Regardless of considering equal mass or
317 equal thickness samples, the soundproofing properties, against aerial sound, of the
318 orange tree pruning fibers reinforced composites were always superior to that of the
319 laminated gypsum boards. Similarly, when comparing the weighted value, the
320 composites showed properties from 2 to 3dB higher than that of the laminated
321 gypsum boards.

322 The mechanical properties of the composite materials, together with the obtained
323 sound insulation properties, make those materials due to be used as light insulation
324 solutions. The application this new material is especially interesting for buildings, but
325 it is possible to use such materials in other fields, as cars or product design.

326 It will be necessary to perform a lifecycle assessment to study the environmental
327 impact of the agroforestry waste reinforced PP as light insulation solutions, in
328 comparison with laminated gypsum boards, to find if its recyclability compensates
329 the energy need to obtain the raw materials.

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