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1 **Emulation of field storage conditions for assessment of energy** 2 **properties of torrefied sugarcane bagasses**

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15 16 **ABSTRACT**

17 Sugarcane Bagasse (SCB) has some characteristics that hinder its direct use as fuel.
18 Torrefaction pre-treatment could be considered to improve the SCB properties for an energy
19 use and to reduce the impact of storage on the reduction of these energy properties.

20 SCB were torrefied at 290°C in a screw reactor, during 5, 7.5 and 10 min. Then, raw and
21 torrefied SCB samples were exposed successively to water-leaching and to white and brown
22 rot fungi, to emulate storage conditions. Mass loss after water-leaching process, moisture
23 content and weight loss due to fungal deterioration after 2, 4, 8, 12, 16 weeks were recorded
24 on each SCB samples. Finally, chemical composition and High Heating Value of the torrefied
25 samples were measured to determine the alterations compared to raw SCB during their
26 storage mimic.

27 Increasing torrefaction duration improves the SCB decay resistance. According to the fungal
28 exposure duration, loss of carbon was recorded, however, HHV remain higher for torrefied
29 SCB than those of raw SCB. Carbon contents and HHV values of raw and torrefied SCB
30 during *Trametes versicolor* exposure [55.20 % > C > 44.55 % and 21.260 MJ.kg⁻¹ > HHV
31 >17.307 MJ.kg⁻¹, results from all tests combined.], and during exposure to *Coniophora*
32 *puteana* [55.20 % > C > 46.25 % and 21.260 MJ.kg⁻¹ > HHV > 17.699 MJ.kg⁻¹] tend to
33 decrease.

34 Severe torrefaction is therefore a good way to improve SCB energy properties while limiting
35 loss of their energy properties during storage.

36 37 **KEYWORDS**

38 Energy, Fungal decay, Leaching, Storage emulsion, Bagasse, Torrefaction.

39 **INDEX SUMMARY**

40

41 **SCB:** Sugarcane bagasse (*Saccharum* spp.) residues sample

42 **SCB-HT-5:** Sugarcane bagasse – Heat-treated at 290 °C - during 5 min (given for example,
43 the same indexing is used for the different biomass and torrefaction residence time).

44 **SCB-Ref:** Sugarcane bagasse reference (raw biomass) sample

45 **TV:** *Trametes versicolor* fungi (white rot)

46 **CP:** *Coniophora puteana* fungi (brown rot)

47 **SCB-HT-5 (TV):** Sugarcane bagasse – Heat-treated at 290 °C - during 5 min and exposed to
48 *Trametes versicolor* fungi (given for example, the same indexing is used for the different
49 biomass, torrefaction residence time and fungi exposures).

50 **ML_{tt}:** Mass loss of the sample due to torrefaction process (in %, oven dry basis)

51 **Ext.:** Extractives content of the sample (in %, oven dry basis)

52 **ML_{leach}:** Mass loss of the sample due to leaching process (in %, oven dry basis)

53 **MC:** Moisture content of the sample due to fungal exposure (in %, oven dry basis)

54 **WL:** Weight Loss of the sample due to fungal degradation (in %, oven dry basis)

55 **C:** Carbon content of the sample (in %, oven dry basis)

56 **H:** Hydrogen content of the sample (in %, oven dry basis)

57 **N:** Nitrogen content of the sample (in %, oven dry basis)

58 **H/C:** Hydrogen / Carbon molar ratio

59 **HHV₀:** Higher Heating Value at constant volume of the dry (moisture-free) sample (in MJ.kg⁻¹,
60 oven dry basis)

61 1. INTRODUCTION

62 Brazil is strongly dependent to the agricultural and forestry production sectors. Brazil is still
63 among the top-countries using the most biomass for energy production with Total Primary
64 Energy Supply (TPES) from solid biomass of 15 GJ/capita [1]. The Brazilian energy
65 production from solid biomass is outranked by those of Finland (62 GJ/capita), Sweden (39
66 GJ/capita), Estonia (GJ/capita), Austria (24 GJ/capita) and Denmark (20 GJ/capita). However,
67 in term of global production, Brazil is the first country in the world using the most solid
68 biomass for energy production followed by the United States [1]. Lignocellulosic biomasses
69 mainly come from plantations dedicated to energy valorization, or as residues from some
70 agricultural production chain. Due to the huge sugarcane planting area in Brazil with 10
71 millions hectares [2], sugarcane ranks first among Brazilian crop wastes (including paddy
72 straw, bean straw, soya straw, maize straw and wheat straw) and agro-industrial residues
73 (including rice husk, coffee husk, sugarcane and fruits bagasse, soybean husk and wood
74 wastes) used for energy conversion [3]. Sugarcane bagasse has been widely used for energy
75 generation. Amongst the domestic energy alternatives in Brazil, biomass from sugarcane
76 bagasse accounts for 16.1% [4]. A large resource of bagasse is available at low cost [5],
77 providing raw material for energy production [6]. Each ton of sugarcane is estimated to
78 produce 140 kg of dry bagasse, giving a world supply of 200 million tons per year with
79 energy values range from 17 to 20 MJ.kg⁻¹ [7].

80

81 Even if sugarcane bagasse (SCB) can be burnt directly to produce heat and electricity, the
82 solid residue after pressing, has some characteristics that hinder its direct use as fuel,
83 compared to fossil fuel, such as high fibrous (43-52%, on a bagasse dry weight basis) and low
84 bulk density (80-120 kg.m⁻³), high moisture content (46-52%, on a wet basis), inconsistent
85 particle size, heterogeneous chemical composition, hydrophilic nature, and relatively low

86 calorific value [8, 9]. Sugarcane bagasse is composed by approximately 32–34% (on SCB dry
87 weight basis) cellulose, 19–24% (on SCB dry weight basis) hemicelluloses, 25–32% (on SCB
88 dry weight basis) lignin, 6–12% (on SCB dry weight basis) extractives, and 2–6% (on SCB dry
89 weight basis) ash [10]. In addition, the storage of the bagasse in humid conditions can cause
90 several damages due to moisture absorption and biological degradation [11]. For example, it
91 has been previously shown that fungal degradation (*Trametes versicolor*) of eucalyptus, pine
92 and coffee husks samples lead to decreases in their HHV values from 17.429 to 17.069 MJ.kg⁻¹,
93 18.801 to 17.377 MJ.kg⁻¹ and 18.823 to 17.194 MJ.kg⁻¹, respectively [12]. Indeed SCB is
94 usually stored in outdoor piles [13] occurring moisture adsorption by wood and the resultant
95 chemical oxidation and/or microbiological activity lead to hydrolysis [14]. This
96 phenomenon increases the internal temperature of the stored biomass stack thereby
97 accelerating the biomass decay degradation [15, 16]. Such degradations reduce the biomass
98 potential to be processed and consequently decrease its market value.

99

100 Some pre-treatment are then necessary like torrefaction, which is a thermal treatment who
101 improves the biomass energetic, physical and mechanical properties in order to make the
102 biofuel more homogenous and stable to biological degradation [17]. The market potential of
103 torrefied biomass is expected to be huge in Central Europe and the Nordic countries, and
104 also around the world, considering the substitution of coal in large-scale power and heat
105 product [18]. Torrefaction means a thermo-chemical treatment of biomass at 200 to 320°C
106 and the process takes place in oxygen free circumstances [19]. Although the torrefaction
107 allows reducing the hydrophilic behavior, it is possible that the torrefied wood nevertheless
108 reaches, during its storage, a sufficient moisture content level so that the rots can develop
109 [20]. Previous studies showed that such a process can be used in order to improve and
110 preserve the energetic properties of torrefied biomasses (eucalyptus, pine and coffee husks

111 residues) during water and fungal exposure [12]. Other past studies highlighted that the
112 improvement the quality of bagasse as feedstock can be also done with torrefaction pre-
113 treatment [21]. The utilization of torrefied bagasse in existing handling and storage facilities
114 and associated issues has been reported recently [22-24] and could be one of the solution to
115 preserve its energetic properties during storage depending on the torrefaction treatment
116 intensity [25]. Despite recent advances over the world, torrefaction technology is still under
117 development and only a few techno-economic assessments regarding the entire logistic chain,
118 including storage, have been made [20].

119

120 The aim of the study was to determine how the various raw and torrefied SCB materials
121 tolerate moisture and fungal decay exposure, simulating by various storage conditions. SCB
122 was torrefied at 290°C with 5, 7.5 and 10 min residence time then leached and exposed to
123 different fungal decay (brown and white rots) during five different exposure durations (from
124 2, 4, 6, 8, 12 and 16 weeks). Both effects of the torrefaction duration and the storage
125 conditions of SCB were investigated. The impact of water-leaching (without decay exposure)
126 and the fungal degradation impact on the chemical (elemental composition) and energy
127 properties (High Heating Values) of raw and torrefied SCB for an energy conversion were
128 thus evaluated. Finally, importance of the torrefaction intensity, depending on the process
129 duration, on these different mechanisms was highlighted. The results obtained through this
130 study could be a great additional data potentially enhancing the models used to predict the
131 energy properties and the degradation level of torrefied biomass during storage.

132

133 2. MATERIALS AND METHODS

134 2.1. Biomass samples

135 Sugarcane bagasse residues (*Saccharum* spp. hybrids) (SCB) were obtained from Jatiboca
136 Sugar and Ethanol industry (Ponte nova, Minas Gerais, Brazil). SCB was first dried outdoors
137 in a drying yard during 30 days, until they reached hygroscopic equilibrium moisture (about
138 20%, on wet basis). Afterwards, milling was done in a knife mill and a classification in screen
139 sieves between 6 and 2 mm, collecting the fraction retained on the 2 mm sieves to diminish
140 the presence of fine particles, which might compromise subsequent analysis. SCB particles,
141 with long size ranged from 10 to 20 mm (and 2-6 mm diameter), were oven-dried at $103 \pm$
142 2 °C for 48 hours with constant air circulation to reach a moisture content near to 0% and
143 avoiding the influence of water on treatments.

144

145 2.2. Torrefaction process

146 SCB was then torrefied at 290° C using 5 kg of biomass for each sample. The three following
147 residence times were evaluated: 5, 7.5, 10 minutes. Two torrefaction replicates were
148 performed for each modality. Torrefaction processes were carried out in an endless screw type
149 reactor, developed in the Panels and Energy Laboratory of the Federal University of Viçosa
150 (Fig. 1). The heating section (with length of 1.375 m) was built in carbon steel main structure
151 with an indirect heating exchange system with a combustion gas followed by an indirect water
152 cooling system.

153 Two timers controlling the gear motor were used to control the residence time of the biomass
154 sample within the reactor. The first of one was used to activate the gear motor for
155 approximately 4 seconds (time required for one complete revolution of the worm screw), and
156 the second timer was used to stop the gear motor for a predetermined time. The sum of these
157 two durations recorded but the both timers, multiplied by the number of revolutions of the

158 worm screw (without end with the roasting section) then defined the residence time of the
159 biomass samples it torrefaction process. A Gulton Gulterm 700-10S digital thermometer,
160 using eight type J thermocouples (Figure 1) was used to continuously record and control the
161 temperature of the torrefaction chamber. More information of the biomasses torrefaction
162 process and reactor can be found in Magalhães et al. 2018 [26].

163 **Figure 1**
164

165 **2.3. Mass loss due to thermal degradation**

166 The Mass Loss (ML%) due to the thermal degradation was determined according to the
167 Equation 1 (Eq. 1):

$$168 \quad ML_{tt} \text{ (in \% , oven dry basis)} = \frac{(m_0 - m_1)}{m_0} \times 100 \quad (1)$$

169 Where :

170 ML_{tt} = mass loss of the sample due to torrefaction process (in %, dry basis)

171 m_0 = initial oven-dried mass before torrefaction

172 m_1 = final oven-dried mass after torrefaction
173

174 **2.4. Chemical composition of raw and torrefied SCB samples**

175 The Chemical Composition was obtained for the determination of the Extractive, total Lignin
176 (insoluble and soluble), and Holocellulose contents. The samples were ground and sieved
177 with mesh of 40 (420 μm) and 60 (250 μm).

178 The total extractive content was determined according to TAPPI 204 cm-97 (TAPPI
179 1996). After a drying step performed at 103 °C until mass stabilization, the sawdust (10 g) was
180 extracted in a Soxhlet with a solution of 1/3 ethanol and 2/3 benzene [from Carlo Erba
181 Reagents Group (France)] during 6 h, and then dried at 103 °C for 48 h to obtain the oven-

182 dried mass of the free extractives sample. Extractive contents were determined by the oven-
183 dried mass difference.

184 The soluble lignin, insoluble lignin and holocellulose contents were determined on free-
185 extractives SCB samples, with some adjustments, according to the guidelines from the
186 methodology used by [Pereira et al. \[27\]](#).

187 Soluble and insoluble lignin contents were determined as following: 500 mg of the three
188 native and torrefied biomasses sawdust, previously extracted (by the 1/3 ethanol: 2/3 benzene
189 solvent extraction process), were separately mixed with 72% H₂SO₄ (10 mL) for 4 h at 30 °C.
190 The mixture was then diluted with distilled water until a H₂SO₄ concentration of 3%,
191 submitted to another hydrolysis at 2 atm pressure and 121 °C for 1 h. The insoluble lignin
192 content was determined by filtration and the soluble lignin content by UV spectrometry. Total
193 lignin was obtained by the sum of insoluble and soluble lignin fractions.

194 Holocellulose content was determined as following: 500 mg of the three native and torrefied
195 biomasses sawdust, previously extracted (by the 1/3 ethanol: 2/3 benzene solvent extraction
196 process), were separately placed in a 100 mL flask containing 30 mL of distilled water and
197 heated at 75 °C. Acetic acid (0.1 mL) and 15% aqueous sodium chlorite (2 mL) were then
198 added each hour for 7 h. The mixture was filtered on a Büchner funnel and the residue washed
199 with water, soxhlet extracted for 2 h with ethanol and dried at 103 °C to a constant mass.

200

201 **2.5. Fungal decay exposure**

202 All samples (raw and torrefied) were firstly submitted to leaching process according to the
203 [NFX 41-568 \[28\]](#) standard. This standardized procedure for accelerated ageing is commonly
204 used for natural durability wood testing. Samples were immersed in distilled water (1 volume
205 of biomass for 5 volumes of water) and subjected for six leaching periods of increasing
206 duration under continuous shaking at 20°C. Water was replaced by fresh water after each

207 leaching period. A first cycle of 3 leaching periods of 1, 2 and 4 hours was performed.
208 Samples were then kept air drying for 16 hours. Leaching was pursued for 3 additional
209 periods of 8, 16 and 48 hours, with change of water between each period. The leached
210 samples were then oven-dried at 103°C and weighed (m_2). Percentage of leaching was
211 calculated following the [Equation 2 \(Eq.2\)](#):

$$212 \quad ML_{leach} \text{ (in \% , oven dry basis)} = \frac{(m_1 - m_2)}{m_1} \times 100 \quad (2)$$

213 Where :

214 ML_{leach} = Mass loss of the sample due to leaching process (in %, dry basis)

215 m_1 = initial oven-dried mass of sample before leaching

216 m_2 = final oven-dried mass of leached sample

217

218 Then, raw and treated biomasses were placed into plastic grid bags (dimensions of 10 x 3 x 1.5
219 cm) in order to expose themselves to basidiomycete attacks. Before starting the fungal
220 exposure experiment, the bags were oven dried 103 °C for 24 h and the initial dry mass (m_3)
221 was measured for further determination of the weight loss of the sample due to the fungal
222 degradation. Each sample was crafted to have an initial dried mass of sample around 3 grams.
223 All samples were then placed in conditioned room (22°C, 70% Relative Humidity) in order to
224 keep humidity prior to the decay exposure tests. After that, all samples were sterilized by x-
225 ray process with a dose of 25.0 (\pm 5.6%) kGy, as recommended by the [NFX 41-568 \[28\]](#)
226 standard, by Ionisos Compagny (France). Contrary to autoclave sterilization, such a Gamma-
227 radiation does not cause significant changes in wood properties and not affect the wood
228 natural durability. In addition x-ray process is more efficient than oven drying to sterilized
229 wood samples.

230 Decay resistance of raw (leached and not leached) and torrefied biomasses after leaching was
231 tested according to an adaptation of [XP CEN/TS 15083-1 \[29\]](#) standard criteria, on both fungi
232 species required by the standard: *Trametes versicolor* (white rot) and *Coniophora puteana*

233 (brown rot). The following fungal exposure duration were tested: 2, 4, 8, 12 and 16 weeks.
234 During these fungal exposures, all test devices were place in dark conditioned chamber (at
235 22°C, 70% Relative Humidity). After the different decay exposure duration, mycelia were
236 removed and each sample was weight (m_4) and then was dried at 103 °C for 24 h and the final
237 dried mass was determined (m_5).

238 The final moisture content (MC) after fungal exposure of the sample was determined
239 according to the following formula (Eq. 3):

$$240 \quad MC \text{ (in \% , oven dry basis)} = \frac{(m_4 - m_5)}{m_5} \times 100 \quad (3)$$

241 Where :

242 MC = Moisture content of the sample due to fungal exposure (in %, dry basis)

243 m_4 = wet mass of fungal decayed sample sample

244 m_5 = oven-dried mass of fungal decayed sample

245

246 The Weight Loss was determined according to the following formula (Eq. 4):

$$247 \quad WL \text{ (in \% , oven dry basis)} = \frac{(m_3 - m_5)}{m_3} \times 100 \quad (4)$$

248 Where :

249 WL = Weight Loss of the sample due to fungal degradation (in %, dry basis)

250 m_3 = initial oven-dried mass of sample before fungal exposure

251 m_5 = final oven-dried mass of fungal decayed sample

252

253 The decay exposure tests were conducted as follows:

254 - The three torrefaction intensities for sugarcane bagasse, after leaching, were tested in order
255 to evaluate the impact of the heat treatment intensity on the biomasses durability;

256 - For both each fungus and fungal exposure duration, 4 replicates of raw biomasses (after
257 leaching) and for each torrefied biomasses (after leaching) were done.

258 - Beech (*Fagus sylvatica*) and Pine (*Pinus sylvestris*) sapwood samples of the dimensions
259 50x25x15mm³ (L, R, T) were used as controls for the virulence of the strains (8 tested blocks
260 for each fungus and for each fungal exposure duration).

261

262 **2.6. Sample characterization**

263 The impacts of leaching and fungal degradation, according to the duration of fungal exposure
264 were evaluated on the following raw and treated biomass properties according to 3
265 characterizations: (i) the Weight Losses (WL) caused by fungal degradation, the Elemental
266 Compositions (EC); and (iii) the High Heating Values (HHV). For each tested modality, the
267 four replicated used for decay test were mixed, ground using a cutting mill Retsch SM 100
268 and sieved. Particle sizes fraction of 0.1 to 0.2 mm was retained. Sawdust was then
269 conditioned at 103 °C for 24 h and stored in air-tight bottle before analysis.

270 **2.6.1. Elemental composition**

271 Elemental analyses were carried out with an ElementarVario Macro tube CHN analyzer
272 according to the [BS EN 15104 \[30\]](#). Prior to analysis, the samples were packed in a tin foil
273 and two tests were performed for each sample. Atomic H/C ([Eq. 4](#)) ratios of samples were
274 determined following [Patch et al. \[31\]](#) and [Ascough et al. \[32\]](#):

$$275 \quad \text{Atomic } \frac{\text{H}}{\text{C}} \text{ ratio} = \frac{\text{Number of H atoms}}{\text{Number of C atoms}} = \frac{\% \text{ H}/1}{\% \text{ C}/12} \quad (4)$$

276 **2.6.2. High Heating Values**

277 The calorific value was measured using an Automated Isoperibol Fixed Bomb Parr 6200
278 bomb calorimeter, following the [CEN/TS 14918 \[33\]](#).

279 1 g of oven-dried biomass, with a particle size < 0.2 mm, were required to perform the
280 calorific test. 2 replicates have been done for each sample and all the results are given with an
281 accuracy of $\pm 5.00\%$.

282

283 **2.7. Statistical analysis**

284 ANOVA (one-way analysis of variance) and Duncan's comparison test, carried out by the
285 JMP 10.0.2 program (SAS Institute Inc., Cary, NC, USA) by applying the Fisher test, were
286 used to evaluate:

287 - The impact of torrefaction duration and leaching process on elemental composition and
288 HHv values of SCB samples. Results were then ranked into several categories from "A" to
289 "D" to evaluate the influence torrefaction duration and from "a" to b" to compare unleached
290 and leached SCB samples (Tab.1).

291 - The impact of torrefaction duration on the chemical composition of SCB samples . Results
292 were then ranked into several categories from "A" to "D" (Figure 5).

293 - The impact of torrefaction duration on ML_{tt} (in %, dry basis) and ML_{leach} (in %, dry basis)
294 of SCB samples . Results were then ranked into several categories from "A" to "D" (Fig. 4).

295 - The impact of torrefaction duration on the chemical composition of SCB samples. Results
296 were then ranked into several categories from "A" to "D" (Fig. 5).

297 - The impact of fungal exposure duration on MC (in %, dry basis) (Fig. 6a) and WL (in %, dry basis)
298 (Fig. 7a) of pine and beech control samples. Results were then ranked into several
299 categories from "A" to "E", "A" to "E", "a" to "e" and "a" to "e" for beech control (CP),
300 pine control (CP) , beech control (TV) and pine control (TV), respectively.

301 - The impact of torrefaction duration on MC (in %, dry basis) (Fig. 6b) and WL (in %, dry basis)
302 (Fig. 7b), according to the fungal exposure duration of SCB samples. Results were then
303 ranked into several categories from "A" to "D", "a" to "d", for CP and TV, respectively.

304

305 The assumption of homogeneity of variance and normal distribution of the sample required
306 for the use of parametric statistics are satisfied. The impact of a parameter on a system not
307 connected by the same letter was considered as no significant at the 5% level.

308

309 For a better comprehension of the study approach, a scheme of the different analysis protocols
310 are presented in Fig. 2. In addition, the sampling and analytical devices used in this study are
311 illustrated in Fig. 3.

312

Figure 2

313

Figure 3

314

315 3. RESULTS AND DISCUSSION

316 3.1. Mass loss determination (ML_{tt} %)

317 According to the exponential kinetic of wood thermal degradation, the treatment duration has
318 an impact on treatment intensity as well as on mass loss kinetic [25]. The results presented in
319 Fig. 4, are in accordance with this statement. For heat treatments performed at 290 °C, ML_{tt}
320 (%) of SCB samples increased with the increasing of the process duration, from 12 ± 0.20 %
321 ($t=5$ min.) to $23,5 \pm 0.21$ % ($t=10$ min.). This phenomenon is due to the fact of for a given
322 temperature, the quantity of each degradation product increases progressively as the treatment
323 duration increases [34].

324

[Figure 4]

325

326 3.2. Chemical composition changes after torrefaction processes

327 According to the Fig. 5, raw sugarcane bagasse sample (SCB-Ref) was composed of 69.15 %
328 holocellulose, 25.49 % lignin (including 24.47 % insoluble lignin and 1.02% soluble lignin)
329 and 5.36 % extractive compounds. Fig. 5 indicated that extractives content of SCB increased at

330 290 °C – 5 and 7.5 minutes and seemed to decrease at 290°C – 10 minutes. These results are
331 consistent with other past studies [35, 36], highlighting that the major increase at low intensity
332 was due to water and ethanol extractives because of polysaccharide degradation. Almost all of
333 the original extractives released and new compounds were generated by hemicelluloses and
334 lignin thermal degradation. These mainly included monosaccharides and their dehydration
335 products, as well as syringaldehyde, syringic acid and sinapaldehyde, as the most prominent
336 lignin derived compounds [36, 37].

337 The proportion of lignin increases considerably after thermal treatment, mainly due to the
338 increase of insoluble lignin content, whereas soluble lignin content decreases according to the
339 heat treatment duration. The ether linkages of lignins are cleaved and new free phenolic
340 hydroxyl groups and a- and b-carbonyl groups arise, which are responsible for crosslinking
341 via formation of methylene bridges [38-40]. The methoxyl content decreases and the new
342 reactive sites on the aromatic ring can lead to further condensation reactions [39]. The
343 decomposition of the holocellulose, mainly due to hemicellulose degradation [41] is
344 statistically significant as the residence time is increasing. The holocellulose is decomposed
345 greatly and the proportion of lignin in the material increases, leaving a highly lignified
346 material [42-44].

347

348

Figure 5

349

350 FT-IR spectra analyses, performed on raw and torrefied sugarcane bagasse by Manatura
351 (2020) [45] indicated the changes in the chemical structures of the hemicellulose, cellulose
352 and lignin, which were associated with the functional groups such as C-O, C=O, O-H, C=C,
353 and C-C-O. Even if the thermal degradation kinetics are different than those of other wood

354 species, the undergone chemicals changes are similar for the sugarcane than those of woods,
355 in despite of their difference in chemical compositions and anatomical structures.

356

357 **3.3. Elemental Composition and High Heating Values of raw and heat-treated SCB** 358 **samples before and after leaching**

359 The objectives of this section are to (i) evaluate the influence of leaching step on the
360 elemental composition and energy properties (HHV) of sugarcane bagasse changes, and then
361 to (ii) determine if torrefaction process, according to the curing temperature and residence
362 time, improves the SCB energy properties and limit the impact of leaching for an energy use
363 (mainly for combustion conversion).

364 As shown in **Tab. 1**, torrefaction duration affects the element compositions of SCB materials.

365 The carbon and nitrogen in the material increased with residence time, while the hydrogen
366 decreased. Hydrogen and oxygen are released as water vapor and carbon dioxide emissions
367 [46]. Hemicelluloses mainly generate carbon monoxide and carbon dioxide through
368 decarboxylation reactions of acid groups linked to hemicelluloses [47], while cellulose
369 generates small amounts of carbon dioxide [48]. In addition, small volatile molecules (as
370 acetic acid, furfural, methylfurfural, vanillin, syringaldehyde, etc.) are released during
371 biomass torrefaction [34].

372

Table 1

373 As shown in **Tab. 1**, HHV increased with the residence time. Overall, **Tab. 1** and **Fig. 4**
374 indicate that the HHV increased with an increase of Mass Loss due to thermal degradation in
375 SCB samples.

376 One of the processes in material usage outdoors is leaching. Leachates of thermally modified
377 wood consist mainly of wood constituents' degradation products (tannic acid, acetic acid,
378 furfural and furfural derivatives), but also extractives [48], that may explain the higher

379 ML_{leach} % of SCB-HT-5 than those of the three other samples (Fig. 4). In addition, due to the
380 thermal degradation of SCB biomass components, the content of hydroxyl groups decreases
381 leading to increased hydrophobity and wettability that is more difficult. According to obtained
382 results of wood mass loss during the thermal treatment, degradation process in SCB-HT-10
383 (ML_{leach} = 2.16%) occurs at greater degree than in SCB-Ref (ML_{leach} = 3.54%), SCB-HT-5
384 (ML_{leach} = 4.88%) and SCB-HT-7.5 (ML_{leach} = 2.78%), thereby reducing the amount of
385 substances accessible to leach (Fig. 5). These results are in accordance with other past studies
386 showing that thermally modified pine and eucalyptus woods (temperature 190, 200 and
387 210°C; durations: 2, 6 and 12 hours are less sensitive to water depending on the treatment
388 intensity, than their respective native woods [49, 50]. Finally, thermally modified SCB treated
389 during 10 minutes (SCB-HT-10) is more environmentally friendly, than the other treatment,
390 due to less water leachable substances (Fig. 4).

391 Tab. 1 also highlights that the HHV of raw and heat-treated SCB samples was decreased by
392 leaching process. In view of the fact that the elemental composition is not significantly
393 modified for all SCB samples following leaching, the decreases of HHV values could be
394 explained by the removal of some extractives compounds during this leaching process. In
395 fact, previous studies shown that extractives content and composition are strongly correlated
396 to the HHV of woody biomass [51, 52].

397

398 **3.4. Decay degradation of raw and heat-treated SCB samples according to the fungus** 399 **exposure duration**

400 The objectives of this section is to simulate the storage of raw and torrefied SCB in order to
401 (i) evaluate the impact of SCB decay degradation on its energy properties and then to (ii)
402 determine if torrefaction process, according to the curing temperature and residence time,
403 allows to limit the impact of decay degradation on the energy properties.

404 **3.4.1. Moisture content**

405 **Fig. 6** shows the final moisture content in % of control beech and pine samples, raw and
406 torrefied SCB samples, after *Trametes versicolor* and *Coniophora puteana* fungi exposures
407 and according to the duration of these decay expositions.

408 Whatever the fungi exposed to the SCB samples, the final moisture content of SCB sample
409 was directly proportional to its hemicellulose content. By comparing **Fig. 6** and **Tab. 1**, it
410 clearly appears that the amount of water absorbed by sugarcane bagasse during fungi test
411 increased when its hemicellulose content increased. The hydrophilic nature of bagasse is
412 mainly due to the high content of hemicellulose. Hemicellulose polymers have many
413 hydroxyl groups (-OH) which drive sugarcane bagasse to be polar and to make easily
414 hydrogen bonds with water molecules [53]. Hemicellulose degradation during torrefaction
415 causes the loss of hydroxyl groups in sugarcane bagasse [51]. Thus, torrefied sugarcane
416 bagasse loses its hydrophilic nature, since there are no hydroxyl groups, which tend to be
417 polar and easily make hydrogen bonds with water molecules. In addition, the changes of
418 lignocellulosic polymers during torrefaction lead to the formation of unsaturated non-polar
419 structures in sugarcane bagasse.

420 The degradation of hemicellulose, cellulose, and lignin in sugarcane bagasse during
421 torrefaction causes shortening of polymer chains. The changes of sugarcane bagasse
422 properties into less fibrous, soft, and brittle is mainly caused by the degradation of
423 hemicellulose and cellulose [54].

424 **Figure 6**

425 The hydrophobic behavior of raw SCB is very similar than those of beech and pine control
426 samples. Their moisture content levels increase highly during the first four fungi exposition
427 weeks, and then continue to increase more slowly for longer tests durations (**Fig. 6**).

428 The moisture content of torrefied SCB samples, after decay exposure, is lower than those of
429 beech and pine control samples and raw SCB samples, whatever the decay test duration and
430 the fungi type. In addition, we can observe a significant moisture content decrease for SCB-
431 HT-7.5 and SCB-HT-10 samples compared to raw and SCB-HT-5 samples. The moisture
432 content of SCB-HT-7.5 and SCB-HT-10 seems to be increased highly during the four first
433 weeks and tend to stabilize for longer fungal exposure duration.

434 **3.4.2. Decay degradation**

435 The overall Weight Loss (WL %) due to *Trametes versicolor* and *Coniophora puteana*
436 exposure of beech and pine control samples, raw and torrefied SCB samples, is shown in **Fig.**
437 **7a.**

438 According to the weight loss values obtained concerning the fungal degradation of beech and
439 pine wood control sample after 16 weeks, the decay resistance test performed through this
440 study has been validated. Indeed, the following minimal degradation levels (according to **XP**
441 **CEN/TS 15083-1 [29]**) of control samples were reached:

442 - *Coniophora puteana* (Brown rot) on Pine: WL = 57.12 % > 30%

443 - *Coniophora puteana* (Brown rot) on Beech: WL = 48.18 % > 30 %;

444 - *Trametes versicolor* (White rot) on Pine: WL = 24.18 % – no requirement;

445 - *Trametes versicolor* (White rot) on beech: WL = 44.24 % > 20 %.

446 For the both tested rots, a similar tendency concerning the decay resistance improvement of
447 SCB was observed after torrefaction. In addition, as for the results observed previously with
448 respect to the moisture content levels, conferred durability to SCB by thermal modification
449 depends on the heat treatment duration.

450 Average values of weight loss due to fungal degradation showed raw and torrefied SCB
451 samples are generally similar to *Coniophora puteana* (Brown rot) than *Trametes versicolor*
452 (White rot). In addition, it clearly appears that SCB-HT-7.5 and SCB-HT-10 samples are

453 significantly more resistant to the tow fungal degradations than SCB-Ref and SCB-HT-5.
454 According to the torrefaction duration, the torrefied SCB samples suffered a destruction of
455 their cell wall [55], a largely hemicelluloses degradation than the other macromolecular
456 components [56], occurring moisture exclusion [57-59] and a limited feed stock, then
457 resulting in a high capacity to limit the colonization and the damages caused by the both
458 fungi.

459 According to the Fig. 7a, the both fungal degradation of beech and pine control samples
460 appears to begin as early as the fourth week of rot exposure. The fungal degradation (TV and
461 CP) of SCB-HT-5 starts as early as the second week of rot exposure, while those of SCB-HT-
462 7.5 and SCB-HT-10 samples is delayed and begins only from the eighth week of fungal
463 contact and then increase very slowly. Finally, SCB-HT-7.5 (WL < 5 %) and SCB-HT-10
464 (WL < 2%) seem to be very promising treatment ways to preserve SCB properties during the
465 biomass storage (Fig. 7b).

466 Figure 7

467 3.5. Impact of fungal exposure on the energy properties of raw and heat-treated SCB 468 samples

469 3.5.1. Degradation by *Trametes versicolor* (TV) – White rot

470 Loss of carbon was recorded in all samples with white rotter *Trametes versicolor* (TV) (Fig.
471 8a). According to the literature, white rot mainly degrades the lignin polymer, causing loss of
472 carbon [50]. Indeed, fungal activity was expected to reduce the carbon content as carbon
473 compounds serve as nutrients and are respired as CO₂ by fungi [60, 61].
474

475 The Nitrogen content increased according to the fungal exposure duration, in all tested
476 materials (Fig. 8c), whereas Hydrogen content decreased for SCB-HT and SCB-HT-5 and
477 increased for SCB-HT-7.5 and SCB-HT-10 (Fig. 8b). Previous study highlighted that fungi

478 have the capacity to scavenge and import nitrogen to decaying wood via a translocating
479 network of mycelium [62]. It results that, for all tested samples, H/C ratio increases according
480 to the *Trametes versicolor* (TV) exposure duration, where the most significant evolution
481 occurs in the first four weeks of fungal exposure (Fig. 8d).

482 As observed in Figures 8e and 8f, the decreases of HHV values, for each SCB samples (raw
483 and heat-treated), according the fungal exposure duration are in agreement with the litterature.
484 Depletion of carbon will lower the calorific value and here, statistically significant changes
485 were detected in carbon content. The heat content is related to the oxidation state of the
486 natural fuels in which carbon atoms generally dominate and overshadow small variations of
487 hydrogen and nitrogen content [63]. Susott et al. [63] and Tillman [64] found a linear
488 relationship between the higher heating value and the carbon content of the natural fuels,
489 chars, and volatiles. Huang et al. [65] and Protàtio et al. [66] have also put highlight that
490 Carbon and Hydrogen are the elements that contribute most to the Heating Values of
491 charcoal. Therefore, low O/C ratio is good to have high HHV values and low H/C ratio is not
492 really desired for the use of wood and charcoals for energy, by combustion process. However
493 high H/C ratios could be suitable for gasification conversion, making more reactive the
494 torrefied biomass.

495 Figure 8

496 3.5.2. Degradation by *Coniophora puteana* (CP) – Brown rot

497 Concerning the changes in Hydrogen (Fig. 9b) and Nitrogen (Fig. 9c) contents during fungal
498 exposure, similar results were observed for *Coniophora puteana* (CP) and *Trametes*
499 *versicolor* (TV). Regarding the Carbon content, different behavior were observed during
500 *Coniophora puteana* (CP) exposure, according to the heat treatment intensity. It results that
501 Carbon content decreases for SCB-HT-7.5 and SCB-HT-10 samples relating to the
502 *Coniophora puteana* (CP) exposition duration, whereas Carbon content increases for SCB-

503 Ref and SCB-HT-5 samples (Fig. 9a). These results could be explained by the remaining
504 cellulose and the hemicelluloses after torrefaction at low temperature (SCB-Ref and SCB-HT-
505 5) are degraded by *Coniophora puteana* (CP), increasing the relative carbon content. In fact,
506 brown-rot fungus (*C. puteana*) causes more weight loss than the white-rot (*T. versicolor*),
507 because it removes the cellulosic fraction and leaves lignin structurally modified. Brown
508 rotter *Coniophora puteana* (CP) prefers coniferous wood species, but seems to cause also
509 carbon loss in SCB. It uses endoglucanases to degrade cellulose and hemicelluloses, but has
510 also been reported to produce cellobiohydrolases that erode crystalline cellulose and could
511 therefore also utilize quite severely torrefied wood [67]. We observe a decrease of H/C ratio
512 and an increase HHV value for SCB-Ref and SCB-HT-5 samples according to the *T.*
513 *versicolor* exposure duration, whereas an increase of H/C ratio and a decrease HHV value for
514 SCB-7.5 and SCB-HT-10 samples were observed in the same conditions (Fig. 9d and Fig. 9e).
515 The increase of HHV in that case (SCB-Ref and SCB-HT-5), may be associated with the
516 degradation of the low-energy content components (e.g., leaf materials or agro-food residues)
517 that were easily accessible to the enzymatic community during storage condition [68].
518 Krigstin and Wetzel [69] explained in their review that changes to the proportion of
519 components in the biomass will affect energy content. For example, the preferential
520 decomposition of hemicellulose will result in increased heating values, but the loss of
521 extractives or the preferential fungal decomposition of lignin would tend to reduce heating
522 values.

523 Figure 9

524 3.5.3 Balance sheet

525 The High Heating Value of SCB was significantly increased according to the torrefaction
526 duration. However, HHVs of raw and torrefied SCB were affected by the decay exposure
527 duration. There was a relationship between the HHV and decay exposure time across all the

528 thermal treatments and fungi, but the change over time was small (Fig. 8e and Fig. 9e).
529 *Trametes versicolor* is a white rot fungus, degrading mainly the lignin polymer, therefore
530 occurred to all SCB samples a decrease in HHV according to the fungal exposure duration. At
531 the opposite, *Coniophora puteana*, a brown rot fungi degrading mainly the hemicelluloses and
532 cellulose fractions, brings an increase in HHV of SCB-Ref and SCB-HT-5 samples and only a
533 small decrease in HHV of SCB-HT-7.5 and SCB-HT-10 samples. Numerous studies on wood
534 storage have recently reported no significant change or a decrease of the higher heating value
535 [70-72], while others have reported a slight increase of heating value [73-75] at the end of the
536 storage.

537 From our laboratory experiment, it was clear that fungi are able to settle on seemingly
538 unfavorable material, which is said to be hydrophobic and suitable for outside storage. The
539 more mildly treated samples experienced more carbon loss, but took in less moisture in
540 outside storage, contrary to the more severely treated samples [20]. The loss of dry matter
541 (WL%) and carbon (C%) as well as the increasing moisture content decrease the calorific
542 value of the fuel and thus increase associated costs [20]. Therefore, torrefaction could be a
543 viable method to eliminate some of the disadvantages of raw biomass as it significantly
544 improves energy content and prevents absorption of moisture during storage [76]. Even if
545 HHV values decrease during fungal exposure, HHV values are still higher for high-intensity
546 treated SCB samples (SCB-HT-7.5 and SCB-HT-10) than those in the raw and or slightly
547 thermally modified (SCB-Ref and SCB-HT-5) (Fig. 8f and Fig. 9f).

548

549 4. CONCLUSION

550 The torrefaction residence time has an important impact on sugarcane bagasse (SCB) mass
551 loss kinetic during torrefaction process, undergoing chemical and elemental composition
552 modifications, High Heating Values and decay resistance improvements of torrefied SCB.

553 The storage emulation tests developed in laboratory show that raw and torrefied SCB are still
554 subjected to biological degradation and dry matter losses. It can be stated that in the most
555 cases, the two tested fungi use mainly the carbon fraction of native and torrefied SCB, which
556 leads to a decrease in calorific value. However, the recorded carbon loss due to fungal decay
557 decreased towards the torrefaction performed with a long residence time (290°C during 7.5 or
558 10 min).Torrefaction pre-treatment performed at severe intensity is therefore a good way to
559 improve SCB's energy properties while limiting lose torrefied biomass quality during storage.
560 Future studies will be needed to simulate the exact fungal degradation dynamics and evaluate
561 the properties changes of different torrefied biomasses during real-life storage.

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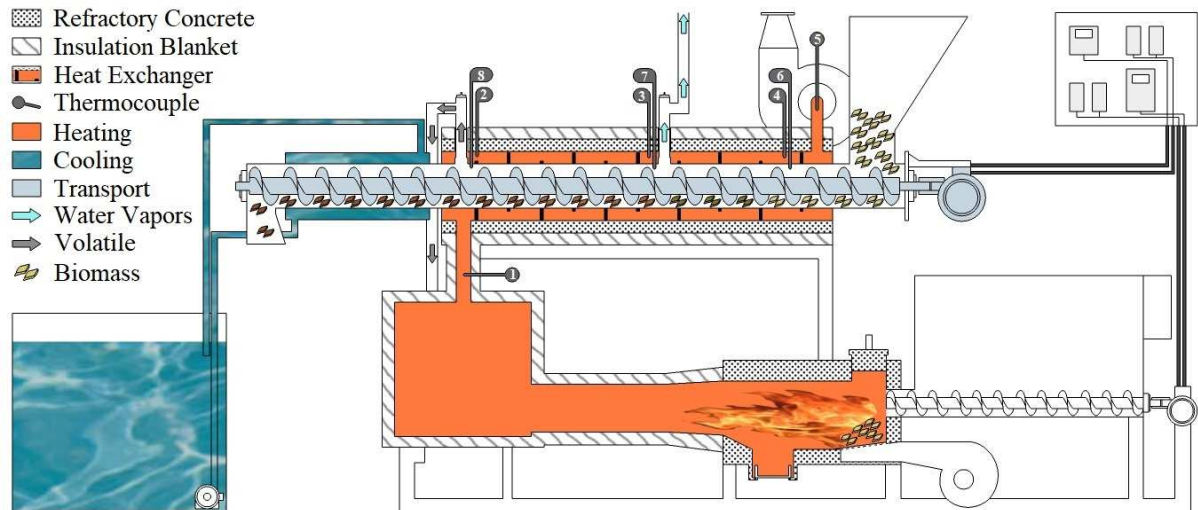
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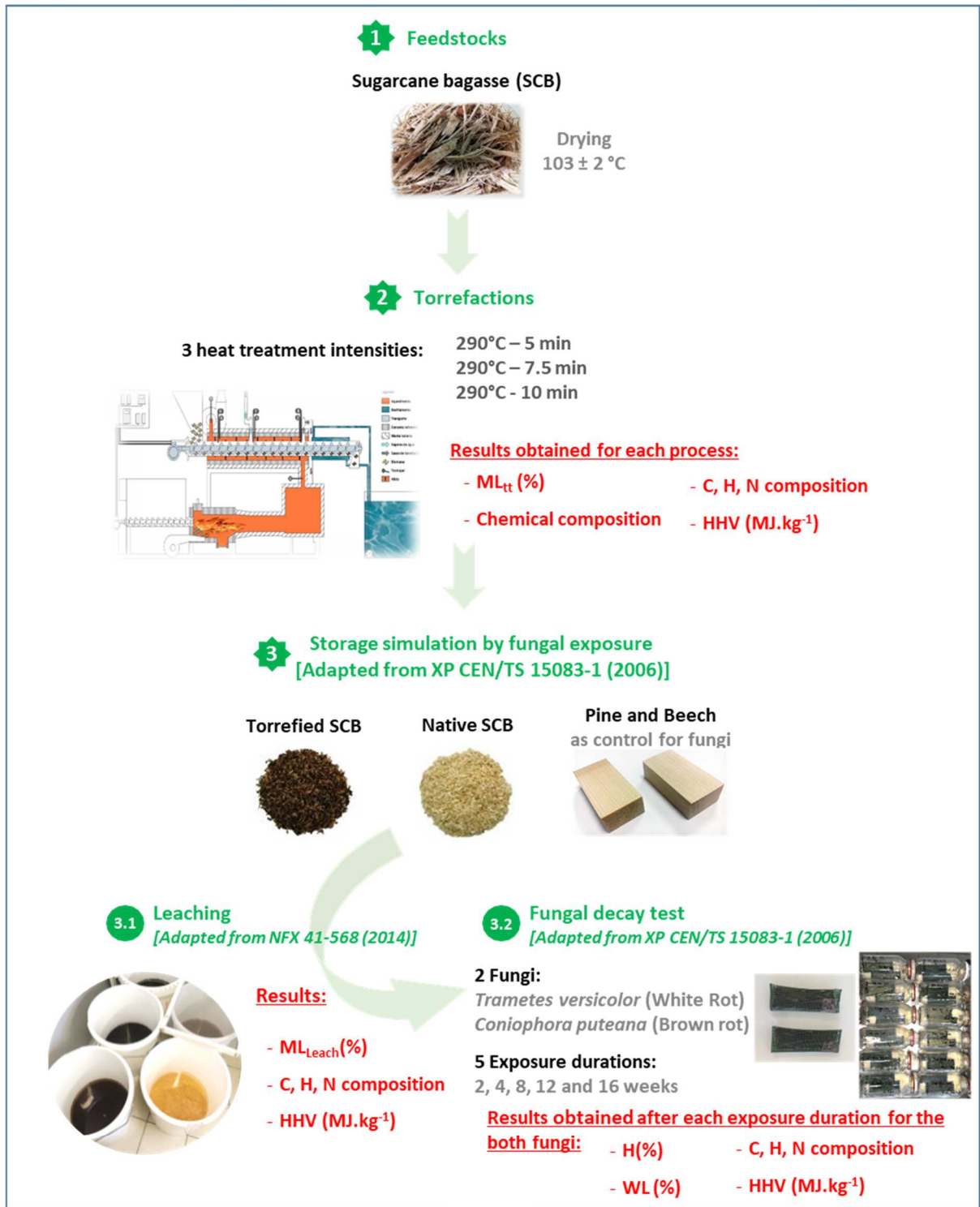
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Torrefaction Reactor Components



① to ⑧ : Thermocouples used in order to control the homogeneity of the temperature in the entire torrefaction chamber

Fig. 1 : Layout in side view of the reactor for biomass torrefaction in semi-continuous flow [18].



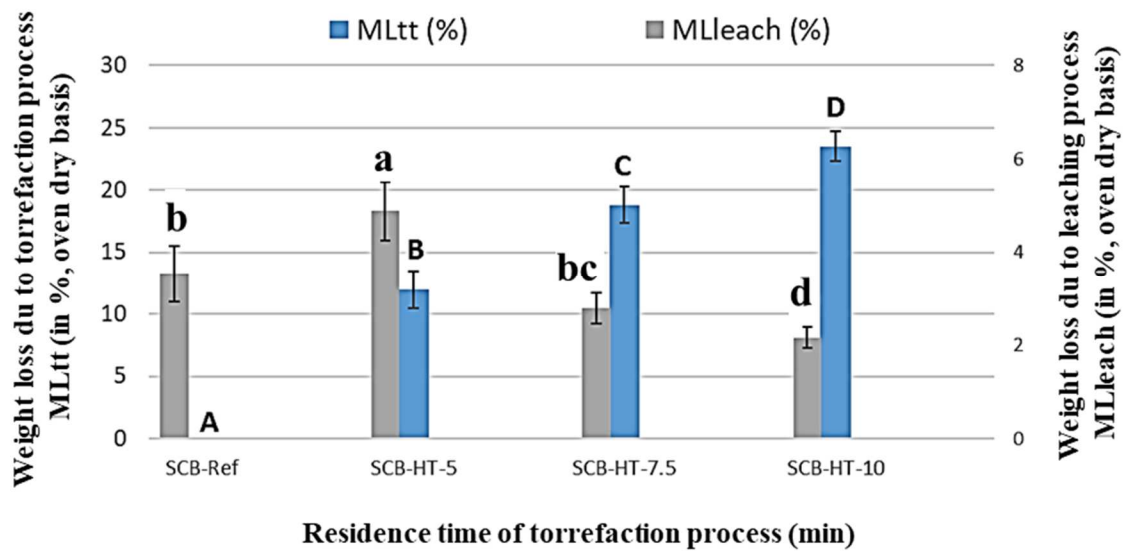
Main results :

- Impact of torrefaction on the SCB chemical and energy properties,
- Impact of leaching on the SCB chemical and energy properties,
- Impact of the fungal degradation on SCB chemical and energy properties.

Fig. 2: Synthesis of the overall approach and the different analysis protocols used for this study.

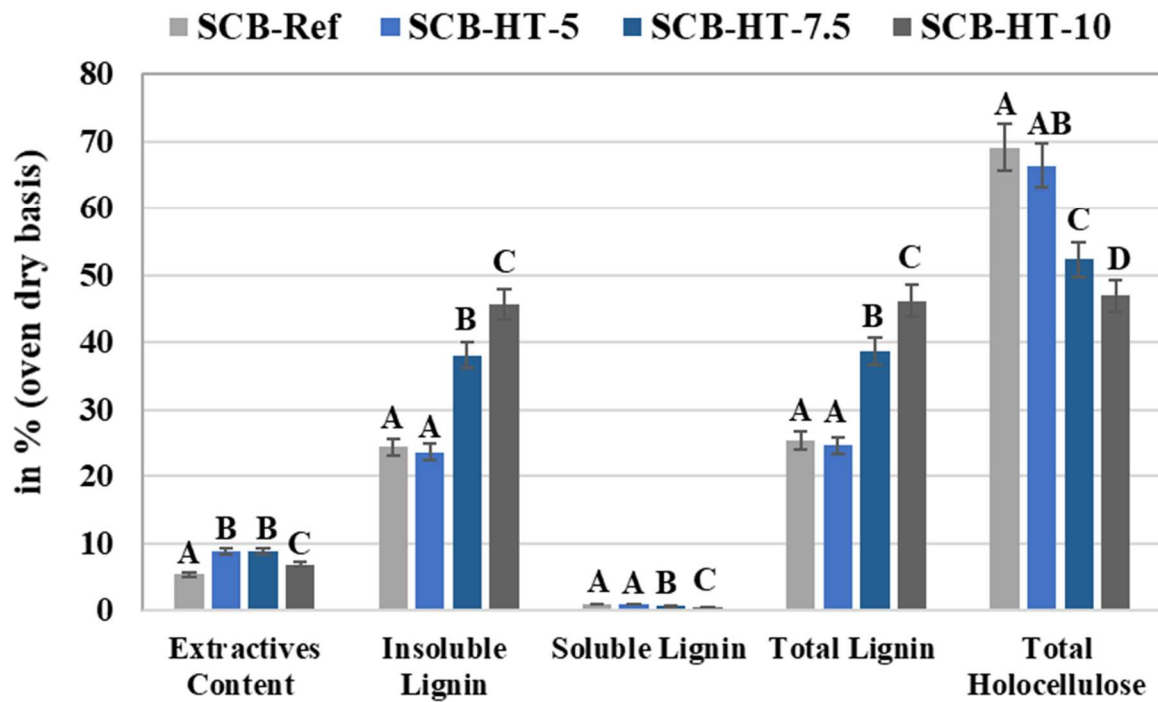


Fig. 3: The various sampling and analysis apparatus used in this study: (a) cutting mill Retsch SM 100, (b) ElementarVario Macro tube CHN analyzer, (c) an Automated Isoperibol Fixed Bomb Parr 6200 bomb calorimeter.



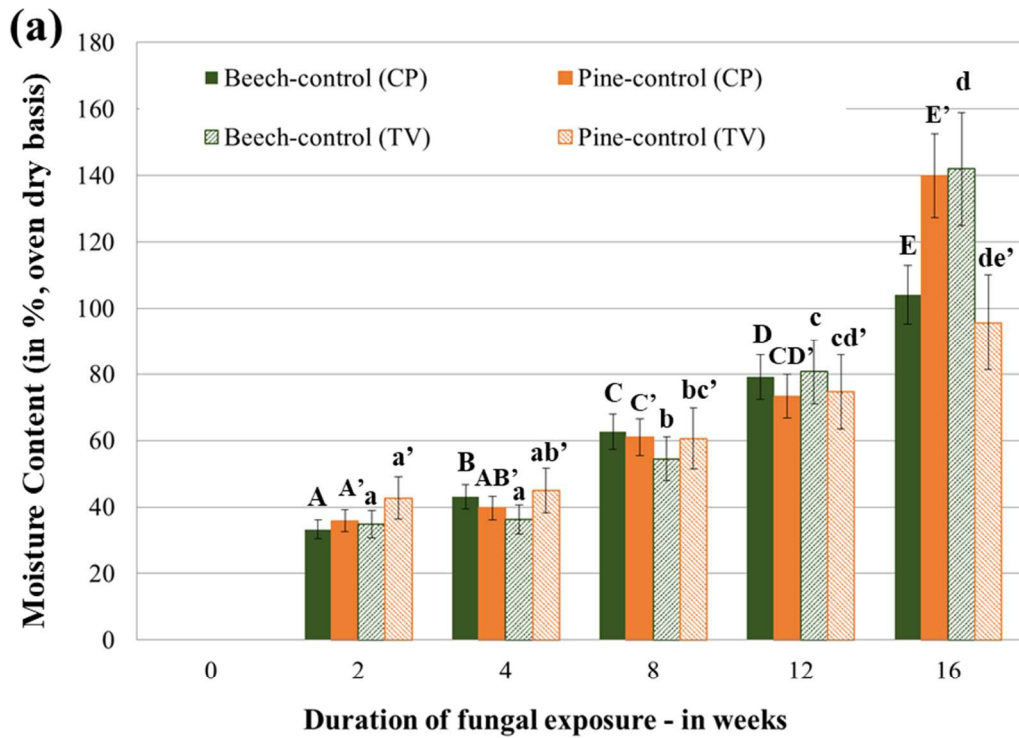
Letters: bar followed by the same letter are not significantly different. Here, the letters from A to D and from a to d indicate the influence of torrefaction duration on the Mass Losses (ML_{tt} %) due to thermal degradation and Mass Losses (ML_{leach} %) due to leaching process of SCB samples, respectively.

Fig. 4: Average values of raw and torrefied SCB Mass Losses (ML_{tt} %) due to thermal degradation and Mass Losses (ML_{leach} %) due to leaching process.

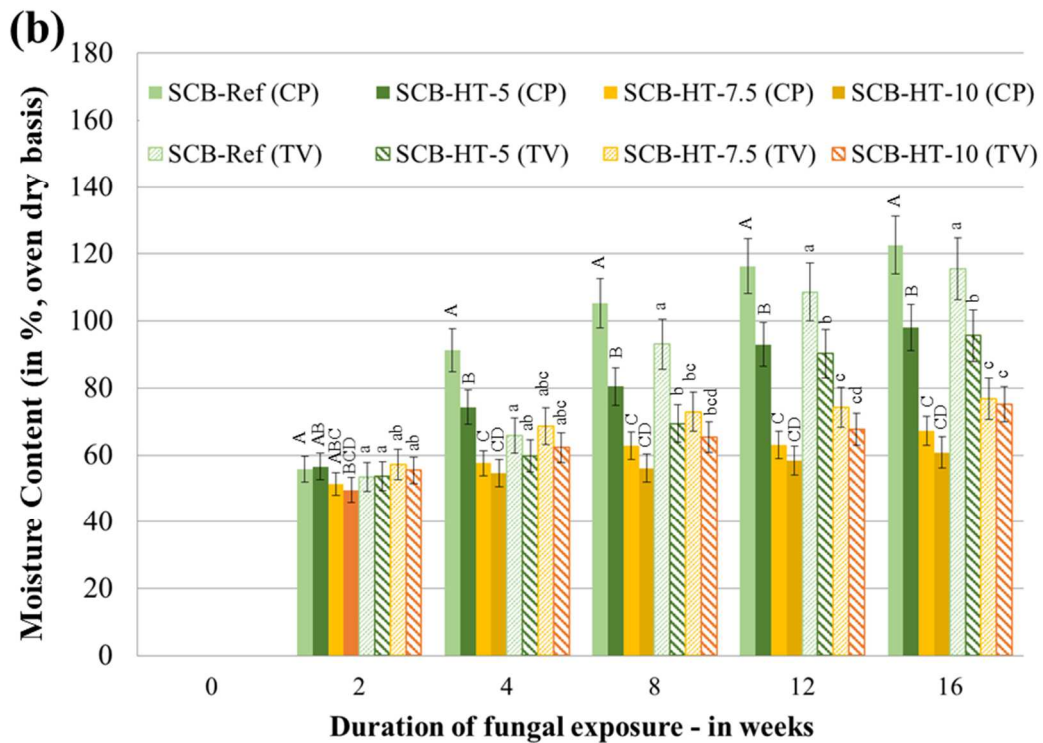


Letters: bar followed by the same letter are not significantly different. Here, the letters indicate the influence of torrefaction duration on the chemical composition of SCB samples.

Fig. 5: Chemical composition of raw and torrefied sugarcane bagasse (SCB). For each group, the means with the same letter were not significantly different at 5% ($\alpha = 0.05$).

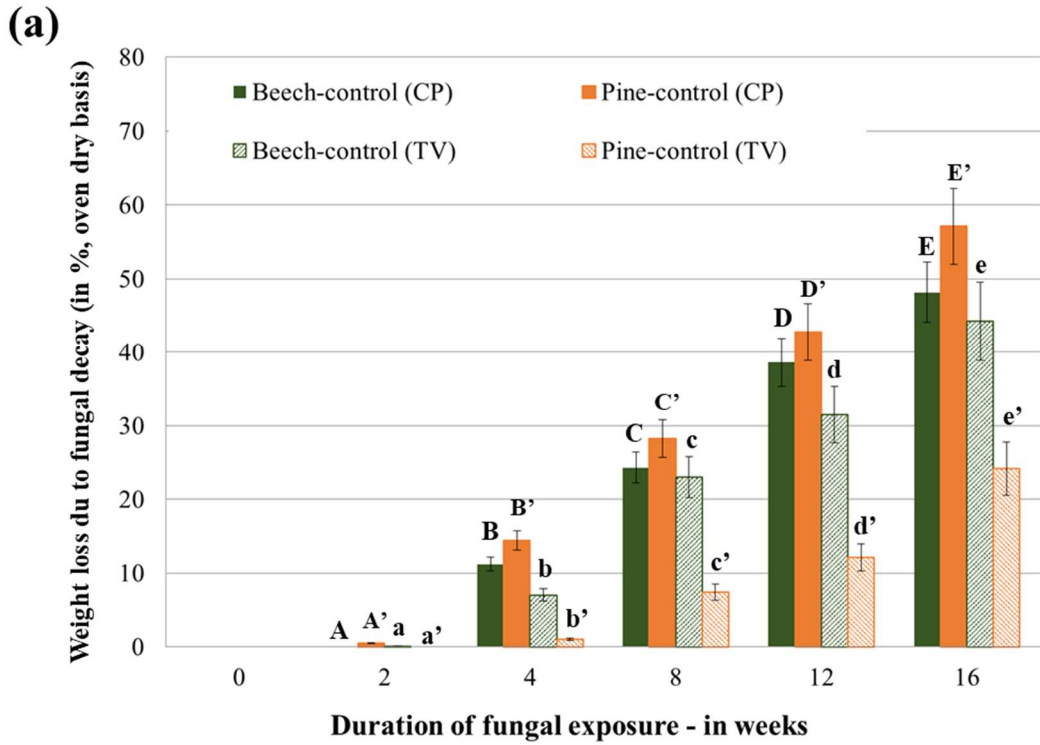


Letters: bar followed by the same letter are not significantly different. Here, the letters indicate the influence of fungal exposure duration on the moisture content of control samples.

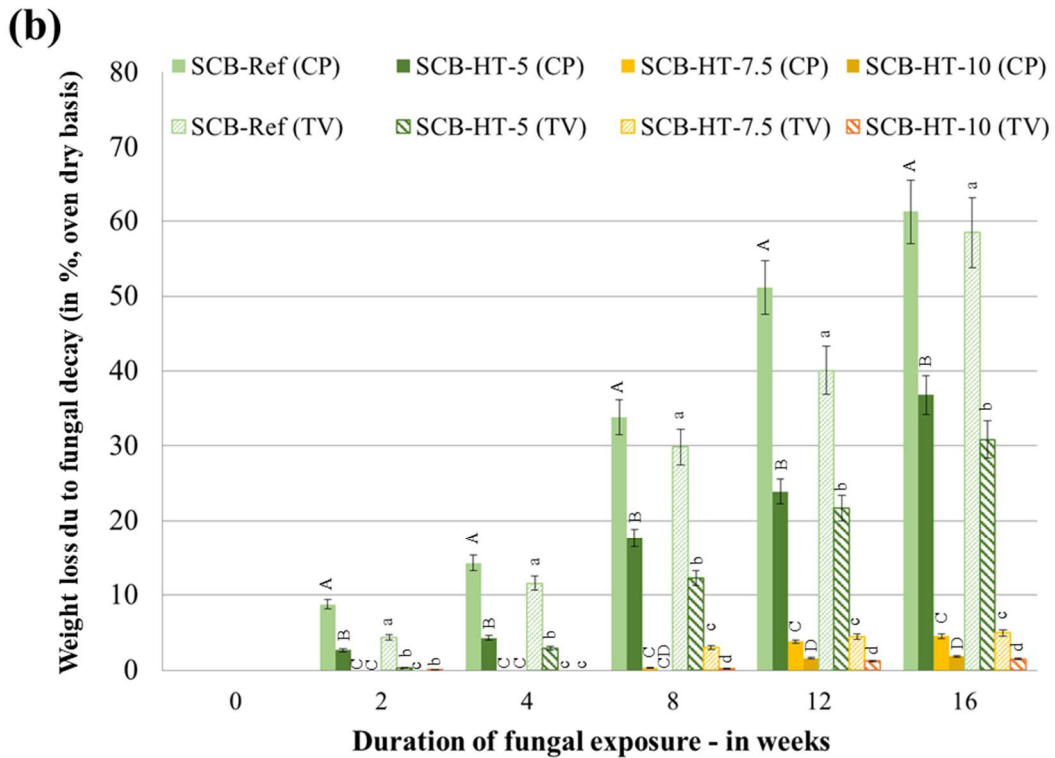


Letters: bar followed by the same letter are not significantly different. Here, the letters indicate the influence of torrefaction duration on the moisture content of SCB samples for each fungal exposure duration.

Fig. 6: Final moisture content (%) after *Coniophora puteana* (CP) and *Trametes versicolor* (TV) exposure of pine and beech control samples (a) and native and torrefied sugarcane bagasse (SCB) (b), according to the fungal exposure duration.



Letters: bar followed by the same letter are not significantly different. Here, the letters indicate the influence of fungal exposure duration on the weight loss of control samples.



Letters: bar followed by the same letter are not significantly different. Here, the letters indicate the influence of torrefaction duration on the weight loss of SCB samples for each fungal exposure duration.

Fig. 7 : Weight losses (WL%) due to *Coniophora puteana* (CP) and *Trametes versicolor* (TV) degradation of pine and beech control samples (a) and raw and torrefied sugarcane bagasse (SCB) (b), according to the fungal exposure duration.

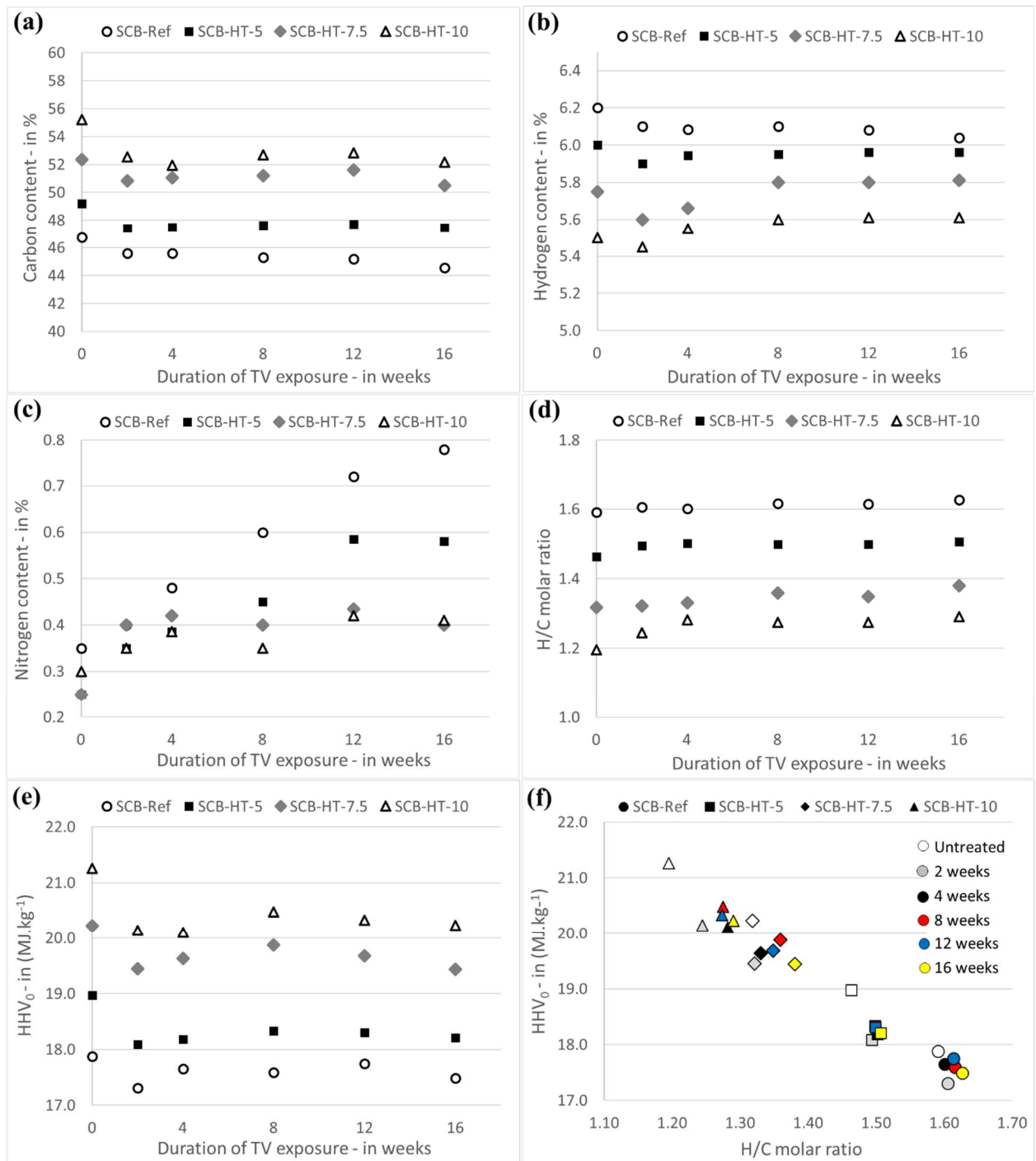


Fig. 8 : (a) Carbon, (b) Hydrogen and (c) Nitrogen contents (%), (d) H/C molar ratio and (e) HHV₀ (MJ.kg⁻¹) of raw and torrefied sugarcane bagasse (SCB), according to *Trametes versicolor* exposure duration; (f) Correlation between HHV₀ (MJ.kg⁻¹) and atomic H/C ratio of the different sugarcane bagasse (SCB) samples.

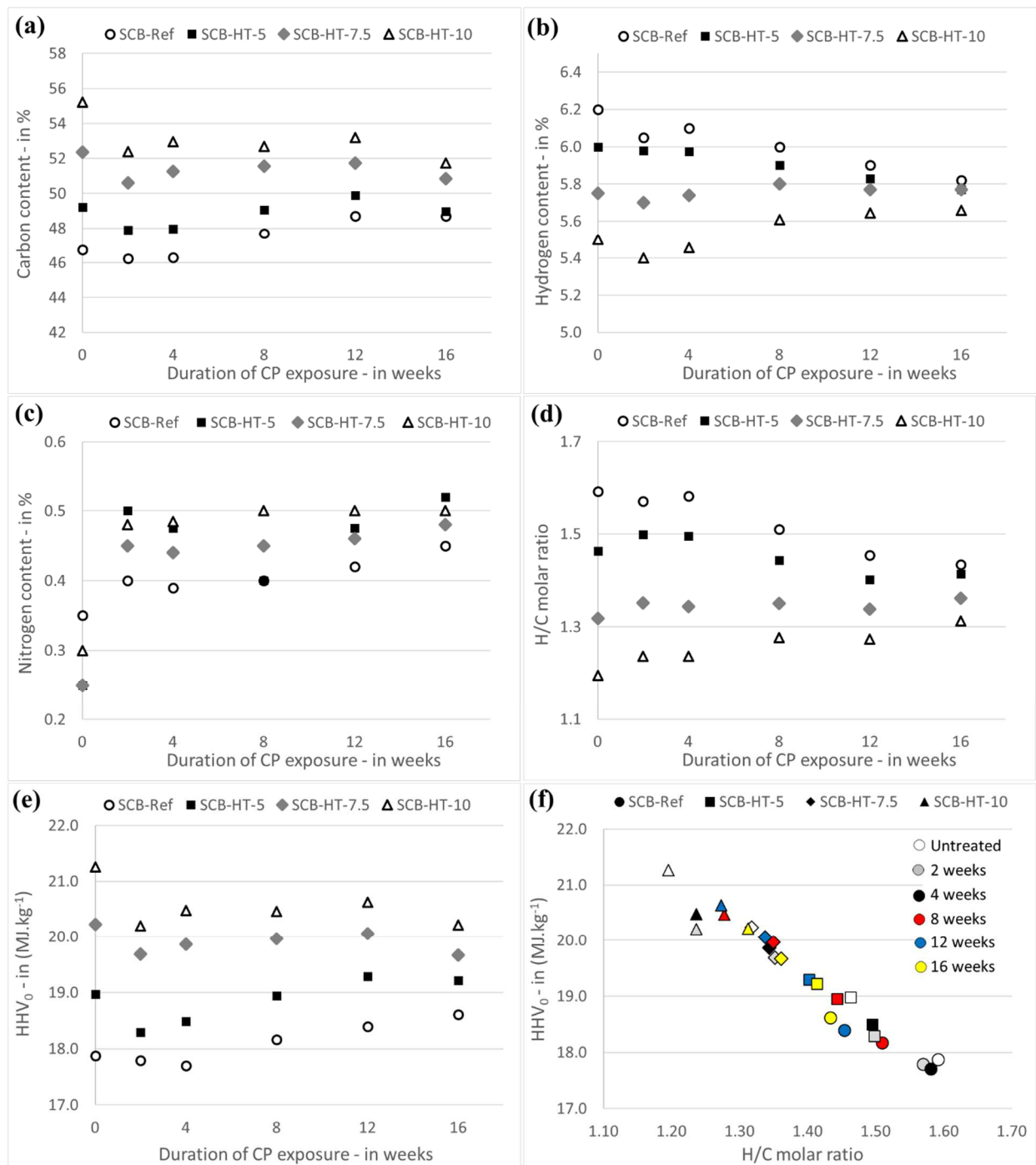


Fig. 9: (a) Carbon, (b) Hydrogen and (c) Nitrogen contents (%), (d) H/C molar ratio and (e) HHV₀ (MJ.kg⁻¹) of raw and torrefied sugarcane bagasse (SCB), according to *Coniophora puteana* exposure duration; (f) Correlation between HHV₀ (MJ.kg⁻¹) and atomic H/C ratio of the different sugarcane bagasse (SCB) samples.

Tab. 1: Elemental Compositions and High Heating Values (HHV), before and after leaching of raw and torrefied SCB samples. For each group, the means with the same letter were not significantly different at 5% ($\alpha= 0.05$)

Reference	Before Leaching process*					After Leaching process*				
	C (%)	H (%)	H/C	N (%)	HHV ₀ ** (MJ.kg ⁻¹)	C (%)	H (%)	H/C	N (%)	HHV ₀ ** (MJ.kg ⁻¹)
SCB-Ref	47.05 (A) (a)	6.00 (A) (a)	1.53 (A) (a)	0.30 (A) (a)	18.479 (A) (a)	46.75 (A) (b)	6.20 (A) (b)	1.59 (A) (a)	0.35 (A) (b)	17.874 (A) (a)
SCB-HT-5	49.60 (B) (a)	5.80 (B) (a)	1.40 (B) (a)	0.35 (B) (a)	19.241 (AB) (a)	49.20 (B) (b)	6.00 (AB) (b)	1.46 (AB) (a)	0.25 (C) (b)	18.975 (AB) (a)
SCB-HT-7.5	53.15 (C) (a)	5.60 (C) (a)	1.26 (C) (a)	0.40 (C) (a)	20.732 (BC) (a)	52.35 (C) (b)	5.75 (ABC) (b)	1.32 (BC) (a)	0.25 (C) (b)	20.228 (BC) (a)
SCB-HT-10	55.10 (D) (a)	5.40 (D) (a)	1.18 (D) (a)	0.40 (C) (a)	21.491 (CD) (a)	55.20 (D) (a)	5.50 (BCD) (b)	1.20 (CD) (a)	0.30 (B) (b)	21.260 (CD) (a)

* Each analysis has been duplicate and all the results are given with an accuracy of ± 0.20 %.

** Each analysis has been duplicate and all the results are given with an accuracy of ± 5.00 %.

Letters: bar followed by the same letter are not significantly different.

Here, the letters from “A” to “D” indicate the influence of the influence torrefaction duration on the elemental composition and HHV values (comparing rows). The letters from “a” to “b” indicate the influence of the water leaching process on the elemental composition and HHV values (comparing columns).

Emulation of field storage conditions for assessment of energy properties of torrefied sugarcane bagasses

GRAPHICAL ABSTRACT

