Substitution of limestone filler by waste brick powder in self-compacting mortars: properties and durability

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# **Credit Author Statement**

Zengfeng ZHAO: Conceptualization, Methodology, Investigation, Writing - Original draft

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Frédéric Michel: Validation;

David Bulteel: Funding acquisition;

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Journal Pre

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# 1 Substitution of limestone filler by waste brick powder in self-compacting

# 2 mortars: properties and durability

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Abstract: The feasibility of using waste brick powder (WBP) in the manufacture of self-14 compacting mortar has been investigated in this study. The limestone filler was partially or 15 totally (0%, 50% and 100%) substituted with WBP. The rheological properties, compressive 16 and flexural strengths, drying shrinkage and durability properties (including carbonation 17 resistance, chloride ion diffusion and sulphate resistance) of self-compacting mortars were 18 evaluated. The WBP-mortars presented a higher yield stress and plastic viscosity than that of 19 WBP-free mortar: the additional water has to be added in order to achieve the equivalent 20 workability. The compressive strength of WBP-mortars slightly decreased after 7 days (the 21 compressive strength of series mortars M-BP decreased 5.6% and 9.3% for 50% and 100% 22 WBP based mortars comparing with the reference mortar, respectively; while the compressive 23 strength of series with similar workability mortar M-BP100WA which was based on 100% 24

WBP decreased 16.7% and it could achieve 26.8 MPa), but the decreasing trend seemed to be compensated by the pozzolanic activity of WBP and remained equivalent after 28 days (the compressive strength of series mortar M-BP100WA decreased 5.3% and it could achieve 35.6 MPa). The substitution of limestone filler by WBP didn't seem to impair the durability behavior of mortars (except for the resistance to carbonation). Therefore, it is possible to manufacture self-compacting mortar by partially or totally substituting limestone filler by WBP.

32 Keywords: waste brick powder, limestone filler, self-compacting mortar, rheology, strength,

33 durability.

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### 39 **1. Introduction**

Concrete is the second most consumed resource after water and is considered as one of the 40 most successful material in the world. The concrete industry's annual global production in 41 42 2010 is approximately 33 billion tons, which consumed nearly 3.7 billion tons of Portlandcement clinker and 27 billion tons of aggregate [1]. The cement industry (global production of 43 cement is 4.2 billion tons in 2019, e.g. ~0.5 tonne per capita) is a major source of greenhouse 44 gas emissions particularly carbon dioxide (CO<sub>2</sub>), which accounts for about 8% of total 45 world's CO<sub>2</sub> emissions [2]. Very large quantities of construction and demolition waste (CDW) 46 are generated yearly around the world. In the European Union (EU), the value was 868 47 million tons in 2014, accounting for 35% of the total waste generation of EU [3]. The main 48 constituents of CDW are the concrete and brick that may come from the construction of new 49

buildings, demolition of old structures, renovation-activities and from natural disasters [4–6]. 50 51 EU Waste Framework Directive (2008/98/EC) has provided a framework for achieving that by 2020 a minimum of 70% (by weight) of non-hazardous CDW shall be prepared for re-use, 52 recycled and other material recovery [7]. The demolition of brick masonry structures produces 53 huge amount of waste, including large quantities of clay brick waste [8-12]. In addition, the 54 ceramic bricks industry generates a significant amount of rejected non-conform bricks. 55 However, the fired clay brick waste (the calcination of raw clay minerals occurs the formation 56 of amorphous material during the production of fired clay brick) generally presents some 57 pozzolanic activity [13-16], which could react with calcium hydroxide and form compounds 58 59 with enhanced strength and durability. Therefore, the waste brick powder (WBP) might be used in cement based materials to decrease the amounts of waste which have to be disposed in 60 landfill and the CO<sub>2</sub> emissions [17–20]. Recently, the use of WBP as a partial substitution of 61 Portland cement in the concrete has been received much attention during the past decades 62 [6,16,21–26]. 63

Liu et al. [6] demonstrated that the hybrid recycled powder from clay brick and concrete waste can be used as a pozzolanic material to replace part of the cement in the concrete when they are well controlled.

<sup>67</sup> Vejmelkova et al. [24] evaluated the mechanical properties, durability characteristics of high <sup>68</sup> performance concrete (HPC) produced with up to 60% of Portland cement replacing by fine-<sup>69</sup> ground ceramics. The mechanical properties and water transport properties were not <sup>70</sup> significantly impaired by ceramic replacement up to 20%, while the resistance against de-<sup>71</sup> icing salts was satisfactory only up to 10% of ceramic replacement, and the chemical <sup>72</sup> resistance (to Na<sub>2</sub>SO<sub>4</sub> and MgCl<sub>2</sub>) were maintained up to 40%.

Ge et al. [26] investigated the fresh and hardened concrete with three different replacement
levels up to 30% and three different particle sizes of ground clay-brick replacing cement. The

results demonstrated that the slump of fresh concrete significantly decreased as the replacement level was over 10%. The early age strength decreased as the replacement level increased, however, the strength of concrete with ground clay-brick had similar 90-day compressive strength to that of the reference concrete. Concrete with ground clay-brick had low chloride ion penetrability and high freeze-thaw resistance.

Schackow et al. [25] studied the durability of Portland cement-lime mortars of partial replacement (10, 25 and 40%) of Portland cement by clay brick waste. The results showed that the mortars with clay brick waste had improved strength and density due to the combined physical pore filling and pozzolanic effect of clay brick waste. The mortar produced with clay brick waste showed low ability to absorb chlorides and the resistance to sulphate was lower than the reference mortar. It should be noted here that all results were obtained based on the constant total water to cement ratio.

Ortega et al. [27] analysed the long-term properties of mortar (including mechanical properties, microstructure and durability) incorporating up to 20% of waste brick powder addition as a clinker replacement. The mortars with 10% and 20% of waste brick powder presented good service properties in long-term (400 days), especially the chloride ingress resistance, even better than reference mortar, which was due to their more refined pore network produced by the pozzolanic activity and the filler effect.

Toledo Filho et al. [23] examined the potential of use crushed waste clay brick as a supplementary cementitious material with the equal workability of mortars in the proportion of 10%, 20%, 30% and 40% of crushed waste clay brick as cement replacement. The results indicated that the optimal percentages of substitution lied between 10 to 20%. The addition of crushed waste clay brick had almost no influence on the compressive strength and elastic modulus up to 20% waste clay brick replacement.

Subasi et al. [28] reported the utilization of waste ceramic powder as filler material replacing cement with the amounts of 5%, 10%, 15% and 20% in Self-Compacting Concrete (SCC). The compressive strength of SCC mix decreased gradually but not a dramatically decrease. The potential to use waste ceramic powder in SCC as cement replacement up to 15% is suitable due to the positive effects on the fresh state properties on SCC.

Bouarroudj et al. [35] estimated the remaining intra granular porosity of a ground powder using modelling approach and experimental approach with the help of mercury intrusion porosimetry. The results showed that porosity of the ground brick is almost eliminated by the grinding procedure (2% and 2.1% with the modelling approach and experimental approach).

108 It should be noted that the references mentioned above are concerned mostly with the partial substitution of cement by WBP in the mortar or concrete. Limestone filler is a commonly 109 preferred filler material to increase the packing of the granular skeleton and maintain the 110 111 cohesion and segregation resistance of SCC [29-31]. Whereas, the source of limestone filler is limited in some region and far transportation is needed for the production of concrete. The 112 WBP, which is locally available, might be used as substitution of limestone filler in SCC. The 113 rejected fired clay bricks waste, straight from the production line, whose effect on the mortar 114 or concrete has not been investigated. Because of the similarities to the bricks waste from 115 116 CDW, preliminary work was conducted with the rejected fired clay brick waste powder as substitution of limestone filler in the self-compacting mortar. 117

The objective of this work is to evaluate the possibility of substituting the limestone filler by waste brick powder (WBP) in self-compacting mortar. The properties of mortars including rheological properties, mechanical properties (compressive and flexural strengths), drying shrinkage, and durability properties (carbonation resistance, chloride ion diffusion and sulphate resistance) were investigated.

# 123 **2. Materials and methods**

### 124 **2.1. Materials**

125 Materials used for producing mortars are presented as follows.

# 126 2.1.1. Cement and natural sand

The cement used in self-compacting mortar was an Ordinary Portland Cement (CEM I 52.5 N) provided by CBR company whose mineralogical composition is shown in Table 1. The density of cement was 3.10 g/cm<sup>3</sup> measured by helium pycnometer (Micromeritics AccuPyc 130). The sand used in this study was a siliceous Standard CEN natural sand in accordance with EN 196-1 [32].

132 **Table 1** 

# 133 Mineralogical composition of cement determined by XRD-Rietveld

	C <sub>3</sub> S	$C_2S$	C <sub>3</sub> A	C <sub>4</sub> AF	Anhydrite	Calcite	Gypsum	
CEM I 52.5 N (%)	66.97	12.08	7.19	9.47	0.02	2.51	1.76	

134

# 135 2.1.2. Waste brick powder and limestone filler

The red waste bricks (high resistance brick block: 288 mm  $\times$  138 mm  $\times$  138 mm with the 136 compressive strength of 35 MPa, Fig. 1 (a)) were collected from a Belgian brick production 137 company [33]. About 200 kg of the waste bricks were crushed firstly by using semi-industrial 138 jaw crusher to obtain two fractions: 0/4 mm and 4/10 mm. The water absorption of brick 139 (fraction 4/10 mm) was 11.3% determined by the IFSTTAR method according to Equation 1 140 141 (WA represents the water absorption coefficient, M<sub>ssd</sub> is the mass of brick at saturated surface dried state using colored absorbent paper, and M<sub>dry</sub> is the mass of brick at 105°C oven dried 142 state) [5]. 143

$$WA = \frac{M_{ssd} - M_{dry}}{M_{dry}} \times 100 \tag{1}$$

145 The fraction 4/10 mm was grinded by using the semi-industrial ball mill (65 litres with 70 kg

146 of specific balls) to obtain waste brick powder (WBP: fraction 0/0.1 mm used in this study,

147 Fig. 1 (b)).



148 **Fig. 1.** WBP preparation: (a) collected red waste bricks; (b) WBP after grinding used in this study [33].

149

A commercial limestone filler (more than 98.1% of calcite) from a Belgian Company was 150 151 used for the production of self-compacting reference mortar. The density of WBP and limestone filler was 3.07 g/cm<sup>3</sup> and 2.73 g/cm<sup>3</sup> respectively, measured by helium pycnometer 152 method. Table 2 shows the chemical composition of WBP determined by X-ray fluorescence 153 (Bruker AXS, S4 Pioneer). The major chemical elements of the WBP were oxygen, silicon, 154 aluminum and iron. Table 3 presents the mineral composition of WBP determined by X-ray 155 156 diffraction (XRD, Brucker AXS D2 phaser diffractometer, 6-80° angle, 0.2° pitch and acquisition time 0.5s). The Rietveld refinement method was used to quantify the crystalline 157 phases and in particular the amorphous phase. The major mineral components of WBP were 158  $\alpha$ -Quartz, hematite and microcline. In addition, the WBP had 16.0% of amorphous phase, 159 which is the most important characteristic with regard to potential pozzolanic activity [6]. 160

161 Table 2.

162	Chemic	cal comp	osition of	crushed b	rick powo	ler (%)							
		SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	MgO	CaO	TiO <sub>2</sub>	SO <sub>3</sub>	$V_2O_3$	MnO	LOI
	WBP	62.8	10.4	16.3	0.6	2.1	2.2	1.7	2.4	0.6	0.1	0.2	0.5

<sup>164</sup> **Table 3.** 

<sup>165</sup> Mineral composition of crushed brick powder (%)

	α-SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	KAlSi <sub>3</sub> O <sub>8</sub>	NaAlSi <sub>3</sub> O <sub>8</sub>	SiO <sub>2</sub>	Amorphous
	Quartz	Hematite	Microcline	Albite	Cristobalite	phase
WBP	55.0	12.8	9.2	4.0	2.7	16.0

167	Fig. 2 presents the particle size distributions (laser granulometry analysis) of WBP, limestone
168	filler and cement used as constituents in the mortars. The WBP had a mono-sized and narrow
169	particle size distribution (PSD): containing a lower proportion of fine particles (1 $\mu$ m to 35
170	$\mu m)$ than limestone filler, and a higher proportion of particle between 35 $\mu m$ and 75 $\mu m,$
171	while limestone filler presented a continuous PSD. Table 4 reports the specific surface
172	determined by BET method, as well as D10, D50 and D90 diameters of WBP, limestone filler
173	and cement. The WBP presented a higher specific surface area value than that of limestone
174	filler according to the BET method.
175	
176	Table 4.

#### Table 4.

177	Physical	properties	of WBP,	limestone f	filler and	cement

	WBP	Limestone filler	Cement
Specific surface area BET (m <sup>2</sup> /g)	1.0	0.8	1.29
Density (g/cm <sup>3</sup> )	3.07	2.73	3.10
D10 (µm)	4.03	1.74	2.53
D50 (µm)	24.80	18.27	11.14
D90 (µm)	60.84	121.67	26.00





179

Fig. 2. Particle size distributions of WBP, limestone filler and cement.

# 181 2.1.3. Mortar design

The mix design was performed according to the method of concrete equivalent mortar (MBE) 182 developed by Schwartzentruber [34]. Table 5 presents the compositions of self-compacting 183 184 mortar. Five self-compacting mortars were manufactured with limestone filler being substituted by WBP at substitution rates (in volume) of 0%, 50% and 100% (noted M-BP0, 185 M-BP50 and M-BP100, respectively). The same water to cement ratio (W/C) of 0.8 was used 186 for the series of mortars M-BP0, M-BP50 and M-BP100 (Here only a high W/C ratio was 187 used to obtain self-compacting mortar and to eliminate the effect of superplasticizers). 188Previous investigations realized on this material [35] showed that the porosity of WBP was 189 190 very low after grinding the brick particle. Preliminary tests showed however that the substitution of limestone filler by WBP had a negative impact on the workability: preventive 191 192 measures (superplasticizers or extra water addition) are required to control the workability of

mixture. It should be noted that the purpose of this study is to estimate the actual effect of
WBP substituting limestone filler, while keeping the other variables constant. Therefore, an
additional quantity of water was used for the series of mortars M-BP50WA and M-BP100WA
in order to achieve similar spread flow index of mixture as reference mortar M-BP0 (WA in
M-BP50WA and M-BP100WA means that water added to produce mortars with similar
workability).

- 199 **Table 5**
- 200 Compositions of self-compacting mortar (g)

Mix Type	Cement	Natural sand	Limestone filler	WBP	Effective water	Additonal water	Total water
M-BP0	448.0	1350.0	298.3	0.0	358.4	0	358.4
M-BP50	448.0	1350.0	149.2	167.7	358.4	0	358.4
M-BP100	448.0	1350.0	0.0	335.4	358.4	0	358.4
M-BP50WA	448.0	1350.0	149.2	167.7	358.4	19.0	377.4
M-BP100WA	448.0	1350.0	0.0	335.4	358.4	37.9	396.3

201

# 202 2.2. Experimental methods

203 The experimental program and test methods are listed in Table 6.

### 204 **Table 6.**

205 Experimental program and test method

Experimental program and test me	Experimental program and est methods.										
Studied properties	Curing time (days)	Samples per test	Standard test method								
Water demand	0	1	Beta P test [36–38]								
Spreading flow index	0	2	MBE cone [34]								
Shear stress and shear rate	0	1	Rheocad 400 [39]								
Density of fresh mortar	0	3	EN 1015-6 [40]								
Air content	0	1	EN 1015-7 [41]								
Flexural strength	7,28	3	EN 196-1 [32]								
Compressive strength	7,28	6	EN 196-1 [32]								
Water absorption	28	3	NBN B15-215 [42]								
Drying shrinkage	1	3	NBN B14-217 [43]								
Carbonation resistance	28	3	EN 13295 [44]								
Sulphate resistance	28	3	ASTM C1012-04 [45]								
Chloride ion diffusion	28	2	Courard et al. [14,36]								

206

# 207 2.2.1. Beta P test

The water demand of limestone filler and WBP was characterized by means of  $\beta_p$  factor [36– 38]. The Beta P test was performed in order to quantify water demand  $\beta_p$  of the mixture corresponding to the rheological behavior of a paste with different water contents. This test

involved the measurement of the spreading of a paste for different water contents and a relative flow area (R) factor calculated from the spreading diameter D (mm) (Equation 2) with the mini-cone (h = 60 mm, d = 70 mm and D = 100 mm) used for European standard EN 1015-3 [46].

215

$$R = \frac{(D^2 - 100^2)}{100^2} = (D/100)^2 - 1$$
<sup>(2)</sup>

216

220

Okamura et al. [47] demonstrated that, for a paste made with any particular powder, the water powder ratio by volume  $(V_w/V_p)$  and the relative flow area (R) are linearly related (Equation 3).

$$V_w/V_p = \beta_P + R \times E_p \tag{3}$$

where  $\beta p$  is the water ratio, which can be considered as comprising the water adsorbed on the powder surface together with that required to fill the voids in the powder system and to provide sufficient dispersal of the particles, needed for mix flowing; Ep is the deformation coefficient, which is a measure of the sensitivity of the fluidity characteristics of the paste to increasing water content.

# 226 2.2.2. Mixing procedure for the manufacture of mortar

A precise mixing procedure was followed according to European standard EN 196-1 [32]. The mixtures protocol was kept the same for all the studied mortars. For the mix of M-BP50WA and M-BP100WA, the total quantity of water (efficient water plus additional water) was used to manufacture the specimens, without any pre-saturating process of WBP.

# 231 2.2.3. Tests on fresh mortars

After mixing, the first rheological test performed was the spread test. The spread flow of fresh 232 233 mortar was measured immediately after the removing the MBE cone [34] (h = 150 mm, d =50 mm and D = 100 mm, approximately 687 ml). The spread value was measured along two 234 perpendicular diameters after 5 min mixing. The second rheological test was performed with 235 Rheocad 400 (CAD Instrumentation) [39] in order to evaluate yield and shear stresses (Fig. 3). 236 This rheometer is a mechanical computer-controlled device, which is developed specially for 237 building materials characterization, such as cement, fresh mortar and concrete. Its principle is 238 based on the measurement of the torque developed in response to the controlled rotation of a 239 measuring device in a bowl. The torque is recorded as a function of the rotation velocity. 240 241 Careful calibration and used program of the system [33] allowed to transfer this correlation to a correlation between the dynamic viscosity of the mortar and the shear gradient (a range 242 between  $0.1 - 60 \text{ s}^{-1}$ ). The density of fresh mortars was determined in accordance with 243 European standard EN 1015-6 [40]. Before casting, the air content of mortars was measured 244 according to the European standard EN 1015-7 [41]. 245





(b)

246

Fig. 3. Rheocad 400: (a) machine with bowl; (b) special impeller.

247

#### 2.2.4. Mechanical behavior of mortars 248

Six specimens of 40 mm  $\times$  40 mm  $\times$  160 mm were prepared for measuring the mechanical 249 behaviour of mortars for each mixture. One day after casting, these specimens were 250

demoulded and stored into water at  $21 \pm 2$  °C until the ages of 7 and 28 days. After each age, both flexural and compressive strengths were evaluated in conformity with European standard EN 196-1 [32]. These two mechanical strength tests were carried out with an INSTRON 5585 (loading capacity of 200 kN). After the failure of the three specimens in bending test, the two parts of each prism were subjected to compressive strength measurement according to European standard EN 196-1.

### 257 2.2.5. Water absorption

Water absorption of mortars was measured after 28 days of curing. Three specimens of 40 mm  $\times$  40 mm  $\times$  160 mm were used for the determination of water absorption according to the standard NBN B15-215 [42]. The specimens were immersed into water until achieving a constant mass. The constant mass was taken as the saturated mass of sample (M<sub>satu</sub>). The dried mass was obtained after drying the saturated sample in an oven at 105°C until constant mass (M<sub>dried</sub>). The water absorption (WA) was then obtained by ratio of the absorbed water amount to the oven-dried mass (Equation 4).

$$WA = \frac{M_{satu} - M_{dried}}{M_{dried}} \times 100 \tag{4}$$

265

# 266 2.2.6. Drying shrinkage

Three mortar specimens (40 mm  $\times$  40 mm  $\times$  160 mm) were used for evaluating drying shrinkage. The prisms were demoulded one day after casting and immediately stored in dry climatic room at 21  $\pm$  2 °C and 60  $\pm$  5% RH, according to the standard NBN B14-217 [43]. The drying shrinkage measurements were continuously performed at ages of 1, 3, 7, 14 and 28 days. Length variances were determined in accordance with the standard NBN B14-217.

# 272 2.2.7. Resistance to carbonation

13295 [44]. The three mortar specimens (40 mm × 40 mm × 160 mm) were cured in water for 28 days and then pre-conditioned at  $21 \pm 2$  °C and  $60\pm5\%$  relative humidity (RH) for 28 days before the carbonation test. Specimens were then stored in the carbonation incubator with 1% CO<sub>2</sub> concentration and relative humidity of  $60\pm5\%$  (21 ± 2 °C). After 28 and 56 days' carbonation, respectively, the carbonation depth of freshly broken faces was measured by means of phenolphthalein indicator following the European standard EN 13295.

# 280 2.2.8. Chloride ion diffusion

273

The chloride ion diffusion coefficient of mortars was determined using two-compartment 281 diffusion cells [14,36] (Fig. 4). After 28 days curing in water, 10 mm  $\pm$  2 mm thick mortar 282 samples were sawed from 8 cm diameter specimens and stored in Ca(OH)<sub>2</sub> saturated solution. 283 Each sample was polished with 600-grade emery paper, rinsed with deionized water and the 284 surface dried with a tissue before being fitted into the diffusion cell. After fitting with epoxy 285 resin and sealing with silicon paste, the cells were filled at one side with 3 M NaCl in 286 saturated Ca(OH)<sub>2</sub> solution (left compartment, e.g. upstream in Fig. 4, the chloride 287 concentration of left compartment was noted as  $C_{up}$ ) and at the other side with Ca(OH)<sub>2</sub> 288saturated solution (right compartment, e.g. downstream in Fig. 4, the chloride concentration of 289 right compartment was noted as C<sub>down</sub>). At periodic intervals (2, 4, 6, 8, 10, 12, 14 and 16 290 weeks, respectively), chloride concentration ( $C_{down}$ ) was determined in right compartment by 291 means of titration from a 10 cm<sup>3</sup> sample of the solution. The chloride ion diffusion coefficient 292  $(D_c \text{ in } m^2/s)$  was determined according to the Equation 5 [48] (where  $C_{up} >> C_{down}$ ). 293

$$D_c = \frac{C_{down}}{C_{up}} \times \frac{eV_{down}}{A(t-t_0)}$$
(5)

where e is the thickness of specimens in m,  $V_{down}$  is the volume of right compartment in m<sup>3</sup>, A is the surface area of specimens in m<sup>2</sup> and t-t<sub>0</sub> corresponds to the testing time in days, respectively.



### 298

Fig. 4. Experimental setup of chloride diffusion cells.

# 299 2.2.9. Sulphate resistance

The sulphate resistance of mortars was performed in accordance with the ASTM C1012-04 [45]. The method involved the determination of length change of prismatic specimens immersed in a standard sulphate solution (with a sodium sulphate concentration of 50 g/litre). After the 28 days curing in the water, the three mortar specimens (40 mm × 40 mm × 160 mm) were measured for length and placed in the standard sulphate solution at  $21 \pm 2^{\circ}$ C. Changes of length of the specimens were measured after storage periods of 1, 2, 3, 4, 5, 6 and 11 weeks, respectively.

# 307 3. Results and discussion

# 308 3.1. Water demand of limestone filler and WBP

The relationship of water powder ratio by volume (Vw/Vp) and relative flow area (R) for 309 310 limestone filler and WBP is presented in Fig. 5. Table 7 shows the results of Beta P test. As can be seen in Fig. 5 and Table 7, the WBP presented a higher βp compared to the limestone 311 filler, which can be justified by mono-disperse and narrower particle size distribution of WBP 312 and higher BET specific surface area [26,28]. This indicates that a higher quantity of water is 313 needed to achieve the same workability of paste for WBP. Thus WBP has a higher water 314 315 demand compared with limestone filler. This finding agrees with the results of other authors [6,25]. Regarding the deformation coefficient (Ep), the WBP had a higher value 316 demonstrating a greater sensitivity to the variation of water quantity [36,37]. 317



Fig. 5. Relationship of  $V_w/V_p$  and R for limestone filler and WBP.

319

318

- **Table 7.**
- 321 Results of Beta P test

Results of Beta I t							
Powder	$\beta_p$	E <sub>p</sub> (deformation coefficient)	$\mathbf{R}^2$				
Limestone filler	0.742	0.038	0.905				
WBP	1.111	0.055	0.935				

322

# 323 **3.2.** Properties of fresh mortars

Fig. 6 presents the spread values of mortars as a function of WBP content. In the series of 324 mortars M-BP (M-BP0, M-BP50 and M-BP100), the spread of mortars decreased when 325 substituting limestone filler by WBP. Other researchers reported that the workability of 326 mortar or concrete decreased as the substitution of the cement by brick powders increased 327 [21,25,26,28]. In the series of mortars M-BPWA (M-BP50WA and M-BP100WA), the 328 substitution of limestone filler by WBP didn't seem to affect the spread flow properties of 329 mortars. The mixture M-BP50WA presented slightly higher or similar spread value as the 330 reference mixture. This outcome is probably due to the higher water demand of WBP 331 compared with limestone filler [25,26] and the higher quantity of water that is added to the 332 mix. 333



Fig. 6. Spread of fresh mortars as a function of WBP content (M-BP refers to the series of mortars M BP0, M-BP50 and M-BP100; while M-BPWA refers to the series of mortars M-BP50WA and M BP100WA).

The flow curves were analysed with the standard Bingham model to study the rheological properties of non-Newtonian fluids such as cement pastes, mortars and concretes (Equation 6).

$$\tau = \tau_0 + \mu \dot{\gamma}$$

(6)

340 where  $\tau$  is shear stress,  $\tau_0$  is yield stress,  $\mu$  is plastic viscosity and  $\dot{\gamma}$  is shear rate.

339

Fig. 7 shows that the flow curve of mortars provided a perfect fit to a straight line for shear 341 rates in the 1-22 s-1 range. The yield stress, plastic viscosity and the correlation coefficient 342  $(\mathbf{R}^2)$  are presented in Table 8. The results showed that the mortars with substitution of 343 limestone filler by WBP presented a higher yield stress and plastic viscosity than that of 344 reference mortar. The trend line equation of the reference mortar presented the lowest yield 345 stress and plastic viscosity. For the series of mortars M-BP, the yield stress and plastic 346 viscosity of mortar increased with the substitution rate of WBP, which is in accordance with 347 spreading flow test. Indeed, these behaviors probably depended on the internal friction 348 349 between the particles and the fluidity of mortars: the WBP presented a mono-size and narrower PSD, a higher surface specific area and probably higher internal friction between the 350

particles than limestone filler. In addition, a few of water was adsorbed by the WBP during the mix, which led to decrease of the availability of free water in the mix: thus the torque and the viscosity of mortar increased with the substitution rate [39,49]. For the series of mortars M-BPWA, the yield stress and plastic viscosity of mortar slightly increased when substituting limestone filler by WBP, which is probably due to higher internal friction of WBP (the additional water was added to produce the mortar with the similar workability level).

On the basis of the values obtained by Rheocad tests, it can be considered that the series of mortars M-BP presented initial yield stress and plastic viscosity higher than the series of mortars M-BPWA. For the series of mortars M-BPWA, the fluidity of the mix increased because of the additional water used, which led to the decrease of the viscosity and the torque or shear stress. These results are consistent with the workability of mortar evaluated by means of spreading flow test.



363

364

Fig. 7. Flow curve of mortars and evaluation of the yield stress.

365

# 367 **Table 8.**

368 Rheological properties of mortar

Mortar	$\tau_0$ (yield stress, Pa)	μ (plastic viscosity, Pa s)	$\mathbf{R}^2$
M-BP0	61.52	5.51	0.99
M-BP50	117.04	7.95	0.99
M-BP100	171.16	12.32	0.99
M-BP50WA	65.60	6.36	0.99
M-BP100WA	76.03	7.55	0.99

369

Fig. 8 indicates the air content of mortars as a function of WBP substitution rate: the higher the substitution rate, the higher the air content. This is probably due to the higher specific surface area and mono-sized and narrower PSD of WBP compared with the limestone filler and induces the inclusion of air bubbles during the mixing procedure. Moreover, the density of the fresh mortars decreased when substitution rate increased. Similar observations were found with waste ceramic powders by Subaşı et al. [28].





Fig. 8. Air content of mortars as a function of WBP content.

377

# 378 **3.3.** Properties of hardened mortars

# 379 *3.3.1. Mechanical properties*

Fig. 9 and Fig. 10 show the results of compressive and flexural strengths of mortars as a 380 381 function of WBP content. When the substitution of limestone filler by WBP increased, the compressive strength of mortars slightly decreased after 7 days (the compressive strength of 382 mortars M-BP decreased 5.6% and 9.3% for 50% and 100% WBP based mortars comparing 383 with the reference mortar, respectively; while the compressive strength of mortars M-BPWA 384 decreased 2.7% and 16.7% for 50% and 100% WBP based mortars, respectively; the 385 compressive strength of series mortar M-BP100WA could achieve 26.8 MPa), which is 386 probably due to the higher air content for mortars with WBP. After 28 days, the compressive 387 strength of mortars with WBP was equivalent to reference mortar with limestone filler (the 388 389 compressive strength of series mortar M-BP100WA decreased 5.3% comparing with the reference mortar and it could achieve 35.6 MPa); the decreasing trend seems to be 390 compensated, probably by the pozzolanic activity of WBP [6,27] (Ortega et al. [27] showed 391 392 the quantity of portlandite for mortar made with 10% and 20% of brick powder did not increase much or even decreased from 15 to 90 days, due to the consumption of portlandite in 393 the pozzolanic reactions based on thermogravimetric analysis) and this effect could be 394 enhanced after 90 days [50]. The compressive strength of the mixture M-BP100 was slightly 395 greater (1.7 MPa) than the mixture M-BP50. The 16% of amorphous phase content present in 396 397 WBP could react with calcium hydroxide and form hydrated products as calcium hydrated silicates (C-S-H) and calcium aluminate hydrates (C-A-H), which progressively enhance 398 strength and durability properties of cement based materials [16,23,24,27]. For the series of 399 400 mortars M-BP, the compressive strength was slightly higher than the series of mortars M-BPWA. The difference between the two series was the highest for the 100% substitution rate, 401 since additional water quantity was added for the series of mortars M-BPWA, and thus the 402 higher water to cement ratio induced the decrease of the compressive strength of mortars for 403 the series of mortars M-BPWA. It is important to notice that all the specimens fulfil the 404

requirements of the European standard EN 998-2 [51] about the specification for mortars for
masonry (the compressive strength of masonry mortars Grade "Md" should be greater than 25
MPa). Similar trends were observed for the flexural strength of mortars.





408

409

Fig. 10. Flexural strength of mortars as a function of WBP content.

411 *3.3.2. Water absorption* 

The water absorption of mortars cured for 28 days are presented in Fig. 11. For the series of mortars M-BP, the water absorption values were similar for the different WBP contents. The

water absorption values of mortars M-BPWA were slightly higher than the series of mortars 414 415 M-BP, which is due to the additional water: this is inducing a higher initial water to cement ratio when compared to the series of mortars M-BP and leading to higher porosity. In 416 addition, the reaction between  $Ca(OH)_2$  and the amorphous compounds (pozzolanic effect), 417 such as silica and alumina present in WBP, will generate supplementary silicate/aluminate 418 hydrates similar to those produced in the cement hydration; it will induce to a refinement of 419 the pore structure and a decrease of the porosity of the cement matrix [16,23,27]. Tests 420 performed at 90 days should confirm even better this trend [50]. Finally, the porosity or water 421 absorption values were similar for the different WBP contents for the series of mortars M-BP, 422 while the slightly higher values were obtained for the series of mortars M-BPWA. 423





Fig. 11. Water absorption of mortars at 28 days.

# 425 *3.3.3. Drying shrinkage*

The drying shrinkage of mortars is presented in Fig. 12. The incorporation of WBP induced a reduction of the drying shrinkage of mortar in comparison with that of limestone filler, which is probably due to their more refined pore network produced by the pozzolanic activity [50]. The results on series of mortars M-BPWA showed a reduction of the drying shrinkage of mortar, but to a lesser extent. It should be noted here that the use of additional water for

431 mortars MP-BPWA increased the initial water to cement ratio and this should contribute to an



432 increase in drying shrinkage compared with the mortars MP-BP [36,52–54].



### 433

# 434 **3.4.** Durability of mortars

# 435 *3.4.1. Carbonation*

Fig. 13 shows the carbonation depth of mortars under accelerated conditions. The substitution 436 of limestone filler by WBP increased the carbonation depth of mortars. In addition, the 437 carbonation depths increased with time. Accelerated carbonation in the mortars is the result of 438 the reaction between carbon dioxide and calcium hydroxide, calcium silicate hydrates 439 440 produced upon hydration [5,54,55]. As discussed earlier in Section 3.3.2, the WBP content slightly increased the permeability of the open pore network, which should promote the 441 penetration of  $CO_2$  and, consequently, an increase in the carbonation rate [25]. The results 442 obtained with the series of mortars M-BPWA showed a higher carbonation depth than that of 443 444 series of mortars M-BP, which is due again to a higher porosity of the mortar.



Fig. 13. Carbonation depth of mortars as a function of time.

446 447

### 448 *3.4.2.* Chloride ion diffusion

Fig. 14 presents the evolution of chloride ion diffusion through mortars versus time. The 449 standard deviations of M-BP0 and M-BP100WA are also presented in Fig. 14. The apparent 450 chloride ion diffusion coefficient of mortars is shown in Table 9. Transport through reference 451 mortar was observed after 16 weeks, with an apparent chloride diffusion coefficient of 452  $4.01 \times 10^{-13}$  m<sup>2</sup>/s. The apparent chloride diffusion coefficients of mortars with WBP were 453 similar to that of the reference mortar (except for the mortar M-BP100WA presenting slightly 454 greater value, which may due to the highest porosity of mortar M-BP100WA [23,26,27]; the 455 mortar M-BP100 presenting lowest value, which may due to the pozzolanic effect and a 456 refinement of the pore structure [50]). But globally, the substitution of limestone filler by 457 WBP didn't seem to significantly impair the chloride ion diffusion resistance of mortars. 458



460



Fig. 14. Chloride diffusion rates for mortars with limestone filler and WBP.

462

### 463 **Table 9.**

464	Chloride o	liffusion	rates f	for 1	nortars	with	limestone	filler	and	WBF	)
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Mortar	Apparent ch	loride diffusion coefficient (m <sup>2</sup> /s)	Standard deviation (m <sup>2</sup> /s)	
M-BP0	$4.01  imes 10^{-13}$		$1.83  imes 10^{-14}$	
M-BP50	$4.00 imes10$ $^{-13}$		$2.81  imes 10^{-14}$	
M-BP100	$3.69 \times 10^{-13}$		$1.49  imes 10^{-14}$	
M-BP50 W.	A $4.03 \times 10^{-13}$		$6.89  imes 10^{-15}$	
M-BP100 W	VA $4.94 \times 10^{-13}$		$3.33  imes 10^{-14}$	

465

### 466 *3.4.3. Resistance to sulphate*

Fig. 15 presents the variation in length of mortars immerged into Na<sub>2</sub>SiO<sub>4</sub> solution. All the 467 mortars exhibited similar expansion after only a few days. The expansion of the mortars M-468 BP50 and M-BP100 after 11 weeks was measured as 314 µm/m and 383 µm/m, respectively. 469 470 The expansion of the series of mortars M-BPWA show similar values as the reference mortar (the expansion was measured as 388 µm/m for the reference mortar). The mortar M-BP100 471 presented slightly higher length variation after the sulphate attack compared with the mortar 472 473 M-BP100WA, which may due to the lower porosity than that of the mortar M-BP100WA, and thus the lower porosity means the lower ability to resist the expansion of the formed ettringite 474 [56]. According to the literature, the resistance to sulphate is related to the pore sizes, as well 475

as the total porosity and permeability of cement based materials. The substitution of limestone
filler by WBP slightly increased the permeability of the open pore network, which was
compensated by the pozzolanic effect of WBP [23,25]. The substitution of limestone filler by
WBP didn't seem to affect the behavior of mortars with regard to sulphate attack.



480

Fig. 15. Length variation of mortars with limestone filler and WBP in sulphate solution.

482

481

### 483 **4. Conclusions**

The possibility of substituting limestone filler by Waste Brick Powder (WBP) in selfcompacting mortar has been evaluated. The main conclusions can be drawn as follows:

The WBP are characterized by a higher βp (a higher water demand) compared to the
limestone filler. This indicated a higher quantity of water is needed to achieve the
same workability of paste for WBP.

The mortars produced by substitution of limestone filler by WBP presented a higher
 yield stress and plastic viscosity than that of reference mortar. In the series of mortars
 M-BP, the yield stress and plastic viscosity of mortar increased with the substitution
 rate of WBP, which is consistent with the workability measured by means of spread

493 test (the spread of mortars decreased with the substitution rate of limestone filler by
494 WBP). In the series of mortars M-BPWA, the substitution of limestone filler by WBP
495 didn't seem to affect the spread properties of mortars.

3) When the substitution rate of limestone filler by WBP increased, the compressive 496 strength of mortars slightly decreased after 7 days (the compressive strength of series 497 mortar M-BP100WA decreased 16.7% and it could achieve 26.8 MPa), but the 498 decreasing trend seemed to be compensated by the pozzolanic activity of WBP and 499 remained equivalent after 28 days (the compressive strength of series mortar M-500 BP100WA decreased 5.3% and it could achieve 35.6 MPa). After 28 days, the 501 502 compressive strength of mortars with WBP was equivalent to reference mortar with limestone filler; the decreasing trend seems to be compensated by the pozzolanic 503 activity of WBP and this effect should be enhanced after 90 days. In the series of 504 mortars M-BP, the compressive strength of mortars was slightly higher than the series 505 of mortars M-BPWA. It is important to notice that all the specimens fulfil the 506 compressive strength requirement of the European standard EN 998-2 for masonry 507 mortars Grade "Md". 508

4) The conclusion can be drawn the choice whether on constant flow index (series of 509 510 mortars M-BPWA) or constant water to cement ratio (series of mortars M-BP). The mortars prepared with additional water (mortars M-BPWA) showed poorer 511 mechanical properties. On the contrary, for the series of mortars M-BP, the WBP's 512 filler or pozzolanic effect will be enhanced by the presence of less water (decrease of 513 flowability). There is a beneficial effect from WBP if no additional water is added. 514 However, in the case of higher WBP substitution rate (100%), it is needed to add extra 515 water or superplasticizers in order to achieve the workability requirement of fresh 516 mortars, especially for the self-compacting mortars. 517

5) The incorporation of WBP induced a reduction of the drying shrinkage. The substitution of limestone filler by WBP however increased the carbonation depth of mortars: the results obtained with series of mortars M-BPWA presented higher carbonation depth than that of series of mortars M-BP, which is due to the higher porosity of the mortar. Finally, the substitution of limestone filler by WBP didn't seem to impair the behavior of mortars in case of sulphate and chloride ions.

The use of Waste Bricks Powder (WBP) as an alternative to limestone fillers seems to be a good opportunity for recycling waste brick and reducing natural resource depletion. Fresh and hardened properties of mortars globally fulfil the requirements for self-compacting mortars. Particular attention should be paid to the use of these materials in the case of the presence of reinforcements and the risks associated with carbonation. However, a more in-depth study, in particular on samples at 90 days for which the manifestation of the pozzolanic effect would be more evident, should still be carried out for future research.

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# Highlights

- Waste brick powder (WBP) presented a higher water demand than limestone filler (LF)
- The mortars produced by WBP presented a higher yield stress and plastic viscosity
- The mortar M-BP presented a higher compressive strength than that of mortar M-BPWA
- The substitution of LF by WBP didn't seem to impair the durability of mortars
- It is possible to manufacture SCC by partially or totally substituting LF by WBP

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# **Declaration of interests**

 $\boxtimes$  The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

□The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

