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5 **Water Absorption of recycled aggregates: measurements, influence of**
6 **temperature and practical consequences**

7

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10

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12

13 **Abstract**

14 Recycled Concrete Aggregates are highly porous and may therefore absorb more water
15 than natural aggregates. Several methods allow for the measurement of water
16 absorption requiring drying at temperature above ambient conditions. We show here
17 that, for temperatures above the ambient temperature ($> 30^{\circ}\text{C}$) and at low relative
18 humidity, the drying of recycled aggregates removes some bounded water contained in
19 hardened cement paste and increases both measured porosity and water absorption.
20 Porosity and XRD suggest that porosity increases due to ettringite dehydration. Thus,
21 the drying step required to characterize recycled aggregates leads to a systematic
22 overestimation of the water absorption. Moreover, this overestimation induces an error
23 in mix-design leading to a higher effective water/cement ratio. If absorption and water
24 content are measured with the same preparation protocol, correction is not needed. We
25 finally propose a correction method for the water absorption measured according to the
26 standard protocol (drying at 105°C).

27

1. Introduction

Due to the limited natural resources, the use of Recycled Concrete Aggregates (RCA) is today a growing practice in the construction industry. These RCA result from the crushing and grinding of demolition wastes from our buildings and our infrastructures. As such, RCA differ from natural rounded aggregates from river in both composition and microstructure and can be seen as a composite granular material made of natural aggregates and hardened cement paste.

Since hardened cement paste is highly porous, RCA have porosity 10 to 20 times higher than the porosity of a natural aggregate; it depends strongly on the volume fraction of cement paste in the aggregate. In contact with water or fresh cement paste, they may therefore absorb more water than natural aggregates. This absorption may affect in turn the properties of concretes prepared with RCA. Depending on the time needed for saturation of these aggregates, this water absorption can be at the origin of a decrease in workability [1–3]. Moreover, by compensating for this water absorbed in the mix-design, high workability can be generated, which can even lead to concrete segregation. In the hardened state, the concrete is more porous, thus increasing the permeability which increases transfers [3,4]. Due to porosity, mechanical strength may decrease compared to natural aggregate concrete [1,3,5–8] and the expected service life based on durability tests was reported to reduce significantly [9,10]. Thus, porosity and the associated water absorption seems in most studies to be a key parameter for predicting both casting and service-life behavior of concretes incorporating RCA.

Several methods allow for the measurement of water absorption of aggregates [11]. European standards for characterizing the properties of aggregates define a specific method in NF EN 1097-6 [12]. In this test, the aggregates are initially dried. Then, the amount of water absorbed is measured after 24 hours of immersion in water. The drying of the aggregates must be carried out at 105°C. However, the standard was designed before the extensive use of recycled aggregates. Indeed, given the presence of hardened

57 cement paste in their structure, drying at high temperatures can affect its microstructure
58 and thus modify its water absorption.

59

60 Nevertheless, several studies on the absorption of recycled aggregates use a drying
61 protocol based on the above standard with a temperature of 105°C [3,13–15]. Some
62 studies recommend a reduction of the drying temperature to 70-75°C [3,4,16] to limit
63 the alteration of the hardened cement paste. However, it has been shown recently that
64 hydrates like Ettringite are very sensitive to temperature changes above 75°C [17–21].
65 Moreover, Baquerizo et al. [20] showed that water content in Ettringite changes
66 significantly with a small variation of temperature and relative humidity. Thus, for
67 recycled aggregates, the drying protocol should be carefully chosen and the influence
68 of temperature and relative humidity on the water absorption measurements need to be
69 addressed.

70

71 In this paper, we show that the drying of recycled aggregates for temperatures above
72 the ambient temperature ($> 30^{\circ}\text{C}$) and at low relative humidity ($< 20\%$) removes some
73 bounded water contained in hardened cement paste and increases both measured
74 porosity and measured water absorption. By measuring in parallel porosity and
75 chemical composition variations with temperature, we suggest that the increase in
76 porosity finds its origin in ettringite dehydration, which released bound water in an
77 amount similar to the measured water absorption increase. Moreover, this absorption
78 overestimation induces a systematic error in mix-design leading to a higher effective
79 water/cement ratio. We finally propose a correction method to be applied to the water
80 absorption measured according to the standard protocol (drying at 105°C).

81

82 **2. Materials and experimental procedures**

83 **2.1. Materials**

84 Four CEM I types of cement from four different factories from Lafarge Holcim with
85 specific density around 3.15 were used in this study. These cements were produced in

86 four different factories (Saint Vigor (SV); Lägerdorf (L); Le Teil (LT) and Saint Pierre
87 La Cour). Their main difference is in their C₃A content. Their chemical composition is
88 given in Table 1. It is obtained through Inductively Coupled Plasma and Atomic
89 Emission Spectrometry (ICP-AES Horiba ultima 2000), analytical technique that
90 allows the quantification of the element traces in liquid solutions by comparison of
91 standard materials (for the cement) and by Differential thermal analysis (ATD-ATG
92 NETZSCH STA 409E). Their mineral composition is also given in Table 1. The W/C
93 of the cement paste in recycled aggregates varies depending on the concrete origin. To
94 study a wide range of recycled aggregates in terms of absorption and with different
95 microstructures, we prepared cement pastes at different water-to-cement ratios (W/C):
96 at W/C=0.5 with cements SV, L, LT and SPLC; W/C=0.2-0.3-0.4-0.6 with cement L. In
97 the case of W/C=0.2, it was necessary to use a superplasticizer. We chose a commercial
98 one, CHRYSO®Fluid Premia 570 at 1.0% in weight of cement in order to have a
99 homogeneous and fluid material for samples casting. All cement pastes were mixed for
100 2 min using a Rayneri mixer at 840 rpm.

101

102

103 **Table 1**

104 Chemical and mineral composition of the cement powders

Chemical composition (% by mass)								
	CaO	SiO₂	Al₂O₃	Fe₂O₃	MgO	SO₃	Cl	Ignition loss
Saint Vigor (SV)	65.4%	21.0%	3.6%	4.4%	0.8%	2.3%	0.1%	1.5%
Lägerdorf (L)	61.9%	19.1%	3.6%	4.2%	0.7%	2.5%	0.1%	1.5%
Le Teil (LT)	68.6%	25.1%	2.5%	0.2%	0.5%	2.2%	0.1%	2.6%
Saint Pierre La Cour (SPLC)	66.5%	20.9%	4.6%	2.6%	0.9%	3.0%	0.1%	1.2%

Mineral composition (% by mass)								
	C₃S	C₂S	C₃A	C₄AF	Gypsum	CaO free	Calcite	
Saint Vigor (SV)	63.2%	12.5%	2.5%	12.6%	5.0%	0.7	1.8	
Lägerdorf (L)	67.2%	5.3%	2.5%	12.7%	5.4%	0.8	0.5	
Le Teil (LT)	58.2%	25.8%	6.2%	0.6%	4.7%	0.8	2.6	
Saint Pierre La Cour (SPLC)	64.5%	11.3%	7.8%	7.9%	6.5%	0.4	0.6	

105

106 After mixing, the cement pastes were poured into 4x4x16 cm³ molds and demolded
107 after 24 hours. The samples were then immersed in water for 3 months to ensure that
108 cement pastes were close to complete hydration. Afterwards, the hardened cement paste
109 samples were crushed using a jaw crusher to obtain aggregates with different sizes. For
110 this study, we sieved the crushed aggregates and only kept the fraction between 6 to 10
111 mm in order to ease the measurement of water absorption. Indeed, in the case of smaller

112 particles (< 4 mm) and highly angular particles such as recycled sand, water absorption
113 according to the standard method induces a high uncertainty on measurements and
114 therefore requires alternative methods [22,23], which are out of the scope of this study.
115

116 **2. 2. Water absorption and porosity measurements**

117 The Water Absorption (WA) is the ratio between the water required to fill a porous
118 sample and its dry mass. The water absorption measurement consists in drying at a
119 specific drying temperature (20°C; 30°C; 45°C; 75°C; 105°C) the samples in order to
120 remove all the water present in the pores (M_{dry}). Then, the samples are immersed in
121 water. The solid-to-liquid ratio is of 100g for 1L, water is not flowing during immersion
122 and hardened cement dissolution is quite slow [24], thus we consider that leaching is
123 neglectable. After 24 hours of water immersion, the samples are removed from water.
124 Finally, the aggregate surface is carefully dried with absorbent cloths until the water
125 films on the aggregate surface disappear to obtain the so-called “Saturated Surface Dry”
126 (SSD) mass (M_{SSD}) in accordance with standard NF EN 1097-6 [12]. To assess its
127 reliability, we also measure the SSD mass with an in-house evaporative method [25].
128 At this stage, the aggregate is saturated but without free water on the surface.

129 From these two measurements, the water absorption is computed as:

$$130 \quad \text{Water absorption (WA)} = \frac{100 \times (M_{SSD} - M_{dry})}{M_{dry}} \quad (1)$$

131 However, in this study, as we will use different cement pastes with varying water to
132 cement ratio (W/C), the volume of sample will also vary for the same mass M_{dry} . To
133 compare samples with different density, the water absorption by volume is more
134 relevant than by mass. Thus, the porosity ϕ (ratio of void to the total volume) is equal
135 to:

$$136 \quad \text{Porosity } \phi = \frac{100 \times (M_{SSD} - M_{dry})}{M_{dry}} * \rho_{Aggregate} \quad (2)$$

137 where $\rho_{Aggregate}$ is the dry density of the cement pastes. This density $\rho_{Aggregate}$ is equal to
138 the ratio between the dry mass M_{dry} of the cement paste and the volume of the sample
139 (taking into account the presence of pores), corresponding to the difference between the
140 M_{SSD} and the mass of the sample immersed in water (measured by hydrostatic weighing

141 in accordance with standard NF P18-459 [26]).
142 Since we use various drying temperatures in this study, we had to choose a criterion
143 defining the end of drying (i.e. when all water has evaporated). In this study, we
144 therefore consider that drying is over when the mass loss between two successive
145 weightings at 24-hour intervals is less than 0.05 %. As the drying time can be very long,
146 for instance, one month in the case of drying at 30°C, soda lime aggregates are present
147 in the drying chamber to avoid carbonation. After treatment, the aggregates were stored
148 at 20°C and relative humidity of 6 %, also to limit carbonation.

149 **2. 3. Mercury Intrusion Porosimetry**

150 Mercury Intrusion Porosimetry characterizes the structure and the interconnected pores
151 of porous material [27,28]. The mercury intrusion porosimeter used in this study is
152 Autopore IV with maximum and minimum applied pressures of 400 MPa and 0.01 kPa,
153 allowing to characterize pore sizes from 5 nm to 60 µm. To estimate this pore size, we
154 consider a contact angle $\theta_{\text{Hg}} = 141^\circ$. The measurement parameters used for this study
155 are 0.0035 MPa for the initial mercury pressure with an exponential mercury pressure
156 increment until 20 MPa and an equilibrium time for each data point of 10 seconds. The
157 sample mass for each test is between 1.0 to 1.5 grams (approximately equal to 2 or 3
158 6/10 mm aggregates). Three different temperatures were used (30-75-105°C) to dry the
159 samples before testing.

160

161 **2. 4. X-ray diffraction**

162 The XRD experiments were carried out in a Diffractometer D8 Advance from Bruker
163 with a Cobalt anode under 35 mA/40 kV without monochromator. We measured the
164 diffraction angles between 3 to 80° with an angle increment equal to 0.01° and an
165 acquisition time of 1 second.

166 To carry out X-Ray diffraction, samples of hardened cement pastes (2- 3 grams) were
167 manually ground (in order to avoid heating the sample during grinding) until samples
168 with a size of less than 63 µm were obtained.

169 The processing of the obtained diffractograms was performed with the Eva software

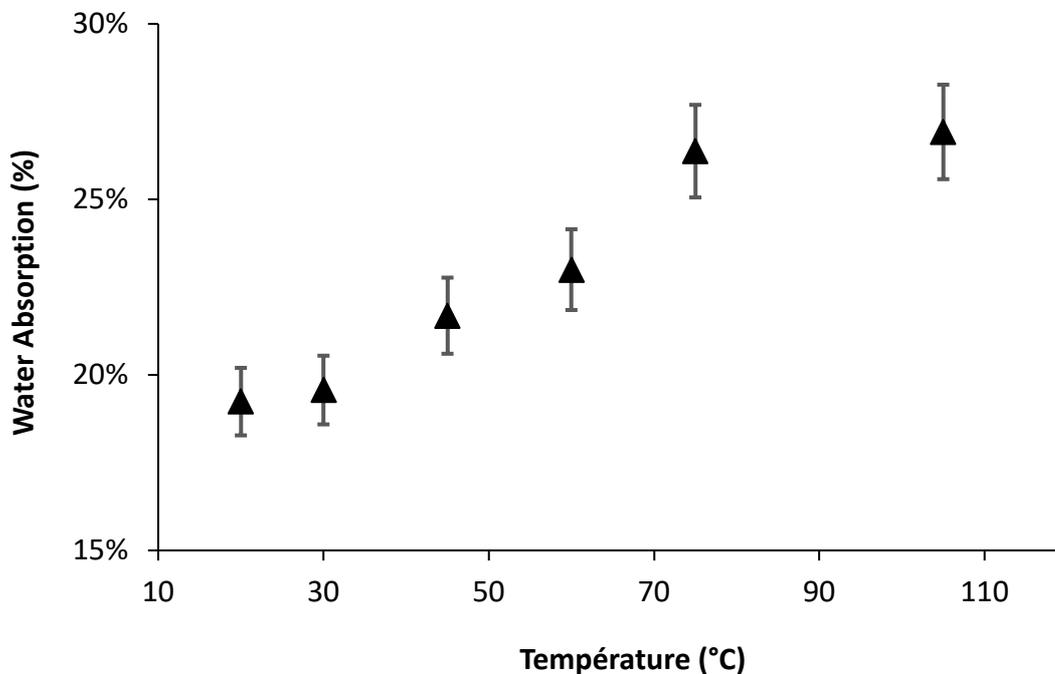
170 coupled to the mineralogical database ICDDPdf2 to determine the presence of
171 crystalline phases typical of a cement paste. Furthermore, to compare their proportion
172 of crystalline phases (Portlandite, Ettringite) quantitatively, the TOPAS software from
173 Bruker was used for quantification by the Rietveld method. The results of Rietveld
174 analyses give the mass percentages of the crystalline phases present in the
175 diffractograms. By knowing the density of each hydrate [27], we then computed the
176 volume fraction of each component.

177

178 **3. Experimental results**

179 **3. 1. Water absorption measurements as a function of drying** 180 **temperature for hardened cement pastes (W/C = 0.5)**

181 In order to evaluate the influence of the drying temperature on water absorption of
182 hardened cement paste, different temperatures were used to prepare the samples before
183 the water absorption measurements.



184

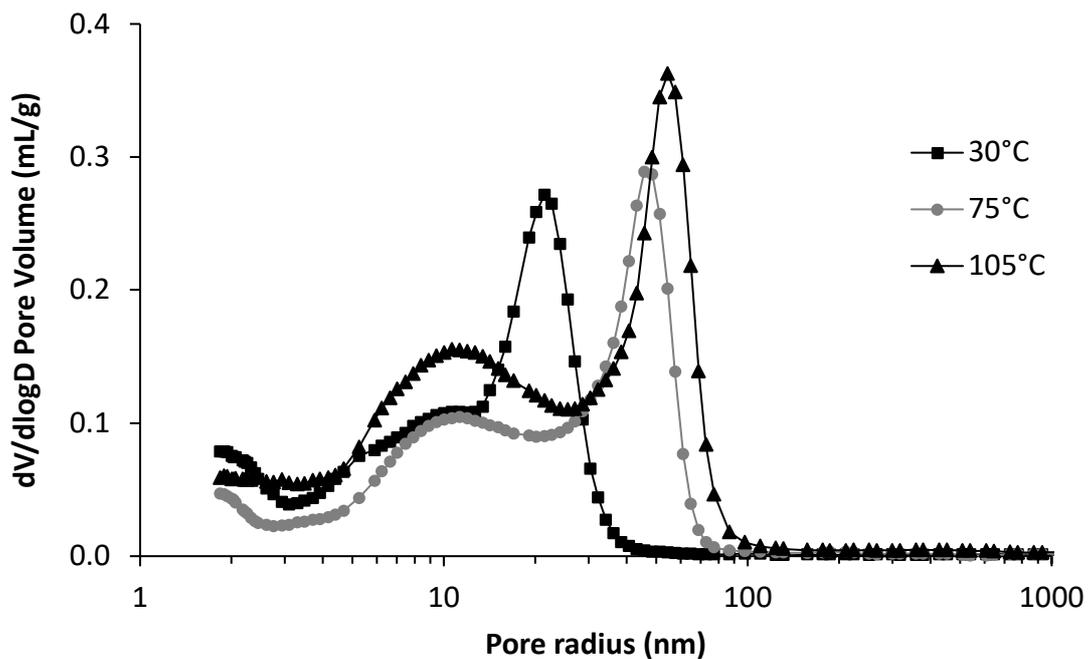
185 **Fig. 1.** Water absorption of hardened cement paste (W/C 0.5 – cement SV) as a function
186 of the drying temperature.

187

188 We plot in **Fig. 1** the water absorption measurements obtained on a hardened cement
189 paste as a function of the drying temperature applied to the samples before the water
190 absorption measurement. It can be seen that water absorption increases with drying
191 temperature. Indeed, we note a linear increase in water absorption between 30 and 75°C.
192 While the absorption measured after drying at 20 or 30 °C is similar, the water
193 absorption measured after drying at 75 °C is 40% higher than the drying at 20/30°C.
194 The water absorption measured after drying at 105°C is the same as the one measured
195 after drying at 75°C.

196

197 **3. 2. Pore size distribution as a function of drying temperature for** 198 **hardened cement pastes (W/C = 0.5)**



199

200 **Fig. 2.** Pore size distribution of hardened cement paste (W/C 0.5 – cement SV) obtained
201 by mercury intrusion porosimetry (MIP) after drying at 30, 75 and 105°C.

202

203 We observed the porous distribution of a W/C 0.5 hardened cement using mercury
204 intrusion porosimetry for drying temperatures of 30, 75 and 105°C. The pore size
205 distribution for 75 and 105°C is similar and two pore populations are visible, a first
206 population at 50 nm and the second around 12 nm (see **Fig. 2**). On the contrary, in the

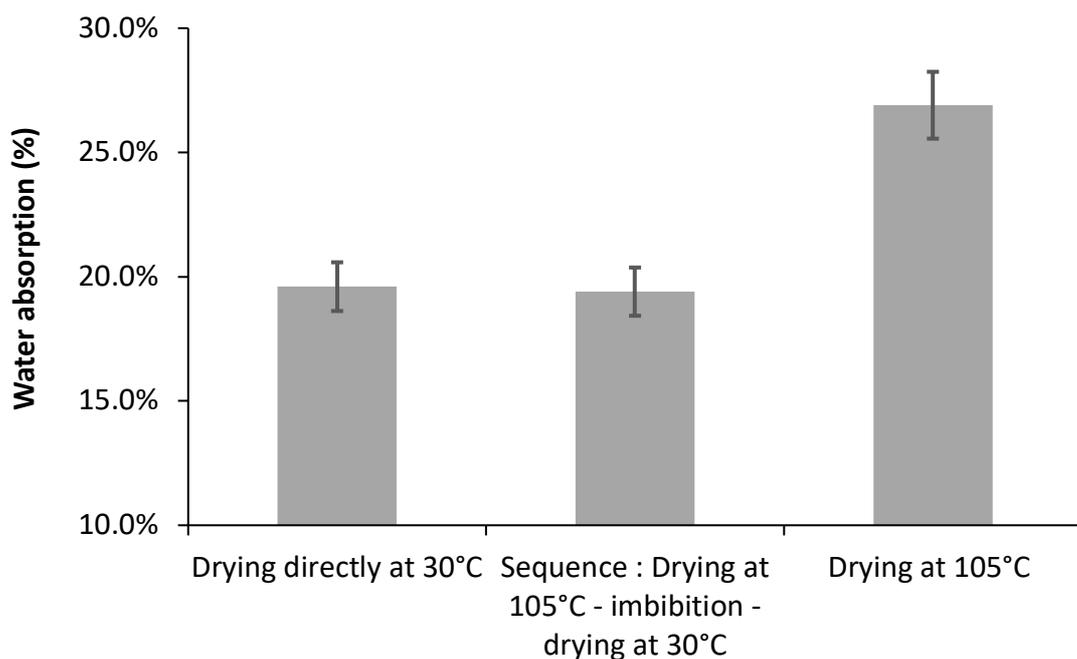
207 case of drying at 30°C, most of the pores are around 10-20 nm.
208 Thereby, the maximum pore size of dried cement pastes increases with the drying
209 temperature. Indeed, the maximum pore radius of the sample for drying at 105°C is 55
210 nm, versus 23 nm for the drying at 30°C. Moreover, the total porosity measured by MIP
211 increases sharply with the drying temperature: at 105°C, the porosity is 32 % compared
212 to 25% at 30°C. Therefore, drying at 105°C has the effect of increasing the porosity of
213 the cement paste and also roughly doubling the pore size of the cement paste compared
214 to drying at 30°C.

215

216 3. 3. Reversibility of porosity increase (W/C = 0.5)

217 In order to better understand the impact of drying on water absorption, a specific drying
218 sequence was tested. It consists of first drying the cement paste at 105°C and then
219 soaking it in water to measure its water absorption. Following this first step, the sample
220 is dried at 30°C. As soon as the mass loss is stabilized, the sample is soaked in water a
221 second time and its water absorption is measured.

222 We compare the water absorption results measured for samples that have undergone the
223 specific drying sequence with the reference, corresponding to water absorption in
224 hardened cement pastes directly dried at 30°C.



225

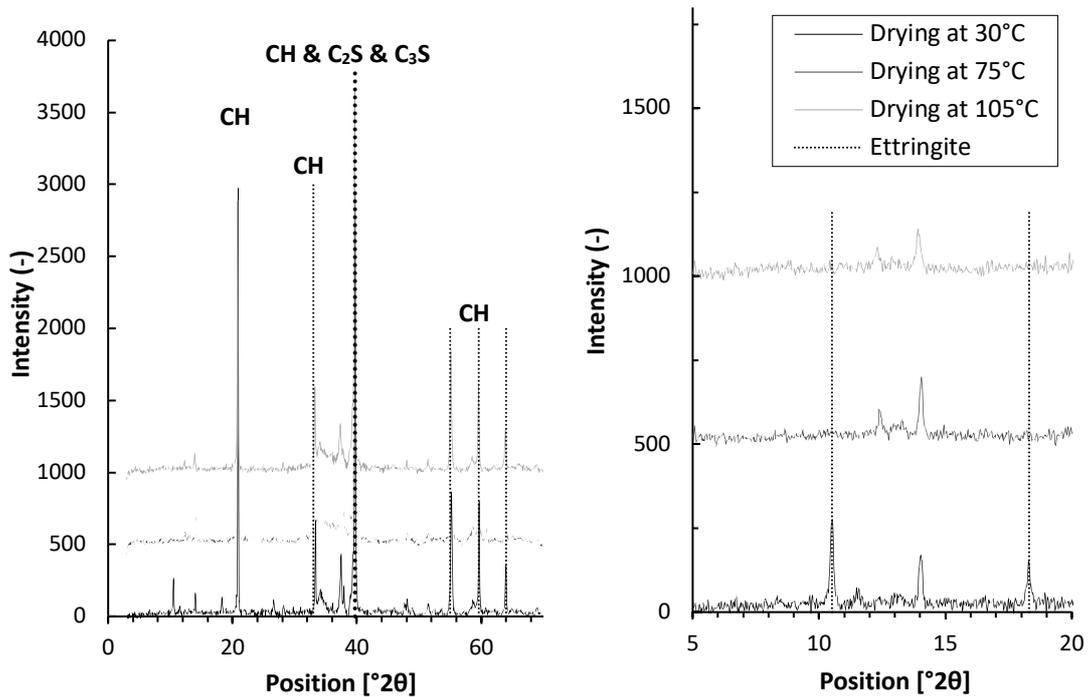
226 **Fig. 3.** Water absorption for samples with different drying history

227

228 The water absorption of the hardened cement paste that underwent the specific drying
229 sequence is identical to that of the reference (19 %) (see **Fig. 3**). Thus, for the
230 investigated samples and conditions (drying at 105°C), the high absorption was not a
231 permanent feature and, after a re-imbibition in water and drying at 30°C, absorption is
232 identical to the one measured on aggregates that were directly dried at 30°C. Therefore,
233 we observe an apparently reversible increase in absorption after drying at 105°C.

234

235 **3. 4. X-ray diffraction results**



236
 237 **Fig. 4.** X-Ray diffractograms of hardened cement paste (W/C 0.5 – cement SV) at
 238 different drying temperatures. a) Diffractograms between 0 and 70° b) Zoom between
 239 7 to 20°

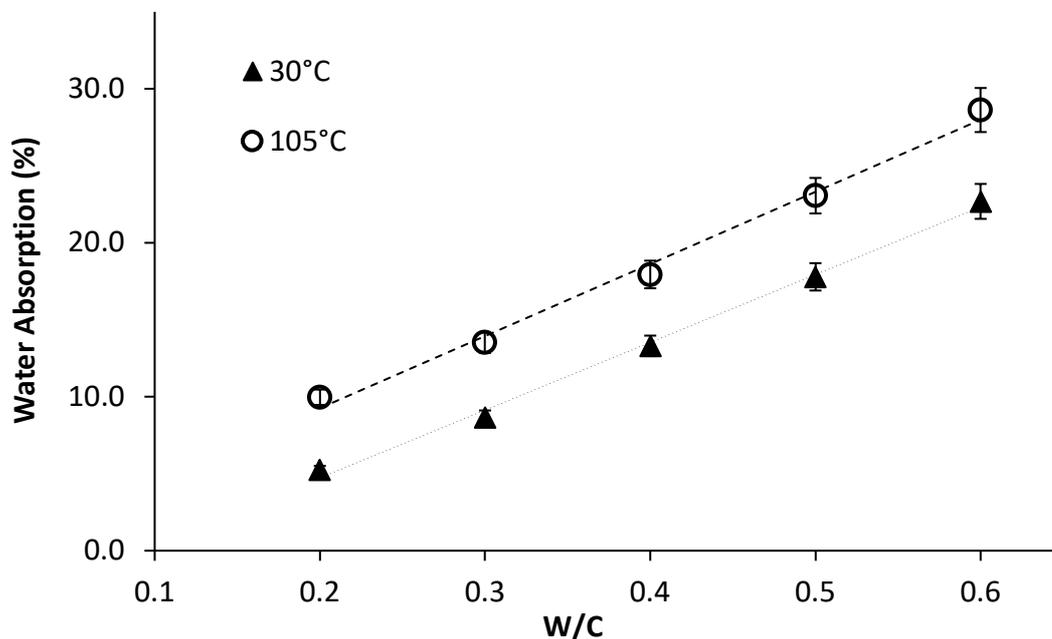
240
 241 In the diffractograms obtained by XRD measurements of a hardened cement paste, we
 242 see the same main characteristic peaks, whatever the drying temperature (see **Fig. 4a**).
 243 Indeed, the peaks of the main hydrates such as Portlandite “CH” (21°, 33°, 40°, 55°,
 244 60°, 64°) or clinker components “C₂S & C₃S” (33-40°) are present.
 245 However, by focusing on small diffraction angles between 7 and 23°, we can observe
 246 differences in the diffractograms according to the drying temperature. Indeed, in the
 247 case of 30°C, the characteristic peaks of Ettringite (11 and 18°) are found. On the
 248 contrary, for a drying temperature of 75 and 105°C, these peaks are no longer present
 249 (see Fig. 4b). These results indicate that the disappearance of Ettringite peaks occurs
 250 between 30 and 75°C, which is confirmed by quantification of the fraction of Ettringite.
 251 By using the Rietveld method and knowing the density of all the hydrates in the cement
 252 paste, we estimated the volume content of crystalline phase hydrates. We obtained a

253 volume fraction of 9.5 ± 2.6 % of Ettringite (measure consistent with state of the art
254 [27,29,30]) when drying was performed at 30°C against 0.1 ± 0.1 % for 75 and 105°C .
255

256 3. 5. Influence of the W/C ratio

257 3. 5. 1. Water absorption

258 In previous sections, the water to cement ratio was kept constant at 0.5. In this section,
259 hardened cement pastes with W/C varying from 0.2 to 0.6 (cement L) were dried at 30
260 and 105°C , and their water absorption was measured.



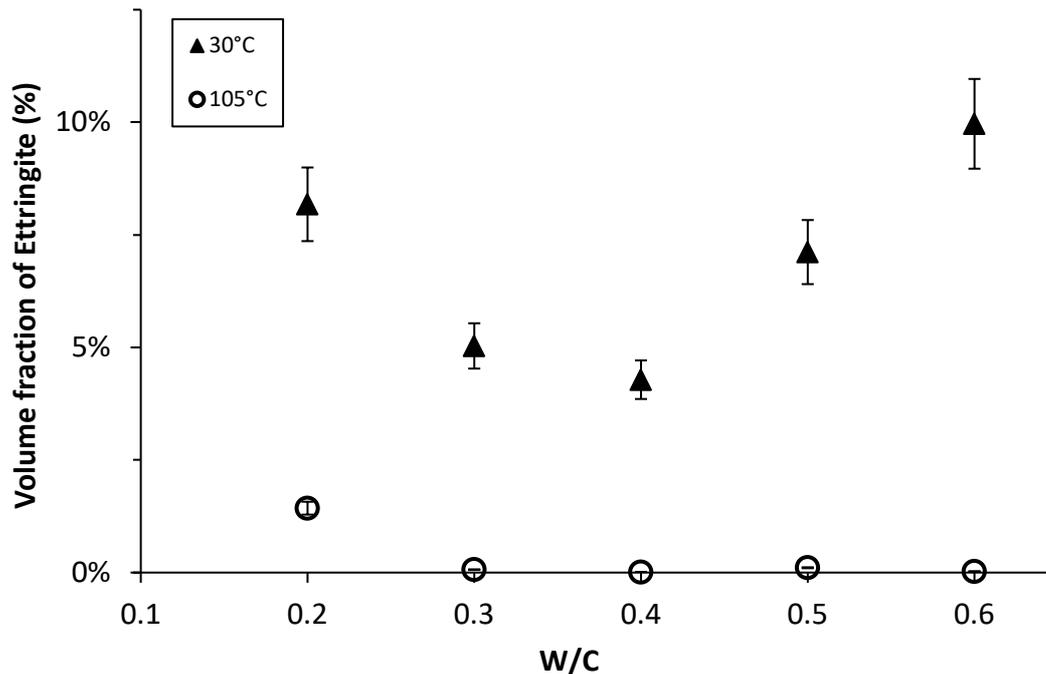
261
262 **Fig. 5.** Comparison of water absorption measured at 30 and 105°C for cement pastes at
263 different W/C ratios (0.2-0.3-0.4-0.5-0.6 – cement L).
264

265 When drying at 30°C and 105°C , water absorption increases linearly with the W/C ratio
266 of the cement paste (see **Fig. 5**). As expected, the higher the W/C ratio of the cement
267 paste, the more porous the aggregate. Moreover, we observe that whatever the W/C
268 ratio of the cement paste, the water absorption measured at 105°C was always higher
269 than at 30°C but follows the same trend along with a parallel linear trend. The difference
270 in water absorption between the two drying temperatures was therefore constant and of
271 the order of 5%.

272

273 3. 5. 2. Ettringite content

274 X-ray diffraction tests were carried out for all cement pastes, with the two drying
275 temperatures. Based on the results of section 3.3, we focused on the volume content of
276 Ettringite estimated by the Rietveld method for 30 and 105°C as a function of the W/C
277 ratio of the cement pastes (see **Fig. 6**).



278

279 **Fig. 6.** Volume fraction of Ettringite obtained by XRD measurements based on Rietveld
280 method for cement pastes at different W/C (cement L) dried at 30 and 105°C.

281

282 It can be seen here that the presence of Ettringite varies with the drying temperature
283 (see **Fig. 6**). When drying the cement pastes at room temperature (30°C), the volume
284 content of Ettringite varies between 5 and 10%, with a complex dependency on W/C.
285 On the contrary, as soon as drying is carried out at high temperature (105°C), the
286 Ettringite content becomes close to 0 %. At 105°C, we observe a small account of
287 Ettringite (around 1.5%) only for W/C=0.2.

288

289

290 **4. Discussion and analysis**

291 **4. 1. Effect of the drying temperature on the absorption**

292

293 When cement paste is dried at different temperatures, we observe that the measured
294 water absorption increases with the drying temperature (see Fig. 1).

295 In this section, we first discard the role of a potential variation in residual water content,
296 as an explanation of the differences in absorption observed as a function of temperature.

297 We then discard the potential influence of an effect of the conditioning temperature on
298 some micro-cracking, which would, in turn, modify the absorption irreversibly. We
299 finally focus on relating the measured Ettringite dehydration to the increase in recycled
300 aggregate absorption with temperature.

301

302 First, we need to verify that the water loss is not due to evaporation of remaining free
303 water but finds its origin in the removal of some bound water.

304 After drying, some liquid water may remain in small pores. For higher temperatures,
305 this residual water content might depend on drying temperature. For each temperature
306 and humidity tested, we estimated the largest pore size at equilibrium with liquid water

307 [31] using Kelvin's law [32]: $\ln(RH) = -\frac{2\gamma V_M}{RT r_{eq}}$ where γ is the surface tension at the

308 Water/air interface (0.072 N/m), V_M is the molar volume of water ($18 \cdot 10^{-6} \text{m}^3/\text{mol}$), R
309 is the ideal gas constant (8.314 J/mol/K), T is the temperature, RH is the relative
310 humidity and r_{eq} is the equilibrium pore radius. These values are reported in **Table 2**.

311

312

313 **Table 2**

314 Experimental conditions (temperature and relative humidity) during the drying and
 315 maximum capillary pore radius r_{eq} .

Temperature (°C)	22.5 ± 0.1	30.2 ± 1.0	45.3 ± 0.1	59.5 ± 0.1	75.0 ± 1.0	105 ± 1.5
RH (%)	6.2 ± 0.4 %	22.7 ± 2.6 %	7.0 ± 1.6 %	2.0 ± 0.1 %	0.9 ± 0.1 %	0.6 ± 0.1 %
r_{eq} (nm)	0.38	0.69	0.37	0.24	0.19	0.16

316

317 For all drying conditions, the equilibrium pore size is of the order of some Angströms
 318 (see in Table 2) around the value of one water molecule. For hardened cement paste,
 319 the smallest pores are in the inter-layer of C-S-H, around one nanometer (1-5 nm)
 320 [30,33–35]. Equilibrium pore sizes are smaller than the smallest pores of cement paste.
 321 This suggests that all liquid water is evaporated at all studied temperatures.

322 Kelvin's law only gives an estimation. We validated experimentally that liquid water is
 323 effectively absent. Indeed, drying conditions at $T=22^{\circ}\text{C}$, $\text{RH}=6\%$ are not directly
 324 comparable to drying at $T=105^{\circ}\text{C}$, $\text{RH}=0.6\%$. For this purpose, we measured the mass
 325 variation of our cement paste at $T=22^{\circ}\text{C}$, $\text{RH}=0.6\%$. A cured cement paste, initially
 326 dried at 22°C and $\text{RH}=6\%$, was dried under vacuum with $\text{RH}=0.6\%$. Decreasing
 327 RH from 6 to 0.6% leads to a mass difference of only 0.3%, negligible compared to the
 328 absorption variations with temperature (5-10%) shown in Fig. 1. Thus, we consider that
 329 all the liquid water is evaporated. We can therefore conclude that the difference in water
 330 absorption measured subsequently is not the indirect consequence of Relative Humidity
 331 variations but the direct consequence of temperature variations.

332

333 Mercury Intrusion Porosimetry confirms the increase in water absorption of the cement
 334 paste (see Fig. 1). Indeed, a significant difference in total porosity between drying at
 335 30°C and 105°C was observed. This increase in water absorption with the drying
 336 temperature may be explained by two phenomena. First, during the drying, because of
 337 the creation of a temperature gradient, microcracking may take place in the hardened
 338 cement paste. This micro-cracking could explain the increase of water absorption with

339 temperature [19,36,37]. The second phenomenon is a dehydration of the hydrates of the
340 cement paste during drying [36–40] potentially causing a collapse of the gel structure.
341 The pore size distributions for the different drying temperatures show that the increase
342 of the drying temperature doubles the pore size in the cement paste (see Fig. 2).
343 However, this result does not allow us to distinguish which phenomenon precisely is at
344 the origin of this increase in pore size. Microcracking creates large pores as could
345 dehydration.

346 However, we submitted a recycled aggregate to multiple drying/humidification steps
347 and showed that the increase in absorption at 105°C is reversible (see Fig. 3). Indeed,
348 after a drying at 105°C followed by a re-imbibition in water and, to finish, drying at
349 30°C, the final measured water absorption was the same as for the reference dried
350 directly at 30°C. This reversible behavior favors the hypothesis of dehydration of
351 hydrates of the cement paste during drying at 105°C. Indeed, in the presence of micro-
352 cracking, the water absorption of the recycled aggregate after the drying cycle would
353 be equivalent to that initially measured during drying at 105°C. We remind the reader
354 here that our paste W/C ratio of 0.5 does not support the idea of a potential self-healing
355 of the micro-cracks during the re-imbibition phase [41,42]. Therefore, we suggest here
356 that microcracking at 105°C is unlikely to create a significant increase in porosity and
357 explain the crease in absorption with temperature.

358

359 We therefore focus now on relating the Ettringite dehydration shown in Fig. 4 to the
360 measured absorption variations. In order to compare the observed increase in porosity
361 with the total volume of Ettringite water, we calculated the volume fraction of water
362 molecules in an Ettringite molecule ($C_6A_3H_{32}$). Considering the molecular weight of
363 each component of Ettringite, the mass proportion of water contained is 46 %. Then,
364 considering that Ettringite density is 1.79 [19,27,43], we estimate that 82 % of the
365 volume of Ettringite corresponds to the volume of the water molecules.

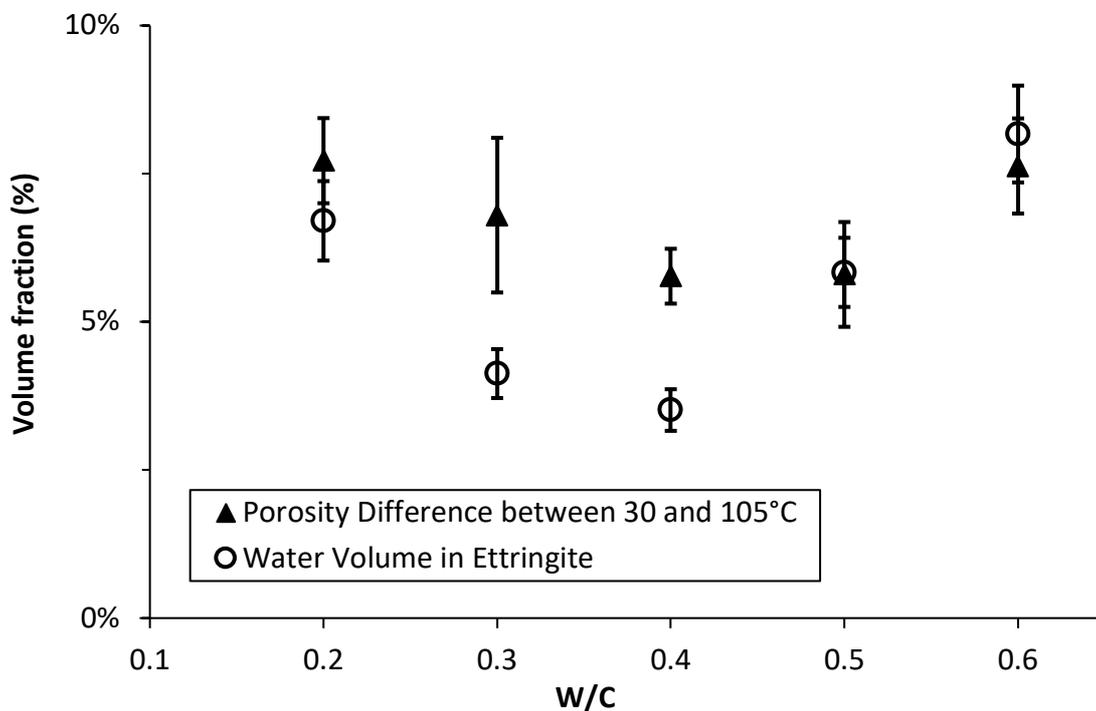
366 The volume content of Ettringite measured experimentally for our cement paste (W/C
367 0.5) is 9.5 %. Thus, the water contained in the Ettringite corresponds to a volume
368 fraction of 8 %. We compare this result with the change of porosity between a drying

369 at 30 and 105°C. The porosity is computed from the water absorption with equation (2):
370 The difference in porosity measured between drying at 30°C and 105°C is also 8 %.
371 Thus, we suggest that the increase in porosity measured when the drying temperature
372 is higher than the ambient temperature is due to the dehydration of Ettringite during
373 drying.

374

375 The same kind of analysis is performed varying the W/C ratio. In **Fig. 5** we observe
376 that whatever the microstructure of the hardened cement paste, drying at high
377 temperature has a very similar impact (on average equal to 5 %) on water absorption.
378 On the other hand, the XRD results presented in **Fig. 6** showed that drying at high
379 temperature leads to dehydration of Ettringite, whatever the W/C ratio of the cement
380 paste. It also showed that the measured Ettringite content did have a complex
381 dependency on the W/C ratio of the cement paste.

382



383

384 **Fig. 7.** Comparison of the volume of water present in the Ettringite for each cement
385 paste (cement L) and the difference in porosity between 30 and 105°C as a function of
386 the W/C ratio.

387

388 From the ettringite content in fig. 6, we computed the volume of bound water and
389 compared this volume and the difference of porosity between drying at 30 and 105°C.

390 In **Fig.7**, we see that the bound water contents in Ettringite follow the same trend as the
391 measured porosity difference between 30 and 105°C.

392 It should be noted that the increase in temperature does not entirely dehydrate Ettringite.

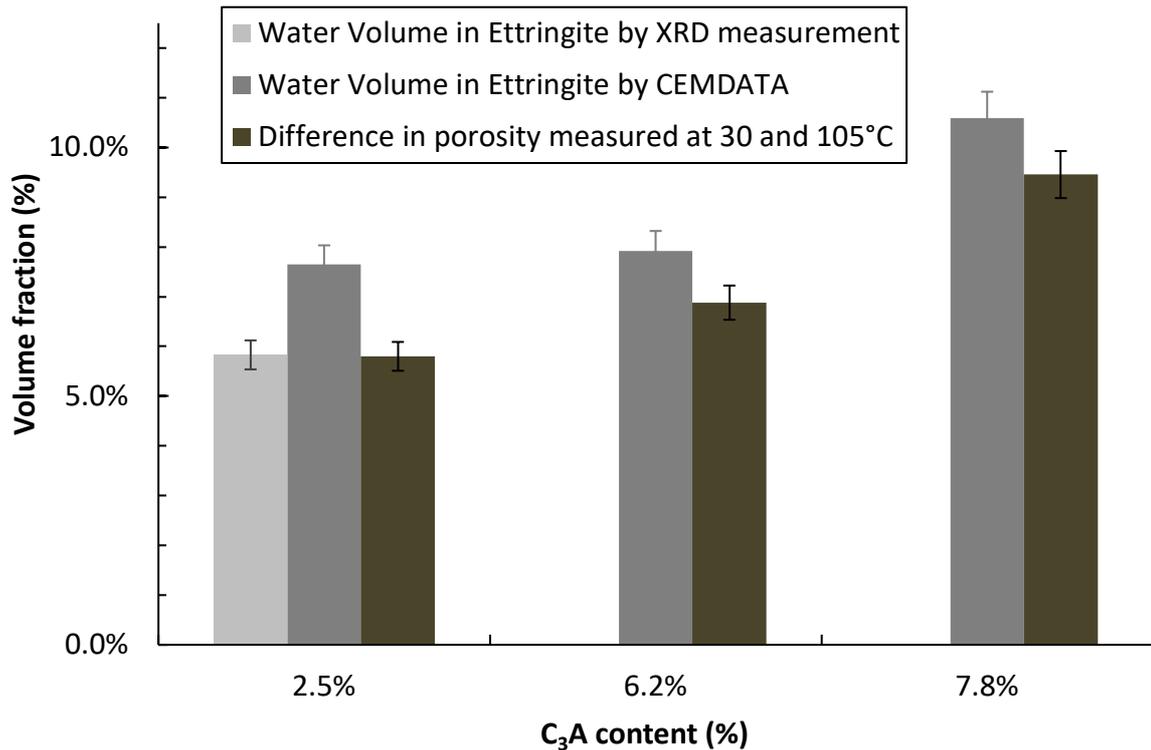
393 It should transform into Metaettringite (12 H₂O) [20,44], which implies that some water
394 molecules remain. Besides, it is likely that other hydrated phases, like Mono-
395 sulfoaluminate, not observed in XRD, may also dehydrate at high temperatures.

396 However, our results show that the contribution of Ettringite dehydration is of the same
397 order of magnitude as the change of absorption.

398

399 To further support the above, we tested cement pastes with a water to binder ratio equal
400 to 0.5 prepared with different cements (cement LT and SPLC) and measured their water
401 absorption at 30 and 105°C. As we use cements with various C₃A content, which
402 influences the amount of Ettringite formed, we assess the influence of the drying
403 temperature on Ettringite content and variation of porosity/absorption. To compare the
404 different cement pastes, we estimated the volume fraction of Ettringite for each cement,
405 using the GEMS software (Gibbs Energy Minimization Software) [45–47] coupled with
406 the CEMDATA database [48,49]. CEMDATA is a database developed specifically for
407 the hydration of Portland cement-based systems. Thus, by knowing the chemical
408 composition of each cement, we modeled the hydration of each of the cement pastes
409 tested. Then, as for the previous results, we calculated the volume of water in Ettringite
410 to the experimental difference of the porosity between 30 and 105°C.

411



412

413 **Fig. 8.** Water volume in Ettringite (by XRD measurement and by CEMDATA) and
 414 porosity difference between 30°C and 105°C, calculated by equation 2, as a function of
 415 the C₃A. Cement pastes at W/C= 0.5 with cements L, LT and SPLC.

416

417 As the C₃A content is increased by a factor of roughly three (from 2.5 to 7.8%), the
 418 variation in porosity between 30 and 105°C roughly doubles (see **Fig. 8**). The Ettringite
 419 contents obtained by simulation follow a similar trend. For C₃A=2.5%, the simulated
 420 Ettringite content is in very good agreement with the XRD measurement. Thus, the
 421 water volume contained in Ettringite, as estimated by thermodynamic simulation, is of
 422 the same order as the difference of porosity between the drying at 30 and 105°C.

423 From the above analysis, we suggest therefore that the increase in water absorption
 424 measured when the drying temperature is higher than ambient temperature is due to the
 425 dehydration of sulfo-aluminates (mostly Ettringite).

426

427

428

429 **5. Practical consequences**

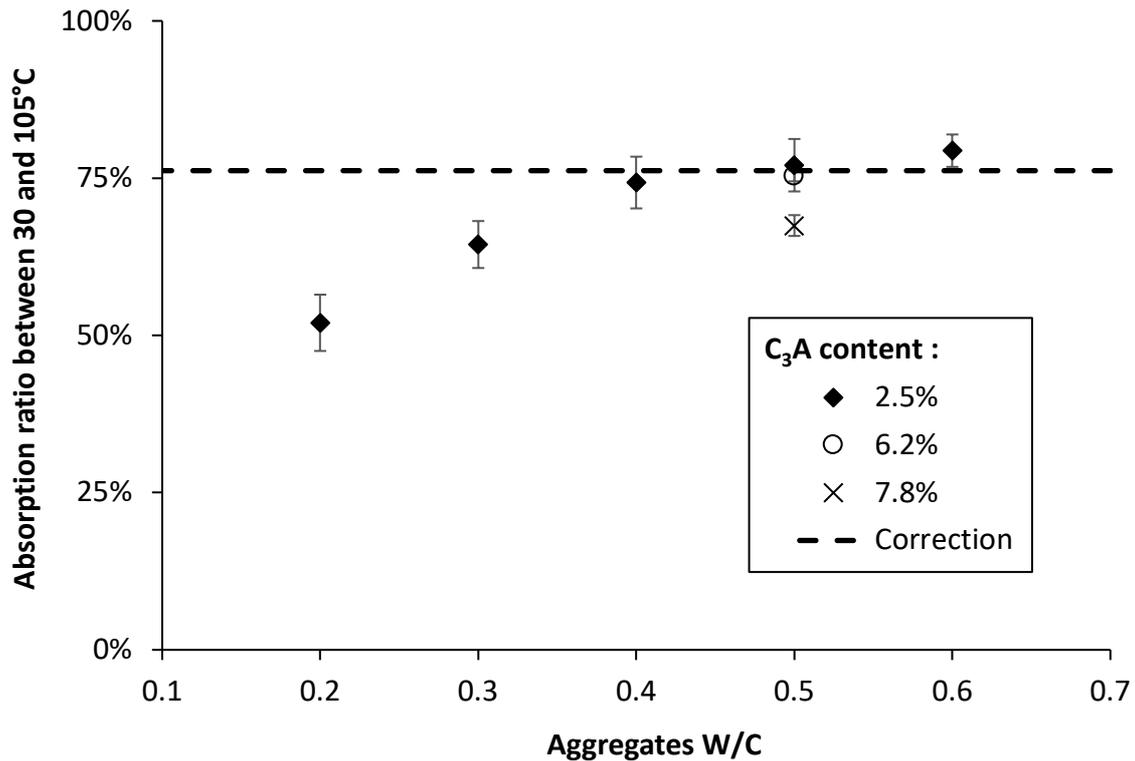
430 **5. 1. Consequences on the measurement of water absorption**

431 As suggested by our results, an increase in the drying temperature of recycled
432 aggregates leads to an overestimation of the actual water absorption of these aggregates,
433 due to the creation of new porosities through sulfo-aluminates dehydration. Such an
434 artefact will occur in all assessment procedures, where aggregates are dried at high
435 temperatures before being tested. This would for instance be the case for pan-drying or
436 oven drying.

437 On the other hand, on an industrial and job-site scale, aggregates are generally stored
438 in silos or hoppers or simply in outdoor heaps. Moreover, in large quantities, thermal
439 inertia is dominant compared to external temperature variations. Thus, even in cases
440 where external temperature exceeds 30°C during daytime, these aggregates should not
441 undergo dehydration before use. However, these aggregates, even if not affected by
442 temperature variations from a microstructural point of view, are exposed to humidity
443 variations and their consequences on water content. It is therefore necessary to dry them
444 in order to measure both their water content and their total water absorption.

445 To solve the above issue, a straightforward approach would lie in drying recycled
446 aggregates at room temperature ($\approx 30^\circ\text{C}$) and low relative humidity. However, drying
447 at this temperature with strict control of relative humidity would result in a very long
448 drying time (around a week), which is not suitable in an industrial context. It is therefore
449 necessary to adapt the determination of absorption by considering the overestimation
450 of the measured absorption value caused by drying at 105°C.

451



452

453 **Fig. 9.** Comparison of the overestimation of the water absorption caused by the drying
 454 at 105°C as a function of the W/C ratio of the cement paste.

455

456 We gather in Fig. 9 the results obtained in this study, i.e. the ratio between the absorption
 457 measured at 30°C and that measured at 105°C. We note that, in the range of standard
 458 concretes (W/C ratio between 0.4 and 0.6), the absorption ratio between 30 and 105 °C
 459 is constant and around 75%.

460 A second-order feature in Fig. 9 is the role played by the C₃A content, which leads to a
 461 slight increase in the overestimation of the absorption value. We however consider here
 462 that, for typical C₃A content and typical W/C ratio, the absorption ratio measured
 463 between 30 and 105°C is fairly constant and equal to 75% with a standard deviation of
 464 3 %.

465 We define the absorption measuring the porous structure of our cementitious materials
 466 as the absorption at 30°C because this temperature does not alter the cement matrix. On
 467 the contrary, the drying at high temperature, in particular the usual preparation at 105°C
 468 degrades the hardened cement paste. Thus, this absorption at 105°C overestimates the
 469 void in the porous media.

470 Based on our results, we propose to estimate the absorption due to the porous structure
471 from the absorption measured at 105°C by: $Abs_{poro} = Abs_{105^{\circ}C} \times 0.75$. The applicable
472 range of W/C is between 0.4 and 0.6 and the initial C₃A content of the cement is less
473 than 6 %.

474

475 **5. 2. Consequences on mix-design and mechanical properties of** 476 **concrete**

477 To emphasize the error in the mix design due to heat treatment in the water absorption
478 measurement, we calculate the differences induced on the effective w/c ratio and the
479 expected mechanical strength. We consider here a cubic meter of an imaginary concrete
480 formulated to reach an effective W/C =0.5 after aggregates absorption. This imaginary
481 concrete contains 400 kg of cement, 700 kg of natural sand 0/4 mm (with negligible
482 absorption) and 1000 kg of dry recycled aggregates (> 4 mm). The water absorption of
483 the recycled aggregates measured at 105°C is equal to 6.0% as in [50].

484 **What is expected to happen:**

485 The volume of water absorbed by the aggregates is expected from the measured
486 absorption value to be 60 L. The total water introduced in the mixer shall therefore be
487 260 L so that the effective water after absorption reaches 200 L and the W/C ratio
488 reaches 0.5.

489 **What effectively happens:**

490 The absorption measurement with drying at 105°C overestimates the absorption of
491 aggregates stored at a lower temperature. Indeed, the absorption due to porosity is
492 $Abs_{poro} = 6.0 \times 0.75 = 4.5$ %. The volume of water absorbed by the aggregates is
493 reduced to 45 L and 15 L of water are therefore not absorbed by aggregates, which
494 results, in turn, into an effective water volume of 215 L instead of 200 L and therefore
495 an increase in the W/C of the concrete from 0.5 to 0.54.

496 In order to roughly assess the impact of this increase in the effective W/C ratio on the
497 mechanical strength, we use the Bolomey formula [51], which predicts compressive
498 strength at 28 days from the W/C ratio. This relation suggests that a decrease in

499 mechanical strength of the order of 10% could be expected.
500 Many studies report a decrease in mechanical strength of the order of 10% when
501 substitution of natural aggregates by recycled aggregates is applied [1,3,6,8]. The
502 question we raise here is whether the systematic decrease in mechanical strength
503 reported in many papers is due to the inherent mechanical properties of recycled
504 aggregates and their interfaces with the cement matrix or could simply be explained by
505 the systematic increase in effective W/C ratio induced by the high temperature artifact
506 for absorption measurement showcased in the present study. However, if the water
507 content is also determined at 105°C, the water content of the aggregates will also be
508 overestimated. Thus, the two errors compensate each other.

509

510 **6. Conclusions**

511 In this paper, we aimed at evaluating the impact of temperature during the drying step
512 on the measurement of water absorption of recycled aggregates. We measured the water
513 absorption of hardened cement pastes at various drying temperatures above ambient
514 temperature of 30°C. We specifically studied the impact of this drying on the pore
515 distribution of these hardened cement pastes as well as on their mineralogical
516 composition. A one-third increase in water absorption was observed between drying at
517 room temperature (30°C and low relative humidity) and drying at 105°C.

518 Our results and analyses suggest that this increase in water absorption with drying
519 temperature increases finds its origin in the dehydration of Ettringite, which results in
520 an increase in the pore size of the hardened cement paste and therefore in an increase
521 in total porosity.

522 Thus, the drying step required to characterize the aggregates, in the case of recycled
523 aggregates and depending on the temperature used, leads to a systematic overestimation
524 of the water absorption. If the absorption and water content are measured with the same
525 preparation protocol, there is no need for correction. However, this overestimation
526 implies in turn a systematic error on the water correction to be applied to the mix design
527 of recycled aggregate concretes. This results in more water being added than necessary

528 to compensate for the volume of water absorbed by the aggregates, thus increasing the
529 effective water to binder ratio of the concrete. This may, in turn, explain the typical
530 decrease in measured mechanical strength when recycled aggregates substitute natural
531 aggregates.

532

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538

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