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1	Bubble stabilisation improves strength of lightweight mortars
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18	
19	Abstract
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21	Lightweight foamed mortars are produced through the addition of foaming agents into the
22	cement blend, so that voids of different sizes are formed within the matrix, reducing the density

of the material, and therefore also its weight. However, the increased porosity of these materials 23 24 usually compromises their mechanical strength, limiting application as a structural material. Modern infrastructure demands high strength lightweight concrete formulations that can be 25 26 adjusted to develop more ambitious projects, both in design and application. In this study lightweight pastes and mortars were produced using Portland cement blended with fly ash and 27 silica fume, with varying water contents, and foamed using aluminium metal powder. To 28 stabilise the bubbles produced through oxidation of the aluminium metal, polyethylene glycol 29 30 was added to the mixes, and proved effective in yielding more uniform bubbles than were observed in the samples with no added stabiliser. This led to improvements in both the bulk 31 density and compressive strength of the materials produced according to this new methodology. 32

34 Keywords: Lightweight construction materials, cement-based foams, aluminium metal,

35 polyethylene glycol

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1. Introduction

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The use of lightweight concretes in structural or semi-structural applications has long been the target of technological developments, with efforts involving the reduction of unit weight of both the aggregate and the binder having been undertaken for more than 100 years (Valore, 1954). The availability of lightweight concrete as a construction material is particularly topical at present, as the combination of good mechanical and thermal insulation properties offers significant improvements in the energy efficiency of buildings in service without excessive increases in the thickness of the building envelope.

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The reaction of metallic aluminium with the alkaline pore solution of the cement, releasing 48 49 hydrogen gas and aerating the binder, has long been favoured as a method of reducing the density of a cementitious binder (Aylsworth and Dyer, 1914), and this method has been shown 50 51 to be applicable to a wide range of binder compositions, ranging from the original work of Aylsworth and Dyer with Portland cement and gypsum (Aylsworth and Dyer, 1914), to the 52 more recently developed foamed geopolymer systems (Zhang et al., 2014). However, the 53 production of foamed concretes with sufficient strength to enable their use in structural 54 55 concrete applications is less straightforward. This material has thus been used largely in applications where value is gained from its insulating properties (thermal and acoustic), and/or 56 57 the reduction of mass which can lead to lower dead load within a structure and greater ease of handling during construction (ACI Committee 233, 2000). 58

59

Recent work on the development of lightweight Portland cement concretes for use as structural 60 materials has shown success in producing 28-day compressive strengths greater than 28 MPa, 61 either by the use of a surfactant as foaming agent in combination with coarse fly ash (replacing 62 fine aggregate) and a small quantity of polypropylene fibres (Jones and McCarthy, 2005), or 63 by using a surfactant and silica fume-blended cement in concretes with standard dense fine and 64 65 coarse aggregates (Lee et al., 2014). In each of these cases, the high strength was provided through the use of a good-quality modern Portland cement combined with appropriately 66 selected supplementary cementitious materials, curing at ambient or near-ambient temperature. 67

In particular, the types of binder formulation which would otherwise be used to produce highperformance dense concretes, including the use of microsilica as a pozzolan, are also likely to give the best strength development when used in lightweight materials, and so provide the most likely targets for optimisation of the strength-density relationship of foamed concretes.

72

73 Elevated-temperature curing is used to form autoclaved aerated concrete, which is often 74 foamed using aluminium metal powder (releasing hydrogen gas as it oxidises under an alkaline environment) (Aroni, 1993), but this material is generally specified only up to strength grades 75 76 less than 10 MPa (Klingner, 2008). Higher-strength materials have been demonstrated through the combination of aluminium metal powder with a microsilica-rich Portland cement blend 77 (Just and Middendorf, 2009). It has generally been considered more difficult to achieve the 78 desired stable, highly uniform, small bubble size required for high strength through the use of 79 metallic foaming agents than by the application of a pre-formed organic foam based on 80 surfactants, but the additional processing steps involved in the pre-foaming route pose 81 disadvantages related to that method. It is therefore considered desirable to develop alternative 82 routes to the production of foamed concrete which are more similar, in terms of the required 83 processing steps, to the methods by which standard dense concretes are produced and placed, 84 85 while yielding materials with strengths that are sufficiently high for structural or semistructural application. This necessitates careful control of the fresh-state properties of the paste, 86 87 particularly flow characteristics, as a foamed paste which requires a high mechanical energy input in pumping and placement is likely to suffer deaeration as a result. 88

89

This study approaches the question of production of moderate-strength, moderate-density 90 91 foamed mortars through an innovative method of bubble stabilisation in a material system 92 foamed by the addition of aluminium metal. A binder is designed based on Portland cement 93 with microsilica and fly ash, with the bubbles generated through hydrolysis of aluminium metal stabilised by the addition of polyethylene glycol as a surface-active agent. This stabilisation 94 mechanism enables the retention of small bubbles within the fresh paste until hardening, 95 resulting in a flowable material which yields a desirable microstructure and good strength 96 97 performance, suitable for future scale-up to use in concretes.

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- 100 **2.** Materials and Methods
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- 102 **2.1.Materials**
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Portland cement of grade CEM I 52.5 N, with a bulk density of 1506 kg/m³, was used 104 throughout this study. A commercial low calcium fly ash, classified as 'siliceous' according to 105 EN 197-1 (European Committee for Standardization (CEN), 2011) and complying with the 106 requirements of EN 450-1 (European Committee for Standardization (CEN), 2012), and silica 107 fume complying with EN 13263-1 (European Committee for Standardization (CEN), 2005), 108 were used as supplementary cementitious materials. Aluminium powder, general purpose 109 110 grade, was used as the foaming agent. As bubble stabiliser, polyethylene glycol (PEG) with an average molecular weight of 20 kDa was used. Quartz building sand, with a particle density of 111 2650 kg/m³ and with 100% passing 2.40 mm, was the fine aggregate in all mortars. 112

- 113
- 114 **2.2. Sample preparation**
- 115

In order to select and optimise the amount of foaming agent and water content, preliminary experiments were carried out using paste mixes. Pastes were produced with water to cement ratios of 0.35, 0.40 and 0.45 to span the range from very stiff to very fluid pastes, and aluminium powder contents of 0, 0.3, 0.6 and 0.9 wt.% relative to the cementitious materials in the paste, according to the process depicted in Figure 1.

- 121
- 122 <Figure 1>

123

The workability of the paste mixes was tested according to a mini-slump radius measurement as described in (Bouvet et al., 2010). Following the mixing protocol as described in Figure 1, the paste was poured into a PTFE cone of 40 mm height, 80 mm lower diameter and 70 mm upper diameter, resting on a Lucite sheet marked with a grid, and compacted by rodding with a spatula. After 1 minute, the cone was lifted clear from the paste, and the diameter of the pat was measured using calipers, than converted to radius values for presentation.

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The paste specimens for analysis were cast in centrifuge tubes, sealed and cured at 21±2°C.
After 24h, bulk density was determined through a measurement of the volume and mass of
cylindrical samples, in an as-cured condition immediately upon demoulding of the specimens.

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Photographs of the longitudinal sections of paste specimens after 28 days of curing were
collecting using a Veho USB optical microscope with a maximum magnification of 20×.

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Based on the results of the study of the paste specimens, the optimal foaming agent and stabiliser doses were selected, and mortar specimens were produced (Figure 2) using a Kenwood mixer. Mortar formulations are given in Table 1, where the addition of fly ash was intended to reduce density and enhance workability, and the addition of silica fume intended to improve early-age strength development. Density was determined through the precise weighing and dimensional measurement of 50 mm cubic specimens. Compressive strength was also determined using 50 mm cubic specimens, in triplicate.

145

146 <Figure 2>

147

148 Table 1. Mix designs for mortars; all quantities in grams.

Sand	Cement	Fly ash	Silica fume	Water	Aluminium	PEG
250	100	0	0	40	0.90	10
250	90	10	0	40	0.90	10
250	97	0	3	40	0.90	10
250	87	10	3	40	0.90	10
250	93	0	7	40	0.90	10
250	83	10	7	40	0.90	10

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152 **3. Results and Discussion**

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3.1. Pastes produced with aluminium powder as foaming agent

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156 The minislump and density results for the paste mixes with different contents of aluminium

157 powder, and different water/cement ratios, are presented in Figures 3 and 4.

159 <Figure 3>

160

161 <Figure 4>

162

It is notable from Figures 3 and 4 that no direct correlation between the workability of the fresh 163 paste and the density of the hardened solids is identifiable, whereas it is well known that air 164 entrainment in concretes can often give an improvement in workability (Lamond and Pielert, 165 166 2006), as the lower density of the aerated pastes can also lead to a reduction in the slump measurement. It is not likely that rodding for 1 minute with a spatula was sufficient to remove 167 the all of the generated gas bubbles from the pastes, and in fact the bubbles remaining in the 168 mix were visible after the minislump tests. All pastes tested were able to demonstrate sufficient 169 170 workability for use in the preparation of mortars.

171

However, it is also clear from the photographs of cross-sections of the pastes, Figure 5, that the networks of bubbles within these materials are not homogeneously distributed, and that the bubbles are neither spherical nor uniform in size. In optimising the strength of a foamed cement-based material, it is considered important to develop a uniform bubble size distribution (Nambiar and Ramamurthy, 2007).

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For this reason, the addition of a surface-active component to stabilise the bubbles was considered desirable; polyethylene glycol was selected for this purpose, as it is water-soluble but a rather weak surfactant (Israelachvili, 1997) (which is desirable to avoid excessive foaming and loss of strength in the hardened materials), retains some stability under the high pH conditions of a fresh cement paste, and showed good performance in preliminary trials of foaming of silicate slurries.

184

185 <Figure 5>

186

By comparison with Figure 5, it is seen from the photographs in Figure 6 that the PEG was effective in stabilising the bubbles; the bubbles are much more spherical, and significantly smaller, than those which are present in the hardened paste in the absence of PEG.

190

191 *<*Figure 6*>*

The results presented in Table 2 show that including 10 wt.% PEG does not modify the 193 workability of the paste but is able to significantly reduce the density of the binder, with 194 approximately a 33% reduction in the bulk density of the hardened paste compared to the paste 195 with Al powder alone; the moulding procedure has clearly removed many of the bubbles 196 generated by the Al in the absence of a stabiliser, whereas the PEG is effective in retaining the 197 bubbles in the paste as it is poured into the mould and progressively hardens during the early 198 stages of curing. Therefore, tests of mortar mixes were carried out using this combination of 199 200 foaming agent and bubble stabiliser.

201

Table 2. Mini-slump and bulk density of foamed pastes with 0.9 wt.% Al powder in the presence of
 PEG as a bubble-stabilising agent

water/cement ratio	Content of PEG (wt.% of cement)	Minislump radius (mm)	Bulk density (g/cm ³)
0.40	0	66.7	1.26
0.40	10	67.8	0.85

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3.2 Mortars produced with Al as foaming agent and PEG as bubble stabiliser

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208 Based on the paste density reduction achievable through the coupled use of Al and PEG to generate and stabilise the bubbles, the next parameter tested was the binder mix design. The 209 210 water/cementitious materials ratio was fixed at 0.40, and fly ash and silica fume were added to 211 the mix in different proportions to determine the influence of each of these components on the 212 bulk density and compressive strength of the hardened mortars. The mortar densities in Fig. 7 are significantly higher than the paste densities in Table 2 due to the use of a regular (dense) 213 quartz fine aggregate in the mortar mixes, at a mass ratio of 2.5 to the cementitious component 214 (Table 1). The addition of fly ash consistently reduces the density of the mortar mixes, whereas 215 silica fume densifies the mixes (Fig. 7A,B). However, fly ash addition reduces the compressive 216 strength at both 7 and 28 days (Fig. 7C,D), and so the additional strength generated through 217 silica fume incorporation was beneficial in enabling the materials to approach the range of 218 strength values which would be required for semi-structural applications, up to 25 MPa for the 219 strongest samples tested here. The positive effect of silica fume on compressive strength is 220 221 more visible in samples without FA, consistent with the fact that the mortars have been dosed

replacing OPC by FA, and therefore, it is expected reduced compressive strength at early timesof curing as the replacement of OPC by FA increases.

224

225 <Figure 7>

226

To clarify the influence of the mix design on key physical properties of the mortars, Figure 8 227 shows the relationship between bulk density and compressive strength, for the mixes listed in 228 Table 1 as well as some additional mixes developed with higher and lower contents of fly ash 229 230 during the preliminary mix design process, all at the same water/cementitious materials ratio of 0.40. The relationship between density and compressive strength displays an increasing 231 trend, as expected based on the extensive literature for foamed concretes produced by various 232 mechanical and chemical foaming methods, and as described by a number of mathematical 233 models, where power-law, logarithmic and linear relationships have variously been proposed 234 for different types of foamed concrete (Kearsley and Wainwright, 2002). Considering the error 235 bars shown in Figure 7, the data presented here could be described by any of these types of 236 237 mathematical relationship with approximately the same degree of precision, and so it is not possible to conclusively state which is the most accurate relationship for this set of mortars 238 239 foamed according to the new methodology presented here. Nonetheless, there is a clear relationship which can be observed by comparison of Figures 7 and 8, whereby the addition of 240 241 silica fume yields a lower compressive strength and a higher bulk density; this curve may be used to design materials with the desired combination of density and mechanical performance, 242 243 through adjustment of the blend of cementitious materials at this particular dose of foaming 244 agent and stabiliser.

245

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248 Conclusions

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This study has presented a methodology by which bubbles can be formed in cementitious pastes through the inclusion of metallic aluminium, and then stabilised by addition of polyethylene glycol. This polymer, although considered a weak surfactant, does display sufficient surface activity to prevent the breakdown of the bubbles and the escape of the entrained gases, while the material is cast and begins to harden and develop strength. This methodology therefore provides a pathway by which an aluminium-foamed cementitious material can be stabilised at

^{246 &}lt;Figure 8>

256	a lower density than would otherwise be possible, while retaining desirable strength
257	characteristics sufficient for semi-structural applications. A mortar compressive strength of 25
258	MPa at 28 days can be achieved by this method for a material with a density of 1.90 g/cm^3 .
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262	Acknowledgements
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265	
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B: Cross-section





