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Determination of the water content of fillerised fine aggregates in the saturated surface dry state

P. Gentilini, O. Yazoghli-Marzouk, V. Delmotte, Y. Descantes¹

Abstract

An experimental setup designed to capture the saturated surface dry (SSD) state of porous fine aggregates incorporating significant amounts of fines is presented. This setup is validated upon testing fine aggregate sources with different water absorption values up to 5.6 %, and at least 8 % of fines. Test duration is found to lie in the 120-to-240-minute-range, depending on test sample initial mass and water content. Several procedures for calculating the water content in SSD state are presented and discussed. One of these procedures uses the drying theory to fit curves depicting water content shift as a function of drying time and identify breakpoints. A more straightforward strategy is focused on the transition point between retained and free water on charts representing the variations of water content as a function of relative humidity. In both cases, fairly repeatable and discriminant water content values in SSD state are obtained. As expected, these values are found significantly higher than those determined in the absence of fines according to the usual cone test [1]. Moreover the lack of reliability of water absorption values determined in the presence of fines according to the cone test procedure is verified. Eventually, several perspectives are pointed out to improve the setup which could easily be automated.

Keywords: fine aggregate, sand, cement concrete, water absorption, test method, experimental setup, drying

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23 1. Introduction

24 Short term and long term performances of cement concrete are known to
25 depend on effective-water-to-cement ratio (W_{eff}/C), where effective water refers to
26 water available for cement hydration. Effective water may be defined as the total mass
27 of water contained in the cement concrete (W_{tot}) less the mass of water retained by the
28 aggregate fractions. For a given aggregate source, this 'retained water' is known to
29 increase with decreasing size fraction, especially in the fine fractions 0/4 or 0/5 mm. The
30 maximum retained water is routinely though disputably [2] assessed by the 'water
31 absorption' value (WA) – mass of water penetrating the voids of particles during a
32 prescribed period of time, routinely 24h, expressed as a percentage of the oven-dried
33 mass of the aggregate sample [1]. For a long time, the large quantities of fine aggregates
34 needed for cement concrete production have been purveyed almost exclusively by
35 natural fine aggregates (NFA) from alluvial or limestone deposits, whose WA values are
36 known to be low (< 1%). In the recent years, the shortage of alluvial sources and waste
37 storage capacity limitations have given rise to a worldwide interest in abundant
38 alternative fine aggregate sources such as recycled concrete fine aggregates (RCFA).
39 However, their use is still limited due to high WA values in the range 6-10 % [3,4].

40 Indeed, adjusting the W_{eff}/C ratio of fresh cement concrete becomes much more
41 difficult when fine aggregates with high WA value are incorporated, since they may add
42 or subtract water allotted for cement hydration, thus impact its short term workability
43 as well as mechanical and durability performances in hardened state [5][6]. Khatib [7]
44 reported the good workability of fresh cement concrete incorporating 100% RCFA with
45 additional water to reach $W_{\text{tot}}/C = 0.5$ (no plasticizer); however, he also noted a
46 compressive strength drop down to 35% after 28 days of curing and a drying shrinkage

47 increase up to 50% when compared with a reference cement concrete incorporating
48 NFA exclusively. Reducing the W_{tot}/C ratio upon using an admixture allows both to
49 maintain sufficient workability and prevent compressive strength drop, but concrete
50 properties in hardened state are still impacted: splitting resistance may decrease by
51 30%, water absorption and sorptivity may increase by 46% and 70% respectively with
52 respect to corresponding values of the reference cement concrete. As a consequence,
53 external fluids may penetrate the concrete microstructure and introduce deleterious
54 substances such as carbon dioxide and chloride ions, causing concrete carbonation and
55 steel corrosion respectively [8].

56 The water absorption of alternative fine aggregates such as RCFA having
57 significant implication on W/C ratios for concrete mixtures, determining it accurately is
58 critical [4]. Therefore, most test methods consist of drying a pre-saturated sample until
59 achievement of the state under which particles surface is free of vaporizable moisture
60 but their accessible pores remain saturated with water, called saturated-surface-dry
61 state (SSD, see Figure 1), then in promptly weighing the sample. To capture the SSD
62 state, some methods focus on the visual detection of test portion color change when
63 particles become free of surface vaporizable moisture [9,10]. Others track the slump of
64 a cone-shaped test portion [1,11,12] or the flow of a test sample off a tilted masonry
65 trowel [10] which occur when between-particles surface tension forces disappear.
66 Others spot slope breakpoints on curves measuring physical characteristics of test

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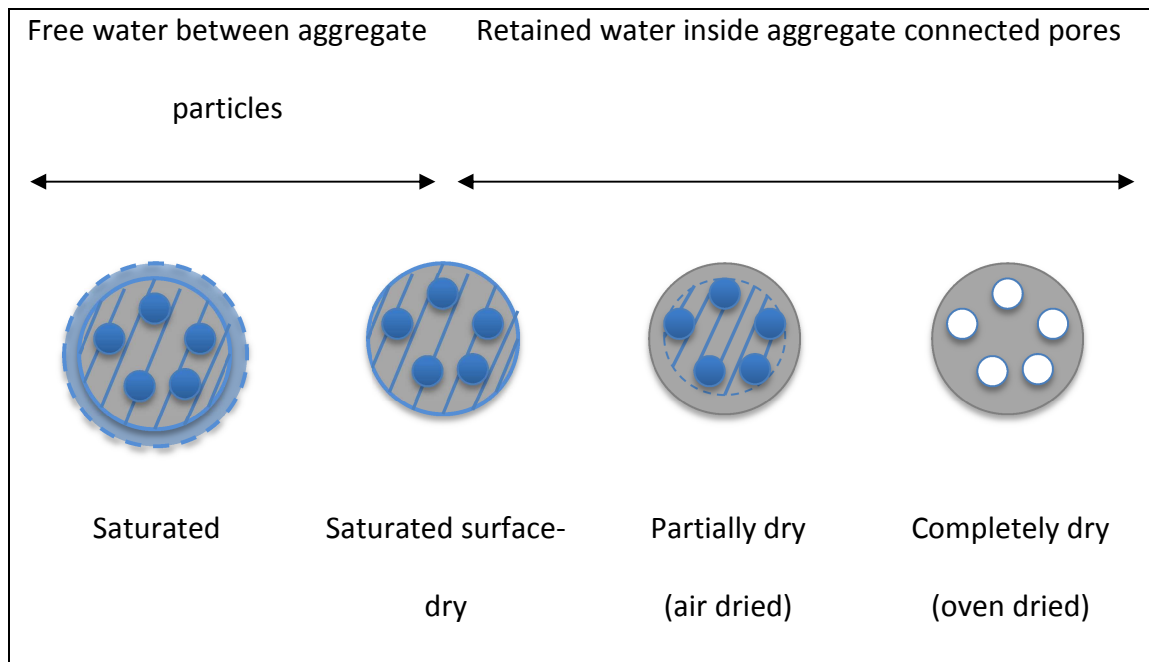


Fig. 1. Schematic view of an aggregate particle for various water content values.

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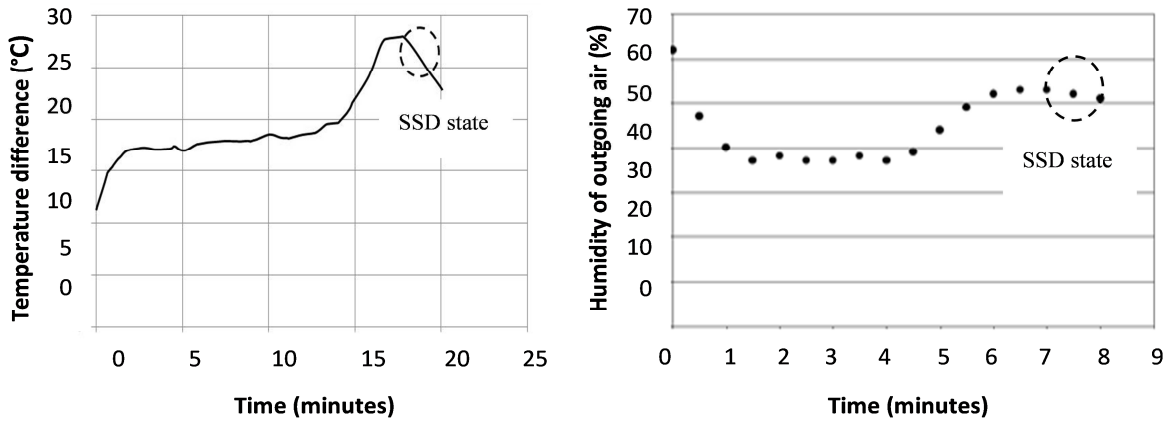
samples as a function of drying time, such as electrical resistivity reflecting the presence of between-particles water bridges [13]. A few test methods attempt to detect the end of the saturation phase while soaking a dry test portion, either from direct measurement of the volume of absorbed water [14], or from detecting the reflection signal of infra-red light rays when particles surface becomes moist [15]. Unfortunately, most of these test methods are unsuitable for fine aggregates such as RCFA, which may incorporate up to 10-13% of fines (particles passing the 0.063 mm sieve) [16,17]. Indeed, Rogers et al [18] have reported a 100% overestimation of WA values assessed from methods using the cone slump criterion [1,11,12], which they allotted to fines adhering to larger particles and forming clusters, thus trapping part of the formerly free water into their inner structure and causing earlier achievement of the sample 'SSD' state than in the absence of fines. Similarly, methods focused on test portion color change yield scattered results in the presence of fines, since homogenously drying a sample becomes delicate [19].

86 Promising WA values of fine aggregates were measured using test methods
87 derived from thermodynamic [19,20] or industrial drying approaches [21]. The former
88 consists in air drying a pre-saturated fine aggregate sample placed into a rotating drum
89 closed at both ends by 0.075 mm square mesh screens, while recording the temperature
90 gradient between drum inlet and outlet as well as relative humidity at drum outlet. The
91 drying phase is stopped and the sample is weighed when the temperature gradient or
92 relative humidity curves evidence breakpoints similar to those shown on Fig. 2,
93 presumably representative of SSD state according to the authors. The latter assimilates
94 the drying of soaked aggregates at rest in a chamber to the evaporation of water from a
95 porous medium at constant air temperature. Fig. 3 depicts sample mass reduction
96 through evaporation as a function of time, which consists of three well-known phases
97 [22]:

- 98 - a transition phase corresponding to the drying chamber conditioning;
- 99 - then, a linear part depicting the evaporation of surface water at a constant rate
100 monitored by environmental conditions (temperature and relative humidity) until
101 achievement of the SSD state;
- 102 - Eventually, a phase at decreasing mass reduction rate evidencing the evaporation of
103 absorbed water from aggregate pores.

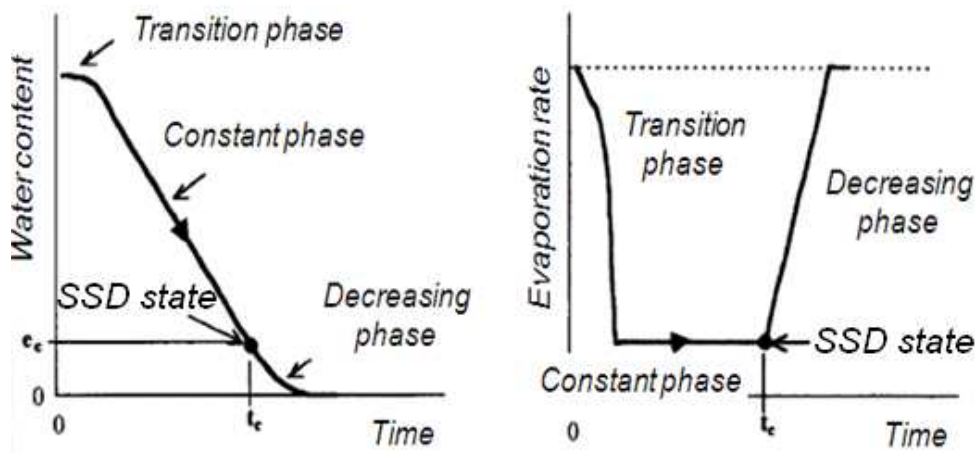
104 Though promising, these two test methods have drawbacks: the drum method is
105 biased by a loss of fines through the screens and disassembly operations which delay
106 the weighing once SSD state is achieved; the drying chamber is unsuitable for uniformly
107 drying polydisperse fine aggregates at rest due to crusting effects [22].

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Fig. 2. Temperature gradient versus time (left) and relative humidity versus time (right) plots [19].



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Fig. 3. Water content (left) and evaporation rate (right) in a porous material [21].

116 In order to determine the water absorption of fine aggregates incorporating
117 fines, the present paper introduces an improved experimental setup designed to
118 capture the SSD state and measure the water content in this state. Section 2 describes
119 the experimental setup and the test program designed for its validation. Section 3
120 investigates the presence of breakpoints on various curves representing recorded data
121 and discusses methods for assessing the water content in SSD state.

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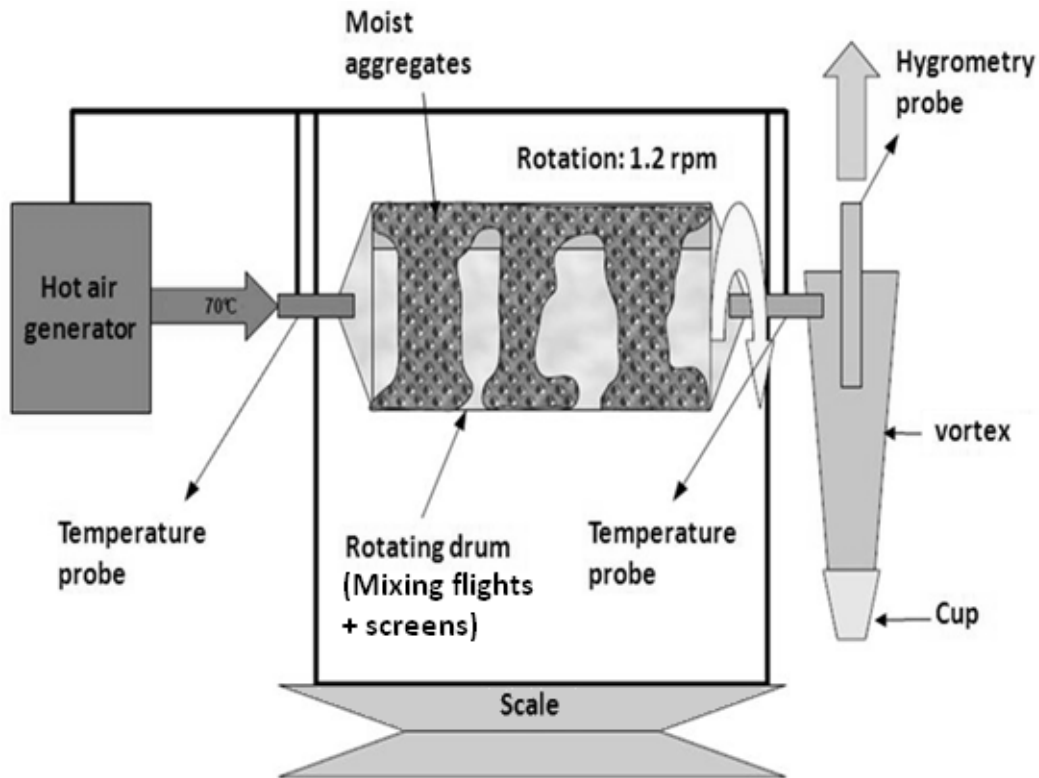
123 **2. Experimental setup and test program**

124 **a. Experimental setup**

125 The experimental setup used to capture the SSD state is inspired from early
126 development by Dana et al [20] and subsequent development by Kandhal et al [19]. As
127 shown in Fig. 4, it is composed of a hair dryer that blows warm air at one end of a
128 rotating drum, which is mounted on caster wheels and driven by a belt through a step
129 motor. The drum incorporates flights to stir the test sample in order to achieve
130 reasonably uniform drying, as well as screens at drum inlet (0.063 mm opening) and
131 outlet to reduce aggregates loss. Temperature is measured by two probes located at
132 either ends of the drum and relative humidity is captured by a hygrometry probe
133 located at drum outlet. However, three major improvements have been implemented:
134 first, both the screen blinding and loss of fines issues reported by Kandhal et al [19] have
135 been sorted out using respectively an outlet screen with a wider opening (0.5 mm), and
136 a fines collector consisting of a simple vortex system equipped with a cup, in which fines
137 will remain trapped till the end of the test (see Fig. 4); second, no more drum removal is
138 needed for weighing as the full experimental setup is mounted on a balance, thus
139 allowing continuous weight monitoring to ± 1 g; third, the air dryer is fed by a voltage
140 regulator to monitor the blown air temperature to roughly 70°C, that is not too high to
141 avoid removing crystallized water in the mortar attached to the aggregate [23,24]
142 though sufficiently high to keep the drying time reasonable. The total mass of unloaded
143 experimental setup mounted on the balance is 13.1 kg.

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Fig. 4. The experimental set-up used for the determination of SSD state.

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b. Test program

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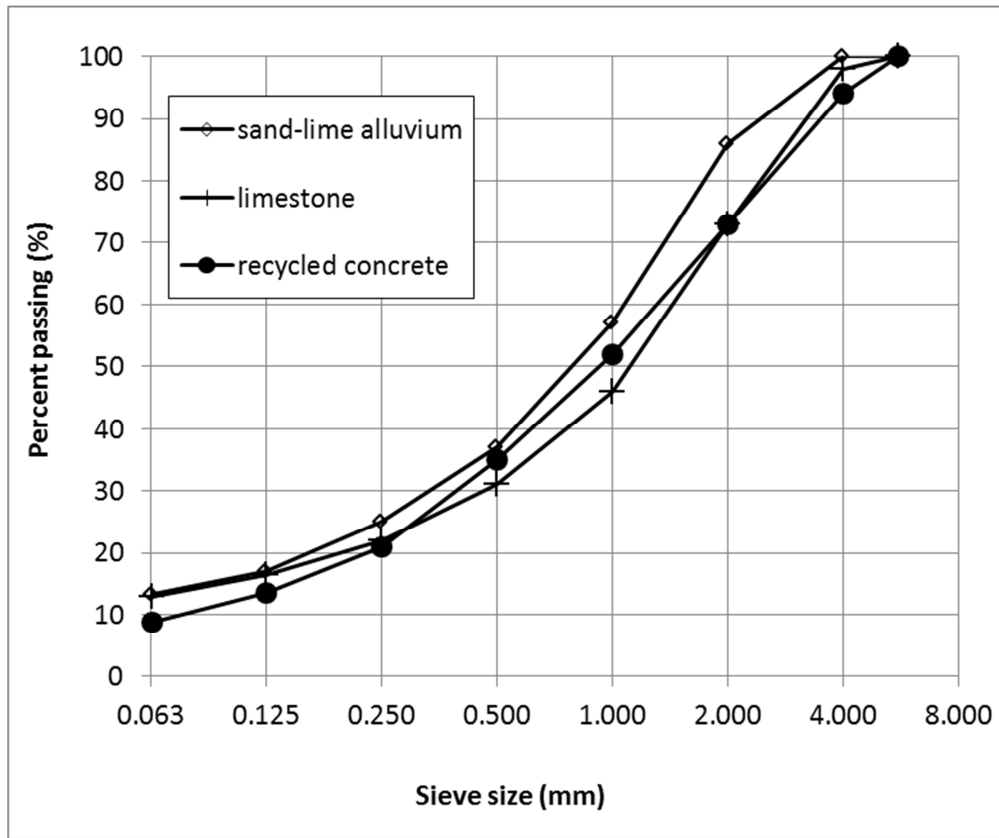
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Tested materials were sampled from three different sources, crushed sand-lime alluvium, crushed limestone and recycled concrete. Fig. 5 depicts the mean grading curve of each material tested, all of them incorporating at least 8% fines. Table 1 summarizes the densities and water absorption values determined for each material according to EN 1097-6 clause 9. The selected materials cover a reasonably wide range of WA values, from 0.8 for NFA up to 5.6% for RCFA, and the corresponding densities vary conversely as already noticed elsewhere [7,25]. Although the test procedure depicted in EN 1097-6 clause 9 prescribes fines discarding, a second set of samples were tested with their fines, supplemented with methylene blue (MB) tests

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Fig. 5. Grading curves of tested materials.

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Table 1. Densities, water absorption and MB values of tested sands.

		sand-lime alluvium		limestone	recycled concrete	
		#1	#2		#1	#2
immersion time (h)		24		24	96	
no fines	ρ_{ssd} (t/m ³)	2.68	2.67	2.61	2.41	-
	WA ₂₄ (%)	0.8	1.1	2.2	5.6	-
with fines	WA ₂₄ (%)	1.5	-	5.4	7.1	7.3
	ρ_{ssd} (t/m ³)	2.65	-	2.50	2.34	2.36
	MB (g/kg)	0.8	-	0.9	-	-

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166 performed on 0/2 mm samples in accordance with EN 933-9 [26] to check the fines
 167 quality. As expected [18], water absorption values in the presence of fines are
 168 systematically higher than those measured without fines, although MB values suggest
 169 low clay contents.

170 For each material source, three 0/4 mm test samples were prepared with
 171 random dry masses and initial moisture contents as mentioned in Table 2. Moisture
 172 content differences were obtained upon immersing each sample for at least 24h (96 h
 173 for RCFA, see Table 1) and then letting it drain at room temperature for some time.
 174 Samples were subsequently introduced into the experimental setup and air dried until
 175 their masses reach equilibrium. Meanwhile, room, drum inlet and outlet temperatures
 176 were recorded every second, as well as relative humidity. Every minute, the drum
 177 rotation was stopped for a few seconds in order to record the mass of the loaded
 178 experimental setup. Once the mass equilibrium was achieved, each sample was further
 179 oven-dried at 105°C until constant mass in order to assess its dry mass according to EN
 180 1097-5.

181 Table 2. Dry masses and initial moisture contents of the test samples.

	sand-lime alluvium			Limestone			recycled concrete		
	#1	#2	#3	#1	#2	#3	#1	#2	#3
Immersion time (h)	24			24			96		
Dry mass (g)	444.1	940.2	430.8	422.0	590.8	909.8	618.4	408.4	371.6
Initial moisture content (%)	20.0	16.6	18.6	21.2	20.3	18.7	16.1	39.3	39.7

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183 **3. Results and discussion**

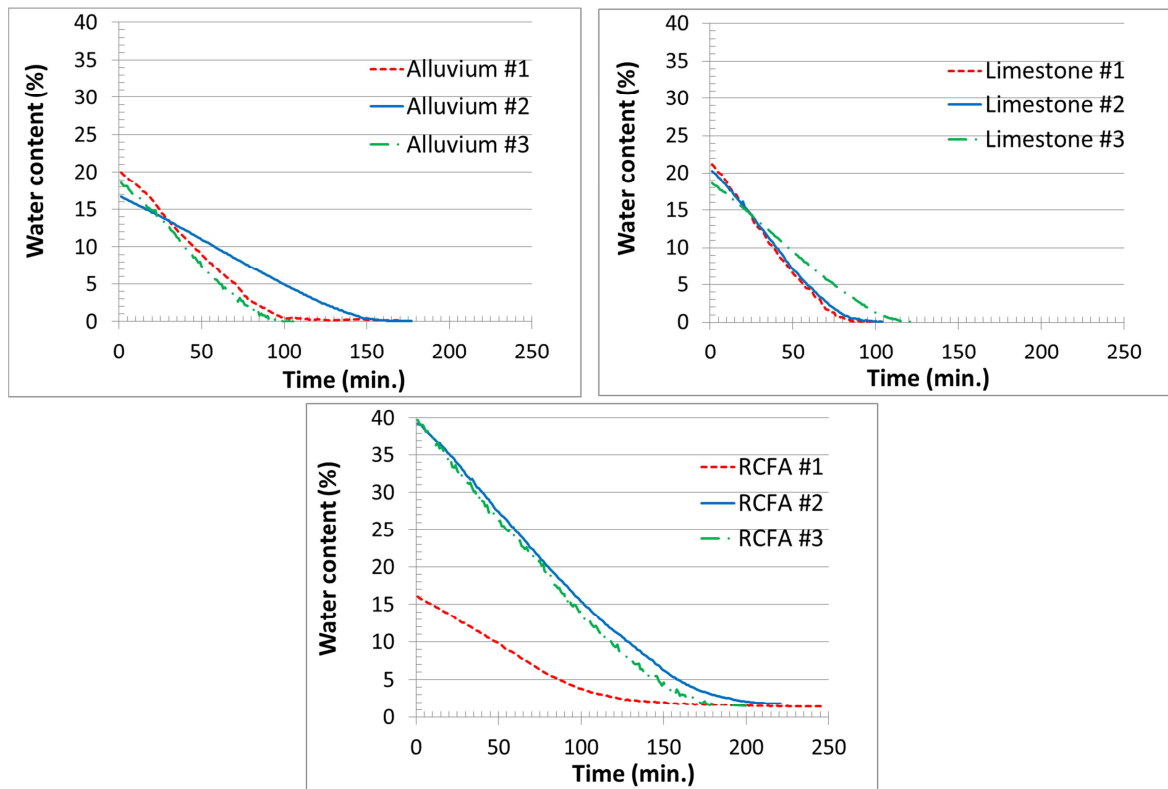
184 **a. Water content as a function of drying time**

185 Fig. 6 represents the variations of the water content of all tested samples as a
186 function of drying time. Several observations arise from this figure.

187 First, the test duration is comprised between 120 and 240 min, which seems
188 much longer than the test duration reported by Kandhal et al [19] with 500-g-samples.
189 However, no objective comparison may be conducted since Kandhal et al [19] discarded
190 particles below 0.6 mm and provided no information on initial water content (the lower
191 the quicker the test) or inlet air temperature (the higher the quicker the test).

192 Next, the residual water content at the end of the non-linear phase is zero
193 except for RCFA, whose residual water content ranges between 1.4 and 1.9 %. This
194 observation suggests that, as expected, the moderate drying temperature used (70°C)
195 efficiently prevents the dehydration of hydrates, with part of the water remaining
196 trapped into micropores or mesopores of the cement paste [27–29]. This aspect will not
197 be investigated further as it falls beyond the scope of the present paper.

198 Furthermore, despite the absence of a transition phase that may be explained by
199 a rather low thermal inertia of the experimental setup, any curve in Fig. 6 evidences a
200 linear phase depicting the evaporation of surface water at constant rate. This phase is
201 followed by a non-linear one at decreasing evaporation rate, which corresponds to the
202 evaporation of absorbed water. These similarities with the drying theory suggest using it
203 in order to assess the SSD state separating these two phases.



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Fig. 6. Water content as a function of time for all tested samples.

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b. SSD assessment from water content versus drying time curves

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Given the experimental setup used to dry a test sample, assessing its SSD state from the water content versus time curve according to the drying theory principles requires the following three assumptions be made: first, the stirring action of flights inside the rotating drum ensures fairly homogenous drying; second, as long as free water (i.e. not retained by the aggregate) may evaporate, the air/aggregate exchange surface has a constant area that remains saturated; last, variations of the laboratory temperature, relative humidity and air generator temperature are neglected.

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216

Under these assumptions, the evaporation rate during the linear drying velocity phase is controlled exclusively by the air/aggregate exchange surface area and the

217 thermodynamic characteristics of the humid air inside the rotating drum (relative
 218 humidity and temperature). Hence it may be expressed as [22]:

$$219 \quad \frac{dW}{dt} = -\frac{hA(T_a - T_s)}{L_v M_s} = -\frac{ha(T_a - T_s)}{L_v \rho_{app}} = -C_1 \quad \text{for } W \geq W_{SSD} \quad (1)$$

220 where W is the test sample water content (%), h is the thermal transfer coefficient
 221 between air and test sample surface ($W \cdot m^{-2} \cdot K^{-1}$), A is the air/test sample exchange
 222 surface area (m^2), $T_a - T_s$ is the mean difference between air and test sample surface
 223 temperatures inside the rotating drum (in K, T_s being equal to the air humid
 224 temperature), L_v is the latent heat of vaporization at test sample surface temperature
 225 ($J \cdot kg^{-1}$), M_s is the test sample dry mass (kg), a and ρ_{app} are the specific surface (m^2/m^3)
 226 and apparent density (kg/m^3) of the test sample respectively, and C_1 is a positive
 227 constant ($\% \cdot s^{-1}$). Observe from equation (1) that the slope of this linear phase shall be
 228 material dependent, since different materials generally have different grading curves,
 229 hence different specific surfaces, and apparent densities. Furthermore, for a given
 230 material source, samples with significantly higher masses shall yield significantly lower
 231 absolute slopes, since A/M_s ratio in a cylinder decreases when M_s increases (this
 232 property, valid as long as the test sample fills no more than half of the rotating drum,
 233 may be mathematically demonstrated upon observing that A may be approximated by
 234 the horizontal free surface area of the test sample at rest in the same cylinder without
 235 flights). Notice that curves depicted in Fig. 6 meet these qualitative predictions.

236 Still under the same assumptions, the capillary theory applicable to granular test
 237 samples allows to express the evaporation rate during the non-linear drying velocity
 238 phase as [22]:

$$239 \quad \frac{dW}{dt} = -C_2(W - W_{res}) \quad \text{for } W \leq W_{SSD} \quad (2)$$

240 where C_2 is a positive constant (s^{-1}) and W_{res} stands for the residual water content of test
 241 sample at the end of the drying operation.

242 Upon integration, equations (1) and (2) yield the following system of equations:

$$243 \begin{cases} W = -C_1 t + W_0 & \text{for } t \leq t_{SSD} \\ W = e^{-C_2 t + C_3} + W_{res} & \text{for } t \geq t_{SSD} \end{cases} \quad (3)$$

244 Where W_0 is the water content at $t=0$ according to the linear fit, t_{SSD} is the drying time
 245 when $W = W_{SSD}$ and C_3 is a constant. For each curve depicted on Fig.6, constants C_1 , W_0 ,
 246 C_2 and C_3 may easily be determined upon fitting the relations of system 3 to their
 247 respective phases using the least squares method. Eventually, W_{SSD} may be calculated
 248 upon eliminating the time in system (3) at $t = t_{SSD}$ and solving the following equation (for
 249 example using the Newton-Raphson's method):

$$250 f(W_{SSD}) = W_{SSD} - \left(e^{-\frac{C_2}{C_1}(W_0 - W_{SSD}) + C_3} + W_{res} \right) = 0 \quad (4)$$

251 For each curve depicted on Fig. 6, Table 3 summarizes the calculated values of
 252 constants C_1 , W_0 , C_2 and C_3 as well as two coefficients of determination R^2 reflecting the
 253 quality of linear and non-linear fits. With R^2 values of 1.00, the linear fits quality is
 254 excellent, and the non-linear fits are also very good with R^2 values ranging from 0.84 up
 255 to 0.99. A consequence of the capillary theory applied to the drying of fine aggregates is
 256 that the rate of evaporation should vary continuously between the linear and non-linear
 257 phases. This may be checked upon equalizing equations (1) and (2) when $W=W_{SSD}$ [22],
 258 which yields the following relation:

$$259 C_1 = C_2(W_{SSD} - W_{res}) \quad (5)$$

260 For each tested sample, Table 3 displays the values of
 261

Table 3. Fit of linear and non-linear phases of drying curves and water content values.

	sand-lime alluvium			limestone			recycled concrete		
	#1	#2	#3	#1	#2	#3	#1	#2	#3
C_1 (% $\cdot\text{min}^{-1}$)	0.227	0.121	0.249	0.306	0.282	0.192	0.133	0.247	0.252
W_0 (%)	20.4	17.1	20.0	21.7	21.4	19.2	16.3	39.9	39.1
R²	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
C_2	0.069	0.054	0.084	0.085	0.095	0.074	0.030	0.041	0.039
C_3	6.3	7.3	6.8	6.3	7.3	7.3	3.9	7.1	6.6
R²	0.98	0.94	0.84	0.94	0.92	0.87	0.99	0.85	0.91
$ [C_1 - C_2(W - W_{res})]/C_1 $	15%	2%	16%	11%	19%	15%	15%	15%	20%
W_{res} (%)	0.1	0.1	0.0	0.0	0.1	0.1	1.4	1.9	1.5
W_{SSD} (%)	2.9	2.3	2.5	3.2	2.5	2.3	6.5	7.0	6.7
W_{trans} (%)	2.7	2.2	2.7	3.5	2.7	2.8	6.4	7.0	6.0
$ W_{trans} - W_{SSD} $ (%)	0.2	0.1	0.2	0.3	0.2	0.5	0.1	0.0	0.7

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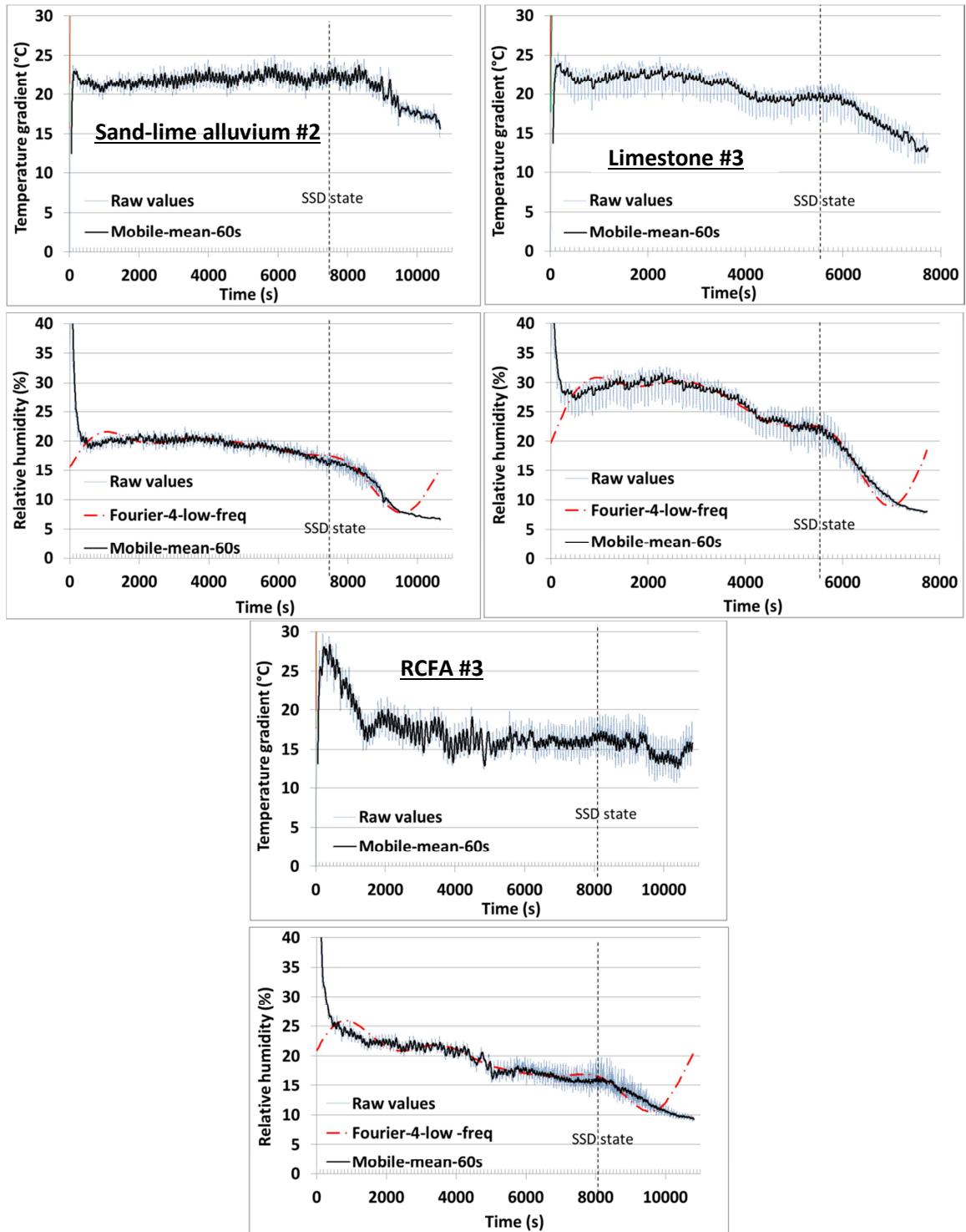
264 $|C_1 - C_2(W_{SSD} - W_{res})|/C_1$ which range between 2 and 20%. These low deviations to
 265 zero might reflect minor diffusion phenomena, whose study falls beyond the scope of
 266 the present paper.

267 Table 3 also gathers water content values calculated for each test sample from
 268 equation (4). Mean water content values in SSD state for sand-lime alluvium, limestone
 269 and RCFA are 2.6, 2.7 and 6.7 % respectively. As expected, observe that these values are
 270 higher than corresponding WA_{24} values reported in Table 1 in the absence of fines. With
 271 fines, comparison between W_{SSD} and WA_{24} values is chancy, which reflects the
 272 irrelevance of the cone slump test in the presence of fines. For any material source but
 273 limestone, the standard deviation of W_{SSD} values is about 0.3 %, which is close to the 0.2

274 % repeatability standard deviation determined on natural fine aggregates in the absence
275 of fines (see EN 1097-6, Table I.4). For limestone, the standard deviation is just below
276 0.5 due to the relatively high value calculated for test sample #1.

277 **c. SSD assessment from temperature or relative humidity versus time curves**

278 Dana et al and Kandhal et al [19,20] have suggested that the SSD state could be
279 captured upon detecting breakpoints on curves depicting temperature gradient ('inlet –
280 outlet' temperature) or relative humidity as a function of time. In order to check this
281 statement, Fig. 7 represents for each material source tested (one test sample per
282 material) temperature gradient and relative humidity variations as a function of time.
283 Depending on test sample initial mass and water content, as well as heat generator
284 fluctuations (± 1.5 °C), these curves acquired every second while both the heat generator
285 and rotating drum were operating appear more or less noisy. To mitigate the noise,
286 mobile means over 60 s (corresponding to the weighing period) have been represented
287 on each chart. These curves are clearly very different from those reported by Kandhal et
288 al [19] (see Fig. 2). Despite a slight downward trend visible mainly on relative humidity
289 curves, the temperature gradient curves and, above all, the relative humidity curves
290 evidence fairly clear drop breakpoints. These are located on each curve past the vertical
291 dotted line depicting the achievement of the SSD state as calculated from the
292 corresponding water content versus drying time curves. The temperature gradient drop
293 is caused by test samples warming once free surface water has totally evaporated.
294 However, temperature charts on Fig. 7 show that this warming is
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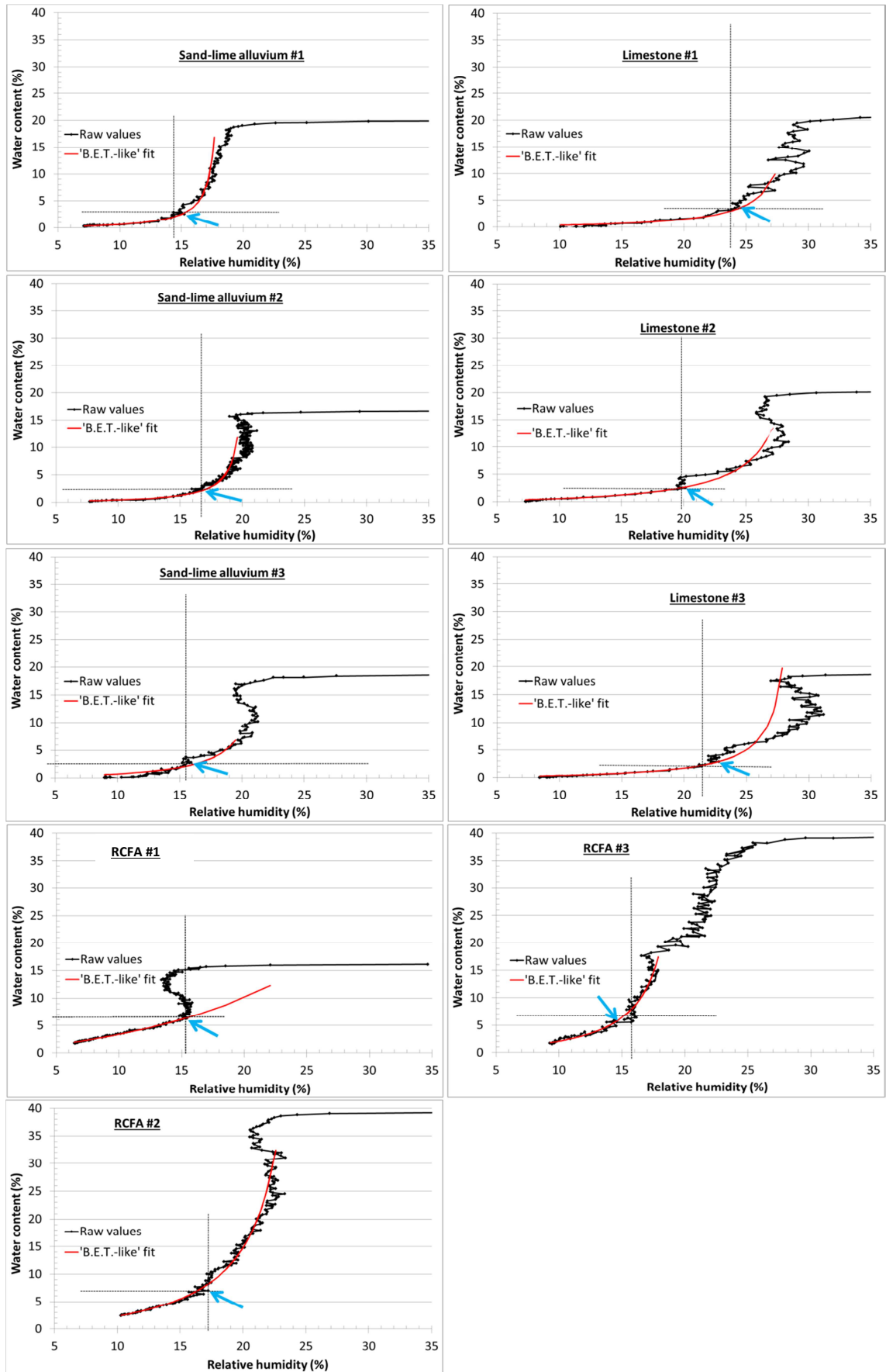
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Fig. 7. Temperature gradient (top figures) and relative humidity (bottom figures) variations as a function of time for all materials tested.

301 offset by 5-25 min. from the *SSD* state, which may be explained by the presence of
302 water trapped in fines porosity and the fairly high specific heat of aggregates (about
303 $1000 \text{ J.Kg}^{-1}.\text{K}^{-1}$). Contrariwise, Fig. 7 suggests that drop on relative humidity curves
304 occurs more promptly once the *SSD* state is achieved, which tends to confirm the
305 assumption of homogenous drying. Upon sampling every 60s and preserving only the 4
306 lowest frequencies, Fourier fits performed on relative humidity versus time curves yield
307 smooth curves whose last local maxima just before the drops superimpose remarkably
308 well with corresponding *SSD* states.

309 **d. *SSD* assessment from water content versus relative humidity curves**

310 In order to improve the straightforwardness and possibly the accuracy of *SSD*
311 state assessment, the two physical properties found to yield the most accurate
312 assessment of the *SSD* state among those recorded were represented one as a function
313 of the other. Hence, for each material tested, Fig. 8 depicts water content variations as a
314 function of relative humidity. Although the inlet temperature is kept reasonably
315 constant all through the drying operations, with the temperature gradient fairly
316 constant until achievement of the *SSD* state (see Fig. 7), curves depicted on Fig. 8 should
317 not be confused with actual water vapour desorption isotherms as defined by IUPAC
318 [30]. Indeed, actual water vapour desorption isotherms are usually determined for a
319 discrete set of relative humidity values, which are successively imposed during several
320 days using various saturated salt solutions until test sample mass achieves an
321 equilibrium [31]. Here, drying operations are quick (less than 4 hours), which partly
322 explains curves fluctuations, and no static equilibrium
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Fig. 8. Water content as a function of relative humidity (3 test samples/material).

326 state is reached. Nevertheless, the outline of curves depicted in Fig. 8 looks similar to
 327 that of water vapour desorption isotherms: a fairly flat phase at the end of the drying
 328 operation (relative humidity about 10-15%) corresponds to minor water content shift;
 329 this phase is preceded by a steep one evidencing important water content change at
 330 higher relative humidity (typically about 15 to 25% depending on material tested). The
 331 transition between these two phases may well be seen as the drying point beyond
 332 which water becomes much harder to eliminate from the material. In other words, this
 333 point is representative of the retained water content assessed by the SSD state. In order
 334 to precisely locate this transition point on each curve shown in Fig. 8, relative humidity
 335 values in the range [5 %; 25 %] may be fit using the mathematical expression of the B.E.T
 336 model [32]:

$$337 \quad \frac{HR}{(1-HR)W} = \frac{1}{CX_{12}} + \frac{C-1}{CX_{12}}HR \quad (6)$$

338 where HR is relative humidity and C and X_{12} are constants. As shown in Fig. 8, fit quality
 339 is very good (R^2 in the range [0.84; 0.97]) for all test samples but alluvium sample #3
 340 ($R^2=0.66$). Fit parameters C and X_{12} range in [-4.5; -1] and [-0.005; -0.249] respectively
 341 (note that these parameters would have positive values for actual water vapour
 342 adsorption isotherms, since parameter X_{12} for example denotes a water content). The
 343 transition point sought may be defined as the point where the fit significantly differs
 344 from actual curve, which may be identified graphically. For each curve depicted in Fig. 8,
 345 a blue arrow spots this transition point, whereas the intersection point of two
 346 perpendicular dotted lines highlights the achievement of the *SSD* state as determined
 347 from Table 3. Observe that the transition point provides a very good assessment of the
 348 *SSD* state, as confirmed by the last three rows of Table 3.

349 **4. Conclusion and perspectives**

350 An improved experimental setup was introduced in order to capture the *SSD*
351 state of porous fine aggregates incorporating significant amounts of fines, such as
352 recycled concrete fine aggregates. This setup was used to test three fine aggregate
353 sources with different water absorption values up to 5.6 % (measured in the absence of
354 fines) and at least 8 % of fines. Test duration was found to lie in the range 120-240
355 minutes, which suggests differences from the shorter duration reported by Kandhal et al
356 [19]. Nevertheless, curves depicting water content shift as a function of drying time
357 were found similar to those reported in the drying theory literature. As a consequence,
358 the drying theory was used to fit these curves and a calculation procedure was
359 explained in full detail to assess the *SSD* state. Fairly repeatable and discriminant water
360 content values in *SSD* state were obtained using this procedure. As expected, these
361 water absorption values were found significantly higher than those determined in the
362 absence of fines according to the usual cone test [1]. Furthermore, the lack of reliability
363 of water absorption values determined in the presence of fines according to the cone
364 test procedure was verified. Besides, as reported by Dana et al and Kandhal et al [19,20],
365 curves representing temperature gradient or relative humidity shift as a function of time
366 were found to evidence drop breakpoints, however the latter appeared to be located
367 closer to actual *SSD* state than the former. Eventually, a more straightforward strategy
368 for assessing the water content in *SSD* state was suggested, which consists in identifying
369 the transition point between retained and free water on charts representing the
370 variations of water content as a function of relative humidity.

371 Although the experimental setup was found able to assess the water content of
372 fine aggregates incorporating fines in *SSD* state - even in the case of recycled concrete
373 fine aggregates thanks to moderate drying -, several perspectives should be pointed out.
374 First, recorded curves tend to be noisy, which causes results scattering that could be
375 avoided by using a more stable hot air generator setup. Second, test duration could be
376 reduced upon optimizing the sample mass. Next, automation of the device would make
377 no difficulty. Eventually, more theoretical work is needed to model the full outline of
378 water content versus relative humidity curves determined using the experimental setup.

379

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384

385 **5. References**

- 386 [1] CEN. Tests for mechanical and physical properties of aggregates - Part 6:
387 Determination of particle density and water absorption. 2014.
- 388 [2] Kasemchaisiri R, Tangtermsirikul S. A method to determine water retainability of
389 porous fine aggregate for design and quality control of fresh concrete. *Constr Build*
390 *Mater* 2007;21:1322–34. doi:10.1016/j.conbuildmat.2006.01.009.
- 391 [3] RILEM. RILEM recommendation I2I-DRG Guidance for demolition and reuse of
392 concrete and masonry. Specifications for concrete with recycled aggregates. *Mater*
393 *Struct* 1994;27:557–9.
- 394 [4] RILEM. STAR 217-PRE Progress of Recycling in the Built Environment (final draft).
395 Springer, ISBN 978-94-007-4907-8. Available at
396 <http://www.springer.com/fr/book/9789400749078>. 2013.

- 397 [5] De Larrard F. Concrete Mixture Proportioning: A Scientific Approach. 1999.
- 398 [6] Zhao Z, Remond S, Damidot D, Weiya X. Influence of fine recycled concrete aggregates
399 on the properties of mortars. *Constr Build Mater* 2015;81:179–86.
- 400 [7] Khatib JM. Properties of concrete incorporating fine recycled aggregate. *Cem Concr*
401 *Res* 2005;35:763–9. doi:10.1016/j.cemconres.2004.06.017.
- 402 [8] Evangelista L, de Brito J. Durability performance of concrete made with fine recycled
403 concrete aggregates. *Cem Concr Compos* 2010;32:9–14.
404 doi:10.1016/j.cemconcomp.2009.09.005.
- 405 [9] Kandhal PS, Lee DY. An Evaluation of the Bulk Specific Gravity for Granular Materials.
406 *Highw. Res. Board, Highw. Res. Rec.*, 1970, p. 44–55.
- 407 [10] Krugler PE, Tahmoressi M, Rand DA. Improving the Precision of Test Methods Used in
408 VMA Determination. *Asphal Paving Technol* 1992;62:272–303.
- 409 [11] ASTM. C 128-07a: Standard Test Method for density, relative density (specific gravity)
410 and absorption of fine aggregate. 2007.
- 411 [12] AASHTO. T84: Standard test method of test for specific gravity and absorption of fine
412 aggregate. 2010.
- 413 [13] Lin P-J, Chuang Y-J. Determination of SSD condition of fine aggregates using AC
414 impedance spectroscopy. *Mater Struct* 2013;46:911–20. doi:10.1617/s11527-012-
415 9943-x.
- 416 [14] Saxer EL. A direct Method of Determining Absorption and Specific Gravity of
417 Aggregates. *Rock Prod* 1956;87:77–9.
- 418 [15] You Z, Mills-Beale J, Williams RC and Dai Q. Measuring the Specific Gravities of Fine
419 Aggregates: An Automated Procedure. *Int J Pavement Res Technol* 2009;2:37–50.
- 420 [16] DRE Bourgogne. Catalogue des matériaux de substitution en Bourgogne (in French).
421 France: 2004.
- 422 [17] Gomes M, de Brito J, Bravo M. Mechanical Performance of Structural Concrete with
423 the Incorporation of Coarse Recycled Concrete and Ceramic Aggregates. *J Mater Civ*
424 *Eng* 2014;26:04014076. doi:10.1061/(ASCE)MT.1943-5533.0000973.
- 425 [18] Rogers C, Dziedziejko T. Fine aggregate density testing, what is the right way to do?
426 15th Annu. Symp. Res. - Int. Cent. Aggregates Res., 2007, p. A2–1 (24 p).
- 427 [19] Kandhal PS, Mallick RB, Huner M. Measuring Bulk Specific Gravity of Fine Aggregates:
428 Development of a New Test Method. In: TRB, editor. *Transp. Res. Rec. J. Transp. Res.*
429 *Board, National Research Council*; 2000, p. 81–90.

- 430 [20] Dana JS, Peters RJ. Experimental Moisture Determination for Defining Saturated
431 Surface Dry State of Highway Aggregates (internal report). 1974.
- 432 [21] Mechling M, Lecomte A, Merriaux K. Mesure de l'absorption d'eau des additions
433 minérales des bétons par vaporométrie (In French). *Mater Struct* 2003;36:32–9.
- 434 [22] Charreau A, Cavaillé R. Séchage. Théorie et calculs (In French) 1991;J 2 480–1 – 23.
- 435 [23] Tam VWY, Gao XF, Tam CM, Chan CH. New approach in measuring water absorption
436 of recycled aggregates. *Constr Build Mater* 2008;22:364–9.
437 doi:10.1016/j.conbuildmat.2006.08.009.
- 438 [24] Mindess S, Young F, Darwin D. *Concrete*. Prentice Hall; 2003.
- 439 [25] Poon CS, Chan D. Feasible use of recycled concrete aggregates and crushed clay brick
440 as unbound road sub-base. *Constr Build Mater* 2006;20:578–85.
441 doi:10.1016/j.conbuildmat.2005.01.045.
- 442 [26] CEN. Tests for geometrical properties of aggregates — Part 9: Assessment of fines -
443 Methylene blue test. 2013.
- 444 [27] Khoury GA, Majorana ĀCE, Pesavento F, Schrefler BA. Modelling of heated concrete.
445 *Mag Concr Res* 2002;54:77–101.
- 446 [28] Zhang Q, Ye G. Dehydration kinetics of Portland cement paste at high temperature. *J*
447 *Therm Anal Calorim* 2012;110:153–8. doi:10.1007/s10973-012-2303-9.
- 448 [29] Gong F, Zhang D, Sicat E, Ueda T. Empirical Estimation of Pore Size Distribution in
449 Cement , Mortar , and Concrete. *J Mater Civ Eng* 2014:1–11.
450 doi:10.1061/(ASCE)MT.1943-5533.0000945.
- 451 [30] Sing KSW, Everett DH, Haul RAW, Moscou L, Pierotti RA, Rouquérol J, Siemieniowska
452 T. INTERNATIONAL UNION OF PURE COMMISSION ON COLLOID AND SURFACE
453 CHEMISTRY INCLUDING CATALYSIS * REPORTING PHYSISORPTION DATA FOR GAS /
454 SOLID SYSTEMS with Special Reference to the Determination of Surface Area and
455 Porosity. *Pure Appl Chem* 1985;57:603–19.
- 456 [31] Baroghel-Bouny V. Water vapour sorption experiments on hardened cementitious
457 materials. *Cem Concr Res* 2007;37:414–37. doi:10.1016/j.cemconres.2006.11.019.
- 458 [32] Brunauer S, Emmett P, Teller E. Gases i n Multimolecular Layers. *J Am Chem Soc*
459 1938;60:309–19. doi:citeulike-article-id:4074706.
- 460