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AGING OF HEMP SHIV USED FOR CONCRETE

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Abstract

Functional properties of vegetal concretes, such as hygrothermal and acoustical properties, are brought by the microstructure of the vegetal particles. However, data are necessary to develop biobased insulation materials by providing results on their long-term behaviour, and only few studies focus on this topic. A first step is to characterize the behaviour of vegetal aggregates in bulk.

In this study, hemp shiv is stored for two years in three different conditions: a static environment used as reference, accelerated aging conditions based on humidification and drying cycles, and an exterior aging. The potential evolution of functional properties of particles is monitored with time and depending on the aging conditions. These evolutions are understood thanks to a study at a microscopic scale, where the microstructure of the particles is investigated.

This study shows that no variation is observed for the reference conditions, whereas four phenomena linked to aging can be identified for accelerated and exterior aging conditions: a mass loss, volume variations of the particles and an opening of the aggregates porosity for

31 both storing conditions. The last one, only observed for exterior aging, is a modification of the
32 pore size distributions.

33 **Keywords**

34 Vegetal aggregate; hemp shiv; hygrothermal and acoustical properties; microstructure;
35 microorganisms.

36 **1 INTRODUCTION**

37 The advent of biobased materials has come about in response to a desire to use products
38 with low environmental impact, made from renewable raw materials. In the building sector,
39 biobased construction materials are in use, such as wood for structural frameworks or
40 biobased insulation materials. Biobased insulators, and notably plant-based concretes, are
41 being used increasingly frequently due to their thermal, hygrothermal and acoustic
42 performances [1]. These functional properties are the result of the vegetal aggregates
43 employed in the formulation [2], whilst mechanical strength is provided by the presence of
44 frame. Numerous studies have been carried out on defining and understanding the properties
45 of the material, or optimising the formulations used. A number of recent studies have looked
46 at the durability and long-term behaviour of these insulators [3], [4], [5], [6], [7], but the
47 accelerated aging processes are much too short or overly severe in comparison to real-world
48 conditions of use, such as the application of freeze-thaw cycles [3]. However, lack of
49 knowledge about the evolution of the properties of plant-based concretes over time is a
50 barrier to the development of these materials. Indeed, the lack of guarantee as to the
51 durability of the desired properties over a given period of time does not inspire confidence in
52 users, architects or insurance companies.

53 It is for these reasons that a study is conducted on the durability of hemp-based concretes,
54 with the aim of identifying the mechanisms which cause the functional properties of the
55 material to change. However, to date, there has been no study done of the change over time
56 observed in the plant aggregates on their own, although it is these particles which are
57 responsible for a large proportion of the properties for which the material is used. In order to
58 understand the mechanisms of aging of plant-based concretes, it seems essential to
59 understand the behaviour of the particles in bulk over time. With a similar chemical
60 composition, made up of cellulose, hemicellulose and lignins for the most part, it is possible to
61 draw inspiration from the numerous studies examining the durability of wood.

62 Durability studies on wood have produced standards which guarantee quality and users'
63 expectations over a given period of time, in set usage conditions and with an amount of
64 maintenance [8]. Depending on the application, specific wood species are chosen according to
65 the applications and the conditions of use. Most of the published work concerns solid wood
66 [9] [10] [11] [12] [13] [14] [15] [16], but some of them are dedicated to wood pellets [17]
67 [18]. Aging conditions correspond to outdoors environments [9] [10] [17] [14] [15] [16] or

68 are accelerated in laboratory [11] [18] [12] [13]. Various aging factors are applied on wood in
69 these studies, such as immersion in water for periods from 10 days at 60 and 90°C [11] to 8
70 and 2000 years in natural conditions [9] [10], variations of relative humidity during 20 days
71 [12] to one year [18] [17], exposure to UV aging until four weeks [13] or natural aging without
72 protection during several years [14] [17]. Therefore, depending on the study in question, the
73 aging conditions are very different in terms of environment or exposure duration. Whatever
74 the protocols used, the functional properties are monitored over time, namely mostly
75 mechanical and/or hygroscopic properties [17], [18] [19] [15] [16], but also acoustic
76 properties [20]. Overall, aging leads to deterioration in the mechanical properties and to an
77 increase in water sorption capacity. Other parameters which characterise the microstructure
78 of the material are also studied, such as porosity, swelling, or variations in weight of the
79 materials. The variations of functional and microstructural properties are often explained by a
80 change in the chemical compositions of the wood, usually due to the degradation of the lignin
81 or hemicellulose [21]. These chemical degradations may result from photo-oxidation or
82 thermo-oxidation reactions, but above all, from the action of micro-organisms which can grow
83 on lignocellulosic materials in high humidity conditions. Indeed, the micro-organisms use the
84 cellulosic materials as a nutrient [22], and they may, selectively or otherwise, attack pectin,
85 lignins, hemicellulose or cellulose. These micro-organisms are present in the air and in water,
86 but also in the plants as they grow. Indeed, in order to separate the fibres from the stalks
87 more easily, the material may be retted for a time, enabling the micro-organisms to consume
88 the pectin which binds the fibres together. Therefore, such retting impacts upon the
89 properties of the materials [23]. There are various tests of resistance to the growth of micro-
90 organisms, placing the plant materials inoculated with moulds in conditions of high
91 hygrometry, as for example 26°C and 85 and 95% RH [24], or 20°C and 70% RH [25]. The
92 result of an attack by micro-organisms is a loss of mass of the materials, which can lead to
93 structural alterations which modify the functional properties, such as mechanical properties.

94 In view of these results, three environments are chosen in this study to assess the durability of
95 hemp aggregates used for vegetal concretes. The first offers stable conditions and is used as a
96 reference point. The second one is an accelerated aging imposing variations in humidity. The
97 relative humidity varies from 40% RH to 98% at a fixed temperature of 30°C. Indeed, in the
98 normal conditions of use of hemp concrete, unlike wood, hemp shiv should never be exposed
99 to direct UV light or to liquid water. This temperature favours the development of micro-
100 organisms, which has been identified as a significant degradation factor. Finally, a harsher,
101 outdoor aging process is used.

102 The objective of this article is to identify the mechanism by which hemp shiv ages in the
103 presence of moisture. The potential evolution of the functional properties of the material
104 (acoustic and hydric properties) is characterised and linked to the microstructural changes
105 (density, porosity, etc.) occurring in the different environments. The discussion focuses on
106 understanding the mechanisms of aging of hemp shiv.

107 **2 MATERIALS AND METHODS**

108 **2.1 Materials**

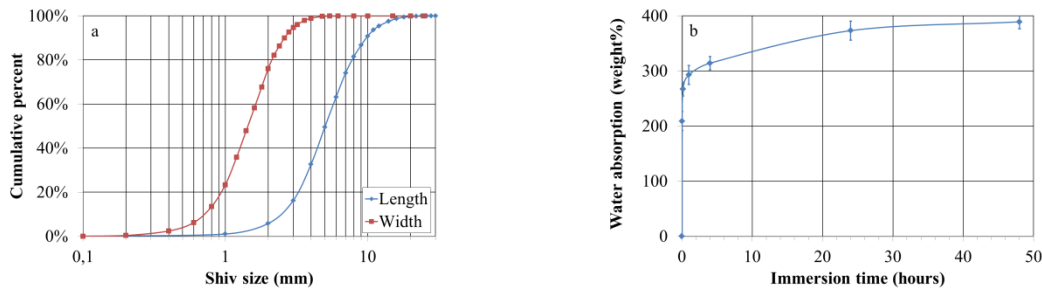
109 A commercial hemp shiv, sold for an insulation application, is selected for this study. To
 110 analyse homogenous samples of particles, batches of 100 g are sampled from an entire 200 L
 111 bag of shiv as recommended by RILEM TC 236-BBM.

112 The initial rate of water absorption, the absorption coefficient, the granulometry (particle size
 113 distribution, PSD) and the quantity of fibres and dust are measured in accordance with the
 114 RILEM recommendations [26].

115 The quantity of soluble compounds is determined by the loss of mass of hemp shiv powder
 116 after 1 hour of immersion at 100°C. 4g of hemp powder are analysed, with a water/hemp ratio
 117 of 100. To obtain the powder, a batch of shiv is crushed and sieved at 500 µm. The results are
 118 presented in Table 1 and Figure 1.

	Initial rate of water absorption IRA (%)	Absorption coefficient K_1 (%/log(t))	Fibre and dust content (%)	Hot water soluble compounds (%)
Hemp shiv	IRA = 197 ± 6	$K_1 = 50 \pm 3$	Fibre: 3.3 ± 0.6 Dust: 0.5 ± 0.2	11.8 ± 0.7

119 *Table 1. Physical properties of hemp shiv*



120 *Figure 1. a) PSD of hemp shiv obtained by image analysis; b) Water absorption of hemp shiv*

121 **2.2 Aging protocols**

122 The hemp particles are submitted to three different environments in this study.

123 The first is used as reference and correspond to static conditions, at 20°C and 50% RH. This is
 124 referred as A_{Ref} , and serves as a control for the potential evolution of the properties of hemp
 125 shiv even when no particular stress is applied.

126 The second is an accelerated aging process. The shiv is placed in a fine-meshed metal basket
 127 in an environmental chamber. At a constant temperature of 30°C, humidification and drying
 128 cycles are applied, with 5 days at 98% RH and 2 days at 40% RH. The choice of these

129 parameters is based on a previous study [6]. The transitions of relative humidity take place
130 over the course of an hour. This aging protocol is denoted A_{HD} .

131 To observe more severe aging of the hemp shiv, a last batch is placed in the same kind of
132 basket, outside (Figure 2), and subjected to various weather conditions, including sunlight,
133 rain, snow and temperature variations. This last aging condition is denoted A_{Ext} .



134
135 *Figure 2. Sample of hemp shiv aged during two years in A_{Ext} conditions*

136 For A_{Ref} and A_{HD} , which are controlled conditions, different measurements are taken from 0
137 (A_0) to two years of aging. In the case of A_{Ext} , where nothing is controlled, fewer
138 measurements are made during the two years.

139 **2.3 Functional properties**

140 The acoustic properties are measured using a Kundt tube (AcoustiTube AFD). Two types
141 of properties are obtained: the acoustic absorption coefficient α and the transmission loss TL.
142 The acoustic absorption coefficient is the proportion of sound energy absorbed by a material.
143 It is expressed for values ranging between 1 (total absorption, no reflection) and 0 (no
144 absorption, total reflection). The transmission loss (expressed in decibels) represents the
145 sound insulation provided by a material. 3 measurements are taken for each batch of bulk
146 shiv over a frequency range [250-2000Hz]. At A_0 , the batches contain 40.7 g of hemp shiv, to
147 obtain a controlled density of $130 \text{ kg}\cdot\text{m}^{-3}$ in the volume of the sample chamber (10 cm in
148 diameter and 4 cm in height). The different batches are aged and reused for the acoustic
149 measurements.

150 The hydric behaviour is determined on the basis of the water vapour sorption isotherms
151 using a DVS machine (Dynamic Vapour Sorption – SMS DVS Advantage). A few granules
152 representing a few tens of mg are suspended in a microbalance within a sealed
153 thermostatically controlled chamber at 25°C . The schedule for the DVS is set to start at 0% RH
154 up to 95%. A given relative humidity is applied until the weight change of the sample is less
155 than $0.0005\% \cdot \text{min}^{-1}$ or during 12 hours if the weight is not stabilised. Sorption isotherms are
156 produced by plotting mass change against relative humidity (RH) and illustrate the water
157 vapour sorption capacity as a function of the relative humidity. The overall sorption
158 behaviour across the whole range of relative humidity values is simulated using the GAB
159 model, as described in an earlier study [2]. The specific surface of water vapour sorption S_m is
160 deduced with this model.

161 **2.4 Microstructure characterisation**

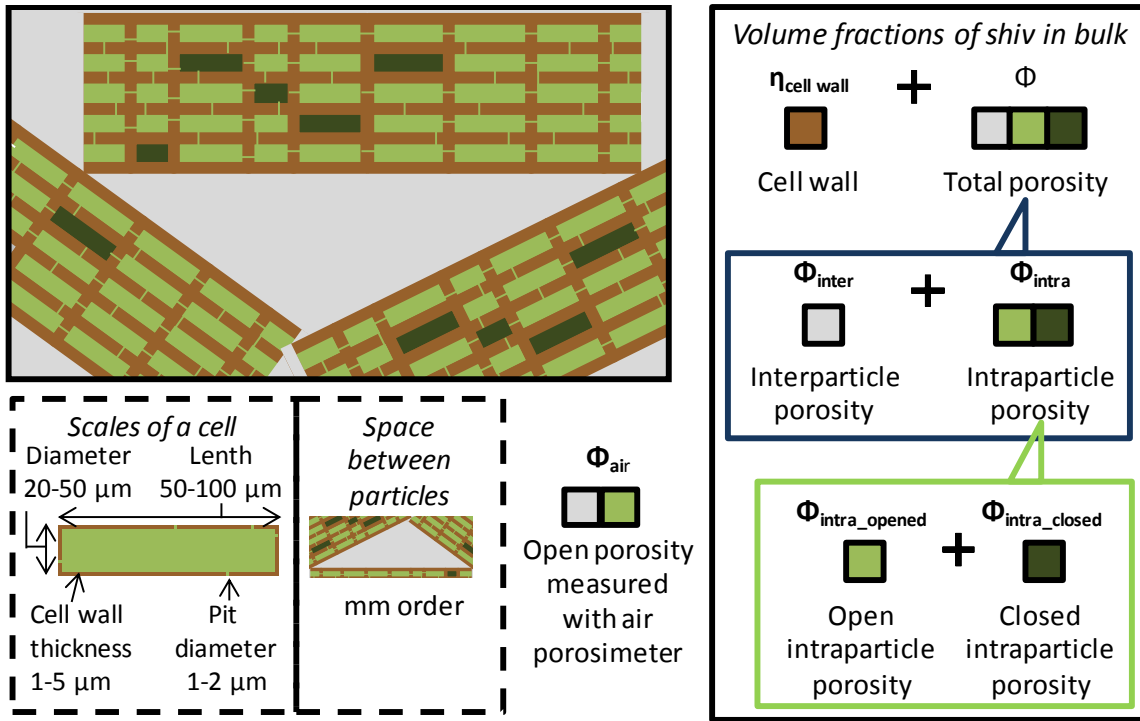
162 Microstructure of hemp shiv is characterised by densities measurements, acoustic
163 parameters and SEM observations.

164 For bulk shiv, three types of volume fractions can be identified [27], and are represented
165 in Figure 3:

- 166 - the volume fraction constituted by the vegetal cell walls of the hemp shiv, noted as
167 $\eta_{\text{cell wall}}$;
- 168 - the volume fraction of air between the particles, or interparticle porosity Φ_{inter} , which
169 is due to an imperfect arrangement of the hemp shiv. These pores are around a
170 millimetre in size;
- 171 - the volume fraction of air within the particles, or intraparticle porosity Φ_{intra} , with
172 pore diameters generally ranging from 3-10 nm for mesopores, to 0.1-1 μm and 20-80
173 μm for macropores [28].

174 Depending on the kind of porosity, it may be open and accessible for measurement, open
175 but not accessible to measurement, or closed. With a pore size in the millimetre range, the
176 interparticle porosity, represented in grey in Figure 3, is always open and accessible. On the
177 other hand, the intraparticle porosity may contain closed pores and pores not accessible to
178 the measurement. For simplicity's sake, no distinction is drawn between intraparticle porosity
179 which is closed or open but not accessible; both are denoted by $\Phi_{\text{intra_closed}}$. This category is
180 randomly represented in dark green in the diagram. The open and accessible intraparticle
181 porosity (in light green) is called $\Phi_{\text{intra_opened}}$.

182



183

184 *Figure 3. Representation of the different types of volume fractions for shiv in bulk, porosity open*
 185 *to the air and pore sizes*

186 By combining different techniques, it is possible to quantify the different volume fractions
 187 corresponding to the vegetal part or to each type of porosity. In all cases, these volume
 188 fractions are expressed as a function of a volume of bulk shiv.

189 The bulk density ρ of the hemp shiv is the ratio of the mass of a batch of bulk hemp shiv to
 190 the volume it occupies in unstressed conditions. The RILEM protocol [26] is applied to around
 191 100 g of particles to obtain this parameter.

192 The density of the plant cell wall $\rho_{\text{cell wall}}$ is an intrinsic value of the material, which
 193 depends on its chemical composition. In this case, the vegetal cell wall is expected not to
 194 contain any porosity. The volume corresponding to the vegetal cell walls of the material is
 195 measured by introducing a fluid into the pores in the material. Knowing the mass of the
 196 sample introduced, the density of the vegetal part can then be calculated. To measure this
 197 density, the hemp shiv is ground to a powder of grain size less than 500 μm in order to
 198 eliminate the porosities of the vegetal cell walls. The volume of the powder is then measured
 199 using a helium pycnometer (Micromeritics AccuPyc II 1340, volume 10 cm^3 , pressure 19.5 psi,
 200 stability criterion 0.005 psi/min).

201 To come closer to real conditions of use of the materials, air porosity has also been
 202 determined on bulk hemp shiv as described by Gourlay [29]. In this case, as O_2 and N_2
 203 molecules are larger than the helium atom, a portion of the pores is closed or inaccessible for

204 measurement. Accordingly, the evolution of the skeleton density ρ_{sk} during the material aging
205 is monitored with this technique. A distinction must be drawn between the density of the cell
206 wall $\rho_{cell\ wall}$, which does not contain any porosity, and the density of the skeleton ρ_{sk} , where
207 closed or inaccessible pores are included into the volume of the vegetal skeleton.

208 The total porosity Φ , which is the combination of Φ_{inter} and Φ_{intra} , can be evaluated on the
209 basis of the density of the plant cell wall and of the bulk density, using the following equation:

$$\Phi = 1 - \frac{\rho}{\rho_{cell\ wall}}$$

210 The volume fraction of solid material $\eta_{vegetal}$ is the remaining volume fraction:

$$\eta_{vegetal} = 1 - \Phi$$

211 The porosity accessible to air Φ_{air} is calculated using the value of the bulk density ρ and
212 the skeleton density ρ_{sk} .

$$\Phi_{air} = 1 - \frac{\rho}{\rho_{sk}}$$

213 Based on acoustic measurements and using the model developed by Zwikker and Kosten
214 [30], it is possible to calculate the acoustic porosity, which represents the porosity causing the
215 dissipation of a soundwave in the material. As the acoustic properties mainly depend on the
216 interparticle porosity [27], it is possible to estimate this porosity using the value of the
217 acoustic porosity Φ_{acou} by assuming that it corresponds to Φ_{inter} .

218 When the interparticle porosity is known, it is possible to deduce the open intraparticle
219 porosity Φ_{intra_opened} using the following equation:

$$\Phi_{intra_opened} = \Phi_{air} - \Phi_{inter} \approx \Phi_{air} - \Phi_{acou}$$

220 Finally, Φ_{intra_closed} is calculated on the basis of the difference between the total porosity
221 and the open porosity:

$$\Phi_{intra_closed} = \Phi - \Phi_{air}$$

222 Other parameters describing the microstructure of the material are obtained from the
223 acoustical measurements [27]:

- 224 - σ , the airflow resistance of the material;
- 225 - α_{∞} , the tortuosity describing the sinuosity of the porous network;
- 226 - Λ , the characteristic viscous length which corresponds to an estimation of the size of
- 227 interconnection between pores.

228 The microstructure is also observed with a scanning electron microscope (SEM) FEI
229 Quanta 400 in secondary electron mode on both longitudinal and transverse slices of hemp
230 shiv.

231 **2.5 Chemical analysis**

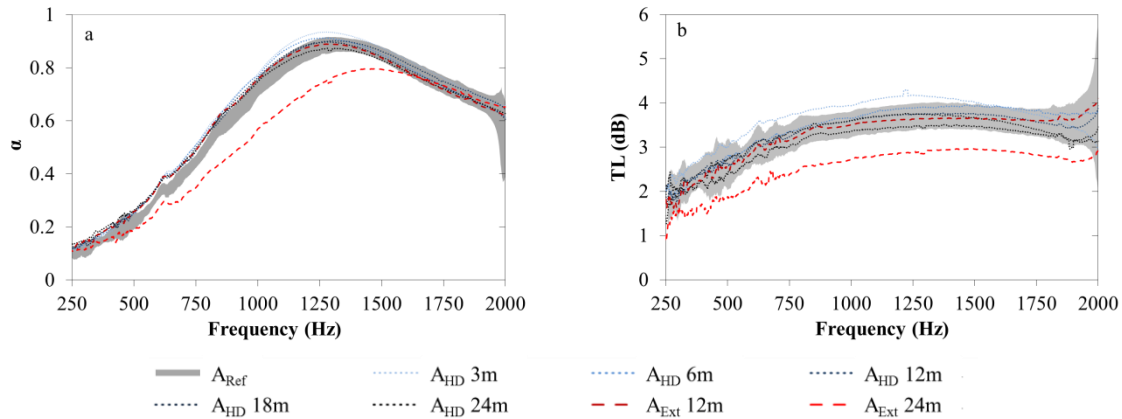
232 The chemical composition of hemp shiv is determined by the Van Soest method, following the
233 standard NF V18-122 [31]. Dry hemp shiv powder is added in a neutral detergent solution in
234 order to extract all kind of molecules excepting cellulose, hemicellulose and lignin. A second
235 immersion in an acid detergent solution extracts hemicellulose. After sulphuric acid
236 treatment, cellulose is hydrolysed and extracted. By mass differences, proportions of cellulose,
237 hemicellulose, lignin and the other compounds are deduced per mass of dry material.

238 **3 RESULTS**

239 **3.1 Functional properties of shiv**

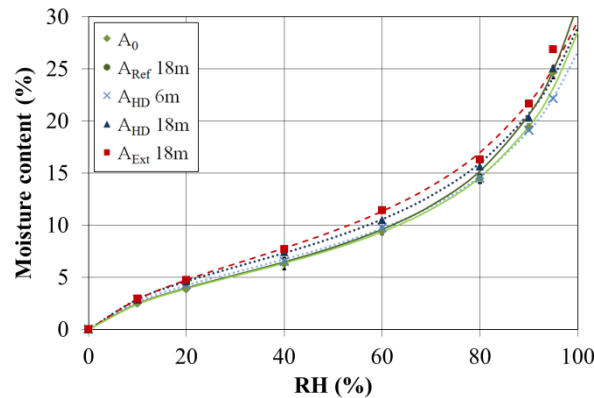
240 First, the results of the monitoring of the functional properties over time and over the course
241 of the aging processes are presented.

242 With regard to the acoustic properties of hemp shiv, the acoustic absorption coefficient
243 and transmission loss are respectively presented, in Figure 4-a and Figure 4-b for the initial
244 batch A_0 and the batches aged with A_{Ref} , A_{HD} and A_{Ext} conditions. For A_{Ref} , the acoustic
245 behaviour does not vary between A_0 and 24 months. The results for that environment are
246 represented in the form of a grey curve beam for α and TL, corresponding to the extant results
247 over two years. The α_{max} of 0.91 is obtained at a frequency of 1350 Hz. For the same frequency
248 the value of TL is around 3 dB. With regard to A_{HD} aging process, a shift toward low
249 frequencies is visible at 1250 Hz for the acoustic absorption curve from 3 months of aging.
250 This value then remains stable for the rest of the aging process. The value of α_{max} also evolves
251 over time, showing an increase at 3 months, from 0.91 to 0.93, and then a gradual decrease as
252 aging continues, to $\alpha_{max} = 0.87$. Finally, with A_{Ext} , the same shift of α_{max} (0.89) is obtained
253 around a frequency of 1250 Hz to 1 year. Conversely, after 2 years of aging in these
254 conditions, a significant variation of the acoustic behaviour is observed, with a sharper drop
255 in the value of α_{max} associated with a shift of the curve towards higher frequencies. The value
256 of 0.80 is obtained at a frequency of 1475 Hz. Thus, a reduction in transmission loss is also
257 measured.



258 *Figure 4. a) Acoustic absorption coefficient and b) transmission loss over a frequency range from*
 259 *250 to 2000 Hz for hemp shiv over time and as a function of the aging processes*

260 Another important functional property of hemp shiv is its water vapour sorption capacity.
 261 The water vapour sorption isotherms are obtained for A_0 and 18 months with A_{Ref} , A_{HD} and
 262 A_{Ext} aging processes (Figure 5). There is no discernible difference for aging A_{Ref} with A_0
 263 between 0 and 80% RH. For A_{HD} , the water sorption increases over a range of relative
 264 humidity between 20% and 80% RH. After 18 months of aging A_{Ext} , the phenomenon is even
 265 more marked. Using the GAB model, it is possible to calculate the specific surface area for
 266 water vapour sorption (Table 2). An increase in specific surface area is observed over the
 267 course of aging A_{HD} , and also for aging A_{Ext} , though it is more marked in the latter case.



268 *Figure 5. Sorption isotherms for hemp shiv A_0 , aged for 18 months by A_{Ref} , A_{HD} and A_{Ext} , with*
 269 *curves generated by the GAB model.*
 270

	A_0	A_{Ref} 18m	A_{HD} 18m	A_{Ext} 18m
ω_m (%)	5.4	5.5	6.3	7.1
C	7.8	7.5	8.7	7.2
K	0.82	0.83	0.79	0.77
Specific surface area (cm ² /g)	182 ± 3	186 ± 3	210 ± 5	237 ± 8

271 *Table 2. Parameters of the GAB model corresponding to the sorption isotherms of A_0 , and 18*
272 *months of A_{Ref} , A_{HD} and A_{Ext}*

273 The changes in the shape of the sorption isotherm curves, the increase in specific surface
274 area and the modifications of the acoustic behaviour with A_{HD} and A_{Ext} aging processes may
275 stem from a modification to the microstructure of the material. Hence, it is necessary to study
276 the potential evolutions of the microstructural and chemical properties of shiv on this scale.

277 **3.2 Microstructure of the hemp shiv**

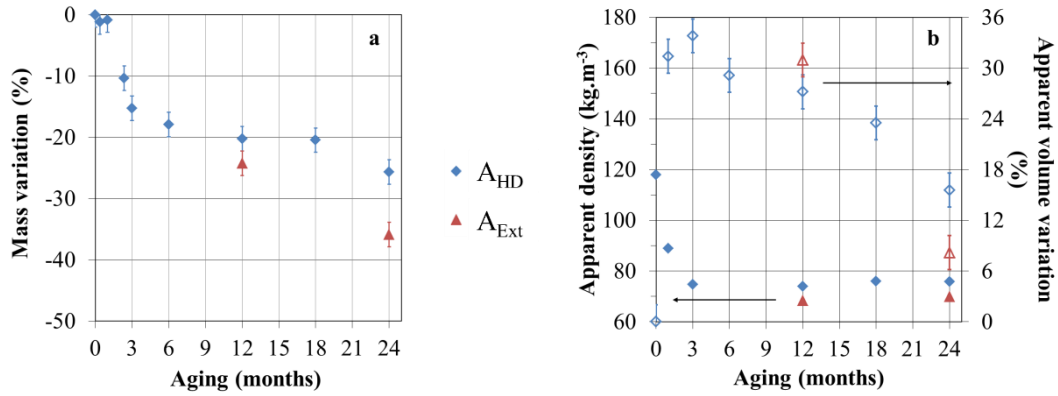
278 *a. Mass and bulk density*

279 The variation in mass and bulk density and volume of the bulk shiv are respectively
280 presented in Figure 6-a and b as a function of time for the A_{HD} and A_{Ext} processes. The results
281 of A_{Ref} are not represented, because no significant variation is observed.

282 The batches of shiv respectively lose 25% and 35% of their initial mass after 24 months of
283 storage in environments A_{HD} and A_{Ext} . For aging process A_{HD} , the majority of the mass loss
284 occurs between one and three months after the beginning of this aging

285 Concerning the bulk density, it decreases by almost 40% after two years of aging, for both
286 A_{HD} and A_{Ext} . Considering values obtained for A_{HD} , the drop is observed during the first three
287 months.

288 From the values of bulk density and mass variations, it is possible to calculate the
289 apparent volume variations of the particles. During the first month of aging A_{HD} , the density
290 falls from 118 kg.m⁻³ to 90 kg.m⁻³ without a significant loss of mass. This can be explained by
291 the swelling of the particles with an increase in apparent volume of around 30%. Between one
292 and three months of A_{HD} , the particles mass decreases, as well as the bulk density, leading to a
293 constant apparent volume. Then, until 24 months of A_{HD} aging, the apparent volume slightly
294 decreases, resulting from a slow decrease of the particles mass whereas their density is
295 constant. However, the volume of hemp shiv after two years of aging remains 16% higher
296 than the initial one. For A_{Ext} , the apparent volume at 12 months rises by 30%, and then drops
297 to a value 8% higher than the initial one.



298 *Figure 6. Evolution of a batch of hemp shiv during A_{HD} and A_{Ext} aging conditions: a) mass of*
 299 *shiv; b) Bulk density (full symbols) and apparent volume variation (empty symbols)*

300 *b. Density of the vegetal cell walls*

301 In order to quantify the vegetal cell wall density $\rho_{cell\ wall}$, helium pycnometer
 302 measurements are performed on powdered hemp shiv (Table 3). In this case, this powder is
 303 not expected to contain porosities anymore. The cell wall density value obtained is 1480 kg.m^{-3} .
 304 This value is not far off the density of the cell wall given in the existing literature for hemp
 305 shiv (1450 kg.m^{-3} [32]), wood (between 1497 and 1529 kg.m^{-3} [33]) or flax and hemp fibres
 306 (between 1480 and 1540 kg.m^{-3} [34] [35]). After two years of A_{HD} and A_{Ext} aging, this density
 307 increases, mainly for A_{HD} conditions.

	Powdered hemp shiv A_0	Powdered hemp shiv 2 years A_{HD}	Powdered hemp shiv 2 years A_{Ext}
Cell wall density (kg.m⁻³)	1480 ± 4	1531 ± 5	1498 ± 5

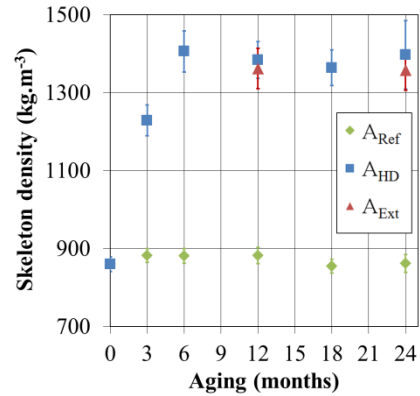
308 *Table 3. Skeleton density of hemp shiv determined by helium pycnometer measurements in*
 309 *powder at A_0 and after 24 months in A_{HD} and A_{Ext}*

310 *c. Skeleton density*

311 The skeleton density is another parameter that can be used to characterise the
 312 microstructure of bulk hemp shiv. The values of skeleton density obtained on particles with
 313 an air porosimeter over time, and over the course of the different aging processes, are shown
 314 in Figure 7.

315 For shiv exposed to the reference environment, a mean value over the 2 years of 872 ± 20
 316 kg/m^3 is measured. However, during six months of A_{HD} aging, the skeleton density increases
 317 significantly until a constant value of $1385 \pm 46\text{ kg.m}^{-3}$. Values of ρ_{sk} of the same order of
 318 magnitude are obtained for A_{Ext} . Comparing these values to $\rho_{cell\ wall}$, the conclusion is that a
 319 portion of the porosity of the shiv is initially closed, and is therefore included in the skeleton
 320 volume of the material when it is measured, leading to a low skeleton density value. As a

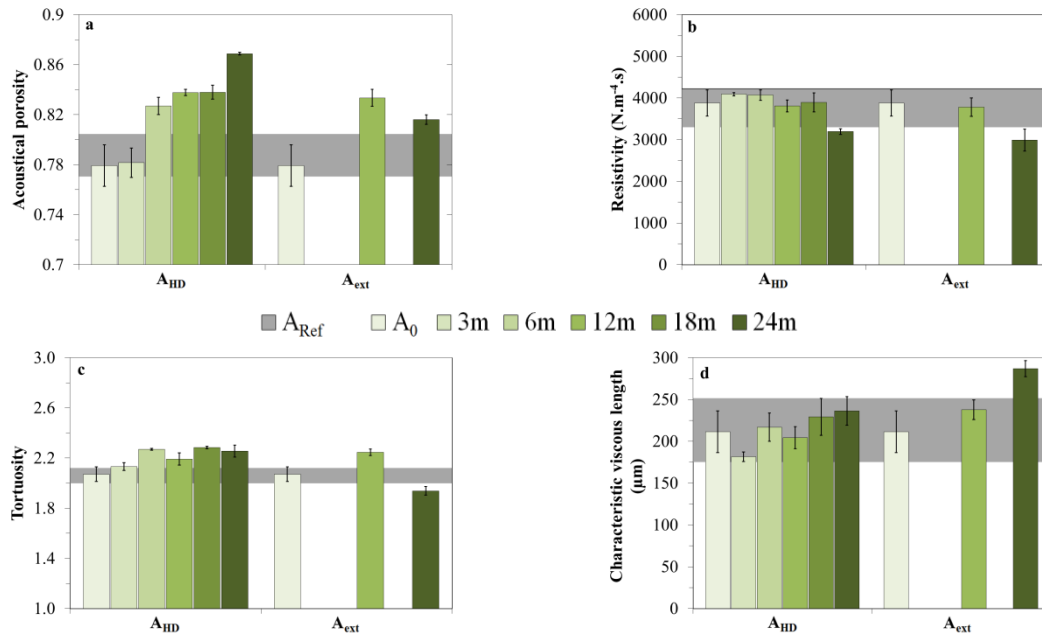
321 consequence, with aging, the increase of the skeleton density can be explained by the fact that
 322 this porosity opens and becomes accessible to air.



323
 324 *Figure 7. Skeleton density of hemp shiv by air porosimetry for hemp shiv under A_{Ref} , A_{HD} and A_{Ext}*
 325 *aging conditions*

326 *d. Microstructural parameters obtained from acoustic analyses*

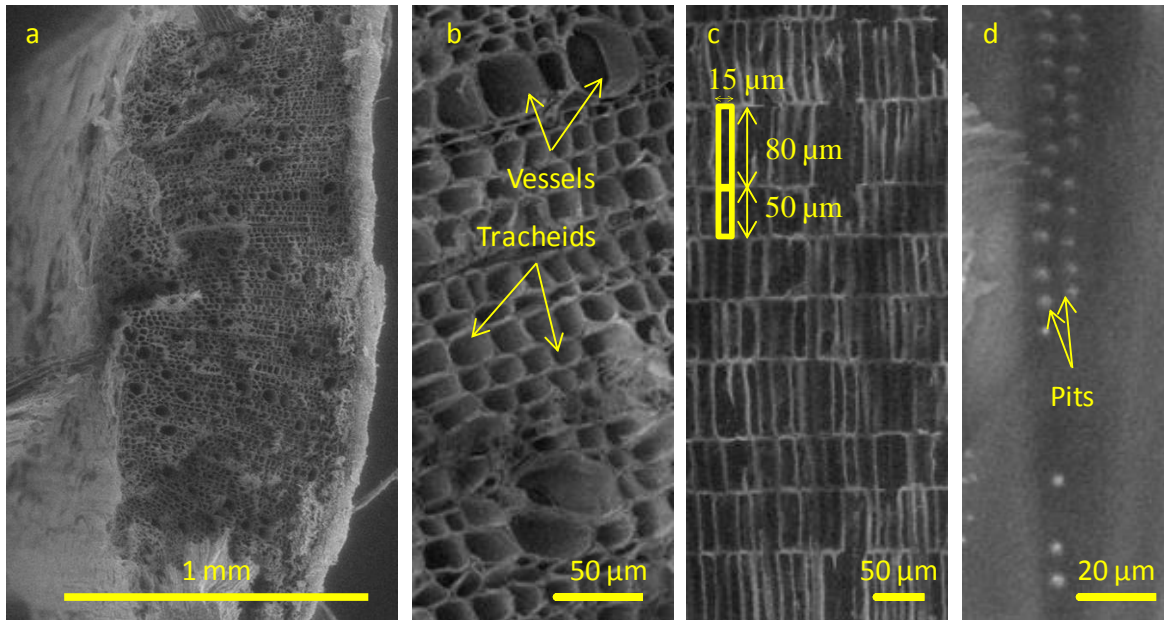
327 The microstructural parameters obtained from the acoustical measurements can also be
 328 used to describe the microstructure of hemp shiv (Figure 8). As with the curves for α and TL,
 329 the results for the reference environment are represented in the form of a beam of values in
 330 grey in the figures for the set of parameters. The acoustical porosity Φ_{acou} increases after 6
 331 months for A_{HD} , and reaches a value of 0.87 at 24 months. For A_{Ext} , a similar behaviour is
 332 observed at 12 months, while the acoustical porosity tends to decrease at 24 months. The
 333 results in terms of air resistivity σ show a slight decrease in the course of both aging
 334 processes. The tortuosity α_∞ increases progressively during aging A_{HD} , reaching a value of
 335 around 2.25. On the other hand, the behaviour of this parameter is different for aging A_{Ext} ,
 336 with a similar increase at 12 months, but a decrease in tortuosity is seen at 24 months. Finally,
 337 the characteristic viscous length Λ decreases at 3 months for A_{HD} , then increases again and re-
 338 joins the values of A_{Ref} . It increases after 24 months in A_{Ext} .



339 *Figure 8. Variation of the acoustical parameters during aging: a) Acoustical porosity Φ_{acou} ; b)*
 340 *tortuosity α_{∞} ; c) resistivity σ ; d) characteristic viscous length Λ for A_{Ref} , A_{HD} and A_{Ext}*

341 *e. Observation of hemp shiv during aging*

342 In parallel with the quantification of the microstructural parameters and the description
 343 of their evolution during the different aging conditions, it is also possible to observe these
 344 modifications by scanning electron microscopy. Firstly, Figure 9 illustrates the structure of
 345 shiv before aging. In Figure 9-a, a transverse slice of a particle is visible. With greater
 346 magnification (Figure 9-b), the tracheids can be better distinguished from the vessels, as the
 347 latter are larger in diameter ($\approx 50 \mu m$). The longitudinal slice in Figure 9-c shows the
 348 alignment of the cells. Finally, Figure 9-d of a vessel shows the pits, the valves between the
 349 cells between 1 and 2 μm in diameter. The observation of these particles during A_{Ref} aging
 350 conditions did not show any significant evolution of plant microstructure.



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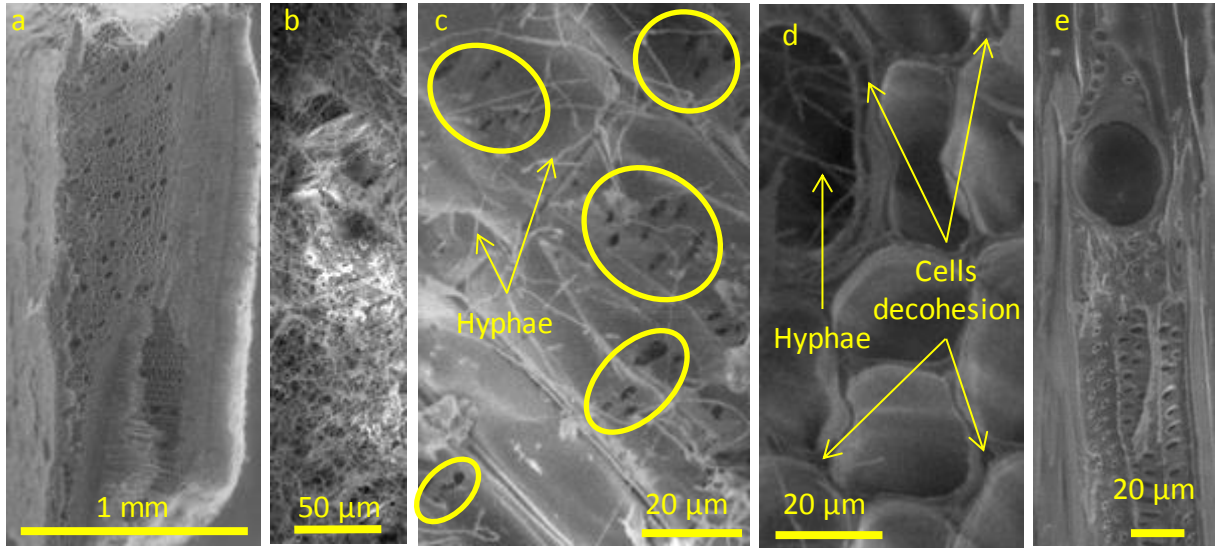
Figure 9. SEM pictures of hemp shiv before aging: a) transverse cross-section; b) transverse cross-section with tracheids and vessels; c) longitudinal cross-section; d) pits in longitudinal cross-section

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The images in Figure 10 are taken on hemp shiv after 15 months of A_{HD} aging. Even though the overall structure of the particles remains unchanged (Figure 10-a), hyphae are present in large numbers on the surface of the particles (Figure 10-b). On other zones of the surface (Figure 10-c), hyphae are present, but also of perforations in the cell wall, which are not as regular as the pits visible in Figure 9-d. The analysis of transverse cross-section (Figure 10-d) shows hyphae, which are thus present within the hemp shiv, and the debonding of the cells from one another. These observations were not made after 15 months of A_{Ref} aging conditions. The perforations and the decohesion of the cellules are also observed in studies regarding the attack of fungi on wood. According to these studies, the attack on the cell wall, leading to a reduction in its thickness, affects hemicellulose and cellulose [36] [37] [38]; the decohesion of cells is due to delignification of the plant matter [39] [40] [41]. Another phenomenon is visible in Figure 10-e: a pit seems to have ripped open, leaving a hole in the cell wall over 30 μm in diameter. That tearing of the alveolus may be due to the cycles of swelling and shrinkage caused by the cycles of humidification and drying.

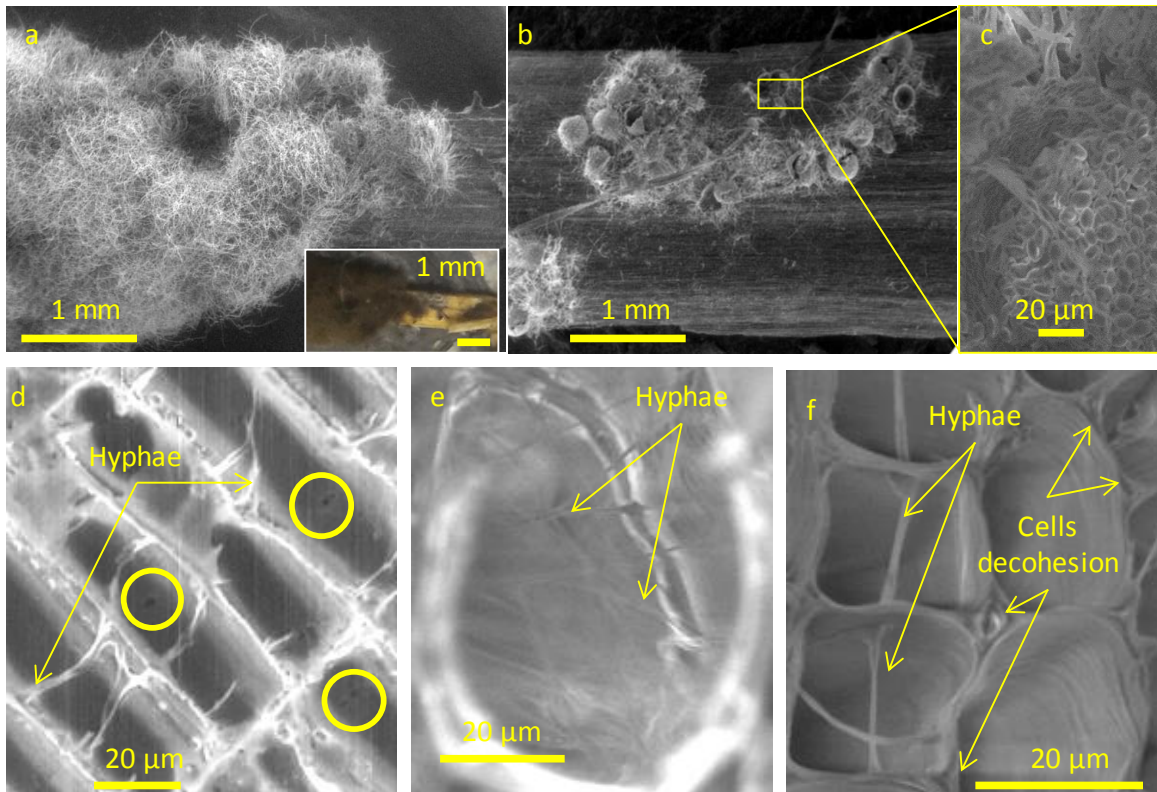
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Overall, the same conclusions can be drawn for hemp shiv 15 months aged by A_{Ext} , with the exception of the fact that micro-organisms are present in a larger quantity (Figure 11-a) and other species of micro-organisms are found (Figure 11-b and Figure 11-c). Perforations in the cell wall are also visible, as well as hyphae and a debonding of cells.



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Figure 10. SEM pictures of hemp shiv after 15 months in HD aging conditions: a) transversal cross-section; b) hyphae on the surface of a particle; c) hyphae and holes in the cell wall on the surface of a particle; d) hyphae and decohesion of cells in transversal cross-section; e) pits and torn pit in longitudinal cross-section



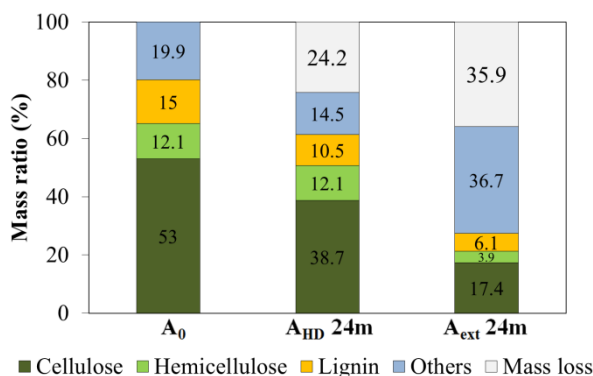
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Figure 11. SEM pictures of hemp shiv after 15 months of exterior aging conditions: a) fungi on a particle with picture; b) sporiferous saccules on the surface of a particle; c) spores and hyphae inside saccule; d) hyphae and holes in cell walls viewed in longitudinal cross-section; e) hyphae in a cell in transversal cross-section; f) hyphae and decohesion of cells in transversal cross-section

384 3.3 Chemical composition

385 The chemical composition of hemp shiv for A_0 , and after 24 months of A_{HD} and A_{Ext} agings
386 conditions are presented Figure 12. The evolution of the mass fraction of cellulose,
387 hemicelluloses and lignin of the vegetal cell walls is visible. Pectin, proteins, lipids, soluble
388 carbohydrates like low molecular mass saccharides or soluble phenolic compounds are
389 extracted in the first step of the Van Soest method [31]. They are labelled as "other" molecules
390 in Figure 12. For aged hemp shiv, mass fractions are presented on the base of the initial mass
391 before aging at A_0 . The mass loss measured as a function of time, plotted in Figure 6,a is also
392 taken into account in Figure 12.

393 Before aging, hemp shiv is mostly based on hollocellulose which corresponds to 65.1 % of
394 cellulose and hemicellulose. The mass ratio lignin/hollocellulose is equal to 0.23. The other
395 compounds represent 19.9% of the mass. This value, obtained after extraction with the
396 neutral detergent used in the Van Soest method is higher than the amount of soluble
397 compounds extracted in water (Table 1). After 24 months of A_{HD} aging, the amounts of
398 cellulose and lignin decrease, whereas the hemicellulose mass fraction is stable. The ratio
399 lignin/hollocellulose decreases to 0.20, lower than for A_0 . The quantity of other compounds is
400 also lower than for A_0 . Finally after two years of exterior aging, a large decrease of the
401 cellulose, hemicellulose and lignin amounts is observed. The total quantity of these three
402 hemp shiv components represents 27 % of the total mass of shiv, whereas they represent
403 more than 80% of the initial mass. Moreover, the amount of other molecules is higher than
404 before aging.



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406 *Figure 12. Chemical composition of hemp shiv (cellulose, hemicelluloses and lignin) at A_0 , and*
407 *after 24 months in A_{HD} and A_{Ext} aging conditions*

408 4 DISCUSSION

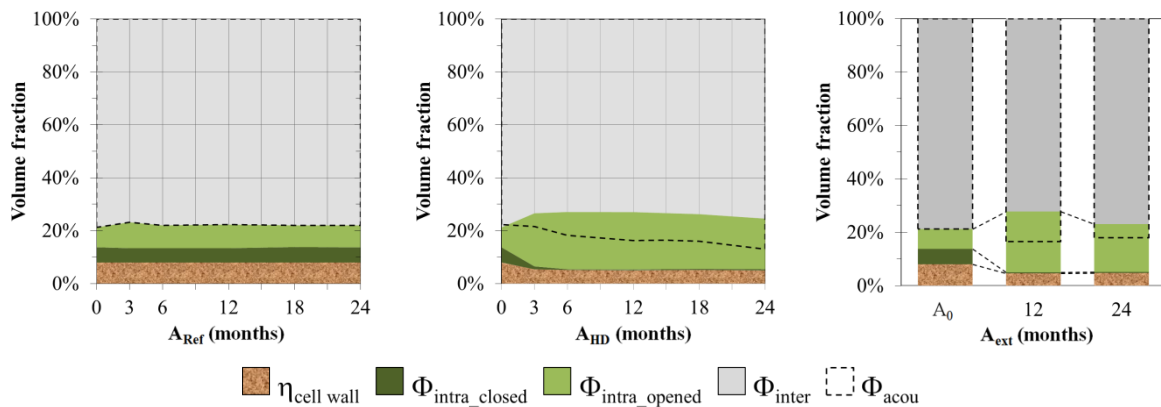
409 The results presented show some differences in the variations of the functional properties as
410 a function of the environment applied to hemp shiv. For the reference conditions A_{Ref} , no
411 significant variation is observed; the shiv retains all of its properties over time. As regards A_{HD}
412 aging, variations in the acoustic and hydric behaviour are visible. This aging induces a shift
413 toward low frequencies in the curve of α with variations of α_{max} over time, and an increase in

414 water vapour sorption capacity (Figure 4-a and Figure 5). With the A_{Ext} aging process, the
 415 modifications in the functional properties are more marked. Indeed, a shift in the acoustic
 416 absorption curve toward higher frequencies is noted, with a significant drop in the maximum
 417 value reached (Figure 4), a reduction in the transmission loss and an increase in water vapour
 418 sorption capacity that is even greater than for A_{HD} conditions (Figure 5).

419 In order to understand the aging mechanisms causing the modifications in functional
 420 properties, the microstructure has been studied using different complementary
 421 characterisation methods. Three major modifications of properties due to A_{HD} and A_{Ext} agings,
 422 and an additional phenomenon for A_{Ext} are observed:

- 423 - a loss of mass;
- 424 - variations in volume with swelling followed by shrinkage;
- 425 - an opening of the porosity that was initially closed;
- 426 - an increase in the distribution of pore sizes for A_{Ext} .

427 An illustration of the evolution of the microstructure in the material over time for the
 428 different aging conditions is given in Figure 13. The different volume fractions of a batch of
 429 hemp shiv are represented by using the values of the volume fractions of the vegetal cell wall
 430 $\eta_{cell\ wall}$, closed intraparticle porosity Φ_{intra_closed} , opened intraparticle porosity Φ_{intra_opened} ,
 431 interparticle porosity Φ_{inter} and acoustic porosity Φ_{acou} . The four aging phenomena identified
 432 are visible in the illustration reported on Figure 13 and are described one by one in the next
 433 paragraphs.



434 *Figure 13. Volume fraction of hemp shiv in bulk for: a) reference conditions; b) HD aging; c) Ext*
 435 *aging*

436 The first phenomenon is a mass loss of 25% for 24 months in A_{HD} and 35% for 24 months in
 437 A_{Ext} conditions (Figure 6-a). The rate of extractible molecules initially contained in the
 438 particles, which is about 20 % (Figure 11) cannot fully explain the mass loss values obtained.
 439 Thus, the structure of the plant matter is also altered during aging by the attack of micro-
 440 organisms, as observed by SEM in Figure 10 and Figure 11. A targeted degradation according
 441 to the type of aging process can be deduced from the density values of the cell wall measured

442 with a helium pycnometer (Table 3). Indeed, cellulose, hemicellulose and lignin respectively
443 have a density of 1559 kg.m⁻³, 1520 kg.m⁻³ and 1260 kg.m⁻³ [42]. As the ratio
444 lignin/hollocellulose after aging is found lower than before aging (Figure 12), the greatest
445 increase in $\rho_{\text{cell wall}}$ after A_{HD} aging may indicate a preferential attack on the lignin. Due to a
446 higher proportion of hollocellulose, the cell wall density increases. Moreover, cells decohesion
447 observed in Figure 10-d, which is the consequence of an attack on lignin, confirms this
448 explanation. For A_{Ext}, the structure of hemp shiv is more severely damaged with less than 30
449 % of hollocellulose and lignin remaining. This severe attack on the structure is also visible
450 during the preparation of hemp shiv for SEM observations. Indeed, most of hemp shiv is too
451 soft to be sliced, making observations on the structure impossible. Pictures on Figure 11 show
452 hemp particles having still enough stiffness to be cut. The impact of the mass loss on the
453 functional properties is primarily visible after 24 months of A_{Ext} aging conditions, as it is
454 directly responsible for the decrease in transmission loss (Figure 4-b).

455 The second modification identified is the succession of two steps, regarding the volume
456 variation of the batches of shiv in A_{HD} and A_{Ext} aging conditions, namely swelling followed by
457 shrinkage of batches of hemp shiv up to 24 months. The swelling is visible thanks to the
458 evolutions of the bulk density and of the mass loss measured (Figure 6-a and b). In the volume
459 set for the acoustic measurements, the swelling of the particles leads to a decrease of the
460 interparticle porosity, as it is necessary to further compact the particles which are placed in a
461 constant volume. However, in the diagram of figure 13, the acoustical porosity increases.
462 Thus, it is no longer possible to make the assumption that Φ_{acou} corresponds to Φ_{inter} . This
463 point is discussed in the next paragraph. The swelling is therefore represented on the basis of
464 the increase in apparent volume. The effect of the swelling is also visible with the increase in
465 air resistivity σ and the decrease of the viscous characteristic length Λ (Figure 8-b and d).
466 Indeed, with the lowering of the interparticle porosity, it becomes more difficult for air to pass
467 through the material, with a smaller noticeable pore size in acoustic terms. The shrinkage of
468 the bulk hemp shiv is observed after 3 months with a stabilisation of the bulk density
469 associated to the mass loss (Figure 6-a and b). Here, once again, the acoustic parameters are
470 affected. With shrinkage, σ progressively decreases and Λ recovers (Figure 8-b and d). For the
471 A_{HD} aging process, the swelling of particles increases from a value of +35 % at 3 months to +18
472 % at 24 months, with respect to the initial volume. The volume variation for A_{Ext} even drops to
473 8% in relation to the initial volume after 24 months of aging. In terms of functional properties,
474 these variations in resistivity account for the increase in α_{max} for the acoustic absorption curve
475 after 3 months, and then the gradual decrease of this value as aging A_{HD} progresses (Figure 4-a
476 and Figure 8-b). These swellings can be explained by water absorption, which causes a
477 deformation of the particles, no longer holds true without a recovering of their initial size
478 after drying. Over the course of aging and the mass loss of the material, the structure of the
479 plant is sufficiently deteriorated by the micro-organisms to cause the particles to shrink over
480 time (Figure 10 and Figure 11).

481 The next phenomenon is the opening of the porosity in the hemp shiv, which, initially, is
482 partly closed or inaccessible for measurement, whether with air or acoustic waves. Indeed,
483 the increase of the skeleton density and its stabilisation over time after 6 months show that a
484 part of the porosity is not initially accessible to the air (Figure 7). The opened porosity at A_0 is
485 86.3 %, when the total porosity is estimated at 92.0 %. This opening of the porosity $\Phi_{\text{intra_closed}}$
486 is visible in Figure 11. Two types of actions lead to this opening of the porosity. The first one is
487 the attack of the cell wall by micro-organisms, which can be seen by the perforations in the
488 structure of the hemp shiv (Figure 9 and Figure 11). The second one is the action of water,
489 with the swelling of the particles leading to the expansion of the pores and the tearing of the
490 pits after cell wall deformation (Figure 10-e). After aging, the originally closed porosity is
491 practically totally open, the values of the open air porosity and total porosity being
492 respectively 94.6 % and 95.0 % after A_{HD} aging, and 94.9% and 95.3 % for A_{Ext} aging. In
493 addition of the opening of the initially closed porosity, there is an increase of the total porosity
494 explained by the loss of matter after the various aging processes. A portion of the porosity
495 also becomes accessible to sound waves. Indeed, the tortuosity increases, as does the acoustic
496 porosity, visible in the diagram (Figure 8-a and c). This means that the hypothesis put forward
497 for A_0 , whereby only the interparticle porosity plays a role in acoustics, no longer holds true.
498 The acoustic wave is then also dissipated by a portion of the intraparticle porosity. The
499 contrast between the two types of porosity remains significant – a multi-porous system is
500 obtained. This phenomenon is observed for A_{HD} aging up to 24 months and A_{Ext} at 12 months.
501 The impact of this opening of porosity on the functional properties is an increase of the water
502 vapour sorption capacity, associated with an increase of the specific surface area S_m
503 determined by sorption measurements (Figure 5). The increase in tortuosity is responsible
504 for the shift toward lower frequencies in the acoustic absorption curve (Figure 4-a).

505 A final modification of structure must be added to the opening of the porosity to explain the
506 behaviour of hemp shiv at 24 months of A_{Ext} aging. In this case, the tortuosity increases at 12
507 months, and then decreases, eventually reaching a value lower than the initial one (Figure 8-
508 c). The acoustic porosity also decreases, whereas the characteristic viscous length increases
509 (Figure 8-a and d). In this case, the openings of pores after the degradation of the hemp shiv
510 are sufficiently significant so that there is no longer a contrast between the interparticle
511 porosity and intraparticle porosity acoustically accessible, a single-porosity system is
512 obtained once again. The path of the acoustic wave is therefore shorter, the tortuosity
513 decreases sharply, and the size of the viscous characteristic length increases. This decrease in
514 tortuosity and the increase of Λ are responsible for the shift towards higher frequencies and
515 for the sharp decrease in the maximum of the acoustic absorption curve (Figure 4-a).

516 **5 CONCLUSION**

517 In this article, different characteristic modifications due to aging of hemp shiv were
518 highlighted. Three different environments have been applied to bulk hemp shiv during two

519 years: reference conditions (A_{Ref}) at 50% RH, hygric aging with variations in relative humidity
520 at 30°C (A_{HD}) and an outdoor aging process corresponding to real weather conditions (A_{Ext}).

521 For the reference conditions A_{Ref} , no significant variation in the functional or microstructural
522 properties was observed. In these hygrothermic conditions which exist in a building, the
523 properties of shiv do not vary over time, so the durability of the material over time is good.

524 For the A_{HD} accelerated aging process, which applies an average humidity of over 80%, with
525 the majority of time spent at 98% RH, three phenomena are part of aging mechanism,
526 inducing evolution of the microstructure and of the functional properties of hemp shiv are
527 observed:

- 528 - a mass loss, which can go so far as to decrease the material's properties in terms of
529 acoustic insulation;
- 530 - volume variations which play a part in acoustic absorption;
- 531 - an opening of the porosity, leading to a higher water vapour sorption capacity, and a
532 change in the acoustic absorption.

533 Finally, for A_{Ext} aging, a fourth phenomenon is added to the first three: the modification of the
534 pore size distribution, which has a significant impact on the sound absorption.

535 The role of micro-organisms as mechanism of the phenomena is demonstrated. Fungal attack
536 alters the microstructure of the hemp shiv, and therefore its functional properties. The micro-
537 organisms develop for the accelerated aging A_{HD} , but not for aging A_{Ref} .

538 The accelerated aging, then, modifies the properties of the hemp shiv, but needs to be
539 compared to conditions of aging in a real-world building in order to be able to estimate the
540 lifetime of the bulk hemp shiv whilst guaranteeing it will retain its functional properties. In
541 the case of hemp-based concrete, it is therefore necessary to monitor fungal development in
542 the material over time, and also any modifications in porosity, which may be indicative of
543 fungal development within the hemp concrete. Moreover, in hemp concrete the aggregates are
544 protected by an alkaline binder. It could be interesting to study if the same aging mechanisms
545 are obtained.

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549 French National Research Agency.

550 **Data availability**

551 The raw/processed data required to reproduce these findings cannot be shared at this time as
552 the data also forms part of an ongoing study.

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