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Materials and Structures

Use of a reference limestone fine aggregate to study the fresh and hard behavior of mortar made with recycled fine aggregate --Manuscript Draft--

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Abstract:	<p>Recycled fine concrete aggregates (RFA) are not enough used in the construction sector, mainly because of their high water absorption capacity. These fine particles are composed of crushed natural aggregate and adherent hardened cement paste. The main goal of this research is to compare the behaviors of mortars made either with RFA or with a model limestone fine natural aggregate (LFA). The LFA is prepared in order to obtain physical properties as close as possible to those of the RFA. A specific characterisation is carried out to compare the density, water absorption, morphology of grains, size distribution and packing density of both aggregates. Mortars are then manufactured with same composition and same volume of LFA and RFA. Different states of moisture of the RFA are studied. The fresh behaviour of the mortar made with saturated RFA is very close to that of the mortar made with LFA which confirms that the latter is a good reference compared to the RFA. Comparison of fresh behaviours of mortars made with RFA of different state of moisture to that of mortar made with saturated sand allows then to determine the water absorbed in the different moisture conditions. Afterwards, a mechanical study is realised, taking into consideration the exact quantity of absorbed water of the RFA in dry or saturated conditions. Knowing the exact effective water value allows us to study both the strength of mortar made with RFA, the strength of the matrix and the adherence between the fine aggregate and the paste.</p>	
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Use of a reference limestone fine aggregate to study the fresh and hard behavior of mortar made with recycled fine aggregate

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Abstract

Recycled fine concrete aggregates (RFA) are not enough used in the construction sector, mainly because of their high water absorption capacity. These fine particles are composed of crushed natural aggregate and adherent hardened cement paste. The main goal of this research is to compare the behaviors of mortars made either with RFA or with a model limestone fine natural aggregate (LFA). The LFA is prepared in order to obtain physical properties as close as possible to those of the RFA. A specific characterisation is carried out to compare the density, water absorption, morphology of grains, size distribution and packing density of both aggregates. Mortars are then manufactured with same composition and same volume of LFA and RFA. Different states of moisture of the RFA are studied. The fresh behaviour of the mortar made with saturated RFA is very close to that of the mortar made with LFA which confirms that the latter is a good reference compared to the RFA. Comparison of fresh behaviours of mortars made with RFA of different state of moisture to that of mortar made with saturated sand allows then to determine the water absorbed in the different moisture conditions. Afterwards, a mechanical study is realised, taking into consideration the exact quantity of absorbed water of the RFA in dry or saturated conditions. Knowing the exact effective water value allows us to study both the strength of mortar made with RFA, the strength of the matrix and the adherence between the fine aggregate and the paste.

Keywords: Recycled fine aggregate- model natural fine aggregate- water absorption- saturation- effective water

1. Introduction

Large quantities of construction and demolition wastes (CDW) are produced each year. For example, the annual production of CDW is around 260 million tons in France [1] and 15 million tons in Belgium [2]. Amongst these wastes, Recycled Concrete Aggregates (RCA) can be considered as inert, and could be used as an alternative source of aggregates for the production

32 of new concrete. So far, only a small fraction of these RCA is used as aggregate in concrete
33 production.

34 RCA are composed of a mix of natural aggregates and hardened cement paste. Comparing to
35 natural aggregates, RCA possess a high water absorption coefficient, (between 4 and 12%) and
36 a lower density (between 2.1 and 2.5g/cm³) [3–5] The cement paste increases the porosity of
37 the material. Cement paste content of RCA is larger in recycled fine aggregate (RFA) than in
38 coarse recycled aggregate[6,7]: this is why RFA represents the most difficult part to valorize.
39 Several research works have been carried out in order to use RFA as aggregate to manufacture
40 mortars or concrete[8,9], as a mineral addition in cementitious materials [10] or as raw material
41 in cement manufacturing [11].

42 RCA are often incorporated into concrete or mortar as substitution of natural aggregates. The
43 influence of RCA substitution rate on the properties of concrete is controversial. Braga et al.
44 [12] studied the incorporation of fine recycled concrete aggregates in mortars with replacement
45 ratios of 5, 10 and 15%. An improvement of rheological and mechanical behaviors was
46 observed when the RCA was incorporated. Neno et al.[13] proposed a substitution of natural
47 fine aggregate (NFA) by RFA in different percentage ratio, and found that to have the same
48 fresh and hardened behavior of mortar, the replacement ratio in mortar composition is limited
49 to 20% by volume of replacement. Vegas et al. [14] studied the performances of masonry
50 mortars made with fine recycled concrete aggregates; a substitution rate of 25, 50, 75 and 100%
51 of the NFA by RFA was investigated. In this study, the quantity of water was adjusted in order
52 to work with the same consistency. The mechanical results showed that the incorporation of up
53 to 25% of recycled aggregate didn't affect the properties of hardened masonry mortars. Pedro
54 et al. [15] studied the simultaneous incorporation of fine and coarse recycled aggregates in
55 concrete. The results show that it is possible to achieve a comparable structural element with
56 recycled material, to those performed with natural one. Carro-Lopez et al. [16] showed that
57 substitution of up to 20% of natural aggregate (NA) by RCA decreased the fresh properties of
58 mortar and concrete. Omary et al. [17] and Fan et al. [18] found that using recycled aggregate
59 decreased the mechanical properties of mortar and concrete. But Hu et al. showed the opposite
60 [19]. Vinay Kumar et al. [20] performed an experimental study on the use of coarse and fine
61 aggregates to design a self-compacting concrete (SCC) : 20% of natural coarse and fine
62 aggregates were replaced by recycled aggregates. No significant difference was observed on
63 the rheological behavior, but an improvement in the mechanical behavior was observed. In
64 similar studies, Omrane et al. [21] and Kou et al. [22] found that the recycled aggregate
65 substitution should be limited to 50% in order to fulfil all recommendations of SCC. Carro-

66 L pez et al.[23] studied the rheology of SCC with RFA. NFA was replaced by 20%, 50%, and
67 100% by RFA. The natural and recycled fine aggregate were sieved and recomposed in order
68 to have a similar particle size distribution. The mixing water was adjusted by adding extra water
69 corresponding to water absorbed after 10 minutes. The results showed a decrease in fresh and
70 hardened properties of concrete for 50% and 100% of replacement of natural fine aggregate by
71 recycled one.

72 The literature review shows contradictory results concerning detrimental (or beneficial) effect
73 of NA substitution by RCA. This variability might first be due to the nature of the used
74 aggregates (both natural and recycled) coming from different sources. But the comparison itself
75 between mortars or concretes manufactured with NA and with RCA might be questioned. In
76 order to find out whether NA substitution by RCA decreases (or increases) concrete properties,
77 the composition parameters of the two concretes have to be as close as possible. In particular,
78 efficient water to cement ratio, volume of aggregates, and physical characteristics of the
79 granular skeleton (particle size distribution, particle's morphology ...) should be similar.
80 Several research works have already compared the behavior of mortars containing NFA to
81 mortars produced with RFA[23]. But to our knowledge, comparison of two mortars possessing
82 very similar mixture proportions and material characteristics has not been carried out yet.
83 In this study, the fresh and hardened properties of mortars manufactured with RFA are
84 compared to those of mortars of identical compositions with NFA presenting very similar
85 physical characteristics to those of RFA. The main objective is to study, when the composition
86 parameters of mortars are almost identical, what is the effect of a total substitution of NFA by
87 RFA.

88 **2. Methodology**

89 Ideally, the simplest way to study the effect of substitution of NFA by RFA in mortars would
90 be to replace a given volume of NFA by the same volume of RFA, keeping the workability of
91 the mortars constant. Then, mechanical properties of the two mortars could be compared and
92 the effect of this substitution (negative or even positive) could be derived. However, replacing
93 NFA by RFA generally leads to changes both in the fresh and hardened properties of mortars.
94 In this study, we need therefore to first define a model NFA, starting from a given RFA, that
95 would lead to the same properties in fresh state for two mortars and where the only difference
96 would be the nature of the fine particles (either RFA or NFA).

97 For this purpose, the rheological behavior has to be defined and can be described approximately
98 by the Krieger & Dougherty model [24] (Eq. 1).

$$\eta_c(\phi) = \eta_c(0) \left(1 - \frac{\phi}{\phi_m}\right)^{-[\eta]\phi_m} \dots \dots \dots (1)$$

99
100 where : $\eta_c(\phi)$ is the viscosity of the suspension, $\eta_c(0)$ is the viscosity of the interstitial fluid, ϕ
101 is the solid volume fraction of particles in suspension, ϕ_m is the maximum packing fraction and
102 $[\eta]$ is the intrinsic viscosity of particles (depending on their shape).

103 Several studies [25–27] have proven that the previous parameters are of first order for the
104 control of mortars and concrete rheological behavior.

105 If one wants the two previous mortars (with NFA or RFA) to possess the same rheological
106 behaviour, the four parameters of the Krieger & Dougherty model should be the same. That is
107 to say:

- 108 - The viscosity of the suspending fluid (the cement paste) should be the same in both
109 mortars. This implies that the two mortars contain the same effective water to cement
110 ratio (W_{eff}/C). As RFA generally possess a high water absorption coefficient, the only
111 way to precisely control the effective water is to use pre-saturated RFA, so that water
112 movements between cement paste and RFA can be prevented;
- 113 - The solid volume fraction of aggregates in the two mortars should be identical. This
114 necessitates using the same envelop volume of particles (i.e. the volume of solid
115 particle plus internal particle porosity should be constant);
- 116 - The maximum packing fraction should be constant, which at least implies that particle
117 size distributions and particle's morphologies would be very similar;
- 118 - The intrinsic viscosity of particles should also be the same. Intrinsic viscosity for real
119 fine aggregate particles is difficult to define; however, this constraint would be
120 fulfilled if particles of the two fine aggregates had approximately the same particles
121 geometry and same particle surface.

122 Based on the previous elements, the RFA and NFA used in this study have been defined as
123 follows:

- 124 - The RFA has been produced from the crushing of a laboratory concrete of known
125 composition.
- 126 - The NFA has been produced by crushing the natural coarse aggregate used for the
127 manufacture of the parent concrete, with the same procedure and same crusher as for
128 the RFA;

- The NFA has then been cut in seven different size fractions (2/4mm, 1/2mm, 0.5/1mm, 0.25/0.5mm, 0.125/0.25mm, 0.063/0.125mm, and <0.063mm) and recomposed to have the same particle size distribution (PSD) as the RFA.

Fig. 1 shows the used procedure. The physical properties of RFA and NFA will be determined and compared in order to verify if the fabrication procedure allows to produce two similar aggregates (section 3). Then, mortars of identical composition will be produced with the two sands and their rheological behavior will be compared in order to verify if the NFA can be considered as a good model for the RFA (section 4). Finally, the compressive strengths of mortars will be determined in order to assess the real effect of the substitution of NFA by RFA on the mechanical behavior of mortars (section 5).

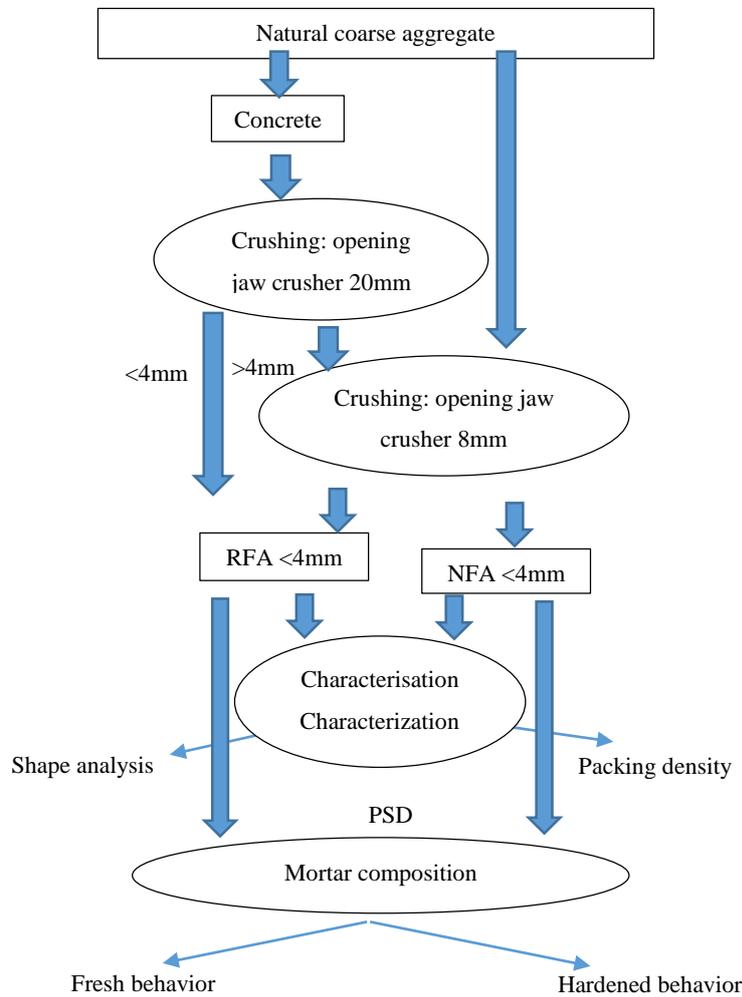


Fig. 1 Followed Methodology – preparation of aggregates – RFA = Recycled Fine Aggregates - NFA = Natural Fine Aggregates

3. Characterisation of fine aggregate

3.1 Materials

A CEM I 52.5N cement from CBR Belgium, complying with standard EN 197-1 is used. The density measured with Helium pycnometer is 3.11g/cm^3 and the specific surface area measured according to the standard EN 196-3 is $3800\text{ cm}^2/\text{g}$. Limestone aggregates 2/7mm, 7/14mm, and 14/20mm are provided by Carmeuse in Belgium. The natural sand 0/4mm is provided by LOVEMAT in Belgium. These materials are used for manufacturing a laboratory concrete which is then crushed for the production of RFA. Table 1 shows the concrete composition. The latter has been designed in order to obtain a consistency class S3 and strength class C30/37. The concrete slump measured according to NF EN 12350-2 is 129mm, and its 28 days compressive strength measured on cubic specimen (15x15x15 cm) is 41MPa.

Table 1 Concrete composition used to prepare the RFA

CEM I (kg)	Water (kg)	Aggregate 2/7 (kg)	Aggregate 7/14 (kg)	Aggregate 14/20 (kg)	Fine Aggregate (0/4) (kg)	Superplasticizer (%)
350	175	216	658	436	612	0.4

The concrete is crushed with a jaw crusher after 90 days curing to insure a high level of hydration; only the fraction smaller than 4mm is recovered. The crushing procedure is performed in two steps: the first step uses an opening jaw crusher of 20mm, and the fraction less than 4mm is recovered. The fraction above 4 mm is crushed a second time with an opening jaw crusher of 8mm. After crushing, the 0/4 mm fraction is carefully homogenized.

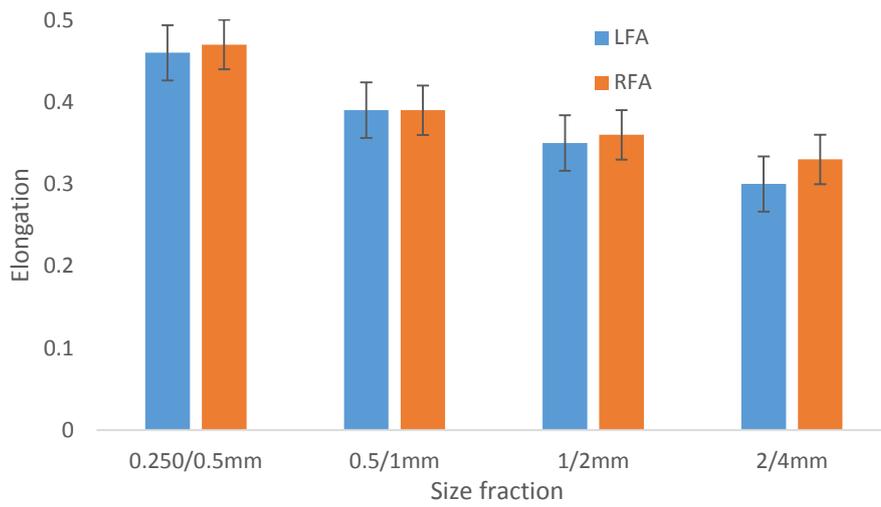
A limestone aggregate 20/32mm from the same quarry than the aggregate use for manufacturing the concrete (Carmeuse in Belgium) is crushed with an opening jaw crusher of 8mm. After that, the limestone fine aggregate (LFA) is recomposed in order to have the same size distribution than the RFA.

3.2 Shape analyses

The shape analyses are carried out with 2d images [28,29] and the measurement is performed with the fraction larger than $250\text{ }\mu\text{m}$ for a mass of sample between 250 and 300 g. This test is performed in order to identify the shape of particles, and to know if the preparation procedure presented in Fig. 1 allows to have a similar shape between RFA and LFA.

189 The sample is gradually laid on a cover belt. Thanks to a camera and a source of light, each
 190 particle is individually evaluated and parameters are calculated. In this work, between 100,000
 191 and 200,000 particles are analyzed. Afterwards, length and width of each particle are recorded.
 192 The morphology is expressed in terms of elongation. The elongation parameter is computed
 193 with (Eq. 2). Fig. 2 shows the results obtained for RFA and LFA: no significant differences are
 194 observed for the different size fractions.

$$\text{Elongation} = 1 - (\text{width}/\text{length}) \dots \dots \dots (2)$$



196
 197 **Fig. 2** Elongations for the different particle sizes of RFA and LFA

198 3.3 Water absorption and density

199 Three methods are investigated for the measurement of water absorption coefficient of RFA:
 200 EN 1097-6 [30], IFSTTAR method N°78 [31] and extrapolation method [6].

201 Using the standard EN 1097-6 or IFSTTAR method, water absorption is determined by
 202 measuring the quantity of water present in the aggregate at saturated surface dry state (SSD).
 203 Firstly, the aggregates are saturated with a certain amount of water for 24 hours. In EN 1097-
 204 6, in order to achieve SSD, the sample is exposed to a warm air flow for evaporating the water
 205 present at the surface of particles. The SSD state is identified using a slump test. A cone is filled
 206 in one time and compacted with 25 pestles shots. The SSD state is determined according to the
 207 shape obtained after lifting the cone. In IFSTTAR method, for achieving SSD state, the sample
 208 is dried progressively by using different colored absorbing papers until there is no more trace
 209 of water.

210 In both methods, the mass of sample at SSD state is recorded (M_{SSD}). Then the sample is dried
211 in an oven at 105°C until constant mass (M_{dried}). The water absorption (WA) is computed with
212 (Eq. 3).

$$WA = (M_{SSD} - M_{dried}) / M_{dried} \dots \dots \dots (3)$$

214 Le et al.[32] have shown that the EN 1067-6 method underestimates the water absorption of the
215 finer granular fractions, whereas the IFSTTAR method overestimates it. The extrapolation
216 method developed by Zhao et al. [6] is therefore also used in this research. This method is based
217 on the relationship between the water absorption and the cement paste content. For determining
218 the water absorption, the sample is divided in different size classes and the adherent cement
219 paste content is measured for each size class. (Eq. 4) shows that there is a linear relationship
220 between the adherent cement paste content and the water absorption. Zhao showed that the
221 measurement of water absorption coefficient is possible only for coarser classes with standard
222 EN 1097-6 or IFSTTAR method (down to 0.5mm) [32]; the water absorption of the finer
223 fraction is then computed by extrapolation with (Eq.4). Knowing the proportion of each size
224 fraction of RFA, the water absorption of the whole 0/4 fraction can be determined.

$$WA_{RFA} = WA_p X_p + WA_{NA}(1 - X_p) \dots \dots \dots (4)$$

226 WA_{RFA} : water absorption coefficient of a given size class of RFA

227 WA_p : water absorption coefficient of adherent cement paste

228 WA_{NA} : water absorption coefficient of natural aggregate in RFA

229 X_p : adherent cement paste content of a given class of RFA

230 In this study, the RFA is sorted into 6 size fractions: (<0.063mm, 0.063/0.125mm,
231 0.125/0.5mm, 0.5/1mm, 1/2mm, and 2/4mm). The water absorption coefficients of the size
232 fractions larger than 0.5mm are determined with IFSTTAR method: by extrapolation, the total
233 water absorption for RFA is then determined. Le et al. [32] showed that using EN 1097-6 or
234 IFSTTAR method lead to very close WA results for the fraction between 0.5/4mm.

235 In the extrapolation method, the adherent cement paste content can be estimated either by
236 soluble fraction in salicylic acid (SFSA) or mass loss (ML) between 105°C and 475°C both
237 methods are investigated here.

238 The SFSA is determined by immersing 0.5g of dry representative sample into a solution of
239 salicylic acid and methanol (14g of salicylic acid and 80 ml of methanol) during 1 hour to
240 dissolve the soluble phases of the cement paste. After that, the solution is filtered to obtain the
241 solid residue. This method is carried out on 2 samples of each granular fraction.

The mass loss between 105°C and 475°C is determined by (1) storing 10g of representative samples for 24 hours in the oven at 105°C and weighing the sample ($M_{105^{\circ}\text{C}}$) (2) putting the sample in the oven at 475°C for 24 hours and weighing the sample ($M_{475^{\circ}\text{C}}$). The mass loss is computed with the (Eq. 5).

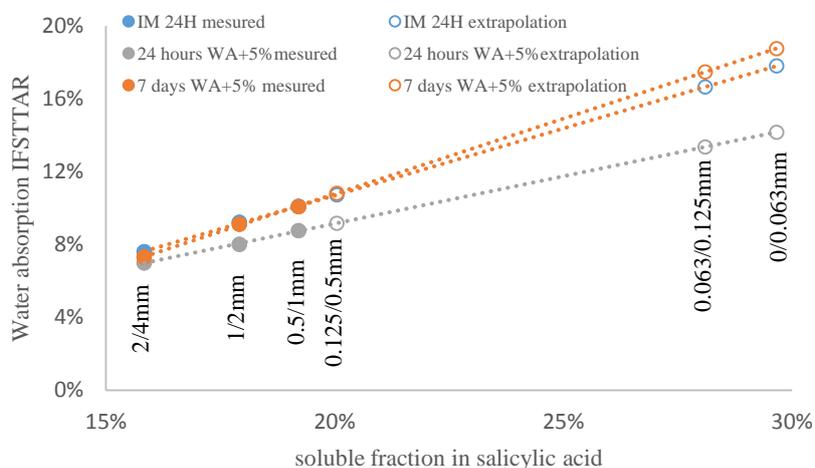
$$ML_{105^{\circ}\text{C}-475^{\circ}\text{C}} = (M_{105^{\circ}\text{C}} - M_{475^{\circ}\text{C}}) / M_{105^{\circ}\text{C}} \dots \dots \dots (5)$$

In this research, the water absorption is determined under the three following different absorption conditions.

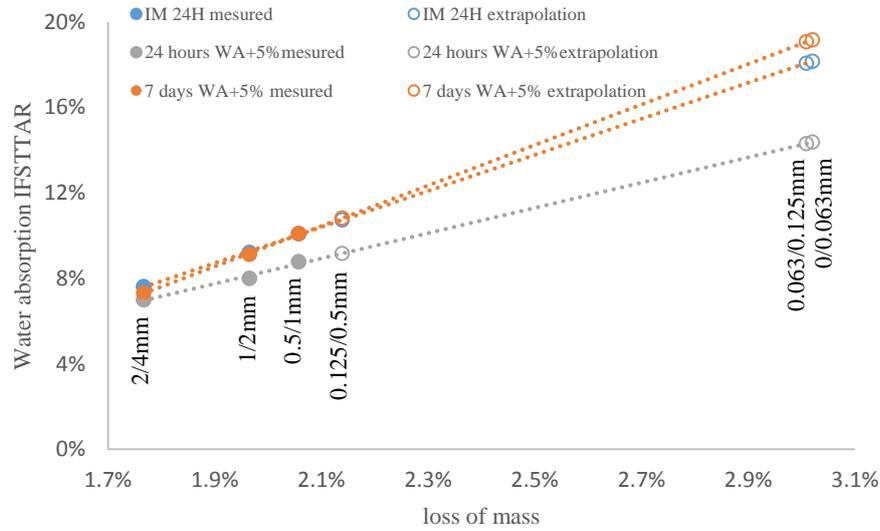
- IM 24 hours: total immersion into water for 24 hours (porosity and real density are computed with this saturation procedure corresponding to EN 1097-6);
- 7 days WA+5%: conservation in sealed bottle for 7 days with a quantity of water equal to WA+5% of the mass of the sample
- 24 hours WA+5%: conservation in sealed bottle for 24 hours with a quantity of water equal to WA+5% of the mass of the sample;

Le et al. [32] showed that conservation of RFA for 7 days in a sealed bottle with a quantity of water equal to WA+5% allowed to saturate the RFA.

Fig. 3 and Fig. 4 show the variation of water absorption for each size fraction and for the different moisture conditions, with the extrapolation method, using respectively the soluble fraction in salicylic acid (SFSA) and mass loss (ML). The results show a linear relation between the water absorption and the mass loss (ML) or the (SFSA). For the different fractions, there is non-significant difference between the samples immersed in water and the one saturated with WA+5% of the mass of the sample for 7 days. It can also be seen that the sample saturated with WA+5% for 24 hours has a lower water absorption comparing to the two others. It also means that, if the capillary rise is not complete after 24 hours, the saturation is completed after 7 days; this result is similar to that presented by Le et al.[32].



267 **Fig. 3** Extrapolation method with the soluble fraction in salicylic acid (SFSA)



269 **Fig. 4** Extrapolation method with the mass loss (ML)

270 Table 2 shows the details of the different water absorption measurements. For the different
 271 moisture conditions, the water absorption coefficient is very close when using ML and SFSA
 272 for the extrapolation method.
 273

274 **Table 2** Water absorption for the different moisture condition of RFA

	IM 24 HOURS	24 HOURS WA+5%	7 DAYS WA+5%
WA EN 1097-6 (%)	7.5	7.4	9.0
WA IFSTTAR (%)	10.8	10.4	12.6
WA IFSTTAR extrapolation SFSA (%)	9.7	8.4	9.6
WA IFSTTAR extrapolation LM (%)	9.8	8.5	9.7

275
 276 Bordy et al.[33] showed that the residual anhydrous crushed paste, when in contact with water,
 277 can harden and agglomerate. To verify if agglomeration of finer particles happens after 7 days
 278 of conservation, the particle size distribution is measured after drying the material at 60°C until
 279 constant mass. The PSD has changed a lot which can be due to the agglomeration of RFA pre-
 280 saturated for 7 days. This can certainly be attributed to anhydrous phases of RFA reacting with
 281 water and changing the granular skeleton. Such a change in particle size distribution can affect
 282 the workability of the mortar manufactured with this mode of saturation.

283 The WA of LFA is measured according to standard EN 1097-6. The value of absolute density
 284 (ρ_{ab}) is determined according to the standard EN 1097-6; the porosity (P) and real density (ρ)
 285 are obtained with (Eq. 6) and (Eq. 7).

$$P = \frac{W_{abs}}{W_{abs} + \frac{1}{\rho_{ab}}} \dots\dots\dots (6)$$

$$\rho = \rho_{ab} \times (1 - P) \dots\dots\dots (7)$$

288 The Apparent density and the water absorption measured for the samples immersed for 24h into
 289 water for LFA are 2.69g/cm³ and 1% respectively. For the RFA, the extrapolation method is
 290 used to compute the porosity and real density, the apparent density and real density for RFA
 291 are respectively 2.4 and 1.94g/cm³.

3.4 Packing density

293 The packing density has an impact on the rheological behavior of mortar or concrete [24]. In
 294 this research, same PSD and very close particle shape are obtained for RFA and LFA. These
 295 two fine aggregates should therefore present similar packing densities.

296 The LCPC protocol [25] is carried out for the measurement of packing density (ϕ_m). This test
 297 is realized by placing a dry sample of 7kg in a cylinder of 160mm of diameter and a height of
 298 600mm fixed to a vibrating table. A flat piston of 20kg is placed on top of the cylinder to exert
 299 a pressure of 10 KPa. The cylinder is vibrated for 1 minute, and the apparent volume after
 300 vibration is measured. ϕ_m is computed with (Eq. 8) where ρ_{app} is the apparent density measured
 301 after the test and ρ is the real density presented in §3.3.

302 Packing density of RFA and LFA are 86% and 84%, respectively. In order to verify that the
 303 measurement did not generate any fine particle, the PSD is also measured after the test.

$$\phi_m = (\rho_{app}) / (\rho) \dots\dots\dots(8)$$

305 The PSD analyses performed before and after the packing density test show a significant
 306 increase in the quantity of fine particles. This is the reason why the value obtained with this
 307 method is not taken into consideration.

308 The generation of fine particles is certainly due to the use of a weight of 20 kg and a vibration
 309 of 1 minute. The same protocol is therefore reproduced without the weight of 20kg. The packing
 310 density of RFA and LFA is in this case 80% and 75%, respectively. PSD analyses are carried
 311 out after the measurement and show no generation of fine particles in this case.

Given the procedure used for the production of RFA and LFA, the physical envelop characteristics of the two sands are very similar (same PSD, similar particle shape, close packing density).

4. Rheological behavior of mortars

4.1 Mortar compositions

Table 3 presents the compositions of mortars with RFA and LFA. The cement used for the manufacture of mortars is the same as the one used for the manufacture of concrete. A limestone filler from Carmeuse in Belgium is also used. Its density measured with the helium pycnometer is 2.72g/cm³ and its specific surface area according to the standard EN 196-3 is 3170 cm²/g. A volumetric substitution of LFA by RFA is performed, and the same amount of efficient water is used. The water quantity is computed by taking into consideration the water absorption after total immersion in water of RFA or LFA.

Table 3 Mortar compositions for the investigation of fresh behavior

	Cement (g)	Limestone filler (g)	Fine Aggregate (g)	Effective water (g)	WA (g)	Weff/C	Weff/P
Reference	1344	895	3968	1209	39	0.9	0.54
Recycled	1344	895	2942.9	1209	288	0.9	0.54

In order to justify whether the difference in packing densities showed in part 3 is significant for the fresh behavior of mortars, the Krieger-Dougherty model is used (Eq. 1). Mehdipour and Khayat [34] consider that the ϕ_m parameter for the application of Krieger-Dougherty model is the packing densities obtained when using LCPC measurement. ϕ_m value for RFA and LFA are given in part 3.4.

Table 4 presents the relative viscosities obtained with the Krieger-Dougherty model based on the characterisation results and mortar compositions. As observed in Table 3, the solid volume fraction of aggregates in the mortar (ϕ) is identical for RFA and LFA, but the packing densities (ϕ_m) present a difference of 5%. A small difference is obtained for the relative viscosities between mortars of LFA and RFA.

Due to the small differences between aggregate properties, and to the small difference obtained for the predicted relative viscosities, the rheology of mortars made with saturated RFA and LFA should be very close.

339 **Table 4** Comparison between the relative viscosities of RFA and LFA

	ϕ	Φ_m	$\eta_c(\phi)/\eta_c(0)$
LFA	43%	75%	5.032
RFA	43%	80%	4.798

340

341 **4.2 Pre-saturation conditions and mixing procedure**

342 Different saturation states of RFA are studied. RFA is first dried at 60°C until a constant mass.

343 The mortars are then produced with 5 different pre-saturation conditions of RFA in order to
344 study the water movement between the paste and RFA:

- 345 - Dry RFA: first mixed with the powders and then the total water is added;
- 346 - Paste + dry RFA: preparation of the paste with water first then add the dry RFA;
- 347 - 5 min WA+5%: add to RFA a quantity of water equal to WA+5% for 5 minutes[35];
- 348 - 24 hours WA+5% : add to RFA a quantity of water equal to WA+5% for 24 hours;
- 349 - 24 hours IM: add to RFA all the quantity of water for preparing the mortar (in order to
350 assure that all the particles are immersed in water).

351 The saturation is achieved by storing the fine aggregates with the amount of water needed in
352 watertight containers. The containers are conserved in air-conditioned room at 20°C.

353 The LFA is used after a saturation of WA+5% for 5 minutes, according to the protocol presented
354 by Schwartzentruber and Catherine [35].

355 Complete immersion of RFA into water for 24 hours (24 hours IM) should allow for the
356 saturation of RFA and should prevent water movements in mortar. Other saturation modes
357 should lead to larger workabilities for mortars [32]. In order to quantify the effective water and
358 estimate the water absorption for a given moisture condition, quantities of water equal to 20,
359 40 and 60% of the water absorption of RFA, respectively, are added to the effective water in
360 mortars manufactured with saturated sand (24 hours IM). The same quantity of water was used
361 for mortars with LFA. Fig. 5 shows the procedure used to produce the different mortars.

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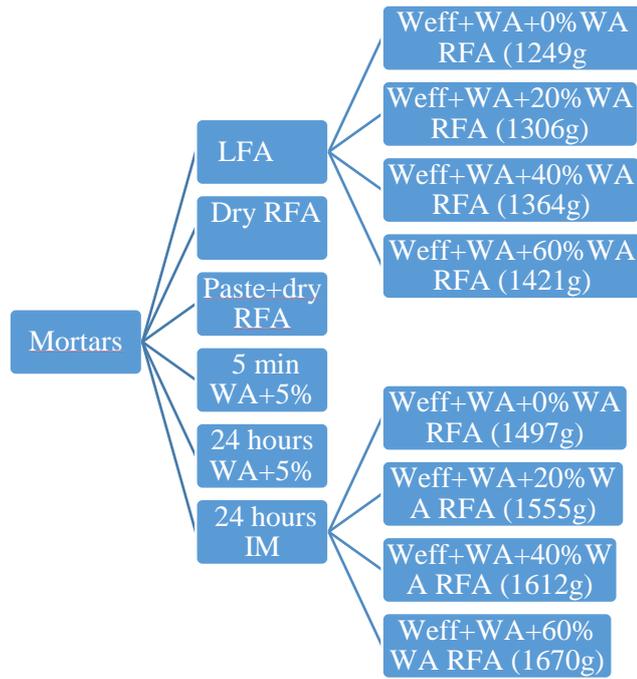


Fig. 5 Mortars manufactured to study the water movements between RFA and cement paste. The total water added in the mixes is put between brackets.

Cement, limestone powder, RFA, LFA and water are first stored at 20°C. Depending on the way of saturation of RFA, cement, limestone powder, fine aggregate, and the total water are mixed at a low speed for 90 seconds; 60 seconds of manual mixing are then following. Finally, 90 seconds of mixing at high speed are performed.

When dry RFA is used, the cement, limestone filler and water are added first and mixed for 60 seconds; the dry RFA is then added and another 60 seconds of mixing are performed. 60 seconds of manual mixing are then following. Finally, 90 seconds of mixing a high speed are performed.

4.3 Fresh state properties

For each mortar, the apparent density, slump (H) and slump flow (D) are measured. All the tests are performed three times to assure a good repeatability. The slump and slump flow tests are performed with a mini cone whose upper diameter is 70mm, lower diameter is 100mm, and height is 60mm.

In order to determine the yield stress, Roussel's model is used [36]. To apply this model, the slump radius should be larger than the height of the slump (D/2>H). The empirical yield stress may then be computed with (Eq. 9).

$$\text{Yield stress} = \frac{225\rho g\Omega^2}{128\pi^2 R^5} \dots\dots\dots (9)$$

- ρ : fresh apparent density of mortar (g/cm³)

- 382 - g : gravity 9.8m/s^2
- 1 383 - Ω : volume of the mini cone (mm^3)
- 2 384 - R : radius of the slump (mm)
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5 385 4.4 Rheological behavior

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8 386 Table 5 shows the apparent density, slump flow and yield stress calculated with Roussel's
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10 387 model. The mortar manufactured with immersed RFA has the closest results to the one
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12 388 manufactured with LFA. The mortar made with RFA saturated with WA+5% for 24 hours
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14 389 presents better workability than the one performed with immersed RFA. This result confirms
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16 390 that conservation of RFA with WA+5% for 24 hours does not allow reaching complete
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18 391 saturation. Referring to the water absorption (Table 2), a difference of 1.3% is observed
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20 392 between this saturation condition and total immersion in water of RFA: this may explain the
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22 393 improvement of workability observed for the mortar made with RFA with WA+5% saturated
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24 394 for 24 hours.

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26 395 Using RFA in dry condition or saturated with WA+5% for 5 minutes gives similar behaviors in
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28 396 the fresh state and leads to the highest workability. This result is due to the incomplete
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30 397 absorption of water by dry fine aggregate (or fine aggregate saturated only for 5 minutes) which
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32 398 leads to maximal amount of effective water for fluidizing the mixture. Adding the RFA in dry
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34 399 condition in the paste gives a lower workability than in the two previous conditions.

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46 400 **Table 5** Fresh properties of the mortars

	Density (g/cm^3)	Slump Flow (mm)	Yield stress Roussel's model (Pa)
LFA	2.22	163	124
Dry RFA	1.88	210	32.1
Paste +dry RFA	1.88	193	47.7
5 min WA+5%	1.87	205	34.1
24 hours + WA+5%	1.92	172	79.3
24 hours IM	1.95	158	130

47 401
48 402 Fig. 6 shows the variations of slump flow and yield stress of mortars made with LFA and RFA
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50 403 after total immersion in water as a function of the efficient water to powder ratio. It is observed
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52 404 that there is only a small difference (in yield stress and slump flow) between the mortars made
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54 405 with LFA and RFA immersed in water.

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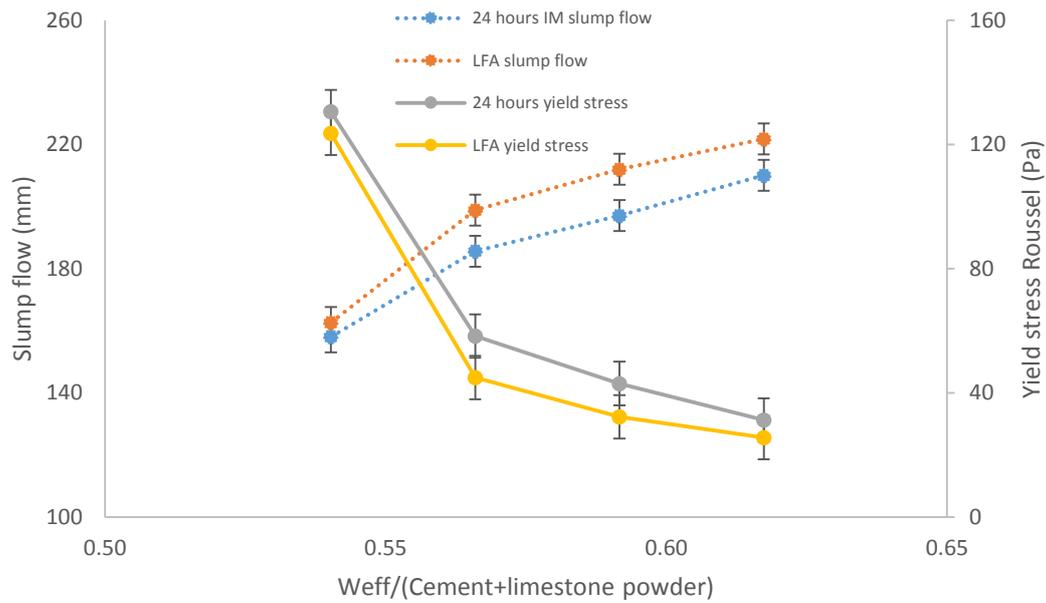


Fig. 6 Workability of the mortar manufactured with LFA and RFA immersed in water for 24 hours

Fig. 7 shows the yield stress measured with Roussel's model as a function of the water in excess. Adding 20, 40, and 60% of the RFA water absorption quantity allows computing the percentage of excess water for the different moisture conditions.

Previous studies show an exponential decrease of yield stress when the W/C increases in the case of pure cement paste [37,38]. In our work, using a parabolic equation gives better regression than using an exponential trend, which may be due to the fact that mortar is used and not a pure paste. Yield stresses obtained with Roussel's model presented in Table 5 are used in order to compute the water in excess and then the water absorption for each moisture condition[39].

The mortar made with RFA saturated with WA+5% for 24 hours presents a yield stress of 79Pa. Based on this result, the water absorption can be computed with the parabolic relation and corresponds to 8.33%. The water absorption presented in Table 2, with a saturation state of WA+5% for 24 hours, shows a similar value which means that the water absorption kinetics are identical for the two tests. This result validates the fact that the rheological study can be used for computing the water absorbed in the different states of moisture.

The use of RFA in dry conditions or after 5 minutes of saturation gives close yield stresses. Based on these values, the water absorption calculated is 5.3% and 5.7%, respectively. The saturation degree which is the ratio of the water absorbed and the standard water absorption (WA, Table 2) is around 54%.

Adding dry RFA to the paste leads to a smaller yield stress; the water absorbed calculated for this condition is 6.81%, which means that the saturation degree of RFA is 70%. In this

condition, RFA absorbs more water than when it is added with the powder and water. Maimouni et al. [39] showed that, after 5 minutes of mixing, the degree of saturation of dry RFA in cement paste with W/C ratio equal to 0.5 is around 70%: this result is similar to the result obtained for the mortar made with RFA in the same condition.

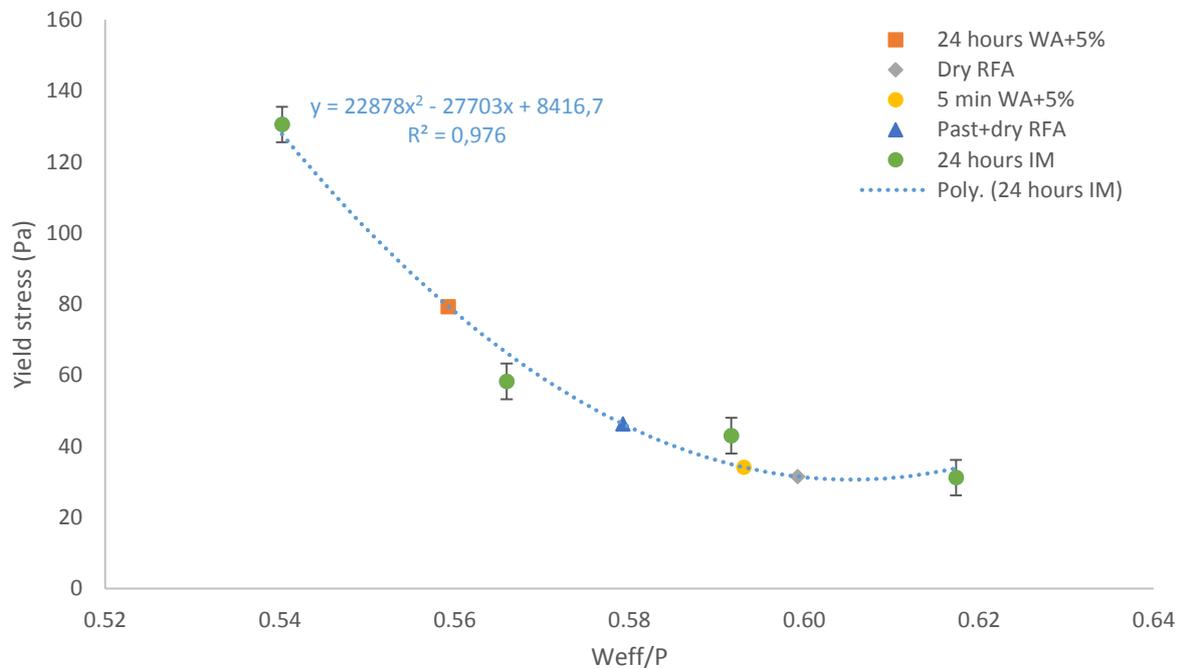


Fig. 7 Measure of yield stress as a function of Weff/P ratio for the different mortars with RFA in different moisture conditions

5. Mechanical behavior of mortars

Zhao et al. [40] showed that the state of moisture influences the interface between the new and old cement paste, the difference having an impact on the mechanical strength. These authors found that using RFA in dry condition leads to better mechanical properties. However, in a similar research Le et al. [41] found no significant difference between the compressive strengths of mortars made either with dry or saturated RFA. In order to better understand the influence of moisture state of RFA on the compressive strength of mortars, different specimen are manufactured with dry or saturated RFA and compared with mortar made with model LFA. Compressive strengths are measured on mortars with RFA and LFA. Prismatic specimens (40mm X 40mm X 160mm) are prepared according to standard EN 196-1. The flexural and compressive strengths are measured according to standard EN 196-1.

Table 6 shows the mortar compositions. For the different mixes, the effective water content - + 0%, 40% and 60% of the water absorption after a total immersion into water of RFA - is

450 considered. The fresh behavior study made in section 4 allows knowing the water absorbed for
 451 the different moisture conditions. In order to assure the same efficient water for the different
 452 mortars, the water absorbed by RFA in dry or in total immersion is taken into consideration in
 453 the water mixes. The water absorption considered in the mortar composition with RFA used in
 454 dry condition is equal to 5.3%, and equal to 9.8% for immersed RFA as shown in Table 6.

455 **Table 6** Mortar compositions for the evaluation of mechanical performances

	Cement (g)	Limestone filler (g)	Fine aggregate (g)	Effective water (g)	Absorbed water	W_{eff}/ (Cement+limestone filler)
LFA 0%	1344	895	3968	1 209	39.7	0.54
LFA 40%	1344	895	3968	1 325	39.7	0.59
LFA 60%	1344	895	3968	1 382	39.7	0.62
DRY RFA 0%	1344	895	2943	1 209	156	0.54
DRY RFA 40%	1344	895	2943	1 325	156	0.59
DRY RFA 60%	1344	895	2943	1 382	156	0.62
24 hours IM 0%	1344	895	2943	1 209	288	0.54
24 hours IM 40%	1344	895	2943	1 325	288	0.59
24 hours IM 60%	1344	895	2943	1 382	288	0.62

456
 457 According to De Larrard [42], the compressive strength of concrete or mortars is related to the
 458 compressive strength of cement matrix and is given by (Eq. 10).

$$R_c = \frac{p \times R_{cm}}{q \times R_{cm} + 1} \dots \dots \dots (10)$$

460 R_{cm} : compressive strength of the cement matrix

461 R_c : compressive strength of mortar

462 p: adhesion quality between the aggregate and cement matrix

463 p/q: characterize the ceiling effect which the limiting effect of the aggregate on the
 464 mechanical behavior of the mortar or concrete.

465 The compressive strength of the cement matrix is computed by (Eq. 11).

$$R_{cm} = 13.4 \times R_{c28} \times \left(\frac{V_p}{V_p + V_w + V_a} \right)^{2.85} \times MPT^{-0.13} \dots \dots \dots (11)$$

467 R_{c28} : represents the compressive strength of the standard mortar made with cement and
 468 limestone powder with the same proportion as showed in Table 3 at 28 days.

469 V_p, V_w, V_a : represent the volume of powder, water and air. The volume of air is
 470 measured with air content CONTROLS/1L.

471 MPT: the maximum paste thickness between two close aggregates computed from
472 (Eq. 12).

$$473 \text{MPT} = D_{\max} \times \left(\sqrt[3]{\frac{g^*}{g}} - 1 \right) \dots \dots \dots (12)$$

- 474 D_{\max} : maximal size of aggregate
- 475 g : the aggregate volume for a unit volume of mortar.
- 476 g^* : the granular packing density, computed with eq (Eq. 13).

$$477 g^* = 1 - 0.45 \times \left(\frac{d_{\min}}{d_{\max}} \right)^{0.19} \dots \dots \dots (13)$$

- 478 d_{\min} : diameter corresponding to 10% passing.
- 479 d_{\max} : diameter corresponding to 90% passing.

480 According to De Larrard [42], for high matrix strengths, the mortar strength is equal to $R_c = p/q$;
481 but for low matrix strengths, it is equal to $R_c = p \times R_{cm}$. In this work, a high W/P ratio is used to
482 allow for a total immersion of RFA in water during the saturation phase. This high W/P ratio
483 leads to low matrix strengths. This is consequently mainly the adherent parameter (p) and its
484 effect on the mechanical resistance which are of interest here.

485 Table 7 presents the compressive strength of the different mortars. Mortars with LFA showed
486 a higher compressive strength than those made with RFA, whatever their saturation state. Also,
487 the compressive strength of the mortar made with dry RFA is larger than that of the mortar
488 made with saturated RFA. The air content was higher for RFA mortars than LFA mortars.
489 Moreover, the air content is larger in the mortar made with dry RFA than in the mortar with
490 saturated RFA. The larger air contents of mortars made with RFA in comparison to those made
491 with LFA can certainly be attributed to differences in surface rugosity of particles, probably
492 larger for RFA than for LFA. The larger air content of mortars made with dry RFA in
493 comparison to saturated RFA is certainly due to the air present in the non-saturated particles.

494 In section 4, a difference of 4.5% of WA is observed between the saturated and dry RFA. This
495 difference in water absorption could increase the air content in mortars made with dry RFA by
496 3.5%. According to the air contents presented in Table 7, a difference of 2.5% is obtained
497 between the mortar made with dry RFA and saturated RFA. This result confirms that the
498 increase in air content for mortars made with dry RFA is due to the air still present in the pores
499 which are not filled with water. So, for the application of the De Larrard model [42], the same
500 air content for the matrix of the mortar made with saturated RFA is going to be used for the
501 mortar made with dry RFA.

502 **Table 7** Compressive strength of mortars, and details for the investigation of the “ceiling effect”

	LFA			RFA dry			IM 24H RFA		
W_{eff}/P	0.54	0.59	0.62	0.54	0.59	0.62	0.54	0.59	0.62
V_{air}/V_{total} in mortar (%)	1	1	1	7	7	7	4.5	4.5	4.5
V_{air} in paste /V_{total} (%)	1	1	1	4.5	4.5	4.5	4.5	4.5	4.5
g	0.43	0.42	0.41	0.41	0.39	0.39	0.42	0.40	0.40
g*	0.76	0.76	0.76	0.76	0.76	0.76	0.76	0.76	0.76
MPT	0.68	0.73	0.75	0.76	0.80	0.83	0.73	0.77	0.79
R_{c28}(MPa)	25.03	25.03	25.03	25.03	25.03	25.03	25.03	25.03	25.03
R_{cm}(MPa)	22.34	18.88	17.42	18.73	15.90	14.69	18.73	15.90	14.69
R_c(MPa)	26.99	24.42	20.29	17.37	15.55	14.89	14.35	13.03	10.34
p	1.222			0.948			0.766		

503
504 As explained above, the De Larrard model is presented only with the p parameter because of
505 the low cement matrix. The adhesion between LFA and paste is better than the one between
506 RFA and paste. Using RFA in dry condition allows for a better adhesion than when saturated.
507 This result is in accordance with the study of Zhao et al. [40]
508 Higher compressive strengths obtained for mortars with LFA than for mortars with RFA can
509 therefore be attributed to two parameters: a higher air content in the paste of mortars made with
510 RFA comparing to the one made with LFA and a better adhesion between the LFA and new
511 paste.

513 6. Conclusions

514 As this is very difficult to study the real effect of substitution, a natural by a recycled fine
515 aggregate, an original method has been proposed for preparing a natural model aggregates and
516 comparing it with recycled one. Specific physical characterisation carried out on both
517 aggregates shows that they present very similar granular characteristics (particle size
518 distribution, elongation, and packing density). Comparison of mortars made with LFA and
519 saturated RFA shows that these two mortars present very close fresh state properties, showing
520 that LFA is a good natural model aggregate for a rigorous comparison with RFA.

521 Using the two previous aggregates, mechanical properties of mortars made with RFA in various
1 522 saturation conditions are compared to those of mortars made with LFA of identical composition
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3 523 (same W_{eff}/C ratio and same paste volume). Results show that the compressive strengths of
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5 524 mortars made with LFA are systematically larger than those of corresponding mortars made
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7 525 with RFA. The lower compressive strengths can be attributed to a lower quality of the bond
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9 526 between aggregates and cement matrix and to a larger air content in mortars made with RFA.
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11 527 Results also show that mortars containing saturated RFA present lower air contents than those
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13 528 with dry RFA, because of the air contained in non-filled voids of aggregates in the latter.
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15 529 However, the compressive strengths of mortars made with dry RFA are systematically larger
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17 530 than those of saturated RFA, because of a better adhesion between aggregates and cement
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19 531 matrix.
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28 535 Wallonia.
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30 536 **Conflict of interest**

33 537 The authors declare that they have no conflict of interest.
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