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## Monitoring of earth concrete damage evolution during drying

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**Abstract.** Earth concrete, constituted mainly of raw earth, is a promising ecofriendly material for future constructions. However, it is composed of fine particles like clay which make it very sensitive to drying. The volume change induced by desiccation shrinkage can be responsible of cracking which can have a detrimental effect on the durability and mechanical properties. Thus, in order to design reliable earth concrete structures, considering a coupling between drying shrinkage and damage is important. The effect of curing conditions on the mechanical properties of earth concrete with different percentages of flax fibers has been studied. The evolution of damage during drying has been monitored continuously using the ultrasound and the acoustic emission (AE) techniques. The mechanical properties of earth concrete decreases for specimens stored at low relative humidity. In addition, the AE results associated with shrinkage and mass loss measurement made it possible to describe qualitatively the drying process.

**Keywords:** earth concrete, shrinkage, drying, damage, Acoustic Emission technique.

### 1. Introduction

Over the past decade, a large amount of natural resources has been consumed by the construction sector accompanied with the emission of a large amount of CO<sub>2</sub> due to the production of cement. Hence, due the increase awareness, many studies are now conducted to find an alternative ecofriendly material. In the case of structures that do not require high mechanical performance, earth concrete can be considered as an acceptable solution in comparison to conventional concrete [1]. In addition, this latter presents many advantages in terms of hygrothermal and acoustical performance [2].

27 However, the presence of a large quantity of fine particles in earth concrete makes it very sensitive to  
28 different types of shrinkage which can induce cracking and affect its strength and durability [3–5].

29 Earth concrete volume change is an unavoidable phenomenon, from very early age to long-  
30 term behavior. At early age (before 24 h), earth concrete goes through negative volumetric variations  
31 due to water evaporation (plastic shrinkage) or internal reactions of chemical origins (autogenous  
32 shrinkage) linked to the hydration of binders. The menisci present at the interface between the water-  
33 filled and empty pores will result in the development of stresses within the liquid phase. These stresses  
34 pull earth concrete matrix particles closer together and lead to a volume reduction and strains within  
35 the solid framework. The autogenous shrinkage component, being mainly dependent on the  
36 composition of earth concrete, develops more rapidly with time than drying shrinkage. Long term  
37 drying shrinkage (after 24 h) depends on the age at the beginning of drying and external parameters  
38 such as relative humidity (RH) and specimen size [6]. The shrinkage of different types of earth  
39 construction have been studied in the literature [7,8]. [9] studied drying shrinkage for un-stabilized  
40 rammed earth walls. Volumetric shrinkage developed in the two direction of the wall with a value that  
41 do not exceed 5%. This value depends on clay content, grain size of the soil and drying rate and process  
42 that is related to the radius of the pores. Shrinkage of rammed earth is much higher than that of  
43 conventional concrete, with a faster evolution in the initial phase, representing 85% of the total  
44 shrinkage recorded during the first 28 days [10]. In addition, specimens stored at a RH of 95% have a  
45 total shrinkage 20% lower than specimens stored at a RH of 35%. Drying shrinkage for cement-  
46 stabilized soils is mainly related to the plasticity index of the soil, the clay content [11] and the clay  
47 minerals present in the soil [12]. Increasing clay content increases desiccation shrinkage [13]. If tensile  
48 strength is less than stress induced by shrinkage restraint , cracking will occur in earth concrete [6].  
49 Micro cracking during shrinkage depends on the type of restraint which can be caused by the structure  
50 (externally applied restraint) or the material (internal restraint). Self-restraining mechanisms can be  
51 related to moisture/shrinkage gradients that develop at different depths in the specimens and by the  
52 presence of aggregates. Those micro cracks increase the permeability of earth concrete and can lead to  
53 stress concentrations during loading. Earth concrete structures are therefore vulnerable to inherent  
54 stresses or shrinkage-induced cracks that can not only cause structural defects, but also reduce the

55 serviceability, durability and aesthetics of earth concrete [14]. Carbonation reactions also occur in the  
56 long term due to the penetration of carbon dioxide into earth concrete, leading to the formation of  
57 calcite [15]. Carbonate crystals have weak cohesion properties and the presence of calcite does not  
58 really improve the mechanical performance of earth concrete [16].

59         Early age shrinkage or plastic shrinkage of earth concrete has been studied in a previous study  
60 [3] and more particularly the effect of flax fibers on cracking sensitivity. In fact, fibers have been  
61 widely used for soil reinforcement and to reduce cracking induced by plastic shrinkage in concrete at  
62 early age [3]. Vegetable fibers have the advantage of being light, ecological, with low-cost as they are  
63 produced as waste materials, in addition to their good acoustic and hygrothermal properties and  
64 compatibility with earth concrete. [3,17] showed that flax fibers enhanced the ductility of earth concrete  
65 by generating a diffused cracking and decreased the horizontal plastic shrinkage. Thus, flax fibers  
66 reduce the cracking risk of restrained earth concrete at early age [3]. The use of natural fibers can be  
67 sensitive in the long term due to their natural decomposition and biological attack [18]. High humidity  
68 can promote this kind of activity within the material and accelerate the biodegradation [19]. Soil  
69 stabilization increases the resistance to biological attacks [20]. Shrinkage is also influenced by the  
70 fiber's type and content [21]. The rate of shrinkage increases with clay and water content but decreases  
71 with fiber content [22]. The addition of fibers improves also the strength and reduces shrinkage  
72 cracking of earth concrete [22,23]. The stabilization of shrinkage occurred earlier with adding straw,  
73 indicating a rapid drying due to the accelerated evaporation through the air channels formed [23]. The  
74 addition of 4% of sisal or coconut fibers reduced tensile shrinkage cracking [24].

75         In this study, long-term shrinkage and the effect of drying on the mechanical properties of earth  
76 concrete have been investigated.

77         In order to monitor damage evolution during shrinkage, the AE technique has been used. This  
78 technique has been widely used to monitor damage and crack development during mechanical loading  
79 [25–29]. Recently, it has been also used to understand some complex phenomena as corrosion [30],  
80 creep [31], hardening of cement paste and concrete [32], drying shrinkage cracking in cement-based  
81 materials [33–36] and drying of ceramics [32]. Different mechanisms have been proposed to explain  
82 the reasons behind the AE activity during the cement paste hydration as the formation and dissolution

83 of hydrates, the consumption of water and the emptying of capillary pores [32]. [37] attributed the AE  
84 activity during self-desiccation to cavitation.

85 A comprehensive study of the damage process during shrinkage of earth concrete is proposed in  
86 this paper using the AE technique. First, the materials and the experimental methods are presented.  
87 Then, desiccation shrinkage and its effect on the mechanical properties are analyzed. Finally, the  
88 characterization of damage evolution under drying is suggested using the AE method.

89

## 90 **2. Experimental program**

### 91 **2.1 Materials**

#### 92 2.1.1. *Soil and Hydraulic binders*

93 The soil of earth concrete mixtures is based on an artificial soil composed of 30% of bentonite clay  
94 (75% of Smectite, 15% of Illite and 10% of Kaolinite) and 70% of sand [3]. This artificial soil has been  
95 chosen in order to avoid the variability of onsite soil. The artificial soil has been stabilized with 3% of  
96 natural hydraulic lime (NHL5/ EN 459-1 [38]) and 8% of cement (CEM 1, 52.5 N PM-CP2 / EN197-  
97 1 [39]) based on the clay and sand mixture mass. Those percentages have been fixed according to other  
98 studies presented in the literature that showed that 2% to 4% of lime are sufficient to stabilize the soil  
99 [40,41], and the optimal percentage of cement to ensure a sufficient strength for earth concrete is 8%  
100 [42]. The pure Portland cement CEM 1 composed mainly of clinker is chosen to prevent the effect of  
101 additional components.

102

#### 103 2.1.2. *Flax fibers*

104 Flax fibers are natural fibers, bio-renewable, recyclable and biodegradable. The fibers were cut at  
105 constant length of 12 mm and their diameter is equal to  $14.66 \mu\text{m} \pm 2.95$  [43]. These fibers are rich in  
106 cellulose, more than 60% of their constitution, which gives them good tensile strength [44]. The fibers  
107 used in this study were not treated in order to keep them natural and easy to use.

108

109 **2.2** Mixtures

110 Three mixtures have been tested by varying the percentage of flax fibers: 0, 0.3 and 0.6%. Clay  
111 and sand were first mixed during 3 minutes to homogenize of the artificial soil. Then, lime and cement  
112 were added to the dry mixture and mixed during 2 minutes. Water and superplasticizer were then added  
113 and mixed for 3 minutes. The superplasticizer Tempo 10 has been used due to its efficiency as a water  
114 reducer admixture in earth concrete [8]. Finally, fibers were progressively added and mixed during 3  
115 minutes. Table 1 presents the mixtures components and composition. The quantity of fibers was  
116 subtracted from the artificial soil mass to keep an equivalent solid mass. The effective water to binder  
117 ratio was kept constant and equal to 0.45. Additional water has been added to take into account water  
118 absorbed by flax fibers. Details about the constituents and mixture properties at early age can be found  
119 in [3].

120

121 **Table 1.** Mixtures components and composition.

Components (kg/m <sup>3</sup> )	Sand	Bentonite	Cement	Lime	Total water	Super-plasticizer	Flax fibers
SA0F	931	405	152	34.2	367	1.6	0
SA03F	929	401	152	34.2	370	1.6	5.7
SA06F	925	399	152	34.2	375	1.6	11.4

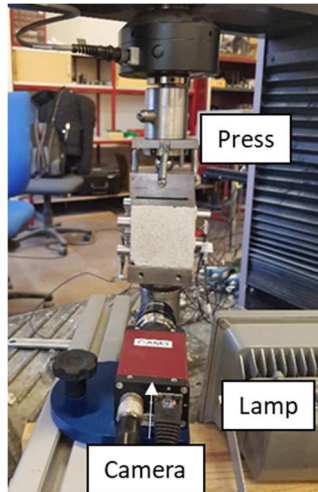
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124 **3. Methods**

125 **3.1** Compressive test

126 Unconfined compressive tests have been conducted using an electromechanical machine with  
127 a capacity of 100 kN (**Figure 1**). A constant loading displacement rate of 0.6 mm/min has been  
128 considered. This test has been realized on cubic specimens of 10×10×10 cm<sup>3</sup> at the age of 28 and 180  
129 days. After pouring earth concrete, specimens were covered with a thin sheet of plastic in order to  
130 prevent water loss and maintained at a temperature of 20°C during 48 h. Then, specimens were  
131 unmolded and stored in two different curing conditions at a RH of 90% and 50% (± 5%) and a  
132 temperature of 20 °C.



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135

**Figure 1.** Compressive test set up

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### 137 **3.2 Ultrasound test**

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### 143 **3.3 Digital image correlation technique**

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The ultrasound system PUNDIT (Portable Ultrasonic Nondestructive Digital Indicating Tester) was used to measure the velocity of ultrasound waves. It consists of a pulse generator with two transducers. An electrical pulse is converted into a longitudinal pressure wave with a frequency of 200 kHz by using a transmitter coupled to the surface with grease.

The compressive tests were monitored with the digital image correlation (DIC) technique. It is a direct and simple method for fracture examination. The usage of this technique implicates an advance surface preparation of earth concrete specimens by using a white and black speckle (white paint at the bottom with small black dots above). The displacement of the tested surface of the specimen is obtained by the analysis of the random speckle at different instances based on the pictures taken during the test. The photos of the specimen's surface have been captured using a digital camera. The reference images have a resolution of  $2452 \times 2056$  pixels. To improve the luminosity of the photos, two lamps have been used in parallel. Images have been taken at a rate of one image per second during the tests and stored

152 directly. The treatment of the images has been realized using the commercial software Vic 2D with a  
153 resolution of 0.05 mm per pixel.

154

### 155 **3.4 Shrinkage and water loss measurement tests**

156 Shrinkage tests were carried out on prismatic specimens of 7x7x28 cm<sup>3</sup> fitted with metal studs  
157 at their ends. Specimens were poured and kept for 24 hours in sealed conditions to avoid drying. After  
158 24 hours, specimens were removed from the molds and shrinkage tests were carried out in an air-  
159 conditioned room at 20 °C ( $\pm 2$  °C) and a RH of 50% ( $\pm 5\%$ ). The measurement of the longitudinal  
160 deformation was carried out using LVDT sensors with a precision of 3  $\mu$ m during several weeks. For  
161 total shrinkage, the ends of the specimens were covered with an adhesive aluminum tape to allow  
162 identical lateral drying of the four sides of earth concrete. For Autogenous shrinkage, specimens were  
163 completely covered with two layers of adhesive aluminum tape to prevent drying. The mass loss was  
164 measured simultaneously on specimens with the same dimensions and stored under the same drying  
165 conditions.

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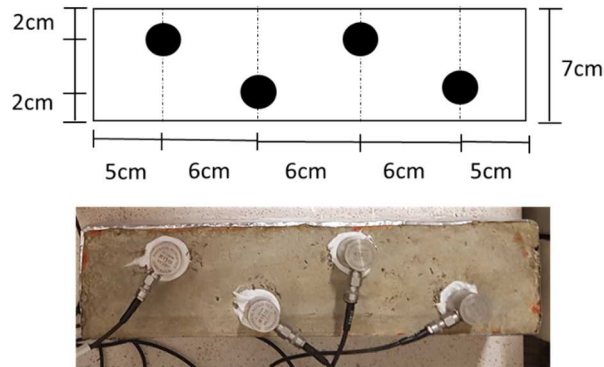
### 167 **3.5 Acoustic Emission technique**

168 The AE method was used to monitor the damage evolution in earth concrete specimens during  
169 shrinkage. The AE system comprised of an eight channel AE Win system, a general-purpose interface  
170 bus (2PCI-DISP4) and a PC for data storage analysis. Four piezoelectric transducers with a resonant  
171 frequency of 150 KHz were used to convert the mechanical waves to electrical signals (**Figure 2**). They  
172 were placed on specimens using silicon grease as a coupling agent. The recorded AE amplitudes range  
173 from 0 to 100 dB. The detected signals are amplified with a 40 dB gain differential amplifier. The  
174 signal detection threshold is set at a value of about 35 dB to overcome the background noise (RILEM  
175 TC212-ACD). The acquisition parameters are set as follows: peak definition time (PDT) = 100  $\mu$ s, hit  
176 definition time (HDT) = 200  $\mu$ s and hit lock time (HLT) = 400  $\mu$ s. A high pass filter with a cut-off  
177 frequency of 20 KHz, and a low-pass filter with a cut-off frequency of 400 KHz are used to eliminate



178 mechanical and electro-magnetic disturbances. Several signal descriptors were calculated by AEwin  
179 system.

180



181

182 **Figure 2.** Prismatic specimen geometry and position of AE transducers

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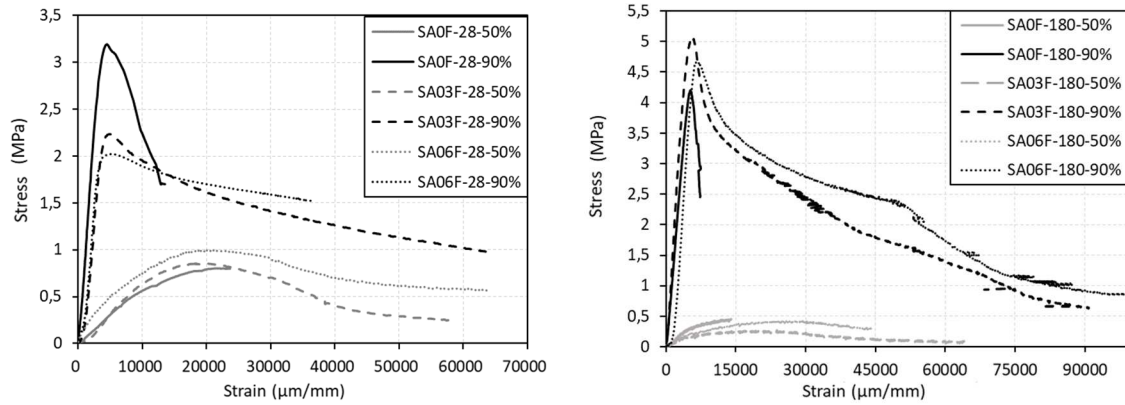
## 184 **4. Results**

### 185 **4.1 Mechanical properties**

186 **Figure 3** shows the stress/strain curves for specimens conserved at two different conditions at  
187 the age of 28 and 180 days. The strain was calculated as the ratio between the average displacement,  
188 obtained using the digital image correlation, and the length of the specimen. In fact, the press  
189 displacement does not reflect the real deformation of earth concrete specimens. The deformation  
190 calculated from the press displacement is higher than that of DIC due to the deformation of the frame  
191 and the crushing of concrete at the contact between the metallic plates and concrete specimen. Different  
192 phases can be distinguished. For mixtures stored at 90% of RH, the stress increased linearly in function  
193 of the strain almost till the maximal strength showing an important elastic phase. However, for  
194 specimens stored at 50% of RH, the elastic phase was observed. The slope of the elastic phase decreases  
195 with an important rate as RH decreases while the strain at the peak increases. After the peak, the load  
196 decreases quickly for SA0F indicating a brittle behavior of earth concrete conserved at 90% of RH in  
197 comparison to that stored at 50% of RH. For specimens with fibers, the post peak region zone was

198 more important indicating an increase of earth concrete ductility due to different softening mechanisms  
199 [19].

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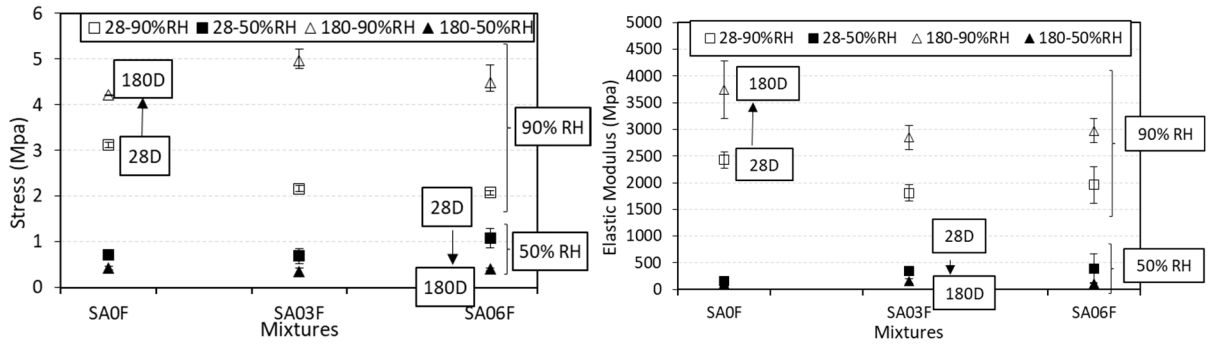
202 **Figure 3.** Stress-strain curves of earth concrete specimens conserved at 50% and 90% of RH for  
203 SAOF, SA03F and SA06F at the age of 28 days (a) and 180 days (b)

204

205 **Figure 4** shows the maximum compressive stress and the elastic modulus of earth concrete  
206 specimens kept at 90% and 50% RH at the age of 28 and 180 days. For the mixtures kept at 90% of  
207 RH, the compressive strength decreases slightly with the addition of fibers at the age of 28 days which  
208 may be due to the higher porosity [3]. The compressive strength of earth concrete increased with age  
209 specially for specimens with fibers which may be due to time-dependent pozzolanic reactions [40]. In  
210 fact, the formed hydrates bind the soil particles together and give them a more compact matrix structure.  
211 This limits considerably the particles rearrangement at the interface and increases the effective  
212 interfacial contact area. In addition, as natural fibers are characterized by their hydrophilicity, hydrates  
213 may cover the surface of fibers due to the presence of water and improve the characteristics of the  
214 interfacial bonding and the frictional force between the fibers and the matrix [45,46].

215 For specimens kept at 50% of RH, a high reduction of strength has been observed due to drying  
216 with a slight effect of the percentage of fibers. This reduction of strength continues with the age of  
217 curing at 180 days.

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**Figure 4.** Maximum strength and Young modulus of SA0F, SA03F and SA06F conserved at 50% and 90% of RH at the age of 28 and 180 days.

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The decrease of the mechanical properties of earth concrete specimens may be due to degradation induced by desiccation shrinkage. This degradation can be observed visually at the surface of the specimens (**Figure 5**). The photos were taken before the compression test at the age of 180 days for specimens stored at 50% of RH. The photos show the flaking of the surface especially for specimens without fibers in addition to visible cracks. Cracks were detected at the 28<sup>th</sup> day for SA0F and spread rapidly over the entire surface of earth concrete, inducing a progressive loss of the surface layer. In fact, micro cracks may develop in earth concrete due to gradient deformation during drying, in addition to the decrease of hydration reactions and the loss of the cohesion between the particles. For the mixtures with fibers, cracks were more visible at the 180<sup>th</sup> day, without leading to a significant loss of earth concrete surface. This slower degradation may be associated to a better crack resistance. In fact, the presence of fibers may prevent the propagation of cracks due to the adhesion between the matrix and the fibers and the bridging effect [47,48]. However, this adhesion may decrease during the drying process due the strain gradient at the interface between the matrix and the fibers which may be responsible of the lower strength at the 180<sup>th</sup> day. This may be also due to a different pore size distribution that can modify the capillary pressure and thus the stress gradient that may impact the mechanical and transfer properties of earth concrete.



240

241 **Figure 5.** Photos of SA0F, SA03F and SA06F before the compression test at 50% of RH at the age of

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180 days

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#### 244 **4.2 Digital image correlation monitoring of fracture tests**

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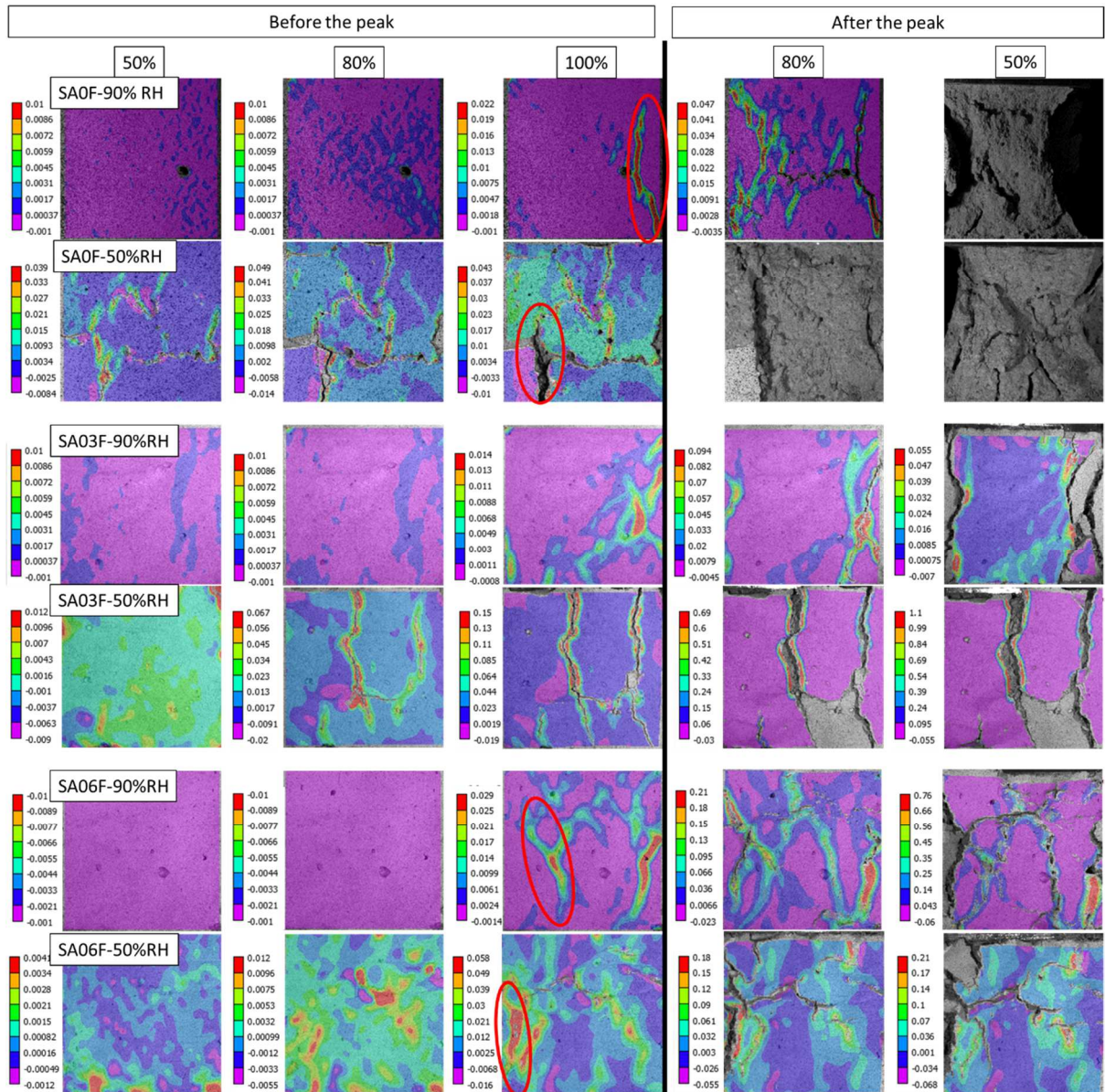
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For a better understanding of the fracturing process, the compressive tests were monitored with the DIC method. **Figure 6** presents the 2D strain fields along the X direction for SA0F, SA03F and SA06F stored at 50% and 90% of RH at different loading levels. For specimens kept at 90% of RH, no cracks have been visualized in the elastic phase and macro cracks started to propagate slightly before the peak. For SA0F, a large crack has been observed with an important width. The developed cracks led to the loss of strength and the total rupture of SA0F at 50% of the maximum strength in the post peak region. The rupture type shows clearly a lateral confinement. In fact, due to friction, a triaxial stress state was created within the material in the areas near the loading plates. Vertical cracks appeared mainly near the corners of the lateral surface and were coupled with the horizontal ones due to bending [49]. Consequently, a big part of the specimen fell indicating a brittle fracture of the specimen [23].

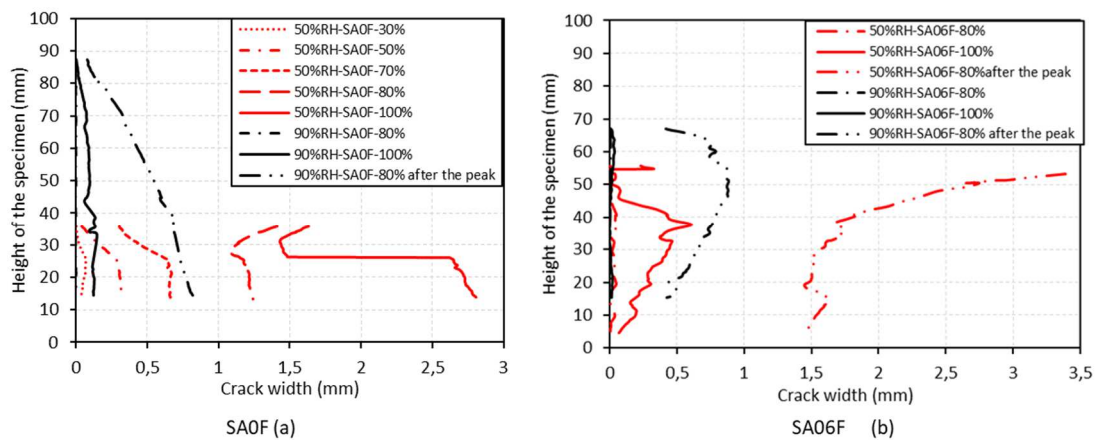
However, for specimens with fibers, a diffuse multi cracking has been observed at the peak with small crack openings. This indicates a more ductile behavior due the presence of fibers that limit the crack propagation and opening by bridging the cracks and redistributing the applied stress during the extension [19,23,49]. Additional cracks have been observed in the post peak region and crack openings increased at the surface with no clear effect of the lateral confinement. This multi cracking is partially responsible of the higher fracture energy and ductility of the material [50].

261 For specimens stored at 50% of RH, cracks appeared earlier at 50% of the maximum strength  
 262 for SA0F and 80% of the maximum strength for mixtures with fibers. This confirms the degradation  
 263 of earth concrete specimens and the pre-presence of micro cracks that developed during drying [8].  
 264 Several cracks were observed for SA0F. The crack openings were also more important for SA0F  
 265 indicating the effect of fibers in increasing the ductility and preventing the brittle rupture.  
 266



267  
 268 **Figure 6.** 2D strain fields along X for SA0F, SA03F and SA06F stored at 50% and 90% of RH at  
 269 different loading levels.  
 270

271 **Figure 7** presents the evolution of a chosen crack width (circled in figure 8) along the height  
 272 of specimens SA0F and SA06F at 50% and 90% of RH. The crack width increases with the applied  
 273 load. It started earlier for specimens kept at 50% of RH at 30% of the maximum strength for SA0F and  
 274 80% of the maximum strength for SA06F which confirms the pre-cracking of specimens subjected to  
 275 drying. In addition, the width of the cracks was much larger for specimens kept at 50% of RH than that  
 276 kept at 90% of RH for the different loading levels. Note that the crack width varied along the crack  
 277 length due to different stress gradient in addition to the effect of flax fibers distribution and orientation.  
 278 The addition of flax fibers slowed down crack openings and increased earth concrete ductility. In fact,  
 279 flax fibers held the soil matrix together by bridging the cracks, in addition to stress transfer and  
 280 redistribution in earth concrete [24]. The good adhesion between fibers and earth concrete matrix  
 281 prevented the spread of large crack opening and improved the fracture energy. The rupture occurred  
 282 due to the debonding of flax fibers from the matrix.  
 283



284  
 285 **Figure 7.** Evolution of the crack width for (a) SA0F and (b) SA06F at 50% and 90% of RH

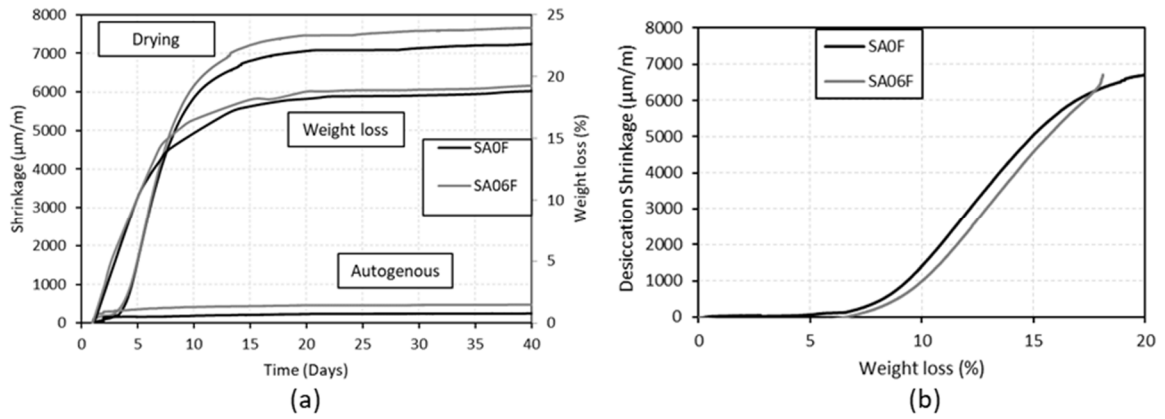
286  
 287 To gain a better understanding of the effect of drying, long term shrinkage was evaluated and  
 288 specimens were monitored with the ultrasound and the AE techniques.  
 289

### 290 4.3 Long term shrinkage

291 To improve the comprehension of the different mechanisms responsible of the degradation of  
292 the mechanical properties of earth concrete due to drying, the evolution of shrinkage in correlation with  
293 the mass loss has been studied. **Figure 8** (a) presents the evolution of the total shrinkage, autogenous  
294 shrinkage and weight loss of SA0F and SA06F in function of time. Autogenous shrinkage occurs  
295 independently of the external water loss and is a result of chemical shrinkage and self-drying shrinkage.  
296 The autogenous shrinkage of earth concrete with and without fibers increases slightly in the first 3 days  
297 after casting and then stabilized. Desiccation shrinkage occurs in earth concrete due to self-desiccation  
298 (internal drying) and mainly due to the loss of water from capillary pores and the gel (external drying).  
299 Total shrinkage is therefore represented as the sum of desiccation and autogenous shrinkage. The  
300 reduction of RH in the pore system causes a water–air meniscus that subjects the pore walls to  
301 considerable stress and leads to substantial drying shrinkage. The shrinkage rate is slow during the first  
302 three days after casting. Then, it increases with an important rate and begins to decrease gradually in  
303 function of time to stabilize after 20 days. Desiccation shrinkage is almost 10 times higher than  
304 autogenous shrinkage. Note that drying shrinkage values are very important in comparison to ordinary  
305 concrete which may be due to a higher capillary pressure and a less rigid solid skeleton.[14].

306 The evolution of desiccation shrinkage in function of the weight loss has been also plotted and  
307 shows two phases (**Figure 8** (b)). During the first 3 days after casting, even though the weight loss rate  
308 is important (about 7%), the generated shrinkage is low. In the second phase, the weight loss and  
309 desiccation shrinkage with increased with an important rate proportionally. The slope variation can be  
310 explained by the modification of the dimension of the pores that are being emptied and the decrease of  
311 the evaporation rate. In fact, during the drying process some of the liquid water cannot reach the surface  
312 and evaporates within the pores.

313



314

315 **Figure 8.** (a) Evolution of total shrinkage, autogenous shrinkage and weight loss in function of time  
 316 and (b) evolution of desiccation shrinkage in function of the weight loss for SA0F and SA06F

317

318 The same behavior has been observed for mixtures with and without fibers. From the beginning  
 319 of the test and till the 6<sup>th</sup> day after casting, the weight loss and shrinkage were the same for the two  
 320 mixtures. However, after this age, the weight loss and the total shrinkage increase slightly for mixtures  
 321 with fibers. This may be due to the higher water absorption with fibers.

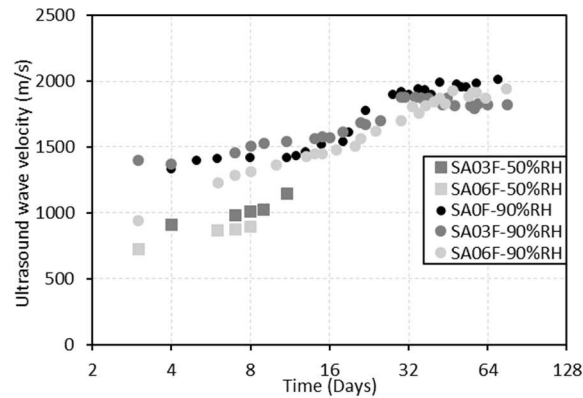
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#### 323 4.4 Ultrasound monitoring

324 **Figure 9** shows the comparison between the ultrasonic waves propagation velocity of  
 325 specimens kept at 50% and 90% of RH in function of the curing age. For specimens kept at 90% of  
 326 RH, the velocity increases with an important rate during the first days and slightly after the age of 28  
 327 days indicating the increase of stiffness [51]. For specimens kept at 50% of RH, the wave propagation  
 328 velocity is smaller. It increases during the first days but the velocity measurement becomes impossible  
 329 after 8 days for SA03F and 12 days for SA06F. This can be related to water loss and premature damage  
 330 of specimens due to drying that may cause cracking and a loss of cohesiveness between particles [52].

331





332

333 **Figure 9.** Evolution of the ultrasonic wave velocity at 50% and 90% of RH in function of time.

334

335 For a better understanding of the different phenomena occurring during drying, shrinkage tests  
 336 have been also monitored using the AE technique.

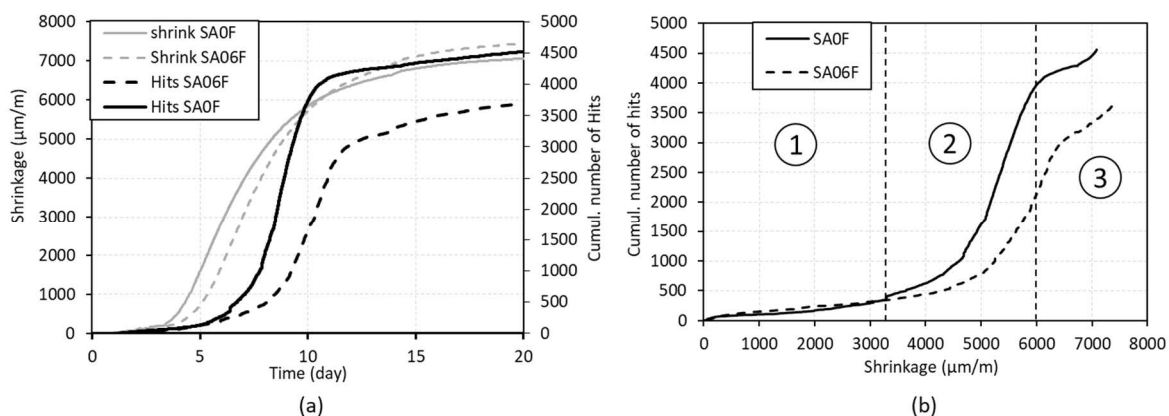
337

#### 338 **4.5 AE monitoring of drying tests**

339 AE hits provide an indication on the number of burst emissions and wave characteristics, such  
 340 as the amplitude, duration and absolute energy. The AE hits detected during shrinkage may be  
 341 generated due to micro cracking or cavitation [37]. In fact, when evaporation occurs at the surface of  
 342 the pore, the radius of the liquid/vapor interface or meniscus decreases. When the radius of curvature  
 343 of the meniscus is small, it retreats into the pore and produces a kinetic energy generating the  
 344 propagation of mechanic waves [53]. Micro cracks can be generated due to moisture gradient that leads  
 345 to non-uniform drying shrinkage. As the surface of the material exposed to drying shrinks faster, micro  
 346 cracks are mainly generated at the surface [54]. Shrinkage can be also restrained by the presence of  
 347 aggregates or fibers inducing micro cracking at the interface with the matrix [55].

348 **Figure 10** shows the cumulated number of AE hits in correlation with shrinkage in function of  
 349 time. For a better analysis, the cumulative number of AE hits has been also plotted in function shrinkage  
 350 for SA0F and SA06F. Three phases similar to those observed while plotting the relationship between  
 351 the weight loss and shrinkage have been obtained. During the first phase, few AE hits were detected.  
 352 They reached a value of 500 for a shrinkage value equal to 3200  $\mu\text{m}/\text{m}$ . This phase is mainly related to  
 353 the departure of water from the surface and larger porosity. The numbers of AE hits was similar for

354 both mixtures. During the second phase between the 6th and the 11th day after pouring, the number of  
 355 the cumulative number of AE hits increases with an important rate (from 500 to 4000 signals). This  
 356 may be related to micro cracks generated by non-uniform desiccation shrinkage at the surfaces exposed  
 357 to drying. This can be also the reason behind the inability to measure the ultrasonic velocity for  
 358 SA06F12 after the 11th day (**Figure 6**). The cumulative number of AE hits was more important for  
 359 SA0F for the same level of shrinkage indicating a higher damage level. This is confirmed with the  
 360 compressive tests where the decrease of strength was more important for SA0F. During the third phase,  
 361 a notable decrease in the acoustic activity was observed indicating the decrease of micro cracks  
 362 generation in parallel with the decrease of the weight loss and shrinkage stabilization.  
 363



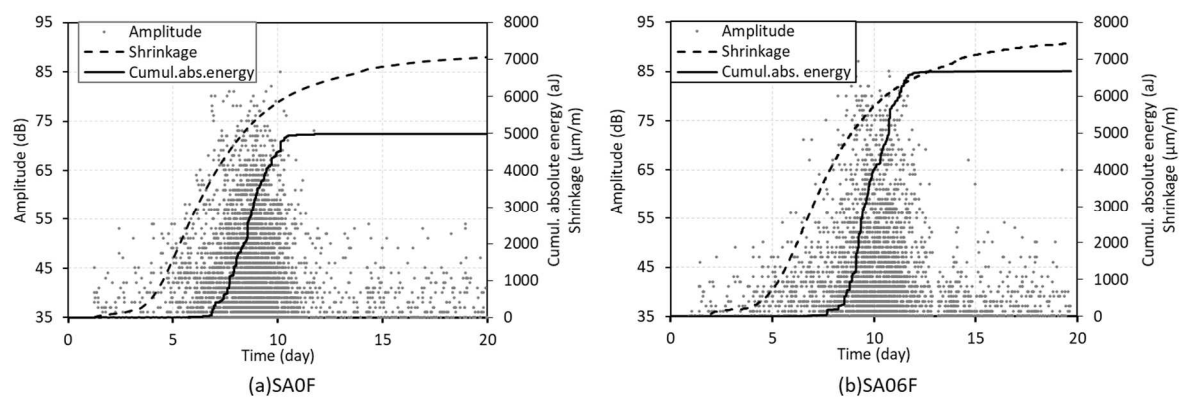
364  
 365 **Figure 10.** (a) Cumulative number of AE hits in correlation with shrinkage and (b) cumulative  
 366 number of AE hits in function of shrinkage for SA0F and SA06F.  
 367

368 Several AE parameters have been also studied during drying. **Figure 11** presents the  
 369 cumulative AE energy in parallel with AE amplitude in function of the time for SA0F and SA06F. AE  
 370 signals with low energy and low amplitude were generated during the first phase. During the second  
 371 phase, the cumulative absolute energy increases with an important rate between the 7<sup>th</sup> and the 11<sup>th</sup> day  
 372 after casting indicating an important damage. The amplitude of the detected AE signals increased also  
 373 and reach a value of 85 dB. In fact, during drying, the water present in the pores of the earth concrete  
 374 evaporates, inducing suction forces. The negative pressure generated in the pores forces the grains to  
 375 rearrange and move closer to each other. When the tensile stress is equal to the cohesive force of

376 attraction between the particles, cracks will be formed [47], [56]. The primary cracks increase in size  
 377 and new cracks are formed until they intersect. The external surfaces of the earth concrete specimens  
 378 are more affected because of the strain gradient. In fact, water loss is faster at the surface than inside  
 379 the specimen according to the drying kinetics which may explain the spalling observed at the surface.  
 380 During the stabilization phase, the generated AE energy and amplitude decrease quickly indicating a  
 381 lower degradation rate.

382 The cumulative absolute energy was more important for mixture with fibers. In addition, this  
 383 energy liberation appeared later. This could be related to a higher toughness with the addition of fibers.  
 384 At one hand, micro cracks can initiate at the interface between earth concrete matrix and flax fibers  
 385 and in the other hand, fibers may be responsible of a different pore size distribution, which can alter  
 386 drying kinetics, stress redistribution and prevent micro cracks from propagating through the specimen  
 387 [17,57].

388



389

390 **Figure 11.** Cumulative AE energy in correlation with AE amplitude and shrinkage in function of  
 391 time for SA0F and SA06F

392

#### 393 **4.6 Evolution of Ib-value**

394 The evolution of the micro cracks dimensions, developed during drying, was carried out by  
 395 measuring the Ib value. The AE peak amplitude is related to the magnitude of the fractures developed  
 396 during desiccation shrinkage. To determine the overall fracture quantitatively on basis of peak  
 397 amplitude, the Ib-value originated from seismology is performed. It is defined as the negative gradient

398 of the seismic magnitude distribution. Statistical values of the amplitude distribution were incorporated  
 399 to the Ib-value in order to apply it for fracture evaluation in civil engineering materials. According to  
 400 the cumulative amplitude distribution, the index (Ib-value) changes following the fracture progress  
 401 [58]. If small-scale fractures are superior to large-scale fractures, Ib-value tend to increase, in the  
 402 contrary if large-scale fractures are superior to small-scale fractures, Ib-value tends to decrease.  
 403 Therefore, Ib-value represents the ratio of weak to strong events [59]. Micro-cracks generate a large  
 404 number of weak acoustic emissions with a high Ib-value. Macro-cracks lead to a relatively low Ib-  
 405 values since more strong events are generated. The Ib-value was calculated using the peak amplitudes  
 406 of 100 successive hits, assuming an average amplitude  $\mu$  and a standard deviation  $\sigma$  of the amplitudes  
 407 and N the number of hits with an amplitude higher than  $\mu - \alpha_1\sigma$  or  $\mu - \alpha_2\sigma$  [60]. The formula used to  
 408 calculate the Ib-value is as follows:

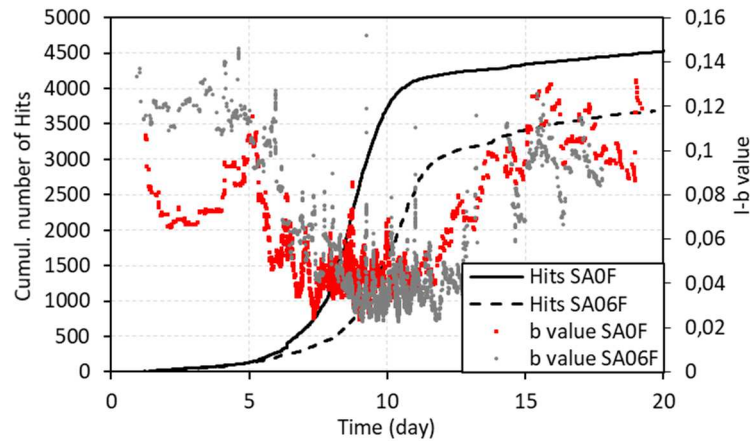
409

$$410 \quad \mathbf{Ib} = \frac{\log_{10} N(\mu - \alpha_1\sigma) - \log_{10} N(\mu - \alpha_2\sigma)}{(\alpha_1 + \alpha_2)\sigma}$$

411 where  $\alpha_1$  and  $\alpha_2$  are constants.  $\alpha_1$  and  $\alpha_2$  values are 0.5 and 1.5 respectively.

412 **Figure 12** shows the evolution of Ib-value during drying in function of time. Ib-value was  
 413 important at the beginning and decreased after the 5<sup>th</sup> day, where damage started to develop with small  
 414 scale fracturing in the specimen. Ib value attended its minimum stability value after 6 days for SA0F  
 415 and 8 days for SA06F where the cumulative number of AE hits increased with a high rate. In this phase,  
 416 micro cracks started to initiate and develop with a larger scale especially near the surface of the  
 417 specimen where drying is important. Ib value increases again showing stability after 11 days for SA0F  
 418 and 12 days for SA06F and attend the same value after 14 days. This indicates the presence of two  
 419 different mechanisms. At the beginning and due to water evaporation, AE hits may be due the retreat  
 420 of water menisci into the pore. Then, micro cracks initiate and develop with an important rate between  
 421 the 5<sup>th</sup> and 12<sup>th</sup> days after concrete casting generating AE signals with higher amplitude and energy.  
 422 The third phase indicates a slow propagation of these cracks already developed in the specimen with a  
 423 lower scale.

424 Note that, Ib value changes not only in function of the loading level or damage rate but also  
425 according to the used concrete, sensors and the applied loading test [35,61].  
426



427

428 **Figure 12.** Evolution of Ib-value during drying.

429

430 Even though damage evolution was monitored continuously with the AE technique, however  
431 the detailed geometry and pattern of the developed cracks are not shown. Additional techniques as the  
432 optical microscopy [35], digital image correlation [62], micro-tomography or others could be used to  
433 obtain additional information on micro cracking.

434

## 435 5. Conclusions

436 The effect of drying on the mechanical properties of earth concrete has been studied.  
437 Autogenous and desiccation shrinkage of earth concrete in parallel with the mass loss have been also  
438 measured. As a part of the investigation, the ultrasound and the AE techniques were applied to monitor  
439 the evolution of damage induced by desiccation shrinkage. AE monitoring allowed a temporal  
440 development assessment of damage and the process of drying was characterized by following the AE  
441 activity and AE parameters. The results show that the AE technique is able to detect low energy  
442 processes during shrinkage as well as damage evolution. The relationship between the AE activity and  
443 shrinkage is illustrated and the effect of fibers was investigated. The AE results associated with

444 shrinkage measurements allow to describe qualitatively the drying process and give wealth information  
445 on damage evolution. Three different phases were distinguished during drying indicating different  
446 mechanisms. Damage increases during the second phase after 6 days of casting indicating the  
447 development of self-restraint cracks. This damage may be partly responsible of the strength reduction  
448 observed with the compression tests.

449 Additional studies will be carried out thereafter to better understand the effect of flax fibers on  
450 shrinkage by the classification of AE signals and conducting addition tests at the microscopic level for  
451 a better characterization of the interface between fibers and earth concrete matrix.

452

### 453 **References**

- 454 [1] N. Kouta, J. Saliba., B. El Oifi, N. Saiyouri, Study of the effect of flax fibers on the fracture  
455 behavior of earth concrete by simultaneous application of digital image correlation and  
456 acoustic emission, 10th International Conference on Fracture Mechanics of Concrete and  
457 Concrete Structures FraMCoS-X, (2019).
- 458 [2] H. Van Damme, H. Houben, Earth concrete. Stabilization revisited, *Cem. Concr. Res.* 114  
459 (2016) 90–102. <https://doi.org/10.1016/j.cemconres.2017.02.035>.
- 460 [3] N. Kouta, J. Saliba, N. Saiyouri, Effect of flax fibers on early age shrinkage and cracking of  
461 earth concrete, *Constr. Build. Mater.* 254 (2020) 119315.  
462 <https://doi.org/10.1016/j.conbuildmat.2020.119315>.
- 463 [4] C.S. Tang, B. Shi, C. Liu, W. Bin Suo, L. Gao, Experimental characterization of shrinkage  
464 and desiccation cracking in thin clay layer, *Appl. Clay Sci.* 52 (2011) 69–77.  
465 <https://doi.org/10.1016/j.clay.2011.01.032>.
- 466 [5] D.C. Ngo, J. Saliba, N. Saiyouri, Z.M. Sbartai, Design of a soil concrete as a new building  
467 material – Effect of clay and hemp proportions, *J. Build. Eng.* 32 (2020) 101553.  
468 <https://doi.org/10.1016/j.job.2020.101553>.
- 469 [6] J. Saliba, E. Rozière, F. Grondin, A. Loukili, Influence of shrinkage-reducing admixtures on  
470 plastic and long-term shrinkage, *Cem. Concr. Compos.* 33 (2011) 209–217.

- 471 <https://doi.org/10.1016/j.cemconcomp.2010.10.006>.
- 472 [7] J. Eid, S. Taibi, J.M. Fleureau, M. Hattab, Drying, cracks and shrinkage evolution of a natural  
473 silt intended for a new earth building material. Impact of reinforcement, *Constr. Build. Mater.*  
474 86 (2015) 120–132. <https://doi.org/10.1016/j.conbuildmat.2015.03.115>.
- 475 [8] J.M. Kanema, J. Eid, S. Taibi, Shrinkage of earth concrete amended with recycled aggregates  
476 and superplasticizer: Impact on mechanical properties and cracks, *Mater. Des.* 109 (2016)  
477 378–389. <https://doi.org/10.1016/j.matdes.2016.07.025>.
- 478 [9] P. Walker, R. Keable, J. Martin, V. Maniatidis, *Rammed earth: design and construction*  
479 *guidelines*, (2005).
- 480 [10] P. Woyciechowski, P.L. Narloch, D. Cichocki, Shrinkage characteristics of cement stabilized  
481 rammed earth, *MATEC Web Conf.* 117 (2017).  
482 <https://doi.org/10.1051/mateconf/201711700178>.
- 483 [11] P. Walker, Strength, durability and shrinkage characteristics of cement stabilised soil blocks,  
484 *Cem. Concr. Compos.* 17 (1995) 301–310. [https://doi.org/10.1016/0958-9465\(95\)00019-9](https://doi.org/10.1016/0958-9465(95)00019-9).
- 485 [12] B.V.V. Reddy, K.S. Jagadish, Influence of soil composition on the strength and durability of  
486 soil-cement blocks, *Ind. Con. J.* 69 (1995) 517–526.
- 487 [13] P. Walker, T. Stace, Properties of some cement stabilised compressed earth blocks and  
488 mortars, *Mater. Struct. Constr.* 30 (1997) 545–551. <https://doi.org/10.1007/bf02486398>.
- 489 [14] J.M. Kanema, The influence of soil content on the mechanical properties, drying shrinkage  
490 and autogenous shrinkage of earth concrete, *J. Build. Eng.* 13 (2017) 68–76.  
491 <https://doi.org/10.1016/j.jobe.2017.07.006>.
- 492 [15] B. Le Runigo, *Durabilité du limon de Jossigny traité à la chaux et soumis à différentes*  
493 *sollicitations hydriques : comportement hydraulique, microtextural et mécanique*, Thèse Dr.  
494 *École Cent. Nantes - Fr.* (2008) 196.
- 495 [16] S. Diamond, E.B. Kinter, Mechanisms of Soil-Lime Stabilization, *Highw. Res. Rec.* 92  
496 (1965) 83–102. <http://onlinepubs.trb.org/onlinepubs/hrr/1965/92/92-006.pdf>.
- 497 [17] N. Kouta, J. Saliba, N. Saiyouri, Fracture behavior of flax fibers reinforced earth concrete,  
498 *Eng. Fract. Mech.* (2020) 107378. <https://doi.org/10.1016/j.engfracmech.2020.107378>.

- 499 [18] T. Sen, J. Reddy, Application of Sisal, Bamboo, Coir and Jute Natural Composites in  
500 Structural Upgradation, *Japanese J. Anesthesiol.* 64 (2015) 1181–1185.
- 501 [19] A. Laborel-Préneron, J.E. Aubert, C. Magniont, C. Tribout, A. Bertron, Plant aggregates and  
502 fibers in earth construction materials: A review, *Constr. Build. Mater.* 111 (2016) 719–734.  
503 <https://doi.org/10.1016/j.conbuildmat.2016.02.119>.
- 504 [20] H.R. Kymäläinen, A.M. Sjöberg, Flax and hemp fibres as raw materials for thermal  
505 insulations, *Build. Environ.* 43 (2008) 1261–1269.  
506 <https://doi.org/10.1016/j.buildenv.2007.03.006>.
- 507 [21] S. Sangma, D.D. Tripura, Experimental study on shrinkage behaviour of earth walling  
508 materials with fibers and stabilizer for cob building, *Constr. Build. Mater.* 256 (2020) 119449.  
509 <https://doi.org/10.1016/j.conbuildmat.2020.119449>.
- 510 [22] Ş. Yetgin, Ö. ÇAVDAR, A. Çavdar, The effects of the fiber contents on the mechanic  
511 properties of the adobes, *Constr. Build. Mater.* 22 (2008) 222–227.  
512 <https://doi.org/10.1016/j.conbuildmat.2006.08.022>.
- 513 [23] M. Bouhicha, F. Aouissi, S. Kenai, Performance of composite soil reinforced with barley  
514 straw, *Cem. Concr. Compos.* 27 (2005) 617–621.  
515 <https://doi.org/10.1016/j.cemconcomp.2004.09.013>.
- 516 [24] K. Ghavami, R.D. Toledo Filho, N.P. Barbosa, Behaviour of composite soil reinforced with  
517 natural fibres, *Cem. Concr. Compos.* 21 (1999) 39–48. [https://doi.org/10.1016/S0958-9465\(98\)00033-X](https://doi.org/10.1016/S0958-9465(98)00033-X).
- 518
- 519 [25] K. Otsuka, H. Date, Fracture process zone in concrete tension specimen, *Eng. Fract. Mech.*  
520 65 (2000) 111–131.
- 521 [26] A.K. Das, D. Suthar, C.K.Y. Leung, Machine learning based crack mode classification from  
522 unlabeled acoustic emission waveform features, *Cem. Concr. Res.* 121 (2019) 42–57.  
523 <https://doi.org/10.1016/j.cemconres.2019.03.001>.
- 524 [27] E. Tsangouri, L. Michels, M. El Kadi, T. Tysmans, D.G. Aggelis, A fundamental  
525 investigation of textile reinforced cementitious composites tensile response by Acoustic  
526 Emission, *Cem. Concr. Res.* 123 (2019) 105776.



- 527 <https://doi.org/10.1016/j.cemconres.2019.105776>.
- 528 [28] J. Saliba, D. Mezhoud, Monitoring of steel-concrete bond with the acoustic emission  
529 technique, *Theor. Appl. Fract. Mech.* 100 (2019) 416–425.  
530 <https://doi.org/10.1016/j.tafmec.2019.01.034>.
- 531 [29] A. Boniface, J. Saliba, Z.M. Sbartai, N. Ranaivomanana, J.P. Balayssac, Evaluation of the  
532 acoustic emission 3D localisation accuracy for the mechanical damage monitoring in  
533 concrete, *Eng. Fract. Mech.* 223 (2020) 106742.  
534 <https://doi.org/10.1016/j.engfracmech.2019.106742>.
- 535 [30] B. Assouli, F. Simescu, G. Debicki, H. Idrissi, Detection and identification of concrete  
536 cracking during corrosion of reinforced concrete by acoustic emission coupled to the  
537 electrochemical techniques, *NDT E Int.* 38 (2005) 682–689.  
538 <https://doi.org/10.1016/j.ndteint.2005.04.007>.
- 539 [31] J. Saliba, A. Loukili, F. Grondin, J.P. Regoin, Identification of damage mechanisms in  
540 concrete under high level creep by the acoustic emission technique, *Mater. Struct. Constr.* 47  
541 (2014) 1041–1053. <https://doi.org/10.1617/s11527-013-0113-6>.
- 542 [32] T. Chotard, A. Quet, A. Ersen, A. Smith, Application of the acoustic emission technique to  
543 characterise liquid transfer in a porous ceramic during drying, *J. Eur. Ceram. Soc.* 26 (2006)  
544 1075–1084. <https://doi.org/10.1016/j.jeurceramsoc.2005.01.048>.
- 545 [33] A.B. Hossain, J. Weiss, The role of specimen geometry and boundary conditions on stress  
546 development and cracking in the restrained ring test, *Cem. Concr. Res.* 36 (2006) 189–199.  
547 <https://doi.org/10.1016/j.cemconres.2004.06.043>.
- 548 [34] L. Assi, V. Soltangharai, R. Anay, P. Ziehl, F. Matta, Unsupervised and supervised pattern  
549 recognition of acoustic emission signals during early hydration of Portland cement paste,  
550 *Cem. Concr. Res.* 103 (2018) 216–225. <https://doi.org/10.1016/j.cemconres.2017.10.019>.
- 551 [35] T. Shiotani, J. Bisschop, J.G.M. Van Mier, Temporal and spatial development of drying  
552 shrinkage cracking in cement-based materials, *Eng. Fract. Mech.* 70 (2003) 1509–1525.  
553 [https://doi.org/10.1016/S0013-7944\(02\)00150-9](https://doi.org/10.1016/S0013-7944(02)00150-9).
- 554 [36] E.D. Dzaye, G. De Schutter, D.G. Aggelis, Monitoring early-age acoustic emission of cement

555 paste and fly ash paste, *Cem. Concr. Res.* 129 (2020) 105964.  
556 <https://doi.org/10.1016/j.cemconres.2019.105964>.

557 [37] P. Lura, J. Couch, O.M. Jensen, J. Weiss, Early-age acoustic emission measurements in  
558 hydrating cement paste: Evidence for cavitation during solidification due to self-desiccation,  
559 *Cem. Concr. Res.* 39 (2009) 861–867. <https://doi.org/10.1016/j.cemconres.2009.06.015>.

560 [38] (2015). EN (European Standards), Lime: Definitions, Specifications and Conformity criteria.  
561 EN 459-1, No Title, (n.d.).

562 [39] (2012) EN (European Standards), Composition, Specifications and Conformity criteria of  
563 common cements. NF EN197-1, No Title, (n.d.).

564 [40] F.G. Bell, Lime stabilization of clay minerals and soils, *Eng. Geol.* 42 (1996) 223–237.  
565 [https://doi.org/10.1016/0013-7952\(96\)00028-2](https://doi.org/10.1016/0013-7952(96)00028-2).

566 [41] S.A. Khattab, M. Al-Mukhtar, J.M. Fleureau, Long-term stability characteristics of a lime-  
567 treated plastic soil, *J. Mater. Civ. Eng.* 19 (2007) 358–366.

568 [42] C.A. Anagnostopoulos, Strength properties of an epoxy resin and cement-stabilized silty clay  
569 soil, *Appl. Clay Sci.* 114 (2015) 517–529. <https://doi.org/10.1016/j.clay.2015.07.007>.

570 [43] J. Page, F. Khadraoui, M. Gomina, M. Boutouil, Influence of different surface treatments on  
571 the water absorption capacity of flax fibres : Rheology of fresh reinforced-mortars and  
572 mechanical properties in the hardened state, *Constr. Build. Mater.* 199 (2019) 424–434.  
573 <https://doi.org/10.1016/j.conbuildmat.2018.12.042>.

574 [44] J. Page, F. Khadraoui, M. Boutouil, M. Gomina, Multi-physical properties of a structural  
575 concrete incorporating short flax fibers, *Constr. Build. Mater.* 140 (2017) 344–353.  
576 <https://doi.org/10.1016/j.conbuildmat.2017.02.124>.

577 [45] V. Anggraini, B.B.K. Huat, A. Asadi, H. Nahazanan, Relationship between the compressive  
578 and tensile strengths of lime-treated clay containing coconut fibres, *Acta Geotech. Slov.* 12  
579 (2015) 49–57.

580 [46] A.S. Muntohar, A. Widiyanti, E. Hartono, W. Diana, Engineering properties of silty soil  
581 stabilized with lime and rice husk ash and reinforced with waste plastic fiber, *J. Mater. Civ.*  
582 *Eng.* 25 (2013) 1260–1270. [https://doi.org/10.1061/\(ASCE\)MT.1943-5533.0000659](https://doi.org/10.1061/(ASCE)MT.1943-5533.0000659).

- 583 [47] X. Qiang, L. Hai-jun, L. Zhen-ze, L. Lei, Cracking , water permeability and deformation of  
584 compacted clay liners improved by straw fi ber, *Eng. Geol.* 178 (2014) 82–90.  
585 <https://doi.org/10.1016/j.enggeo.2014.05.013>.
- 586 [48] C. Barrier, Effects of Fiber Additives on the Desiccation Crack Behavior of the Compacted  
587 Akaboku Soil as A Material for Landfill, (2008) 141–149. [https://doi.org/10.1007/s11270-](https://doi.org/10.1007/s11270-008-9703-2)  
588 [008-9703-2](https://doi.org/10.1007/s11270-008-9703-2).
- 589 [49] E. Quagliarini, S. Lenci, The influence of natural stabilizers and natural fibres on the  
590 mechanical properties of ancient Roman adobe bricks, *J. Cult. Herit.* 11 (2010) 309–314.  
591 <https://doi.org/10.1016/j.culher.2009.11.012>.
- 592 [50] G. Srikar, G. Anand, S. Suriya Prakash, A Study on Residual Compression Behavior of  
593 Structural Fiber Reinforced Concrete Exposed to Moderate Temperature Using Digital Image  
594 Correlation, *Int. J. Concr. Struct. Mater.* 10 (2016) 75–85. [https://doi.org/10.1007/s40069-](https://doi.org/10.1007/s40069-016-0127-x)  
595 [016-0127-x](https://doi.org/10.1007/s40069-016-0127-x).
- 596 [51] G. Ye, P. Lura, K. Van Breugel, A.L.A. Fraaij, Study on the development of the  
597 microstructure in cement-based materials by means of numerical simulation and ultrasonic  
598 pulse velocity measurement, *Cem. Concr. Compos.* 26 (2004) 491–497.  
599 [https://doi.org/10.1016/S0958-9465\(03\)00081-7](https://doi.org/10.1016/S0958-9465(03)00081-7).
- 600 [52] Ş. Erdogdu, U. Kandil, M. Nas, Ş. Kurbetci, S. Nayir, Yüksek Sıcaklığa Maruz Uçucu Kül  
601 İçeren Betonun Ultrases Geçiş Hızı ve Basınç Dayanımının Değerlendirilmesi, *Nevşehir*  
602 *Bilim ve Teknol. Derg.* 6 (2017) 314–325. <https://doi.org/10.17100/nevbiltek.322416>.
- 603 [53] E.D. Dzaye, G. De Schutter, D.G. Aggelis, Monitoring early-age acoustic emission of cement  
604 paste and fl y ash paste, *Cem. Concr. Res.* 129 (2020) 105964.  
605 <https://doi.org/10.1016/j.cemconres.2019.105964>.
- 606 [54] L. Topolář, L. Pazdera, B. Kucharczyková, J. Smutný, K. Mikulášek, Using acoustic emission  
607 methods to monitor cement composites during setting and hardening, *Appl. Sci.* 7 (2017).  
608 <https://doi.org/10.3390/app7050451>.
- 609 [55] J. Bisschop, J.G.M. Van Mier, How to study drying shrinkage microcracking in cement-based  
610 materials using optical and scanning electron microscopy?, *Cem. Concr. Res.* 32 (2002) 279–

- 611 287. [https://doi.org/10.1016/S0008-8846\(01\)00671-8](https://doi.org/10.1016/S0008-8846(01)00671-8).
- 612 [56] A. Aldaood, M. Bouasker, M. Al-mukhtar, Impact of wetting – drying cycles on the  
613 microstructure and mechanical properties of lime-stabilized gypseous soils, *Eng. Geol.* 174  
614 (2014) 11–21. <https://doi.org/10.1016/j.enggeo.2014.03.002>.
- 615 [57] B. Kim, W.J. Weiss, Using acoustic emission to quantify damage in restrained fiber-  
616 reinforced cement mortars, *Cem. Concr. Res.* 33 (2003) 207–214.  
617 [https://doi.org/10.1016/S0008-8846\(02\)00978-X](https://doi.org/10.1016/S0008-8846(02)00978-X).
- 618 [58] D.G. Aggelis, D. V. Soulioti, N. Sapouridis, N.M. Barkoula, A.S. Paipetis, T.E. Matikas,  
619 Acoustic emission characterization of the fracture process in fibre reinforced concrete,  
620 *Constr. Build. Mater.* 25 (2011) 4126–4131.  
621 <https://doi.org/10.1016/j.conbuildmat.2011.04.049>.
- 622 [59] J.H. Kurz, F. Finck, C.U. Grosse, H.W. Reinhardt, Stress drop and stress redistribution in  
623 concrete quantified over time by the b-value analysis, *Struct Heal. Monit.* 5 (2005) 69–81.
- 624 [60] T. Shiotani, K. Fujii, T. Aoki, K. Amou, Evaluation of progressive failure using AE sources  
625 and improved b-value on slope model tests, *Prog. Acoust. Emiss VII.* 7 (1994) 529–534.
- 626 [61] D.G. Aggelis, T. Shiotani, S. Momoki, A. Hirama, Acoustic emission and ultrasound for  
627 damage characterization of concrete elements, *ACI Mater J.* 106 (2009) 509–514.
- 628 [62] F. Lagier, X. Jourdain, C. De Sa, F. Benboudjema, J.B. Colliat, Numerical strategies for  
629 prediction of drying cracks in heterogeneous materials : Comparison upon experimental  
630 results, 33 (2011) 920–931. <https://doi.org/10.1016/j.engstruct.2010.12.013>.
- 631