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Effect of bio-stabilizers on capillary absorption and water vapour transfer into raw earth

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44 **Conflicts of interest**

45 The authors declare no conflict of interest.

46 **Availability of data and material**

47 The data that support the findings of this study are available from the
48 corresponding author, S. G., upon reasonable request.

49 **Authors contributions**

50 Conceptualization, S.G., D.R., A.P.; Validation, S.G., D.R., A.P., T.C., F.C., S.P.;
51 Investigation T.C., S.G., D.R., A.P., F.C., S.P.; Writing-original draft, S.G.;
52 Writing-review and editing D.R., A.P., F.C., S.P., T.C.

53 **Abstract**

54 While raw earth can be used as a structural material, its water sensitivity limits its
55 use in modern construction. Earth has a strong affinity with water. Both water
56 imbibition and water vapour transfer have to be taken into consideration. This
57 affinity can be a positive point to regulate indoor ambience, but it can also
58 negatively affect earth's mechanical properties. To avoid this possible detrimental
59 effect, bio-based additions are used all around the world to stabilize, protect from
60 water and enhance the mechanical properties of earth-based building materials. Bio-
61 based stabilizers have to limit liquid water absorption and maintain mechanical
62 properties without acting as a water vapour barrier that can lead to durability issues.
63 In this article, capillary water absorption and water vapour permeability

64 measurements are performed for combinations of various earths and bio-based
65 additions (linseed oil, xanthan gum, casein, alginate and tannins). Linseed oil and
66 xanthan gum help to protect the earth-based samples from absorbing liquid water
67 without affecting the water vapour transfer in these materials.

68 **Keywords:** Earth-based building materials; Bio-based additions; Capillary
69 absorption; Water vapour permeability; Mechanical strength

70 **1. Introduction**

71 Earthen construction, which is a global traditional building technique that has
72 been used for thousands of years, has been, to some extent, neglected in the last
73 century due to the development of the steel and concrete building industries, even
74 if this vernacular technique has survived in both industrialised and developing
75 countries. In the context of global warming that forces humans to reconsider the
76 global economic growth paradigm, this construction technique is now regaining
77 interest in the building industry, mostly because of its low environmental impact,
78 its recyclability and its availability locally [1–7].

79 However, even if raw-earth seems to be a promising building material, it also
80 presents some disadvantages, such as its significant variability and durability
81 issues that limits the democratization of its use [8, 9]. Such a large variability of
82 resources (using various processing routes) is also leading to unguaranteed
83 minimal mechanical performances [10–15]. Furthermore, the water sensitivity of
84 earth, in both liquid and vapour phases, is often quoted as its main weakness
85 related to durability because it can alter the material and strongly reduce its
86 mechanical properties. On the contrary, these hygroscopic characteristics of raw-
87 earth are also strong assets, as this material is able to adsorb or release moisture
88 from the atmosphere of a building and thus naturally regulate the indoor hygro-
89 thermal conditions [8, 9, 16–19]. Hence, it seems to be important to aim to keep
90 the initial mechanical properties of this building material, by protecting it from
91 water in a liquid and vapour phase without constraining the diffusion of water
92 vapour.

93 Several options have been studied to optimize, preserve and maintain the
94 mechanical properties and enhance the water durability of this material. One
95 option is to stabilize it with hydraulic binders [20–26] but it is commonly agreed

96 that their use should be avoided to aim for the lowest embodied energy building
97 material [27–30]. Another option is to deflocculate the clay particle's
98 microstructure to optimize the rheology of the material and thus its final
99 mechanical properties [4, 25, 31–33] by using a dispersant such as Sodium-
100 hexametaphosphate (Na-HMP) that acts as a superplasticizer on cement particles
101 in concrete. Since these dispersants are chemical and industrial products, it seems
102 wise to minimize their content in the mix.

103 Other options could have less impact on the environment. Thus, the optimization
104 of the processing method could systematically improve the final mechanical
105 properties of the material by increasing its dry density [14, 34–37]. In addition, to
106 substitute the previously cited additions that increase the water durability and the
107 mechanical properties of the material during its life, some bio-based additions
108 could be used. From empirical knowledge, several bio-based additions have been
109 used around the world to enhance earthen building materials [38, 39]. The
110 potential of bio-polymers to enhance mechanical properties, water durability and
111 the rheological behaviour of raw earth building materials has also been
112 highlighted in recent research works [13, 38, 40–49].

113 Firstly, in this study, the impact of selected bio-based additions on the water
114 durability of different earthen materials is assessed by using a capillary absorption
115 test. Discussion is supported by apparent contact angle observations carried out on
116 compacted earth samples. Then the effect of some of the most promising additions
117 on the water vapour permeability of the material is investigated. The main aim of
118 this study is to highlight that some bio-polymers and bio-based additions, by
119 various mechanisms, can strongly enhance the water durability, and therefore the
120 mechanical properties of earthen materials during in-service life without affecting
121 the transfer of water vapour in their pores, which is one of their most useful
122 physical properties.

123 **2. Materials and methods**

124 **2.1. Materials**

125 *2.1.1. Tested earth materials*

126 This study is part of a regional project in Brittany, France that aims to develop
127 earthen construction on a wider scale. Three kinds of earth (K, R and S)
128 representative of the variability of the local resources were studied. All of these
129 resources have already been studied in previous works [13, 14, 46, 47].

130 **Kaolin based soil: K**

131 The kaolin based soil (K) is a model earth reconstituted from quarry materials and
132 composed of kaolin coming from Ploemeur, with the size of the particles ranging
133 from 0 to 10 μ m ($d_{50} = 4\mu$ m), EN-196.1, standard sand (with particles having a
134 diameter of up to 4 mm) and a finer river sand provided by the same supplier as
135 the standard sand and having a particle size distribution ranging from 63 to 200
136 μ m. The kaolin has a specific gravity of 2.65 and a specific surface area of 10
137 m²/g. The final mix obtained using a Dreux and Gorisse method [50] in order to
138 optimize the granular compacity is composed by mass of 17% of kaolin, 23% of
139 river sand and 60% of standard sand. This soil K has a small methylene blue value
140 of 0.2 and can be qualified as low active earth. The Plasticity Index of the soil K
141 (<200 μ m) is about 13.4 with a liquid limit of 32% and a plastic limit of 18.6%. It
142 has already been shown that with these types of low-active soils, the effect of a
143 bio-based stabilizer is significant [46, 47].

144 **Local soil extracted in Redon, Brittany (rammed earth): R**

145 This studied material R is usually processed using a rammed earth technique. It is
146 a clayey sand with 50% of its particles finer than 10 μ m and 95% of its particles
147 finer than 2 mm. The soil particles are a mix of quartz and various types of clay:
148 kaolinite, illite and chlorite. The Plasticity Index of the soil R is about 14.5 with a
149 liquid limit of 36.5% and a plastic limit of 22%. This soil has a methylene blue
150 value of 0.7.

Local soil extracted in St Sulpice, Brittany (cob earth): S

The studied earth S is usually processed using the cob earth technique. It is a fine clayey soil with more than 70% of its particles finer than 20 μm . The soil particles are a mix of quartz and various types of clay: kaolinite, illite and some traces of vermiculite (swelling clay) as determined by XRD analysis. The Plasticity Index of the soil is about 16.5 with a liquid limit of 46% and a plastic limit of 29.5%.

This soil has a methylene blue value of 1.1.

The particle size distribution (PSD) of the three tested soils obtained by laser diffraction (LD) and sieving or hydrometer analysis (HA) and sieving, are plotted in Fig. 1. It can be seen that the PSD slightly varies between methods. This is due to the fact that both methods assumed that the particles are spherical and that this assumption affects the results of each technique differently (sedimentation velocity for HA and light diffraction for LD) leading in both cases to slight measurement errors.

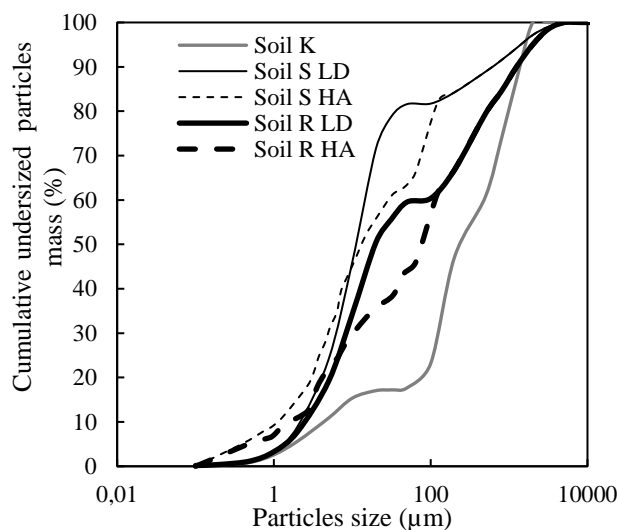


Fig. 1 Particle size distribution of soils K, R and S

2.1.2. Bio-based additions

Several bio-based additions, the biopolymers, were also tested. After a review of previous studies and traditional recipes [13, 38, 40–49] different bio-based additions, that can be classified into 4 different families were chosen. Their potential to enhance raw-earth based materials are described in the following subsections. As in the antecedent studies [13, 47], the dosages of the different additions were determined in order to optimize; the dry mechanical strengths of different earthen materials, their rheological behaviour, and their mechanical

175 strengths while their water content increases. The tested dosages were also
176 determined through preliminary tests in order to choose the range of biopolymer
177 content that provides significant improvement.

178 **Polysaccharides: Alginates (Alg), Xanthan Gum (Xan), Cellulosic Glue (CG)**

179 Polysaccharides are long chains of sugars that can react with water and form a gel.
180 Combined with earthen materials, they can form microscopic bonds between the
181 clay layers [38]. In this study Alginates (Alg), Xanthan Gum (Xan), Cellulosic
182 Glue (CG) have been selected because of their availability locally.

183 The chains of alginate (Alg) can attach themselves and form a cross-linked
184 isotropic insoluble gel [51]. Alginates have already been tested in past studies as a
185 stabilizer for raw earth material [13, 41, 43, 46, 47]. In this study, the alginate
186 used is a white powder of sodium alginic salt Cimalgin HS3[®] provided by
187 Cimaprem (Redon, France). The chosen dosage of alginate is 1% of the mass of
188 the dry earth.

189 Produced by fermentation of glucose or sucrose by the “*Xanthomonas campestris*”
190 bacterium, Xanthan gum (Xan) is a highly stable branched heteropolysaccharide
191 [49, 52, 53]. This gum is mostly used for its unique rheological properties in the
192 food industry and several other sectors. Recent studies have highlighted that
193 Xanthan gum (Xan) could strongly increase the mechanical properties of soils and
194 earthen materials [49, 54]. In addition, these long macromolecules could adsorb
195 onto the surface of clay particles and thus protect earthen materials. For this study,
196 two dosages of xanthan gum, provided by the “wake-up-easy sportfood”
197 company, were retained at 0.5% and 1% of the mass of the dry earth.

198 According to Anger et al. [38], many different vegetal glues are used all around
199 the world to enhance the properties of earthen constructions. Thus, a vegetal
200 cellulosic glue addition (CG) was also tested. In this study, an industrial 100%
201 vegetal wallpaper glue was used with a dosage of 6.25 grams per litre of mixing
202 water.

203 **Lipids: Linseed Oil (LO) and Vegetal-based Varnish (VV)**

204 Lipids are the fat content of living systems. These biopolymers are insoluble in
205 water and are used in earthen construction for their hydrophobic nature, notably in
206 outside coatings of vernacular buildings [39].

207 Linseed oil (LO) is made up of 90% of polyunsaturated fatty acids that are part of
208 the lipids. These fatty acids are amphiphilic with a polar head and a non-polar
209 unbranched chain. During drying, the oil molecules oxidise and polymerise by
210 creating a cross-linked network. This phenomenon, called siccation, is very
211 slow and can be catalysed by the presence of oxygen, the presence of metallic ions
212 and the temperature or the light energy [55–57]. For this study, two doses of
213 linseed oil mixed in the mass of the earthen material were chosen at 1% and 2% of
214 the mass of the dry earth. The effect of a brush application of this oil on the
215 surface of the earthen materials was also tested.

216 A vegetal-based varnish (VV) consisting of a vegetal oil emulsion without any
217 volatile organic compounds or formaldehyde was also tested. It was coated with a
218 brush on the surface of the different earthen materials.

219 **Proteins: Casein (Cas)**

220 The proteins are macromolecules of living systems that are usually amphiphilic.
221 Casein (Cas) has both hydrophilic and hydrophobic properties. It has been used
222 previously in vernacular raw earth construction as a coating or mixed up in the
223 mass of the material to reinforce its water durability [38, 39]. As in a previous
224 study [13], micellar casein powder was used at a dosage of 1% of the mass of the
225 dry earth.

226 **Tannins: Chestnut Tannins (Tan), Oak Seed Extract (OSE)**

227 Tannins are complex polyphenols that can be encountered in most plants [44].
228 Tannins have been used since antiquity to make leather from hides. Also, in raw
229 earth traditional construction, tannins are used to strengthen coatings of earthen
230 walls in relation to their water durability [39, 45]. According to other studies,
231 under certain conditions, tannins can also act as a dispersant for clay particles [13,
232 38, 42] and thus could increase the mechanical and water durability properties of
233 earthen blocks.

234 In this study, the first type of tannins used were chestnut tannins (Tan) originally
235 sold for wine making applications. Here, the tested dosage was 0.4% in the mass
236 of the dry earth as in previous works [13].

237 Also, Hafshejani et al. [44] have highlighted the fact that an Oak Seed Extract
238 (OSE) could be used as a dispersant for bentonite drilling mud. In this study, the
239 addition of an OSE powder sieved at 315 μm , was studied. In the case of the
240 addition of OSE powder, the dosage was 0.8% in the mass of the dry soil.

241 2.1.3. *Other materials*

242 **Sodium-hexametaphosphate (HMP)**

243 As previously described, Hexametaphosphate (HMP,) when added at a small
244 concentration, can be an efficient dispersant for clay particles [4, 25, 32, 33]. The
245 use of a dispersant can be efficient to minimize and optimize the porosity of
246 earthen materials, and thus help to increase their mechanical and water durability
247 properties. In order to highlight the effect of an efficient dispersant on the water
248 durability of earthen materials, Hexametaphosphate (HMP) was used as it has
249 been used in previous studies [13, 14, 32, 47]. Also, some of the bio-based
250 additions could have a thickening effect on the rheological properties of the raw
251 earth-based materials [13, 47]: thus, the addition of an efficient dispersant in the
252 mix design is important. The dosage chosen here was 0.0425% in the mass of the
253 dry soil. In this study, it is assumed that HMP acts only on clay particles to ease
254 the sample's processing [47]. As a consequence, it is here considered as a first
255 approximation that HMP does not interact with bio-based additions.

256 **Hydrated Lime (HLime)**

257 One of the aims of this study is to highlight the fact that the use of a mineral
258 binder is not the only option to protect raw earth materials from water. Therefore,
259 for comparison purposes, tests were also conducted on earth samples stabilized
260 with hydrated lime at a content of 5% in the mass of the dry soil.

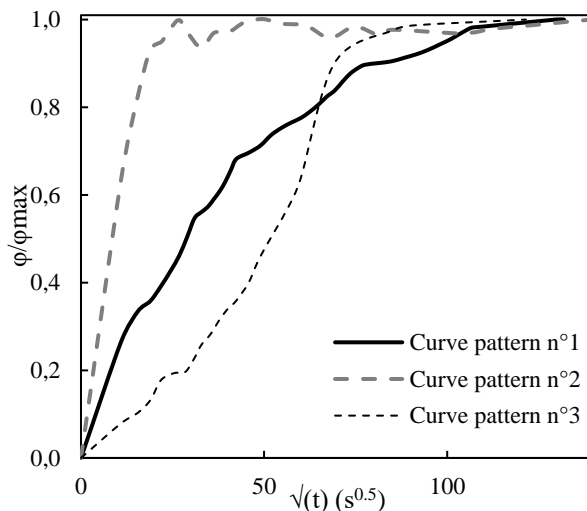
261 **2.2. Methods**

262 *2.2.1. Capillary water absorption tests*

263 As several studies have already reviewed [38, 39], some bio-based additions can
264 have the ability to reduce the water sensitivity of earthen materials. In order to
265 highlight this property, a capillary absorption test was developed, inspired by the
266 AFNOR NF EN 13057 [58] French standard and the AFNOR XP P 13-901 [59]
267 French standard for cement stabilized compressed earth blocks.
268 20*40*40mm³ cast samples of earth-based material, mixed with the previously
269 listed different bio-based additions were placed on: a filter paper, a nylon grid, on
270 a water-soaked sponge and in a flush water bath. All of the samples were cast at
271 the same consistency showing the same plastic behaviour. To obtain such targeted
272 consistency, the water content of each mix (Earth+addition) was adapted,
273 depending on the impact of the bio-based addition on the earth's rheology. The
274 sample weight variation was recorded during the experiment. The aim here was to
275 highlight the effect of bio-based additions on the liquid water capillary absorption
276 of earth-based samples using a simple laboratory test. The tests were conducted
277 on dried (after one week in a 50°C heat chamber) samples. Hence, the initial water
278 content of the tested samples was zero as Hall [60] suggests. The test started as
279 soon as the dry sample was placed on the filter paper/nylon grid/sponge device.
280 The atmosphere in the test room was maintained at a temperature of 20°C and a
281 relative humidity of 50%. The water level in the water bath remained constant
282 during the test. The test stopped when the water content of the samples no longer
283 evolved.

284 As already described earlier, the sample weight change versus the elapsed time t
285 was followed. By plotting the cumulative absorption inflow ϕ (kg/m², weight of
286 absorbed water by unit area) versus the square root of time ($t^{0.5}$), an absorption
287 curve with two phases was obtained (Fig. 2). During the first phase, the absorption
288 kinetic was mainly governed by the capillary forces: the mass of absorbed water
289 in the sample quickly increased. Then, the second phase started when the water
290 level reached the top of the sample. During this phase, the diffusion of the
291 entrapped air in the sample's pores governed this kinetic [61]. In Fig. 2, the
292 transition in the second phase is visible at $t^{0.5}=50 \text{ s}^{0.5}$.

293 Following these observations, two different criteria were used to describe the
 294 water capillary absorption of the tested samples. First, a capillary absorption
 295 coefficient A_{cap} ($\text{g.m}^{-2}.\text{s}^{-0.5}$) was determined to highlight the capillary absorption
 296 velocity during the first phase. In order to take the variation of the dry density of
 297 the samples into account, the value of A_{cap}/n was considered, where “n” is the
 298 apparent porosity of the sample. Then, the capillary moisture content w_{cap} , that is
 299 the moisture content of the sample when the second phase of the absorption curve
 300 started, was determined. In order to determine the A_{cap} value, different methods
 301 that stem from several studies [59–62] can be used: Hall’s method, the 1-tangent
 302 method and the fix-time method.
 303 More than 80 capillary absorption tests were conducted on different formulations
 304 of different earths. As described earlier, the absorption cumulative inflow ϕ
 305 (kg/m^2) versus the square root of time ($t^{0.5}$) was obtained. These curves
 306 highlighted the capillary absorption pattern of each sample. During the data
 307 analysis of these tests, three distinct absorption kinetics were observed. The three
 308 types of pattern are plotted in Fig. 2. Here it is important to find the most efficient
 309 and accurate method to derive the capillary absorption coefficient from each type
 310 of pattern. For a better illustration, the dimensionless cumulative inflows (inflow
 311 divided by maximal value for each case) are plotted. On the first pattern, phases 1
 312 and 2 are not clearly distinct. On the second pattern, the two phases are clearly
 313 distinct. In both the first and second patterns (most of the time) the shape of the
 314 first phase is slightly concave. On the third pattern, the two phases of absorption
 315 are distinct but the shape of the first phase is convex.



316
 317 **Fig.2** Different patterns of capillary absorption

318 The 1-tangent method consists in performing a linear regression on the linear part
319 of the first phase of absorption. This method consists in the estimation of the slope
320 of this tangent and is based on the diffusivity equation [62, 63]. A simplified
321 expression of this equation is shown in Eq. (1).

$$\frac{m_t - m_0}{A} = A_{\text{cap}} \times t^{0.5} + k_1 \quad (1)$$

323
324 In this experiment, this method was chosen because it seems to be the most
325 versatile for the data's interpretation. This method seems to provide accurate A_{cap}
326 values for the second and the third type of patterns, but approximate results [64]
327 for the first type of pattern. However, the obtained A_{cap} values for the first type of
328 pattern can remain consistent if the time range used for the computation is
329 carefully chosen.

330 This 1-tangent method efficiently describe the material's average behaviour
331 during the first absorption phase and can be used for each type of pattern.
332 In order to take into account density variation between samples, A_{cap} values were
333 divided by the apparent porosity (n) of the tested samples before comparison. In
334 each case, w_{cap} was also determined at the end of the second phase, in order to
335 take into account of the different capillary absorption behaviours.

336

337 2.2.2. Contact angle measurements

338 Each impact of a bio-based addition on the water durability of different earthen
339 materials was induced by a protection mechanism at the microscopic scale of the
340 material. In the case of some identified efficient bio-based additions, contact angle
341 measurements of water droplets on the surface of different formulations of the soil
342 S were conducted, in order to highlight those mechanisms. Soil S was chosen
343 because it presents the highest clay content, the highest water affinity and is
344 supposed to react strongly with water droplets. Thus, if an addition is able to limit
345 the interactions between clays and water, it would be easily noticeable for the soil
346 S. Hence, the contact angle of a sessile droplet of water on the surface of earthen
347 materials was studied using a DSA30 drop shape analyser (Krüss GmbH). Tests
348 were performed on dry samples of the soil S combined with selected additions in a
349 room at a temperature of 20°C and a relative humidity of 50%. Sessile droplets of

15 μ L of water were created using a vertical syringe (needle with an outer diameter of 1.835 mm). The contact angle was thus measured from a shape analysis based on the Young-Laplace equation at the interface between the water droplet and the material. Such a measurement is usually performed on a non-porous surface in order to obtain surface properties of the solid. In this study, the observation of the kinetic of infiltration of a single droplet obtained evidence of potential infiltration resistance, for example due to hydrophobic properties, provided by a bio-stabiliser. Tests were conducted on samples having the same densities, that had been processed by extrusion, the different earth mix being at a same given plastic consistency. To obtain this constant consistency, the water content of each mix was slightly modified, since each addition is modifying the rheological behaviour of the mortar at the fresh state. The evolution of the contact angle versus time was followed for different formulations of soil S. The analysis of the different contact angles obtained could be a strong indicator of the change in infiltration behaviour caused by the addition of the bio-based stabiliser that strengthens earthen materials in relation to their water durability.

2.2.3. *Water vapour permeability tests*

Some bio-based additions used to limit the water capillary absorption and protect the materials may also limit the water vapour transfer. The water vapour permeability of a material characterises its ability to transfer moisture under a vapour pressure gradient. It includes vapour transfer by diffusion, vapour transfer by effusion and liquid transfer (linked to capillary condensation)[65–67]. In this study, the water vapour permeability was measured following the recommendations of the cup method international standard [68] described in the Collet et al. study of hemp concretes [65]. The test was performed under isothermal conditions (23°C) for a given set of relative humidity (0/50 %RH). Before the measurement, the samples were firstly dried in an oven at 60°C and then stabilised at (23°C, 50%RH). The stabilisation was reached when the variation of mass was lower than 0.01% between three consecutive weighing's at a 24 hours interval. Five samples were tested for each formulation. Four formulations were tested in order to study the effect of additions on water vapour permeability. The selected soil was chosen according to its strength and its ability to be shaped into cylinders of 2cm in height and 11 cm in diameter,

383 representative of the material, that were cast at the same “suited for casting”
384 consistency. The chosen additions were selected according to their potential to
385 strongly reduce the water sensitivity of earthen materials. Thus, the selected
386 formulations were: Soil S without additions, Soil S-HMP, Soil S-HMP-Xan(0.5%)
387 and Soil S-HMP-LO(2%).

388 2.2.4. *Compressive strength tests*

389 Finally, in order to highlight the fact that an increase of water durability leads to
390 an increase of the in situ mechanical properties of earthen materials, the
391 compressive strength of several cast samples of chosen formulations of the soil S,
392 were put into a wet chamber (20°C, 98%HR) for different durations. The samples
393 were cast at the same “suited for casting” consistency in order to obtain similar
394 densities. Compressive strengths at the dry state were also measured for all of the
395 formulations. The compressive tests were carried out with a sample of an aspect
396 ratio following the adopted procedure of previous studies [13, 14, 47].

397 **3. Bio-based stabilizers influence on capillary** 398 **water absorption of raw-earth based materials:** 399 **different mechanisms**

400 First, as some of the studied bio-based additions can enhance the water durability
401 of earthen materials, several capillary absorption tests, previously described, were
402 conducted on different formulations of the three studied soils. In the following
403 subsections, an analysis of these tests is conducted, focusing on the impact of the
404 bio-based additions on these absorption behaviours and on the distinct
405 mechanisms involved in the capillary absorption limitation.

406 **3.1. Capillary absorption tests: results and bio-polymer effects**

407 3.1.1. *Various capillary absorption behaviours for different earths*

408 Firstly, the results obtained for the three different soils without any addition
409 highlighted their variability of behaviours regarding the capillary absorption due
410 to their various compositions. The clay content, the particle size distribution and

the nature of the clays of each tested soil leads to distinct A_{cap} and w_{cap} values that could help to refine the understanding of earthen material behaviours. The capillary absorption curves of each of the three different earthen materials are plotted in Fig. 3. The A_{cap} (determined with the 1-tangent method) and w_{cap} values, as well as the apparent porosity value n , of each material are shown in Table 1.

<i>Soil</i>	<i>K</i>	<i>R</i>	<i>S</i>
$A_{cap} \text{ (g.m}^{-2}.s^{0.5})$	26	167	231
$W_{cap} \text{ (kg/m}^3)$	285	513.7	665
Apparent porosity n	0.26	0.32	0.36
$A_{cap}/n \text{ (g.m}^{-2}.s^{0.5})$	100	514	647

Table 1 Capillary absorption results for soils K, R and S.

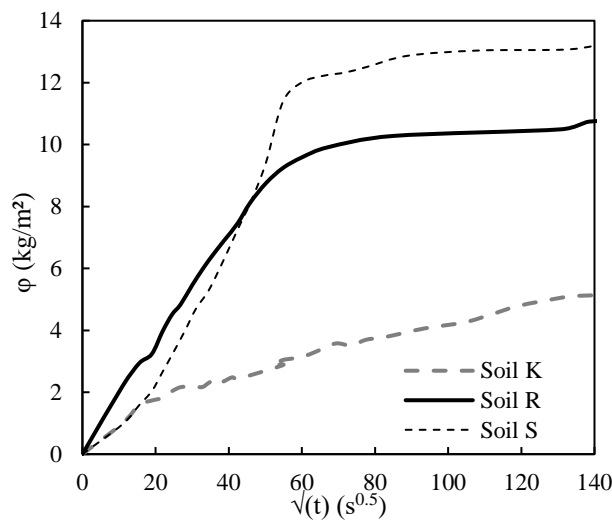


Fig. 3 Capillary absorption behaviours of soils K, R and S

The soil K displays two slightly different absorption phases (Pattern n°1/Pattern n°2), and a rather slow capillary absorption (low A_{cap}) with a low w_{cap} . In this soil, the low content of clay particles (approximately 10% of particles finer than 2 μm) and their limited specific area (pure kaolinite) can explain the low values of A_{cap} and w_{cap} .

The soil R has a rather low clay content and its clayey part is composed of kaolinite, chlorite and illite that present higher specific areas than kaolinite. These types of clay are relatively stable clays. The absorption curve of the soil R highlights two clearly distinct capillary absorption phases (Pattern n°2), the shape

of the first phase also highlights a faster but constant capillary absorption (Higher A_{cap}) that leads to a stable second phase that displays a higher w_{cap} value.

Secondly, the finest soil S has a higher clay content than the soil R, and its clayey part is composed of kaolinite, illite and some traces of vermiculite (swelling clay) that presents a high specific area. Again, the capillary absorption curve displays two distinct main phases. However, during the first phase, the absorption flow was initially quite slow and then a strong acceleration of the absorption was easily noticeable (Pattern n°3). This is probably due to the nature of the fine fraction of the soil S, these clay particles being more active and reacting more strongly with the initially adsorbed water after a first period of absorption. Vermiculite clays are expanding clays that are composed of thin layers linked by exchangeable cations (Mg^{2+}) that present high hydration capacities [42, 69]. In this case, during the first phase of absorption it is likely that the first period of slower absorption is linked to the only “common” capillary forces as already observed for soils K and R.

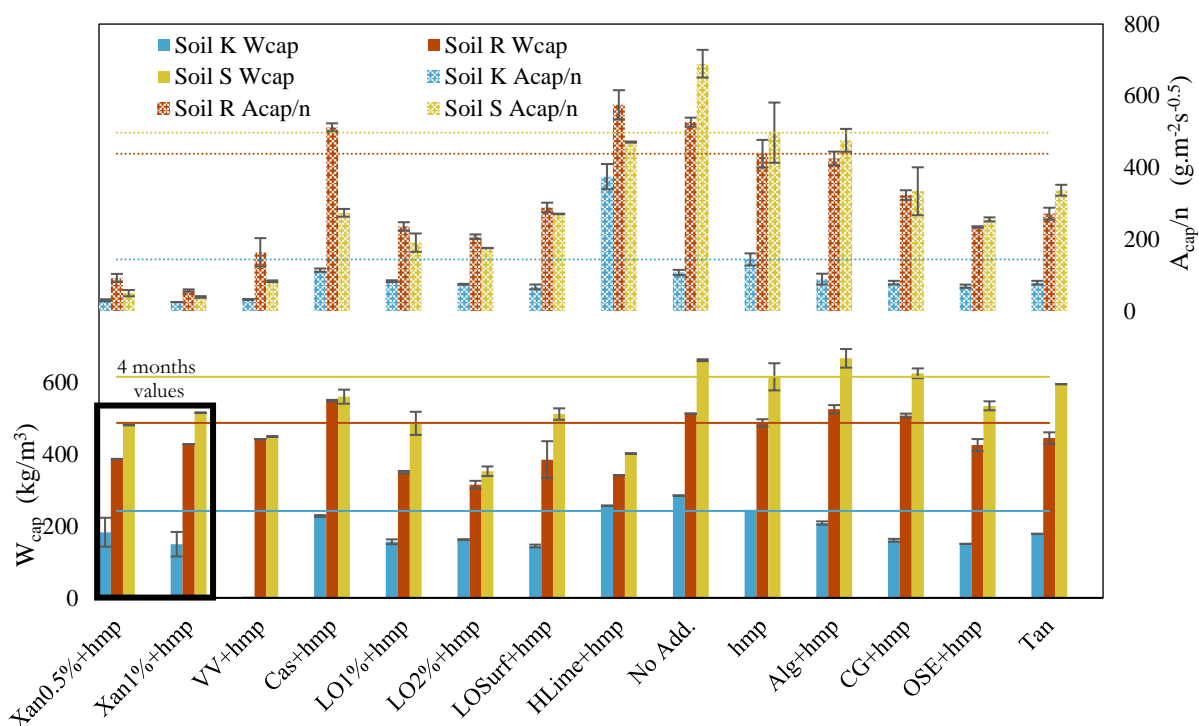
Then, when the acceleration is noticeable on the absorption curve, it is likely that the absorption is driven by both the capillary forces and the hydration of the interlayer cations. That could explain the acceleration of capillary absorption during the first phase, with the highest value of A_{cap} . The soil S is also the finest of the soils tested, and it is likely that its pores are very small in a strongly ramified porosity. That could also explain the higher velocity of the capillary absorption in the first phase, in view of the compositions and behaviours of the two other soils (K and R, coarser soils, with a lower porosity likely to be less ramified). It would be interesting to link those results with estimations of the mean size of the pores of each soil because capillary forces are driving the absorption phenomenon [70]. Note that the properties of the porous network can be linked to the clay content, the nature of the clayey part and the specific area of each soil and can partly explain the absorption kinetic ranking between tested soils [10, 71, 72].

Finally, these capillary absorption curves are linked to two main physical processes: the capillary absorption in a porous construction material directly linked with the porosity of the material, and the adsorption of water on the surface of the clay particles that could catalyse this capillary absorption of water, and affect this phenomenon.

3.1.2. *Effect of bio-based additions on capillary absorption*

As stated previously, the impact of several additions on the capillary absorption behaviours of the studied soils were followed. For each combination (Soil + addition), at least 2 samples were studied, and the mean values of w_{cap} and A_{cap} balanced by their apparent porosities were obtained and plotted in Fig. 4. In this Figure, the w_{cap} and A_{cap} values of each combination are compared to reference values obtained for the soils combined with HMP only, since in almost all of the studied combinations HMP was added. HMP was added in the mix for most of the studied bio-based additions in order to ease the fabrication of the samples and to optimise their densities as in previous studies [47]. It also appears that HMP seems to have a limited effect on the capillary absorption properties of the three studied soils. If some interactions between biological stabilisers and HMP may have occurred, they have not been taken into account in this study. HMP was just supposed to act as a dispersant for the clay particles and to help to densify each sample. Thus, for a combination of a soil with HMP and a given bio-based addition, it was considered that the change observed in the capillary absorption properties could be attributed to the effect of the bio stabiliser. The w_{cap} and $A_{cap/n}$ values obtained for the soils without any addition are also plotted in this Figure. First, it is easily noticeable that each addition seems to have the same effect on the capillary absorption behaviours of the three soils. Apart from a few exceptions, for each of the studied combinations, the $A_{cap/n}$ and w_{cap} values are the highest for soil S and the lowest for soil K. The previous observations and conclusions are thus borne out. It also appears that the effect of the bio-based additions that are slowing the water capillary absorption are more noticeable on soil S. For these additions (Xan, VV, LO, CG, OSE, Tan) an average of a 61% reduction of the $A_{cap/n}$ value is visible for soil S, when the average reduction is about 58% for soil K and 52% for soil R. Since the bio-based additions are probably interacting first and foremost with the clayey parts of the soils, their influence should be more visible on the finest soils that present the highest clay content and specific areas. “Soils+HLime”, “Soils with no additions” and “Soil R+Cas” present higher $A_{cap/n}$ values higher than the reference soil (Soil+HMP). It can be noted that these formulations are presenting the lowest dry densities, potentially showing the porosity effect on capillary absorption and the $A_{cap/n}$ value. Thus, other tests will

495 be conducted in order to better understand the influence of the dry density of a
 496 given material on its capillary absorption behaviour in further studies.
 497 For some additions that are thickening the material (Linseed Oil, Caseine,
 498 Xanthan gum, Alginate, Cellulosic glue), a variation in the final dry density
 499 cannot be avoided. The analysis of these A_{cap}/n values is thus relevant to highlight
 500 the effect of the selected bio-based additions on the capillary absorption behaviour
 501 of earthen materials. The w_{cap} values are also giving some hints on those capillary
 502 absorption behaviours. For all of the tested samples, the w_{cap} values were obtained
 503 after 24 hours of the experiment, except for the addition of xanthan gum: in these
 504 cases, the w_{cap} was never reached (no mass stabilisation) and the given values
 505 were calculated after 4 months of the experiment.



506
 507 **Fig. 4** A_{cap}/n values and w_{cap} values for several combinations of soils K, R and S
 508 For the addition of OSE and Chestnut tannins, the water capillary absorption
 509 seems to be slowed (A_{cap}/n value is decreased by 20% in comparison with the
 510 reference value A_{cap}/n obtained with only HMP), but their effect on the water
 511 capillary absorption behaviours of earthen materials appears to be quite low. The
 512 same observations can be made for the additions of casein, alginates and
 513 cellulosic glue.
 514 Concerning the addition of a Vegetal Varnish on the surface of the earthen
 515 materials, at first sight, it caused a strong reduction of the water capillary

516 absorption for all of the samples (A_{cap}/n values are divided by a factor ranging
 517 from 3 to 5). However, and despite a reduction of the w_{cap} values, it also appeared
 518 that as soon as the absorbed water had crossed the varnish layer, the sample
 519 started to swell and crack. The vegetal varnish could thus increase the water
 520 durability of earthen materials until a certain limit.
 521 For the addition of linseed oil (LO 1% or 2% in the mass of the material, or
 522 LOSurf on the surface of the samples), all of the obtained A_{cap}/n values are at least
 523 halved (Fig. 4). For all the tested proportions and cases, this observation can be
 524 made. This strong reduction of the A_{cap}/n values is probably linked to the highly
 525 hydrophobic character of dried linseed oil that repels water from the material. The
 526 w_{cap} values decrease by around 30% for all the tested proportions and cases.
 527 However, throughout the duration of the tests, the samples that stabilized with
 528 linseed oil in the mass (1% or 2%) do not display any signs of swelling and
 529 maintain a strong apparent cohesion, visible after 72 hours of testing. These
 530 observations cannot be made with the addition of linseed oil on the surface of the
 531 materials: after 24 hours of testing, a strong swelling of the samples is visible and
 532 their cohesions are highly jeopardized, such as observed for the reference samples
 533 (Soils+HMP). For the addition of linseed oil in the mass, it resulted as if the
 534 linseed oil had covered the surface of the pores of the material and thus could
 535 protect the clayey binder of the material from interlayer water infiltration. That
 536 could also explain the low w_{cap} values and the lack of swelling of the materials
 537 stabilized with linseed oil in the mass.
 538 Then, the addition of different contents of Xanthan gum was also tested and this
 539 bio-based addition seemed to be the strongest water capillary absorption reducer
 540 for earthen materials. In all of the different cases, the A_{cap}/n values were divided
 541 by a factor ranging from 5 to 10. The absorption kinetic in the first phase was
 542 strongly reduced in this way. In addition, the water capillary absorption was so
 543 much reduced, that the transition of this phenomenon in phase 2 was never
 544 reached, even after 4 months of the experiment. Because of phase 2 never having
 545 being reached, the w_{cap} value cannot be precisely determined. The calculated
 546 “ w_{cap} ” values after 4 months were still lower than the references values.
 547 Furthermore, after 4 months of testing, the samples did not display any signs of
 548 swelling and maintained a strong apparent cohesion. It is very likely that the
 549 Xanthan gum was forming microscopic bonds between the clay layers [38] in the

550 material, creating another network of links between the particles of the soil.
551 However, the risk of fungal proliferation in this kind of material exposed to
552 constant moisture should be taken into consideration [73].

553 Finally, the addition of Hydrated Lime in the materials was tested and it appeared
554 that this addition did not reduce the A_{cap}/n values. However, all of the tested
555 samples of earthen materials combined with hydrated lime presented w_{cap} values
556 divided by 1.5. In addition, during all the experiments, the samples did not display
557 any signs of swelling and kept their initial shapes. As the effect of lime on the
558 soil's microstructure depends on their different natures [30], the carbonation of
559 lime created a rigid skeleton in the material [74]. Hence, the protected clays could
560 avoid to adsorb water on their surface and that could prevent the earthen material
561 from swelling.

562 **3.2. Insight provided by water droplet penetration: highlighting** 563 **hydrophobic properties**

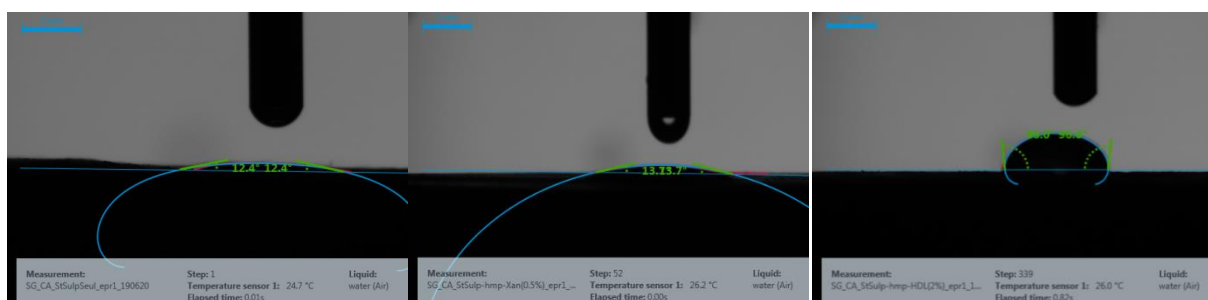
564 For some of the tested bio-based additions (Xan, LO, LOSurf and VV), a strong
565 reduction of the water capillary absorption has been observed and those additions
566 seem to be promising in order to protect earthen materials from the effects of
567 liquid water. However, the protection mechanisms at the microscale seem to be
568 quite different depending on the type of bio-based addition.
569 First, there is likely to be a difference between the coating additions (VV,
570 LOSurf) and the mass treatments (LO in the mass, Xan) that would seem to imply
571 distinct absorption behaviours. For the mass treatments, the A_{cap}/n values are
572 strongly reduced (divided by a factor ranging from 2 to 10) and the w_{cap} values are
573 slightly reduced for the 1% linseed oil addition, further reduced for the 2% linseed
574 oil additions and strongly reduced for all of the xanthan gum additions.
575 Furthermore, all of these mass treatments imply that the raw earth samples do not
576 display any signs of swelling and maintain a strong apparent cohesion. In the case
577 of the coating additions, if the A_{cap}/n values are strongly reduced (divided by a
578 factor ranging from 2 to 5) and the w_{cap} values are slightly reduced, but after a
579 certain time of water capillary absorption, the protected earth samples start to
580 swell and crack. There is thus an apparent difference between coating additions
581 and mass treatments. The mass treatment appears to provide a global protection

that prevents interlayer adsorption of water between the clay particles of the soil while the coating only seems to create a protective layer on the surface of the sample that stays effective until a certain point. Consequently, in the case of a coating, as soon as the water has crossed this protective layer, the capillary absorption behaviour becomes similar to the behaviour of an unprotected earth sample.

Then, by focusing on the two most effective mass treatments (LO in the mass and Xan), and more precisely on their interaction with liquid water, it seems that there are strong differences in their implied mechanisms that protect the microstructure of earthen materials.

Contact angle measurements were conducted for the soil S combined with the above-mentioned additions and on soil S without additions. Other tests were also conducted on this soil combined with other additions (Tan, Cas or Alg). For all of the studied combinations, HMP was added in the mix, except for the additions of tannins (Tan 0.4%) and for the reference (Soil S without addition). In Fig. 5, pictures of the contact angle of a 15 μ L sessile droplet of water on the surface of the different tested samples after 0.01 seconds of contact are shown. In Fig. 6 the evolution of the mean contact angles versus the time are plotted. For the addition of linseed oil in the mass, the contact angle of the sessile droplet of water is much larger than for the other studied samples and remains at the surface for a much longer period of time. In this case, the water droplet is not absorbed before 30 seconds, whereas it is absorbed within a second for an unprotected sample.

Considering these observations, it can be concluded that the linseed oil mass treatments seem to be very effective in creating a distributed hydrophobic layer on the surface of all of the soil particles of the sample. Hence, it strongly reduces the water capillary absorption of the sample by protecting the surface of the clay bonds of the material's microstructure.



(a) Soil S without addition

(b) Soil S+HMP+Xan(0.5%)

(c) Soil S +HMP+LO (2%)

Fig. 5 Pictures of contact angles at $t=0.01s$ for a 15 μL sessile droplet of water for different formulations of Soil S

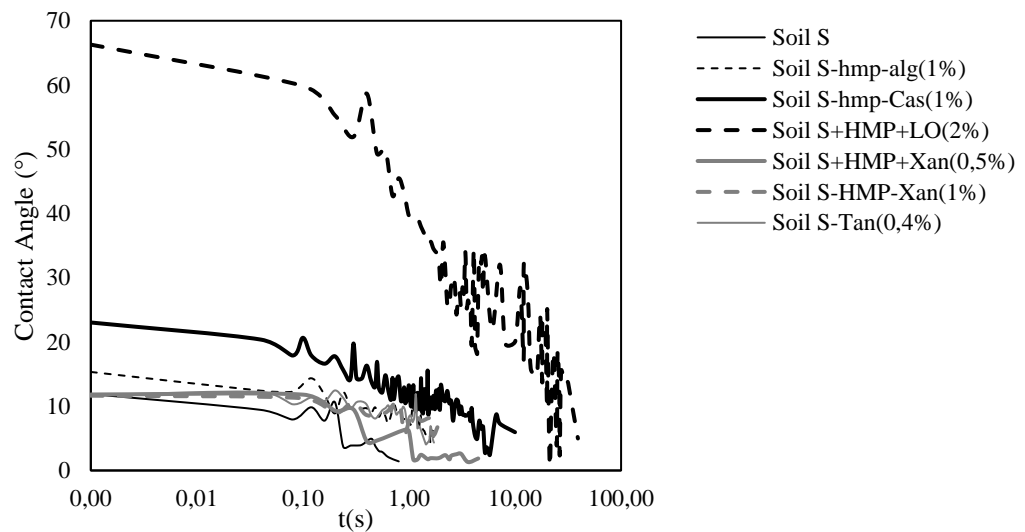


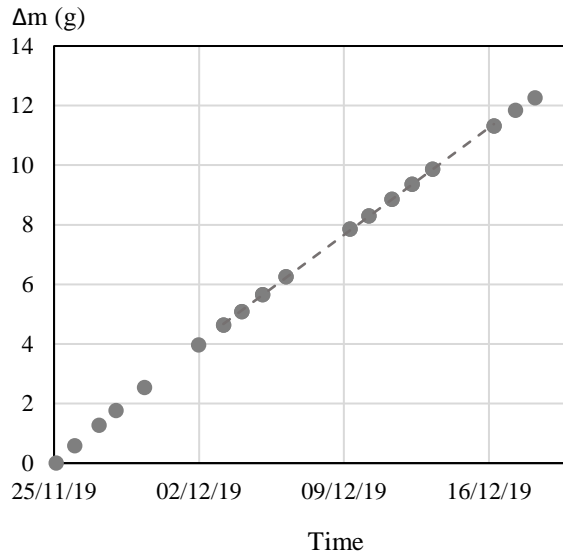
Fig. 6 Evolution of contact angle values for a 15 μL sessile droplet of water for different formulations of Soil S

However, for the different additions of xanthan gum (0.5% in the mass of the dry soil or 1%) there is no incidence on the measured contact angles, the water being absorbed within a second in the porosity of the samples in the same way as in the unstabilized samples. In this case, the mechanism that is limiting the capillary absorption is different. Some previous studies have observed several samples of different soils stabilized with xanthan gum by means of a scanning electron microscope (SEM) [54, 75], and have highlighted the fact that the xanthan gum is reinforcing the soil microstructure by creating a Xanthan gum–fine soil network via electrostatic and hydrogen bonding. Xanthan gum is not hydrophobic, but it can be bonded with the clayey parts of the studied soil and thus create another bonding network between the particles of the soil. This allows the sessile droplet of water to be absorbed in the material within a second (Fig. 5 and Fig. 6). However, this contact angle measurement is mostly evidencing the hydrophobic properties of the additions, even if the overall macroscopic behaviour of the sample remains hydrophilic (contact angle lower than 90°) due to the porous nature of the material. Also, it is not directly correlated to the capillary absorption, because the absorption of the sessile droplet is also affected by a gravity effect and surface roughness.

633 Finally, these two efficient mass treatments seem to strongly improve the
634 durability of earthen materials regarding the water capillary absorption by
635 significantly modifying their microstructure. That being said, it would be
636 unfortunate to redesign the raw earth material regarding its water durability to
637 such a point that it could affect the transfer of water vapour in its pores, which is
638 one of its most interesting properties. Thus, it is relevant to focus on the water
639 vapour permeability of a given soil combined with linseed oil and xanthan gum in
640 its mass and to compare the obtained results to the water vapour permeability
641 properties of this material without addition and combined with HMP. These
642 results could give some indications about the ability of these stabilized materials
643 to transmit water vapour, to regulate the indoor humidity of buildings and to
644 improve ambient comfort conditions.

645 **4. Water vapour permeability of selected** 646 **stabilized materials**

647 This section details the results of the water vapour permeability and water vapour
648 resistance factor studies performed on four formulations: Soil S without addition,
649 Soil S + HMP, Soil S + HMP + LO(2%) and Soil S + HMP + Xan(0.5%).
650 Fig. 7 illustrates an example of mass uptake Δm along time. This kinetic is
651 representative of the results obtained on all specimens. The steady state was
652 reached after 8 days and the slope was calculated from linear regression between
653 day 8 and day 21. The correlation coefficient is very high (> 0.999). Table 2
654 shows the average water vapour permeability calculated from the 5 samples of
655 each formulation.
656



657

658 **Fig. 7** Mass uptake versus time during water vapour permeability test – example of soil S

<i>Soil mix</i>	Mean water vapour permeability (π, kg/(m.s.Pa))	Water vapour diffusion resistance factor (μ)	Mean dry density (kg/m³)
<i>Soil S</i>	9.9×10^{-12}	19.8	1665
<i>Soil S + HMP</i>	9.6×10^{-12}	20.4	1677
<i>Soil S + HMP + LO(2%)</i>	7.8×10^{-12}	25.0	1658
<i>Soil S + HMP + Xan(0.5%)</i>	10.5×10^{-12}	18.7	1605

659

Table 2 Water vapour permeability mean results for four formulations of soil S

660

The measured water vapour permeability and water vapour resistance factor are of

661

the same order of magnitude for all of the tested formulations, around $9.5 \cdot 10^{-12}$

662

kg/(m.s.Pa) and 21 respectively. Thus, these earthen samples present good

663

moisture transfer capacities for structural materials [66, 76]. Furthermore, for a

664

given formulation, the density influences the water vapour resistance factor as it

665

affects the porosity. So, the variation of the water vapour resistance factor is

666

plotted versus density in Fig. 8 for all samples of all the formulations. As

667

expected, the water vapour resistance factor increases with density. Results show

668

that Soil S, Soil S + HMP and Soil S + HMP + Xan(0.5%) presents similar

669

relationships between water vapour diffusion resistance and dry density with

670

measurements almost grouped on a same line. They are slightly greater for the

671

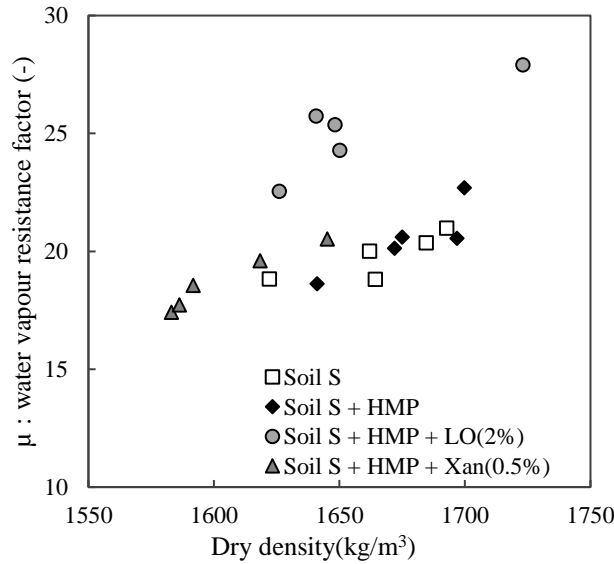
Soil S + HMP + LO(2%) formulation along the investigated density range.

672 The addition of HMP does not influence the transmission of water vapour in the
 673 soil S.

674 The addition of linseed oil in the mass of the material produces an increase in the
 675 water vapour resistance factor along the density range, by 25 % in average. In
 676 fact, the linseed oil coats and partially fills the pores. In addition, it is highly
 677 hydrophobic so it reduces the transfer of water vapour.

678 The addition of xanthan gum does not affect the water vapour resistance for a
 679 given density of soil. However, as it induces lower density, it leads to lower
 680 resistance.

681



682

683 **Fig. 8** Evolution of water vapour diffusion factors resistance factors (μ) versus the dry densities
 684 for four formulations of soil S

685 Finally, the addition of xanthan gum in the mass of earthen materials protects
 686 earth samples from the absorption of liquid without affecting their water vapour
 687 permeability while the use of linseed oil reduces it.

688 In order to complete this study on moisture transfer, it would be interesting to
 689 characterise the ability to adsorb and store moisture from the atmosphere, with the
 690 measurement of a Moisture Buffer Value (MBV) [77]. This ability widely impacts
 691 indoor comfort.

5. Effect of water uptake on the mechanical behaviour of earth – A practical discussion

It is known that the mechanical properties of the earthen materials are decreasing with their moisture content [10, 11, 15] and several studies have already highlighted the fact that bio-based additions could increase the mechanical properties of earthen materials [13, 38, 40–49]. In this study, it has been highlighted that two bio-based mass treatments (Linseed Oil in the mass and Xanthan Gum) of earthen materials could limit their capillary absorption of water, increasing their durability regarding water without affecting the transfer of water vapour in their pores. Four formulations of the soil S were thus studied: Soil S+HMP, Soil S+HMP+LO(1%), Soil S+HMP+LO(2%), Soil S+HMP+Xan(0.5%). The evolution of the compressive strength of these formulations versus their moisture content is plotted in Fig. 9. The evolution of the moisture content of these formulations versus the time they have spent in the wet chamber is plotted in Fig. 10. Once again, these results highlight the two different protecting mechanisms of linseed oil and xanthan gum.

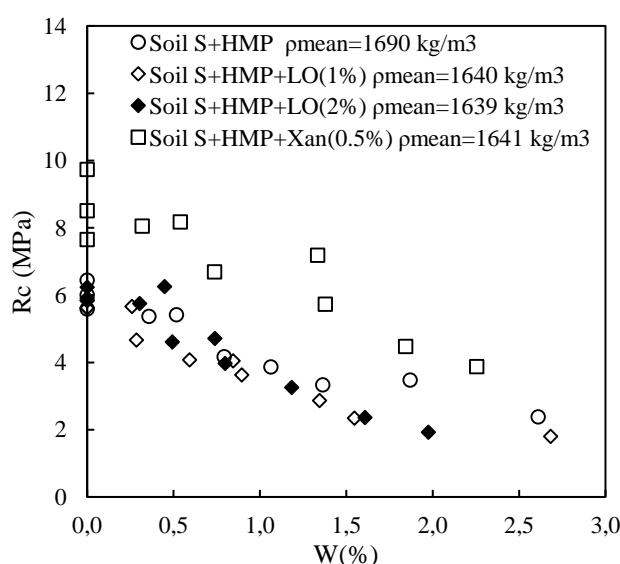


Fig. 9 Evolution of compressive strength versus the moisture content for four mixes of soil S

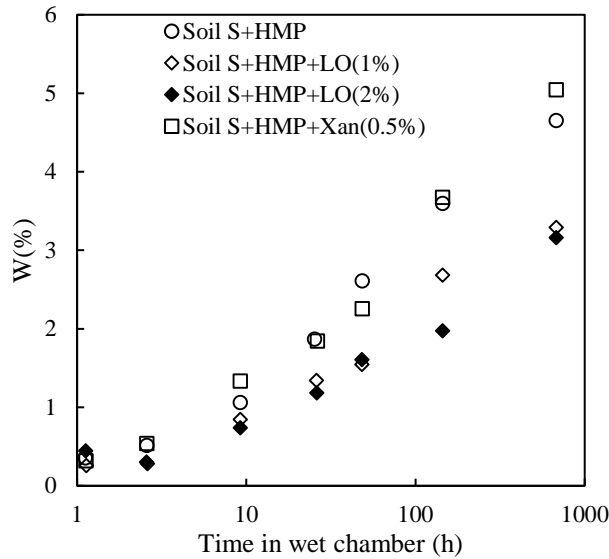


Fig. 10 Evolution of the moisture content versus the time spent in wet chamber for four mixes of soil S

In Fig. 9 it is clear that the xanthan gum is limiting the decrease of the compressive strength of the earth samples while their moisture content is increasing. However, in Fig. 10 it is also noticeable that the moisture content of the samples stabilized with xanthan gum is increasing at the same rate as the moisture content of the unstabilized samples, after having being put in wet chamber. These observations could confirm that xanthan gum is probably creating a bonding network of gel gluing the soil particles, as has been highlighted in previous studies [54, 75], increasing the sample's in-situ mechanical properties. The hydrophilic properties of xanthan gum are well known and this gum is reacting with water to create a network of gel that strengthens the soil when drying [54, 75]. It is also justifiable to question whether xanthan gum is still hydrophilic after bonding with the soil's particles or not. As previously stated, in Fig. 9 it also seems that the moisture content of the samples stabilized with xanthan gum is increasing at the same rate as the moisture content of unstabilized samples, after having being put in wet chamber. Therefore, it can be assumed that this stabilisation method is not absorbing an additional amount of water after drying. This potential change of hydrophilic nature still has to be thoroughly studied. On a wider scale it still seems that the addition of xanthan gum in an earthen sample could help to increase its water durability, leaving the clay particles available for water adsorption without in any way affecting its affinity with water vapour and its hygric properties.

734 At first sight, by focusing again on Fig. 9, the addition of linseed oil at different
735 given contents does not seem to limit the decrease of the mechanical properties of
736 the tested earth samples while their moisture is increasing. However, in Fig. 10 it
737 is clearly noticeable that these additions allow the earth samples to decrease their
738 adsorption of ambient humidity and thus to present a lower moisture content
739 increase. These observations are also confirming the foregoing conclusions about
740 the effect of linseed oil on earthen materials. If linseed oil is not creating a new
741 network of bonds in the material that provides better mechanical strengths, it is
742 coating all of the pores of the material, limiting the interactions of the dry earthen
743 material clay particles with the ambient humidity by its highly hydrophobic
744 properties. Thus, the increase of the moisture content of the material is slowed.
745 Even if the interactions between the material and the ambient humidity are
746 limited, the material stabilised with linseed oil is still able to store moisture and is
747 open to water vapour transfer. However, it would be interesting to determine its
748 moisture buffering capacity.

749 **6. Conclusion**

750 The aim of this study was to highlight that some selected bio-based additions are
751 able to enhance the water durability of earthen materials. For mass treatments
752 such as linseed oil or xanthan gum and coatings with linseed oil or vegetal-based
753 varnish, the water capillary absorption of earthen structural materials is strongly
754 reduced. By limiting this capillary absorption, the mechanical properties of the
755 tested samples can be maintained when exposed to in-situ conditions. Hence, their
756 water durability was enhanced. Several patterns of capillary absorption were
757 observed for the different studied soils and additions, enabling a better
758 characterization and understanding of the studied soils as well as an identification
759 of distinct protection mechanisms regarding the water durability of the different
760 bio-based additions. Furthermore, for the most efficient additions, the water
761 vapour permeability of stabilized earthen samples was monitored. The results
762 show that these additions can enhance the water durability of earthen materials
763 without affecting the transfer of water vapour in their pores and thus their hygric
764 properties. These results are promising and xanthan gum or linseed oil, combined
765 with efficient processing methods, can be good alternatives to hydraulic binders in

766 order to stabilize earth-based building materials and produce raw earth blocks that
767 achieve compliance with modern standards regarding strength and durability.

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