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Properties of cement pastes and mortars containing recycled green glass 1 powder 2 Samir NAHI^{a, b, *}, Nordine LEKLOU^a, Abdelhafid KHELIDJ^a, Mohamed N OUDJIT^b, 3 Abdelfatah ZENATI^b 4 ^a University of Nantes - IUT Saint-Nazaire, GeM, CNRS UMR 6183, Research Institute in 5 Civil Engineering and Mechanics, 58 rue Michel Ange (BP 420), 44606 Saint Nazaire Cedex, 6 7 France. 8 ^b Built Environment Laboratory « LBE », Faculty of civil engineering, University of Sciences and Technology HOUARI Boumediene, (BP 32) El-Alia, Bab Ezzouar, Algiers, Algeria 9 10 16111, Algiers, Algeria. 11 *Corresponding author e-mail: n.ss.n@outlook.com (S. NAHI)

12 Abstract

This work deals with the characterization of the properties of mortars and cement pastes made with 13 14 powdered soda lime waste glass. Different rates of cement replacement by weight are used: 0%, 10%, 15 25%, 35% and 60%. Both fresh and hardened properties are assessed. The influence of glass powder on the workability of mortar is determined using flow table tests. Chemical shrinkage and hydration 16 heat of early age blended cement paste are evaluated using gravimetric methods and isothermal 17 18 calorimetric respectively. The dynamic Young's and shear moduli of mortar are evaluated using nondestructive methods. Mechanical performances of mortars are measured using flexural and 19 compressive strength tests. Pore structures of different mortars are investigated with mercury intrusion 20 21 and water porosity tests.

Results from this investigation indicate that the amount of cement replacement by glass powder plays a critical role in determining mortar characteristics. Chemical shrinkage and heat assessment show that high glass powder replacement of cement paste increases the hydration reaction rate. Compressive strength and dynamic Young's and shear moduli values decrease with increasing glass powder content especially for mortar made with 60% glass powder.

27 Mortar made with 60% glass powder has bigger pores due to the dilution effect, i.e., the reduction in28 the amount of hydration product at 7 days hydration. Significant compressive strength is gained

between 7 and 90 days for mortar with glass powder, especially with 60% replacement showing pozzolanic reaction taking place during this period. The pozzolanic reaction that occurs between glass powder and calcium hydroxide (Ca(OH)₂) to form more calcium silicate hydrate gel (C-S-H) leads to the refinement of pores and the reduction of porosity.

Key words: glass powder, pozzolanic reaction, chemical shrinkage, porosity, dynamic Young's and
shear moduli.

1. Introduction

Glass is considered to be one of the most used materials in numerous quotidian applications. Glass can 36 be found primarily in the form of containers such as beer and soft drink bottles, wine and liquor 37 38 bottles, and bottles and jars for food, or in the flat form for windows and automotive industries. It can 39 also be found in cosmetics, appliances, electronics, fiber and other products. Ideally, waste glass should either be reused or remanufactured to produce new glass products [1]. Though glass can 40 theoretically be completely recycled, there are still limitations in terms of meeting the quality criteria 41 for glass remanufacturing [2]. Just a small amount of the waste glass that is generated yearly is 42 43 recycled. Global recycling volumes in 2018 were estimated to be around 27 million tons, which represents only 21% of the amount of glass produced [3]. A large amount is discarded and occupies 44 45 space in landfill or thrown and abandoned in nature. Its presence is not always aesthetically pleasing and can represent a real danger to the safety of people and animals as well. Glass can cause serious 46 47 environmental pollution (air, soil and water pollution) because of being non-biodegradable. In addition 48 to landfill space, environmental and human constraints, glass is a non-renewable resource and its 49 recycling reduces non-renewable natural resource consumption, energy consumption and carbon 50 footprint.

Glass can be divided into several major families, depending on the field of application: soda lime, borosilicate, aluminosilicate, lead glass, silica glass, zirconia glass, bio glass, fluorinated glass, vitroceramic, chalcogenides, and metallic [4]. Among those families, soda-lime glass is the most frequently used [4, 5] and the most investigated due to the large number of containers collected in urban areas [6]. The particle size of glass has been found to have major impact on its usability in concrete mixes [7]. Glass was first used as aggregate in cementitious material, but this attempt wasn't successful due to the occurrence of alkali-silica reactions (ASR) [8].

Indeed, ASR can be mitigated by adding pozzolanic materiel such as fly ash [9, 10, 11], ground granulated blast furnace slag [11, 12], silica fume [11], and even glass in powder form (GP) [11, 13, 14], or by adding steel fiber, lithium chloride or lithium carbonate compounds [11]. Conversely, mortars with ground glass immersed in water for a period of seven years did not show any sign of degradation due to ASR [15].

Finely ground glass is an amorphous material. When it is compared to other pozzolanic materials, we can see that the overall content of SiO₂, Al₂O₃ and Fe₂O₃ is similar or slightly lower than that of the other mineral additions such as silica fume, fly ash or natural pozzolans [15, 16, 17].

Nowadays, ground glass pozzolans have been introduced as new supplementary cementitious 66 materials in Canadian standard A3018-18 [18]. The reactivity of glass powder is a compromise of 67 several parameters such as water-to-binder ratio, fineness, colors, temperature, etc. Much previous 68 69 work has been done in this field to understand the effect of particle size (or fineness), on glassy SCM reactivity [1, 17, 19, 20, 21, 22, 23, 24]. Most of these tested glass powder below a certain grain size 70 71 or between two limited intervals of grain size so that it is difficult to assess the contribution of the 72 particle size on the Pozzolanic reactivity. This is due to a continuous gradation and flocculation of 73 particle size leading to difficulty of separation. In order to investigate the effects of particle size on 74 glass powder reactivity, Mirzahosseini et al. [19], prepared mortar and paste using glass powder with very narrow particle size distributions 0-25 µm, 25-38 µm and 63-75 µm. Results indicate that mortar 75 76 with particle sizes in the range of 0-25 µm shows the highest compressive strength but that it is lower 77 than reference mortar even at 90 days. Patel et al. [20], studied the effect of glass powder particle size 78 decreases from 75 µm to 63 µm on mortar compressive strength. Results indicate that 20% 79 replacement of cement by GP with particle size smaller than 63 μ m, gives compressive strength comparable to reference mortar at 90 days and satisfies the Strength Activity Index (SAI) at 7 and 28 80 days. Whereas, in the case of particles smaller than 75 μ m, just 10% to 15% of cement replacement by 81 GP can be used to reach satisfied SAI. Aliabdo et al. [21], found that the use of 10% glass powder 82

with particles size smaller than 75 µm as cement replacement enhances the mortar compressive 83 strength by about 9.0 % at 7 days. Parghi et al. [22], found that compressive strength of mortar made 84 85 with 5%, 10%, 15%, 20% and 25% glass powder having particle size smaller than 75 µm, as cement 86 replacement increases compared to the control mortar at 7, 28 and 90 days. Results done in this field show that the pozzolanic properties of glass powder are highly influenced by the particle sizes of glass 87 or rather by its fineness of grinding and the amount of glass used. The reactivity and compressive 88 89 strength of cementitious materials increase as the particles size of the waste glass decreases and 90 fineness increases. According to Idir et al. [1], each kind of glass should be assessed in order to 91 evaluate its reactivity.

92 The optimum glass replacement partly depends on the particles sizes that govern pozzolanic reaction. 93 According to Du et al. [25], when using particles with mean diameters of about 3.4 μ m, the optimum 94 GP replacement ratio in term of compressive strength is between 30% and 45%. Below the ratio of 30%, there is redundant portlandite in the cement hydration products, while when exceeding a ratio of 95 45%, insufficient portlandite is available for the GP pozzolanic reaction. Conversely, when it comes to 96 97 durability parameters such as chloride diffusion, water penetration depth and sorption, it is found that concrete made with 60% GP shows better performance. According to Neithalalath et al. [26], the 98 incorporation of GP results in an increase of the hydration degree of the cement grains. The hydration 99 100 degree of glass powder depends partly on the cement substitution level. Increasing the GP content 101 decreases the calcium hydroxide content and leads to a decrease in the Ca/Si ratio, which becomes less than 1 in the C-S-H formed due to less available Ca²⁺ and a lower pH in the pore solution. Thus, it will 102 103 weaken the hydration degree of the glass because of the instability and the easy decomposition of the 104 hydrates formed [5, 25].

Notwithstanding that the chemical composition of colored glass seems to be similar, there are great differences in the structure depending on the role of each element as network former, modifier or intermediate [4]. Among the divergent results found concerning the influence of glass particle diameter on the reactivity of cementitious materials and the diversity of existing glass (type, colors), just a few works, e.g. Du et al. [5, 25, 27],Liu et al. [28], have studied the influence of high glass content (60%) on cement pastes, mortar and concrete characteristics. With high glass, the literature

does not show any results on chemical shrinkage of cement paste or dynamic Young's and shear 111 moduli of mortar. Additionally, there is limited available literature characterizing the microstructure of 112 113 mortar made with different rates of GP content taking into account their colors and particle size. In order to bring more clarification to fresh and hardened characteristics especially concerning hydration, 114 microstructure and porosity further study is required. The purpose of this work is to investigate the 115 influence of cement replacement with soda lime Green Glass Powder (GR GP) on paste and mortar 116 117 characteristics. Increasing rates of GP replacement of cement by mass (10%, 25%, 35% and 60%) are used. Material characteristics are evaluated by measuring flow, heat of hydration, chemical shrinkage, 118 compressive strength, and dynamic Young's and shear moduli, water evaporation and gives more 119 explanation about mortar microstructure and porosity. This study is done to determine the optimum 120 percentage of green glass powder and the possibility of high green glass usage. 121

122 **2.** Materials and methods

123 **2.1. Materials**

124 The main components of cement pastes and mortars used in this study are:

-Standard sand (0/2 mm), consisting of non-reactive siliceous rounded particles, conforming to
European Standard EN 196-1 [29].

-Ordinary Portland cement (OPC) CEM I 52.5 N which complies with European standard test EN 1971 [30].

-Green glass powder coming from various types of wine and liquor bottles collected from the seaside.

In order to study just the effect of glass as a SCM without the influence of contamination and impurities caused by the different liquid content remaining in the bottles, plastic or paper labels stuck on the bottle, and metal or plastic security rings, glass bottles are first cleaned and washed before grinding. After that, they are dried and crushed. After removing plastic and metal impurities, they are ground in a ball mill. The powder resulted is poured through a 0.063 mm sieve and the powder passing through was used for this study. According to a previous study [23], ground glass powder consists mainly of elongated and plate shaped particles. Fig 1 Shows the surface texture of the finely ground GP using a scanning electron microscope (SEM). As we can see from Fig. 1, glass consists of irregular and plate shaped particles with smooth surface.



140

141

Fig. 1. SEM of green glass powder.

142 Particle size distribution of the cement and glass powder used for this work was determined by using a

143 laser particle size analyzer as shown in Fig. 2. Cement particles are finer than glass particles, the

average particle size for cement is $9.8 \,\mu m$ compared to $17.68 \,\mu m$ for glass.







Fig. 2. Particle size distribution of cement and glass powder.

Chemical composition (%)	OPC	GP
SiO ₂	20.07	73.64
Al ₂ O ₃	4.86	1.42
Fe ₂ O ₃	3.07	0.38
CaO	64.25	9.08
MgO	0.95	1.43
SO ₃	3.55	0.27
K ₂ O	1	0.01
Na ₂ O	0.18	11.62
P ₂ O ₅	-	0.01
TiO ₂	-	0.05
LOI	1.09	2.09





Table 1 Chemical composition of the cement and glass powder.

149 The cement bogue's composition and physical properties of the cement and glass powder are

150 summarized in Tab.2.

151

152

Bogue's composition				
C ₃ S 60.8				
C_2S	14.	6		
C ₃ A	8.2	2		
C ₄ AF 9.8				
Physical characteristics	OPC	GP		
Specific gravity	3.16	2.5		
Blaine (cm ² /g)	4290	3206		
Particle size distribution (µm)				
Diameter at 10%	1.26	3.6		
Diameter at 50%	9.80	17.68		
Diameter at 90%	30.42	46.73		

Table 2 Cement Bogue's composition of and physical characteristics of cement and glass powder.

155 **2.2.** Mix proportions and mixing procedure

156 Cement paste and mortar mixes having the same binder composition, noted by 0 SCM, 10 GR GP, 25 157 GR GP, 35 GR GP and 60 GR GP, according to their cement replacement rates of 0%, 10%, 25%, 158 35% and 60% respectively, are prepared for this study (see Tab. 3). Followability, compressive and 159 flexural strength, water porosity, mercury intrusion porosimetry, mass variation, and dynamic Young's 160 and shear moduli tests are performed on simple mortar. Hydration heat assessment and chemical 161 shrinkage are evaluated for cement pastes. A water-to-binder ratio of 0.5 is used and kept constant for 162 all mixes used for tests except the chemical shrinkage mix, where a water-to-binder ratio of 0.4 is used 163 in order to avoid the risk of bleeding and its potential influence on chemical shrinkage. The mixing is done according to the standard NF EN 196-1 [29]. Fresh pastes are directly tested after mixing for 164 chemical shrinkage and heat hydration assessment at 20 °C. Each mortar mix is composed of three 165 166 parts sand and one-part binder (cement and glass). For each mortar mixture, $40 \times 40 \times 160$ mm specimens are manufactured. After 24 hours (h) curing in a moist room at 20 °C and 90% relative 167 humidity (RH), the mortar bars are demolded and cured in a conditioned room at 20±2 °C. Specimens 168 designed for mechanical tests, water porosity and mercury intrusion porosimetry tests were 169 170 conditioned in water. Specimens for mass variation and dynamic Young and shear moduli tests are 171 conditioned in a climatic chamber at 50±5% RH.

Mix	0 SCM	10 GP GR	25 GP GR	35 GP GR	60 GP GR
Cement	100	90	75	65	40
Glass Powder	0	10	25	35	60

Table 3 Binder composition of paste and mortar.

173 **2.3.** Testing methods

174 **2.3.1** Followability of mortar

In order to access consistency for unconfined and consolidated fresh mortar made with different rates of cement substitution by glass powder, a flow table test is performed according to EN 1015-3 [31]. A standard conical mold, having an internal diameter of 100 mm at the base, 70 mm at the top and 60 mm height, provided with a riser, is used. Tests consist of 15 vertical shocks in 15 seconds to a mold in which was placed a sample of fresh mortar. This latter is introduced on the mold in two layers, each layer compacted with at least 10 strokes to insure uniform filling. After removing the mold, two perpendicular spread diameters are measured and the flow diameter is expressed by the average.

182 **2.3.2** Heat evolution assessment

Early age hydration kinetics of cement paste made with different levels of glass powder substitution (0%, 10%, 25%, 35%, and 60%) is evaluated by using a TAM Air calorimeter device [32, 33]. Solid raw materials are mixed in advance and then mixed with water for three minutes. Immediately after mixing, samples of 4 to 6 g are taken and placed into standard plastic vials and then loaded into the isothermal calorimeter. The heat release and heat flow results are normalized by the mass (g) of total binder (cement + glass powder) and by mass of cement. The hydration heat measurement is continued for approximately 96 h (4 days) under a constant temperature of 20 °C.

190 **2.3.3** Chemical shrinkage of cement pastes

191 The chemical shrinkage of different cement pastes made with and without glass powder is measured 192 by gravimetric methods [34, 35]. Immediately after mixing, about 10 grams of mixed paste are placed 193 into a plastic cylindrical vial of about 2 cm in diameter and 5 cm in height. In order to ensure complete 194 percolation, a sample paste thickness of 7.0 mm to 8.0 mm is used. Water is added drop by drop to 195 minimize the disturbance of the paste top layer. Cement paste is introduced into the vial without 196 trapping of air bubbles in the paste. The vial is hung on a balance (accuracy 0.0001 g) by a nylon 197 thread and immersed in a water bath at 20 °C.

Tests start 30 minutes after initial water-cement contact for all cement pastes and last 48 h. The apparent mass of the vial with cement paste is recorded automatically every 5 minutes. The hydration process lead to volume changes of the cement paste. Water penetrate into the paste and fill the porosities generated by Le Chatelier's contraction. Chemical shrinkage of cement paste *CS* (*t*, *T*) (mm³/g of initial anhydrous cement) is calculated as follows:

203
$$CS(t,T) = \frac{MS(t) - MS(t_i)}{MC.\rho_W(T)}$$
 Eq. (1)

where MS(t) is the apparent mass of both the vial and the sample as recorded by the balance at time *t* (*g*); $MS(t_i)$ is the apparent mass of both the vial and the sample at initial time $t_i(g)$; $\rho_w(T)$ is the water density (*g/mm³*); *T* is the water bath temperature (°C); *MC* is initial anhydrous cement mass of the sample (g).

208 **2.3.4** Compressive and flexural strength of mortar

The compressive and flexural strength tests were conducted according to EN 196-1 [29] at 7, 28 and 90 days using $40 \times 40 \times 160$ mm³ cubes. Three flexural strength tests and six compressive strength tests were conducted for each mix, where the average and the standard deviation were reported.

212 **2.3.5** Mercury intrusion porosimetry of mortar

In order to investigate the pore network of the mortars, a classical mercury intrusion porosimetry (MIP) technique was used. It is based on the premise that when a nonwetting fluid (one having a contact angle greater than 90°: mercury) is forced into the pores of the sample by increasing pressure, the higher the pressure, the smaller the pore that the fluid can enter. The relationship between the pressure and capillary diameter is described by Washburn's equation as follows:

218
$$d = \frac{-4\gamma\sin\phi}{p}$$
 Eq. (2)

219 Where *d* is the size of an equivalent cylindrical pore that requires a given pressure *p* to be filled, γ is 220 the surface tension of the mercury, and \emptyset is the contact angle between the mercury and the pore wall. 221 The values for γ and \emptyset are assumed to be 0.485 N/m and 130°, respectively.

Mercury porosimetry is an extremely useful characterization technique for porous materials [36]. It is a widely recognized technique for characterizing the distribution of pore sizes in cement-based materials [37].

MIP tests are carried out on specimens with an apparent volume of about 3 cm³, cut from the core of the mortar cube after 28 and 90 days of curing. In order to make the small specimens devoid of moisture, they are dried in an oven at 40°C until the mass stabilized. The dried specimens are kept in the oven until the start of the test. The pressure range used was from sub-ambient up to 375 MPa, covering a pore diameter ranges from about 200 µm to 3 nm.

230 2.3.6 Dynamic Young's modulus, dynamic shear modulus of mortar

231 Dynamic Young's modulus and dynamic shear modulus of the mortar specimens are measured using a Grindosonic apparatus following ASTM E 1876-01[38] and NF EN ISO 12680-1[39]. Tests are 232 performed on prismatic specimens with standard dimensions of $40 \times 40 \times 160$ mm³. The test is based 233 on exciting vibrations in the mortar prism by striking them with a small tapping device. A vibration 234 235 sensor detects the flexural or torsional vibrations, which are then analyzed by the Grindosonic device. The resonant frequency is manually recorded from data acquisition related to excitation system. 236 237 Elastic dynamic modulus is determined from the density and resonant frequency of the mortar. For each mixture, dynamic Young's and shear moduli tests are performed on three specimens and the 238 239 average is reported. Specimen mass is recorded for each specimen as well.

240

2.3.7 Water porosity of mortars

Water porosity testing consists of measuring the percentage of voids that are connected to the surface of hardened mortar and accessible by water. Tests are carried out using the French standard NF P 18-459 [40], modified by adding new steps consisting of half immersing the sample in water for 24 h under vacuum pressure to ensure complete saturation of the specimens after total immersion. Water porosity tests are carried out on three prismatic specimens at 7, 28 and 90 days. Samples are first put under the vacuum pressures until reaching a pressure of 25 milibar. This vacuum is maintained for 4 h.
After that, the specimens are half submerged in water for 24 h under pressure followed by total
immersion in water for 48 h to suck water into the small pores. The porosity is obtained by weighing
the specimen in water and saturated surface-dried. The same specimen is dried in an oven at 105 °C
until constant mass. The water porosity is calculated as follows:

251
$$n = \frac{M_{air} - M_{dry}}{M_{air} - M_w}$$
 Eq. (3)

Here M_{air} is mass of the mortar specimen at the saturated surface-dried state, M_{dry} is the mass of mortar specimen at oven dried state, and M_w is the mass of mortar specimen saturated in water.

3. Results and discussions

Fig.3. shows the flows of mortars made with different amounts of glass powder. It can be seen from this figure that there is no variation in the flow of mortar made with 10% and 25% replacement of cement by glass powder compared to the reference mortar in both vibrated or non-vibrated form (0 hits). However, a slight decrease in the flow is seen with mortar made with 35% and 60% replacement in vibrated form contrary to non-vibrated form, which remains the same as the reference. The increase of glass powder replacement from 35% to 60% decreases the flow value.





Fig. 3. Flowability of mortar made with different amounts of glass powder.

A previous study by Sadiqul Islam et al. [41], shows that a minor increase in flow mortar is achieved 264 with an increase of glass powder replacement (10%, 15%, 20%, and 25%); they reported that this 265 266 increase might result from the effect of glass material, which is cleaner in nature. According to Taha et al. [42], when using glass powder with particles size below 45 µm, the slump value of concrete is not 267 affected by 20% replacement. Schwarz et al. [43] evaluated the workability of plain cement and 268 cement replaced with glass powder at rates of 10%, 20%, and 30% by mass. Results indicate that 269 270 increasing glass powder content leads to increased flow due to the nonabsorbent nature of glass 271 increasing free water content. Aliabdo et al. [21] found that the use of glass powder as cement 272 replacement (5%, 10%, 15%, 20%, and 25%) increases concrete slump. This behaviour may be due to 273 the glassy surface and low water absorption of glass powder or may be attributed to the coarser 274 particles of glass powder compared with cement. In contrast, the same authors [21] reported that the 275 use of glass powder as cement addition (5%, 10%, 15%, 20%, and 25%), which increase the binder 276 amount (cement and glass powder) compared to the amount of binder in the case of using glass 277 powder as cement replacement, decreases concrete slump. This trend may be due to the increase of 278 fine material content which increases the cohesion of concrete mix and thus decreases the concrete 279 slump. Lu et al. [44] studied the effect of 20% replacement of cement by GP with different particles 280 sizes (204 µm, 88.5 µm, 47.9 µm, 28.3 µm and 18.8µm). Results indicate that bigger particle size and 281 irregular shape of glass particles hinder the movement of fresh mortar and result in a reduction of 282 flow. Liu et al. [28] prepared mortar with a low water-to-binder ratio (0.25) and the same dosage of 283 super plasticizer, using increasing content of glass powder as cement replacement (10%, 30% and 284 60%). They reported that the flowability of mortar increases with increasing glass content. However, 285 when preparing the same mixtures with a high water-to-binder ratio (0.5) and without super 286 plasticizer, results show no significant change on the flowability or even a slight decrease with high 287 volume recycled waste glass content.

Based on the literature and our results, there are contradictory results about the influence of GP as cement replacement on the flowability of cementitious materials. This can be explained by counteracting effects of several parameters such as the shapes of particles, their size, the nonabsorbent nature of glass, the smooth surface of glass particles, the influence of particle wettability and the amount of cement replacement. Irregularly shaped particles with high aspect ratios and sharp edges decrease fluidity. However, due to the grinding effect, the shape of the particles is more favorable to fluidity. The non-absorbent nature of glass particles increases the effective water to cement (w/c) ratio and enhances the flowability. Small particles need more water to wet their surface. Smooth surfaces promote particle movement; this increases fluidity. The rate of replacement is an important parameter since the increased amount of glass leads to an increase of water in the mix. Moreover, there is not much cement paste in the mix, which can decrease the particle's friction and increase the fluidity.

299

3.2. Heat of hydration assessment

The heat rate and the total achieved heat of cement pastes containing GP under a constant temperature of 20 °C over 96 h, normalized by the amount of binder and the reference cement paste are shown in Fig. 4. Their peaks' age and intensity hydration value are presented in Tab. 4. The relative values of the principal heat rate peaks of 10 GR GP, 25 GR GP, 35 GR GP and 60 GR GP pastes to the principal heat rate peak value of reference cement paste 0 SCM were found to be 0.903, 0.774, 0.692, 0.455 respectively.



- 307 Fig. 4. Hydration heat assessment of glass powder blended cement pastes, normalized by the amount
- 308

of binder	(cement	and g	lass	powder).
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	Age of hydration peaks		Intensity of hydration peaks	
Mixture	Measured Value	Value relative to	Measured Value	Value relative to
	(hours)	reference paste (%)	(hours)	reference paste (%)
0 SCM	10.37	100	4.42	100
10 GP GR	10.34	99.7	3.99	90.3
25 GP GR	10.22	98.6	3.42	77.4
35 GP GR	9.91	95.6	3.05	69.2

6	0 GP GR	8.65	83.4	2.01	45.5
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Table 4 Characteristic values of isothermal calorimeter tests.

The relative values indicate that the replacement of cement by GR GP caused a reduction in the heat rate. This result is in accordance with previous literature [28, 44]; they reported that the rates of heat evolution of the waste GP modified pastes are lower than that of the pure cement pastes. This demonstrates that the waste GP has a lower reactivity at the early stages of hydration. It is important to note that the heat rate of paste with 60% replacement by GP is less than the half that of the reference.

315 The principal peaks of hydration for cement pastes containing glass powder occur earlier than that of 316 cement paste without glass powder, 0 SCM. The relative times of the apparition of the peaks heat rates 317 of 10 GR GP, 25 GR GP, 35 GR GP and 60 GR GP pastes to the time of the peak heat rate for the 318 reference cement paste 0 SCM are found to be 0.997, 0.986, 0.958, 0.834. This indicate that the time 319 to reach the first heat flow peak decreases with the increase of glass powder content. This acceleration of the hydration process of cement can be explained by the increase of the effective water to cement 320 321 (w/c) ratio due to the replacement of cement by glass powder and the high alkali content of glass 322 powder. Huang et al. [45], reported that the alkali in cement accelerates the hydration at early ages. High alkali addition of cement shortens the induction period and promotes the dissolution of C₃S and 323 324 the formation of Ca(OH)₂. According to Du et al. [27], fine glass powder can accelerate hydration via 325 the adsorption of calcium ions from the liquid phase and acts as nucleation and growth sites for C-S-H 326 and other hydrates. Moreover, the higher content of alkalis (Na₂O) may act as catalysts in the 327 formation and growth of C-S-H at an early age [46, 47].

When calorimetry results are expressed as the heat evolution normalized to the cement component as shown in Fig. 5, we can see that the replacement of recycled waste glass increases the reaction rate for all paste made with glass powder except the 10 GP GR paste, which remains the same. The reaction of the cement component is enhanced due to the physical presence of glass powder. This so-called filler effect is attributed to two main factors: the available space for the hydrates of the clinker phases to form in and the surfaces grains, which act as sites for the heterogeneous precipitation and growth of hydrates [48].



Fig. 5. Hydration heat assessment of glass powder blended cement pastes, normalized to the cement
 component.

338 **3.3.** Chemical shrinkage

Fig. 6 shows chemical shrinkage test results over 48 h, normalized by cement mass, of the control 339 paste and pastes modified with 10%, 25%, 35% and 60% glass powder. It is clear from this figure that 340 341 chemical shrinkage, normalized by cement mass, for all cement paste samples increases throughout the hydration process. Chemical shrinkage is related to the volume change during the time. The volume of 342 the resulting hydrates during the hydration process is lower than that of the initial reactants (cement, 343 glass powder and water) [34]. It is also seen from Fig. 6 that increased glass powder in cement paste 344 345 increases the normalized chemical shrinkage, especially with 60% cement replacement with GP, where cement contains high amounts of C₃A and glass powder has high alkali content (Na₂O). 346 According to Yodsudjai. [49], the chemical shrinkage of paste increases with the C₃A and C₄AF 347 348 content and equivalent alkali content. Moreover, increasing glass powder content causes more 349 available water for the hydration of cement particles, which is expected to increase chemical 350 shrinkage.





352

Fig. 6. Evolution of chemical shrinkage versus time for cement pastes with different glass

powder contents (w/b = 0.4).

354 3.4. Compressive and flexural strength:

355 Fig.7 and Fig.8 show the compressive and flexural strength variation of cement mortar samples with

and without glass powder after 7, 28 and 90 days of moist curing.





358

Fig. 7. Effect of GP replacement ratio on the compressive strength of mortar at 7, 28 and 90

days.



361 362

Fig. 8. Effect of GP replacement ratio on the flexural strength of mortar at 7, 28 and 90 days.

363 From these figures, it is clear that the compressive and flexural strength of cement mortar specimens 364 increase with age. Increasing the glass powder amount as cement replacement decreases the 365 compressive strength at all curing periods in this study. In general, flexural strength follows the same 366 trend as compressive strength, low difference is noticed for all mortars made with glass powder 367 compared to the reference. Higher reduction in compressive strength is seen at an early age than at the 368 latest age, which is in accordance with previous findings [25]. The lowest value is obtained at 60% GP 369 GR. A substantial difference is observed between the control mix 0 SCM and the 60 GP GR mix at 7 370 days. The reduction in the strength values at the early age can be explained by the reduction of the amount of clinker compounds responsible for strength development at early age. The compressive 371 372 strength hardly changes throughout the 90-days period for mortar with 60% of glass powder. This can be explained by the long-term activity of pozzolanic reactions. With longer curing, the amorphous 373 silica in glass powder slowly dissolves under the alkaline environment and reacts to form Ca(OH)₂ in 374 375 the pore solution C-S-H gels [5, 25].

At 90 days, the compressive strength is very similar for mortar with 25% and 35% glass powder substitution. This may be due to the pouzzonanic activity of the additional glass powder (10% by weigh of cement) from 25% to 35% that still shows pozzolanic activity. Enough late strength is provided to compensate for the lower early strength. These results can be emphasized by the slow rate of glass powder pozzolanic reaction since the compressive strength at 7 days is greater for mortar with
25% cement substitution by glass powder that in mortar with 35% replacement.

382 Using a water-to-binder ratio of 0.487, Du et al. [25] studied the effect of compressive strength on concrete made with GP as cement replacement at 15%, 30%, 45 %, and 60% by weight of the total 383 cementitious materials. The mean diameter of the glass particles is 3.4 µm. Results indicate that with a 384 longer curing age (28 and 90 days), the compressive strength continuously increases with GP 385 386 replacement level up to 45%, and the concrete mixture with 60% GP exhibits a comparable strength to the reference mixture. Two main factors could explain the contradictory results found on concrete 387 388 compared to the current results for mortar compressive strength: the interfacial transition zone between 389 cement paste and aggregate on concrete, which is more densified using glass powder, and the particle size used, that is 3.4 μ m compared to the 17.68 μ m mean diameter in the present work. 390

391

3.5. Mercury intrusion porosimetry

In this section, the pore structure results of mortar made with cement substituted with glass powder at 0%, 10%, 25%, 35%, and 60% after 28 and 90 days' curing are discussed. The cumulative intrusion versus pore diameter curves and differential curves are shown in Fig. 9.A, Fig. 9.B, Fig. 9.C, Fig. 9.D and Fig. 9.E.





Fig. 9.A. Cumulative and differential pore size distribution of mortar made with 10% glass powder at

398

28 and 90 days.





400 Fig. 9.B. Cumulative and differential pore size distribution of mortar made with 25% glass powder at

28 and 90 days.



402

403 Fig. 9.C. Cumulative and differential pore size distribution of mortar made with 35% glass powder at

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28 and 90 days.



405



28 and 90 days.





409 Fig. 9.E. Cumulative pore size distribution of mortar made without and with different glass powder
410 content at 90 days.

We can see from these figures that the pore size distribution is affected by the glass powder rate substitution. The mortar containing glass powder shows higher porosity than the reference at 28 and 90 days. The highest value is obtained with 60% replacement. This finding supports the compressive strength results, which are low.

The main peak intensity increases with 10% and 25% replacement of cement by glass powder at 28 415 416 and 90 days. Mortar with 35% replacement of cement by glass powder shows the same peak intensity as reference mortar at 28 days. In contrast, at 90 days, the main peak intensity decreases compared to 417 418 the reference. The same trend is shown with 60% replacement of cement by glass powder. The main 419 peaks denote remarkable decreases compared to the reference whether at 28 or 90 days. The main peak is also shifted towards bigger pores at 28 days. At 90 days it moves towards smaller pores; at the same 420 time new peaks with small intensities reflecting bigger pores are created. This can be explained by the 421 422 dilution effect of cement, which leads to much more available space reflecting that higher porosity and the hydration process and pozzolanic reaction that lead to the densification and the refinement of the 423 424 microstructure. Results of the same nature were found by Liu et al. [28] while studying the microstructure of normal strength sample mortar containing 60% mixed colored waste glass. They 425 426 associated the higher porosity to the lower gel-to-space ratio, which is caused by the higher water-to-427 binder ratio.

Another important parameter that should be taken into consideration to explain the porosity increase,
is the increased volume of the binder (cement + glass powder) for mortar made with glass powder
compared to the reference mortar due to the lower bulk density of glass powder (2.5 g/cm³) compared
to cement (3.16 g/cm³).

432 From 28 to 90 days, we can see that the porosity reduces and the microstructure becomes more 433 refined. An increase in the volume of mercury intruded at higher pressure is seen for all mortar made 434 with glass powder compared to the reference whether at 28 or 90 days, indicating more refined 435 microstructures. This can be explained by pozzolanic reactions occurring between glass powder and 436 calcium hydroxide to form more C-S-H gel leading to the refinement and densification of the mortar's 437 pore structure. These results are in accordance with previous MIP research done by Du et al. [5]. They studied the effect of glass powder on cement paste and mortar samples collected from the fractured 438 439 surfaces of concrete, free of large aggregate particles. Portland cement is replaced at different contents: 15%, 30%, 45%, and 60% by weight. Their results indicate that glass powder pastes have 440 more refined pores. Results indicate that the total porosity only marginally varies, and the pore size 441 442 distribution is more refined, with increasing GP content.

The cumulative intrusion-versus-pore diameter curves for 25 GP GR and 35 GP GR are very close
especially at higher pressures. In addition, total porosity is the same at the same age, 28 and 90 days.
This is consistent with compressive strength results and water porosity results.

	Porosity (%)		Critical pore diameter (nm	
Mixture				
	28 Days	90 Days	28 Days	90 Days
0 SCM	11.22	10.74	39.99	41.75
10 GP GR	12.04	11.67	36.91	34.21
25 GP GR	13.60	13.08	39.44	37.79
35 GP GR	13.63	13.26	42.30	43.84
60 GP GR	15.96	15.58	62.98	37.84

446 **Table 5** Critical pore size and total porosity of mortar made with different glass powder content.

Critical pore size corresponding to the steepest slope of the cumulative intrusion curves and total 447 porosity corresponding to the maximum volume intruded are shown in Tab. 5 for 28 and 90 days. At 448 449 28 days, the critical pore size remains constant for mortar with glass powder or changes slightly compared to the reference, except for the 60 GP GR where the critical pore size increases due to the 450 dilution effect. From 28 to 90 days, the critical pore size marginally changes for all mortars made with 451 glass powder except 60 GP GR where the critical pore size significantly decreases. As said before, the 452 453 dilution effect leads to bigger pores with 60 GP GR. However, pozzolanic reactions that occur between 28 and 90 days lead to much more refinement of the mortar pore structure. According to Du 454 et al. [5], concrete made with 60% replacement of cement by glass powder shows higher volume 455 fraction of pores in the range of 0.1 µm to 10 µm at 91 days due to insufficient calcium hydroxide 456 reacting with glass powder to further fill the pores. 457

458

3.6. Dynamic Young's and shear moduli of mortar

459 Dynamic Young's and shear moduli are determined using impulse excitation by vibration. Results 460 shown in Fig.10 and Fig.11 indicate that Dynamic Young's and shear moduli increase with age from 461 the time of demolding until 12 days. After that age, i.e. from 13 days to 90 days, all mortar seems to 462 show stable values of dynamic Young's and shear moduli or indicate very small increases.

Fig.10 shows also that increasing GP content in mortar reduces the dynamic Young's modulus. A
significant decrease is seen for 60 GP GR mortar. The same trend is shown in Fig.11 for dynamic
shear modulus.

466



468 Fig. 10. Dynamic Young's modulus of mortar made with different rates of glass from the time of



470

demolding to 90 days.



471 Fig. 11. Dynamic shear modulus of mortar made with different rates of glass powder from the time of472 demolding to 90 days.

473 Fig. 12 shows the weight loss of different mortars from the time of demolding to 90 days.

Results from Fig.12 indicate that weight loss increases with increasing glass powder content. Very
slight variation in weight loss can be seen for mortar made with 10% glass powder compared to the
control mortar. A considerable part of the weight loss occurs before seven days for all mortar studied.
After 27 days very slight variation can be seen on mass loss for the different mortars, which is due to

478 variation in humidity curing conditions.



- 479
- 480
- 481

Fig. 12. Mass loss of mortar made with different content of glass powder from the time of demolding to 90 days.

When comparing compressive strength to dynamic Young's and shear moduli results, we note that compressive strength increases from 28 days to 90 days but that the dynamic Young's and shear moduli show very little variation. This can be explained by the probability of microcracks

development on mortar structure, that slows down the increase in the dynamic Young's and shear 485 moduli with time. The microcracks existence is most likely due to the water lowering caused by 486 487 evaporation at early age and by consumption during the hydration process including pozzolanic reaction that lead to microstructure refinement. The evaporation of free water from pore capillaries 488 leads to shrinkage due to capillary tension. This turn leads to the apparition of microcracks [50]. As 489 shown by mercury intrusion porosimetry, the hydration process from 28 to 90 days led to the 490 491 refinement of mortar structures. According to Baroghel-Bouny [51], the hydration processes induces a liquid water content decrease in cementitious materials while the microstructure is hardening and the 492 493 pore structure is refining. It is well known that capillary tension is inversely proportional to pore 494 radius. Hence, the probability of microcracks appearing is higher with increasing glass powder content because the increased content leads to the refinement of the pores by pozzolanic reactions. 495

496

3.7. Water porosity

Fig.13 shows the evolution of the pores accessible to water for mortar samples after 7, 28 and 90 days 497 of curing in water at 20 °C. 498

499 It can be clearly seen from this figure that the control mortar is less porous than the mortars containing 500 glass powder.

501 The water-accessible porosity of a cementitious material is influenced by its constituents, the hydration process that leads to the formation and change of the microstructure, and testing conditions 502 503 (vacuum pressure and drying at 105 $^{\circ}$ C). The replacement of cement by glass powder increases the water porosity due to the dilution effect. There are less cement and glass particles and much more 504 water in the matrix. The drying effect causes water departure and makes the mortar more porous. 505 Moreover, glass powder has a lower specific gravity than cement and the replacement by mass lead to 506 507 much more volume of paste (water + cement + glass) in the mortar.



Fig. 13. Water accessible porosity of mortar with different glass powder contents at 7, 28 and 90 days. We can see from Fig. 13 that at 7 and 28 days with 10% cement replacement by glass powder, the porosity remains stable compared to the reference mortar at the same curing ages. By increasing the rate of cement replacement with glass powder, the water porosity increases for all curing periods, reaching the highest value at 60% replacement.

The water porosity doesn't show great change between 7 and 28 days except the mortar with 60% replacement. During the hydration process between 7 and 28 days, the hydrates fill parts of the voids caused by the dilution effect, especially for 60 GP GR, and the porosity decreases slightly.

518 During water porosity testing, samples are exposed to vacuum pressures to evacuate void space. As

said previously, the evaporation of free water can lead to microcracking, especially with high glasscontent.

521 Between 28 days and 90 days, all mortars with glass powder show slight increases in water porosity. 522 This can be explained by the pozzolanic reaction, which occurs; leading to an increase in C-S-H gel in 523 the matrix and the refinement of the microstructure. The change occurring in water porosity is an 524 effect of several parameters: filling the void, caused by the dilution effect with newly formed hydrates, 525 pozzolanic reactions that lead to the refinements of the microstructure and the test conditions (vacuum 526 pressure and heating at 105°C).

527 When comparing water porosity and mercury porosity values for the same mixture and the same 528 hydration's age, it is clear that water porosity is higher than mercury porosity. It is well known that 529 mercury porosity qualifies restricted range of the pores structure (from 200 μ m to 3 nm in our case), 530 however water can cover large game of porosity than mercury. Moreover, mercury intrusion porosity 531 is determined on sample dried at 40°C and water porosity is calculated based on sample dried at 532 105°C.

533 **4.** Conclusions

Waste glass powder from green glass bottles has been investigated for use as cement replacement on cement paste and mortar. The main conclusions that can be taken out from the present work are the following:

-The flow of mortars made with glass powder is influenced by several counteracting effects:

- The nonabsorbent nature of glass particles that increase the effective water to cement (w/c)
 ratio and enhances the flowability.
- The irregular shape of particles with high aspect ratio and sharp edges, which decrease the fluidity.
- The particle sizes where the shape of finer particles increases fluidity but more water is needed
 to wet their surface.
- The smooth surface of glass promotes particle movements and increases fluidity.
- The decrease of the cement paste in the mix arising from high-level replacement increases
 particle friction and decreases fluidity.

-For a water-to-binder ratio (W/B) of 0.5, increased cement replacement by glass powder decreases the compressive strength of mortar at all curing periods in this study (7, 28 and 90 days). This reduction is mainly due to the dilution effect and the non-absorbent nature of glass powder, which increases the effective water-to-binder ratio. The long-term pozzolanic reactions using glass powder is proven through the change in compressive strength from 7 to 90 days especially for mortar made with 60% of glass powder.

-It is evidenced by heat evolution normalized relative to the cement component and chemicalshrinkage that increasing glass powder content in cement paste increases the reaction rate. The rate of

hydration is increased by the physical presence of glass powder. The time to reach the first heat flowpeak decreases with an increase of glass content.

-The porosity is reduced and the microstructure became more refined from 28 to 90 days for all mortars. The pore size distribution is affected by the glass powder substitution rate. The mortar containing glass powder shows higher porosity than the reference at 28 and 90 days. The highest value is obtained with 60% replacement. This finding is in accordance with the low compressive strength and high water porosity.

-High rate of replacement of cement by glass powder leads to bigger pores due to the dilution effect. In contrast, the pozzolanic reaction that occurs between glass powder and calcium hydroxide leads to an increase in C-S-H gel, which leads to the refinement and densification of the pore structure of mortar made with glass powder.

-The change occurring in water porosity is an effect of several parameters: filling the void, caused by
the dilution effect with newly formed hydrates, pozzolanic reactions that lead to the refinements of the
microstructure and the test conditions (vacuum pressure and heating at 105°C).

569 -The Dynamic Young's modulus increases with age from the time of demolding to 12 days. Between 13 and 90 days, the Young's modulus for all mortars seems to be stable or shows a very small 570 increase. This can be explained by the probability of microcracks development on mortar structure, 571 572 that slows down the increase in the dynamic Young's and shear moduli with time. The microcracks 573 existence is most likely due to the water lowering caused by evaporation at early age and by 574 consumption during the hydration process including pozzolanic reaction that lead to microstructure 575 refinement. The increase of glass content in mortar reduces dynamic Young's modulus. A significant 576 decrease is seen for 60 GP GR. The same trends are shown for dynamic shear modulus.

We can see from this study that the presence of high glass content in cementitious materials enhances the rate of hydration. This is evidenced by chemical shrinkage and heat assessment. Due to the dilution effect and the nonabsorbent nature of glass, high glass content mortar increases mass loss and porosity. However, pozzolanic reactions increase the refinement of the pore structure. Results also show a significant decrease in the dynamic Young's and shear moduli of mortar made with high glass content. Taking into account economic consideration of using waste glass and the characteristics studied in the present paper, we conclude that 35% cement replacement by green glass powder, can be considered as the optimum for mortar uses. The use of high glass powder content as cement replacement is possible for a water-to-binder ratio of 0.5. It is important to note that further studies related to durability characteristics are needed especially for high glass content on using such mortar as construction materials. It is also recommended to reduce the water-to-binder ratio, which is expected to enhance the durability characteristics of cementitious materials made with green glass powder.

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