

Low energy pre-blended mortars: Part 2 – Production and characterisation of mortars using a novel lime drying technique

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HUGHES, D.C., ILLINGWORTH, J.M. and STARINIERI, Vincenzo (2015). Low energy pre-blended mortars: Part 2 – Production and characterisation of mortars using a novel lime drying technique. *Construction and Building Materials*, 101 (1), 710-720.

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25 characteristics. It is shown that optimally dried mortars are not subject to degradation
26 following storage of both mortar types.

27

28 **Keywords:** mortar, sand drying, ggbs, slaked lime, storage, Roman cement

29

30 **1 Introduction**

31

32 The first part of this paper [1] described a technique by which the addition of quicklime to
33 wet sand could be controlled to produce Formulated Lime mortars which are dry and
34 suitable for long-term storage in silos or bags prior to final wet mortar production. The
35 principal process-control factors to achieve optimal drying have been identified as lime
36 addition based upon a fraction of the stoichiometric requirement for complete slaking of
37 the quicklime (i.e. 3.113 g of CaO being required to combine with 1 g water), free
38 moisture content of the sand and mixing time of the combined sand and quicklime blend
39 followed by a 24 hour “rest period”. The philosophy can be equally applied to other
40 factory produced mortars in which free water may remain following the formulation
41 process.

42

43 A fundamental feature of this concept is that water which is not contained within the pore
44 structure of the sand, i.e. free water, rather than the total water content is the one which
45 must be controlled. In this scenario the datum state of the aggregate is its saturated
46 surface dry (SSD) condition in which the sand pores are completely filled whilst the free
47 moisture is zero. Trials of mortars produced with natural hydraulic lime (NHL 3.5)
48 showed that mortars in which the binder was mixed with SSD sand and stored for 8

49 weeks in a sealed box exhibited no degradation when compared to companion mortars
50 which were immediately produced without the storage period.

51

52 This part of the paper describes the application of the technique in the production of two
53 types of mortar. The first are Formulated Lime mortars, M1 and M5, produced with the
54 addition of ggbs as the hydraulic phase. Formulated Limes are specified in BS EN 459-
55 1:2010 [2] as being limes with hydraulic properties and comprising lime and added
56 material with hydraulic and/or pozzolanic properties whilst the strength classes M1 and
57 M5 (compressive strengths of 1 MPa and 5 MPa respectively) are specified in BS EN
58 998-2:2010 [3]. The mix design process has to ensure that the quicklime addition is
59 sufficient to yield both the dry mortar for storage and also the correct amount of ggbs,
60 principally the ggbs/slaked lime (CH) ratio, to yield the desired mortar type whilst
61 maintaining the required volumetric proportions of binder to aggregate. The properties of
62 mortars made immediately after addition of the hydraulic component are compared to
63 those in which the dry blended mortar was stored for 10 weeks prior to mortar
64 production. The second application is in the production of De-Activated Roman Cement
65 (DARC) mortars suitable for long-term storage. A characteristic of Roman cements is
66 their rapid setting such that retardation is required for the production of practical mortars.
67 A workable life of 1 – 2 hours has been specified for render mortars [4] for which a pre-
68 hydration process has been developed [5]. In essence, this process involves adding an
69 initial controlled amount of water to the Roman cement such that sufficient reaction takes
70 place to retard the setting of the mortar when the fresh mortar is subsequently
71 manufactured. During that study it was observed that DARC mortars stored for 1 – 12
72 weeks exhibited some degradation during storage, thought to be due to the presence of
73 residual water following the de-activation process. Thus, the situation is akin to that of
74 the presence of excess water in the Formulated Lime mortars which needs to be

75 removed, although in this case the water results from a factory-based process rather
76 than from as-delivered materials. Mortars with the addition of quicklime following the de-
77 activation stage are compared with samples which were oven-dried at this point as well
78 as control samples.

79

80

Part A: Formulated Lime Mortars

81

2 Materials and Methods

83

2.1 Materials

85

86 A siliceous sharp sand was used throughout and was oven dried to constant weight at
87 110°C prior to use. Two different quicklimes, C1 and E, were used for the preparation of
88 the lime-dried mortars. The chemical and physical properties of these materials, together
89 with properties of the slaked limes, were reported in Part I of this study [1]; Table 1
90 summarises the key properties of the slaked limes and a commercial CL90 which has
91 been used as a control. ggbs was supplied by Civil & Marine Slag Cement from the
92 Frodingham plant, UK.

93

Lime	C1	E	CL90
Free lime (% CaO)	70.9	68.7	71.3
Insolubles (wt%)	5.8	4.8	3.8
CaO (wt%)	0.3	0	0.9
Ca(OH) ₂ (wt%)	93.2	90.8	93.0
CaCO ₃ (wt%)	2.9	6.9	2.3
A _{BET} (m ² /g)	15.19	18.16	11.26
V _{TOT} (cm ³ /g)	0.098	0.109	0.072
Bulk Density (kg/m ³)	585	559	575

Table 1: Properties of limes

94

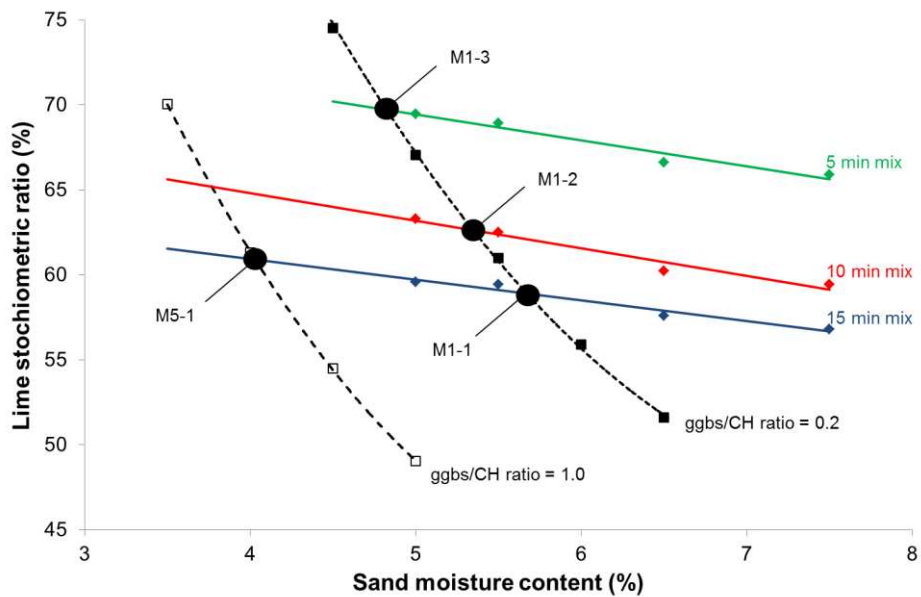
95

96 2.2 Mortar Production

97

98 2.2.1 Mix design of lime-dried mortars

99



100

101

Fig 1: Example of mix design for optimum mortars using lime E.

102 Figure 5 of ref [1] shows that the optimum stoichiometric ratio for quicklime addition to
103 dry the sand decreases with increases in either the sand moisture content or the mixing
104 time of the wet sand and quicklime. This is represented for lime E in Figure 1 by the 3
105 linear relationships. The design of the lime-dried mortar formulations was intended to
106 produce mortars of M1 and M5 classification according to BS EN 998-2 [3]. The
107 approximate ggbs/CH ratio (by weight) required to achieve the stated classification was
108 0.2 and 1.0 respectively where the mortars were produced to a common flow of 170 mm
109 [6]. Obviously, the precise ratio required to yield a specified strength for any mortar will
110 also be related to the purity of the quicklime, grading of the sand as it affects workability,
111 the sand density and the density of the Ca(OH)_2 (also referred to by its cement chemistry
112 notation of CH) generated during the drying process as they affect volumetric mix
113 proportions. Once a sand:binder ratio has been selected it is possible to calculate how
114 much Ca(OH)_2 is required for a specified ggbs/CH ratio. Hence, knowing how much
115 quicklime would be required to chemically combine with all of the free water for a given
116 free moisture content of the sand, the stoichiometric ratio of quicklime addition may be
117 calculated which generates the correct amount of Ca(OH)_2 for the specified ggbs/CH
118 ratio, after accounting for the purity of the quicklime. For instance, for a particular mortar
119 batch size and mix proportions, if 1381 g of Ca(OH)_2 is required to yield the desired
120 ggbs/CH (equivalent to 1045 g of CaO), whilst theoretically requiring 1771 g of CaO to
121 chemically combine with the free water in the sand, this would represent a stoichiometric
122 ratio of 59%. In this procedure the density of the ggbs and the CH have been measured
123 but no allowance was made of any interactions between the two phases which might
124 affect their packing efficiency.

125

126 The associated values of stoichiometric ratio and sand free moisture content for both
127 ggbs/CH ratios have been superimposed on Figure 1 and the intersection of the

128 relationships indicates the optimum drying conditions for each mortar; the 4 mortars
 129 using quicklime E detailed in Table 2 are indicated on Figure 1. The same approach was
 130 adopted for mortars using quicklime C1 In accordance with the terminology previously
 131 described [1] all mortars are considered optimally dried. All mortars were produced at a
 132 constant sand:binder ratio of 2.25:1 by volume.

Code	Lime type	Stoich ratio (%)	Sand m/c (%)	Mix time (min)	ggbfs/CH ratio	w/b ratio
M1-1	E	58.87	5.69	15	0.2	1.35
M1-2	E	62.62	5.35	10	0.2	1.36
M1-3	E	69.72	4.81	5	0.2	1.36
M1-4	C1	61.20	5.71	15	0.2	1.37
M1-5	CL90	~	~	~	0.2	1.41
M5-1	E	60.9	4.03	15	1	1.03
M5-4	C1	61.2	4.08	15	1	1.05
M5-5	CL90		~	~	1	1.06

133 Table 2: Mix design

134
 135 The experimental programme was designed to determine the effect of various
 136 parameters of the lime-drying process on the physical properties of the resulting
 137 hardened mortars. The formulations allow assessment of the influence of the drying
 138 process, the drying time and the quicklime source; mortars using CL90 provide control
 139 data.

140

141 **2.2.2 Preparation of lime-dried mortars**

142

143 Ten kg batches of sand were prepared containing known amounts of free water (wt.%
144 basis). The sand was then mixed in a Hobart mixer with appropriate quantities of
145 quicklime for the specified period of time (i.e. 5, 10 or 15 min). Following mixing, the
146 lime-dried materials were stored in sealed containers for 24 hours to ensure complete
147 hydration of the quicklime; this step was included after previous observations indicated
148 the presence of free water in the blend at the completion of the initial mixing [1]. The
149 required quantity of ggbs was then added in order to provide the desired mix
150 proportions. At this stage, the dry mortar mix was divided into two equal portions. One
151 portion was stored in a sealed plastic container and retained under laboratory conditions
152 (20°C, 50% RH) for 10 weeks; the other portion of the mix was prepared immediately.
153 These mortars have been termed “stored” and “fresh” respectively.

154

155 **2.2.3 Production of plastic mortars**

156

157 Lime dried mortars were produced by adding mix water to the “dry mortar” whereas the
158 control mortars were produced by adding the water to a blend of CL90 and SSD sand.
159 All plastic mortars were produced in a Hobart mixer with a mixing time of 15 minutes.
160 Appropriate quantities of water were added to give a flow table value [7] of 170mm (+/-
161 5mm). This value was considered to provide materials with workability consistent with
162 that observed in typical construction practice [8]. The required free water:binder ratio to
163 achieve the specified flow value for each mortar is displayed in Table 2.

164

165 **2.2.4 Curing of mortars**

166

167 Following preparation, the plastic mortars were cast into steel moulds of the required
168 geometries (see section 2.3). The samples were initially cured within the moulds for 3

169 days at 20°C and covered by a polythene sheet to minimise evaporation. The moulds
170 were then stripped and the samples cured for a further 4 days under the same
171 conditions. Unless stated otherwise, extended curing was then carried out at 20°C (\pm
172 2°C) and 65% RH (\pm 5%), hereafter referred to as air curing. The level of CO₂ in the
173 curing room was assumed to be the standard atmospheric concentration (0.033 \pm
174 0.001% by volume). For comparison, some samples (for strength testing only) were
175 subject to extended curing under water, also at 20°C.

176

177 **2.3 Analytical Methodology**

178

179 **2.3.1 Compressive and Flexural Strength**

180

181 The test specimens were prepared in steel moulds of 40 x 40 x 160 mm dimensions.
182 Mortar was placed in 2 layers and compacted using a vibration table. The three-point
183 flexural and compressive strengths of the hardened mortars were determined using an
184 Instron 4206. Crosshead speeds of 0.5 mm/min and 1 mm/min were used for flexural
185 and compressive testing respectively. Measurement of compressive strength was
186 carried out at 7, 28, 91 and 365 days. For air-cured samples, flexural strengths were
187 determined at 7 and 91 days whereas water-cured materials were tested at 28 and 91
188 days.

189

190 **2.3.2 Density and Porosity**

191

192 The bulk density and total open porosity of the hardened mortars were assessed in
193 accordance with BS EN 1015-10 [9] and BS EN 1936 [10] respectively (vacuum
194 saturation method). The reported values are the mean of three individual samples.

195 **2.3.3 Sorptivity**

196

197 The water sorptivity of the hardened mortars was determined according to the
198 methodology described in detail by Hall [11]. The test specimens were prepared in 70
199 mm steel cubes and cured as described in section 2.2.4. Prior to testing, the samples
200 were conditioned at 60°C to constant weight in an atmosphere circulated over silica gel
201 and soda lime. The sorptivity was measured through the moulded bottom surface; the
202 vertical faces were sealed by the application of water-resistant tape. The reported values
203 represent the mean of three individual samples.

204

205 **2.3.4 Water Vapour Permeability**

206

207 The water vapour permeability of the hardened mortars was determined using a
208 modified version of the methodology described in BS EN 1015-19 [12]. The test
209 specimens were cast in circular steel moulds, producing specimens of 180 mm diameter
210 and 20 mm thickness. The mortar discs were then subjected to the curing regime
211 specified in section 2.2.4. After curing, the samples were prepared for analysis at 60°C in
212 an atmosphere circulated over silica gel and soda lime. The samples were then placed
213 in stainless steel test cups containing a saturated solution of potassium nitrate and
214 sealed in-situ with molten paraffin wax. The air gap between the base of the samples
215 and the solution was 12 mm (± 2 mm). The KNO_3 solution generates a relative humidity
216 within the air gap of 93.2% at 20°C. The samples were then placed in a fan-assisted
217 storage chamber maintained at 20°C ($\pm 2^\circ\text{C}$) and 50% RH ($\pm 5\%$) and weighed every 24
218 hours until steady state vapour transmission was achieved. The reported values
219 represent the mean of three individual samples.

220

221 **2.3.5 Mercury Intrusion Porosimetry (MIP)**

222

223 The pore size distributions of the mortars were determined using MIP at an age of 91
224 days (air cured). The measurements were carried out with a Micromeritics 9400 series
225 instrument over a range of pressures between 3.9×10^{-3} and 410 MPa. Assuming a
226 contact angle of 140° and a mercury surface tension of 484×10^{-3} N/m, pore diameters
227 ranging from 375 μm to 3.8×10^{-3} μm were characterized. Specimens with an approximate
228 volume of 2 cm^3 were cut from the mortar prisms using a low speed saw. Prior to
229 analysis, the samples were dried to constant weight at 60°C in an atmosphere circulated
230 over silica gel and soda lime.

231

232 **3. Properties of “fresh” mortars**

233

234 **3.1 Pore structure**

235

236 Values of the total open porosity and dry bulk density are shown in Table 3. The general
237 trend observed for all mortars is a slight decrease in porosity, allied with an increase in
238 bulk density, as the curing time was extended, and is more marked in the M1 mortars.
239 The M5 series of mortars show higher density and lower porosity than the M1 series, as
240 would be expected from the higher ggbs/CH ratio. The difference is more marked in
241 mortars produced using either quicklime C1 or the CL90.

Mortar	Open Porosity (%)			Dry Bulk Density (kg/m ³)		
	28 d	91 d	365 d	28 d	91 d	365 d
M1-1	32.2	31.6	31.0	1757	1781	1791
	<i>32.7</i>	<i>31.8</i>	<i>31.4</i>	<i>1689</i>	<i>1717</i>	<i>1729</i>
M1-2	31.8	31.4	31.0	1769	1794	1799
	<i>32.9</i>	<i>31.7</i>	<i>31.5</i>	<i>1684</i>	<i>1719</i>	<i>1735</i>
M1-3	32.1	31.4	30.8	1761	1795	1797
	<i>32.8</i>	<i>31.9</i>	<i>31.4</i>	<i>1689</i>	<i>1714</i>	<i>1737</i>
M1-4	33.5	32.8	32.3	1720	1749	1767
	<i>34.2</i>	<i>33.1</i>	<i>32.6</i>	<i>1660</i>	<i>1700</i>	<i>1709</i>
M1-5	34.9	34.2	33.7	1693	1686	1701
	<i>35.1</i>	<i>34.2</i>	<i>33.6</i>	<i>1651</i>	<i>1679</i>	<i>1700</i>
M5-1	29.3	29.3	29.1	1767	1805	1805
	<i>29.9</i>	<i>28.5</i>	<i>27.8</i>	<i>1733</i>	<i>1746</i>	<i>1779</i>
M5-4	29.4	29.4	29.1	1755	1777	1784
	<i>30.1</i>	<i>29.1</i>	<i>27.7</i>	<i>1729</i>	<i>1741</i>	<i>1775</i>
M5-5	29.5	29.8	29.3	1745	1759	1767
	<i>30.6</i>	<i>29.0</i>	<i>28.2</i>	<i>1721</i>	<i>1733</i>	<i>1766</i>

242 Table 3: Porosity and density of mortars at ages of 28, 91 and 365 days (air curing in
243 Normal font, water curing in Italics).

244

245 Within the M1 range of mortars M1-1, M1-2 and M1-3 possess the same porosity at a
246 given age and curing condition; all these mortars were dried with the same quicklime E,
247 the only difference being in the process parameters of stoichiometric ratio and mixing
248 time. The influence of lime type may be seen in the higher porosities of M1-4 (quicklime

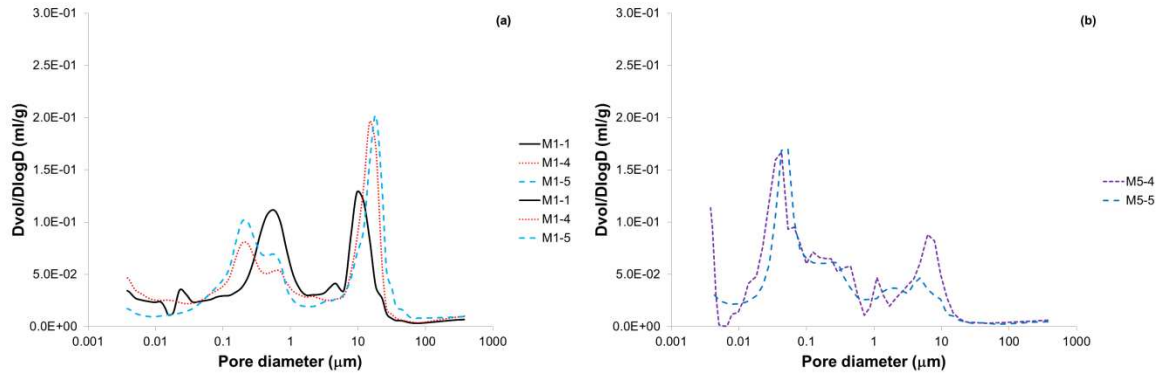
249 C1) and M1-5 (CL90). However, any influence of lime type in the M5 mortars (M5-1, M5-
250 4 and M5-5) is reduced to a minimum, probably as a function of the reduced lime content
251 of these mortars.

252

253 The influence of curing regime is small for the M1 mortars although water curing yields
254 the slightly higher porosity at all ages. In contrast, water-cured M5 show higher porosity
255 at 28 days but lower porosity at 365 days, presumably reflecting additional hydraulic
256 activity at later ages.

257

258 The pore size distributions of the mortars (91 days of air curing) are shown in Figure 2.
259 The data highlight significant differences in the pore size distributions between the M1
260 (Fig 2a) and M5 (Fig 2b) mortar series. The lime-rich M1 series contain a large
261 proportion of pores in 2 distinct zones, i.e. 10 – 18 μm and 0.2 – 0.6 μm . Mortars M1-4
262 and M1-5 exhibit similar pore structures; both mortars are based upon lime from the
263 same source. Mortar M1-1 shows a slightly lower threshold pore diameter and a lower
264 volume of the coarsest pores which accompanies the lowest open porosity (Table 3). In
265 contrast, for the M5 mortars, the bulk of the porosity is made up of pores in the region of
266 0.05 μm diameter. This is a function of the higher ggbs/CH ratio and lower w/b ratio of
267 the M5 series. As with the M1 mortars, the pore distributions of M5 mortars using
268 quicklime C1 and CL90 are similar.



269 Fig 2: Pore size distribution of (a) M1 and (b) M5 mortars after 91 days of air-curing.

270

271 A similar pattern of small differences in open porosity accompanied by marked
 272 differences in pore size distribution has been previously reported by Arandigoyen and
 273 Alvarez [13] for cement-lime mortars.

274

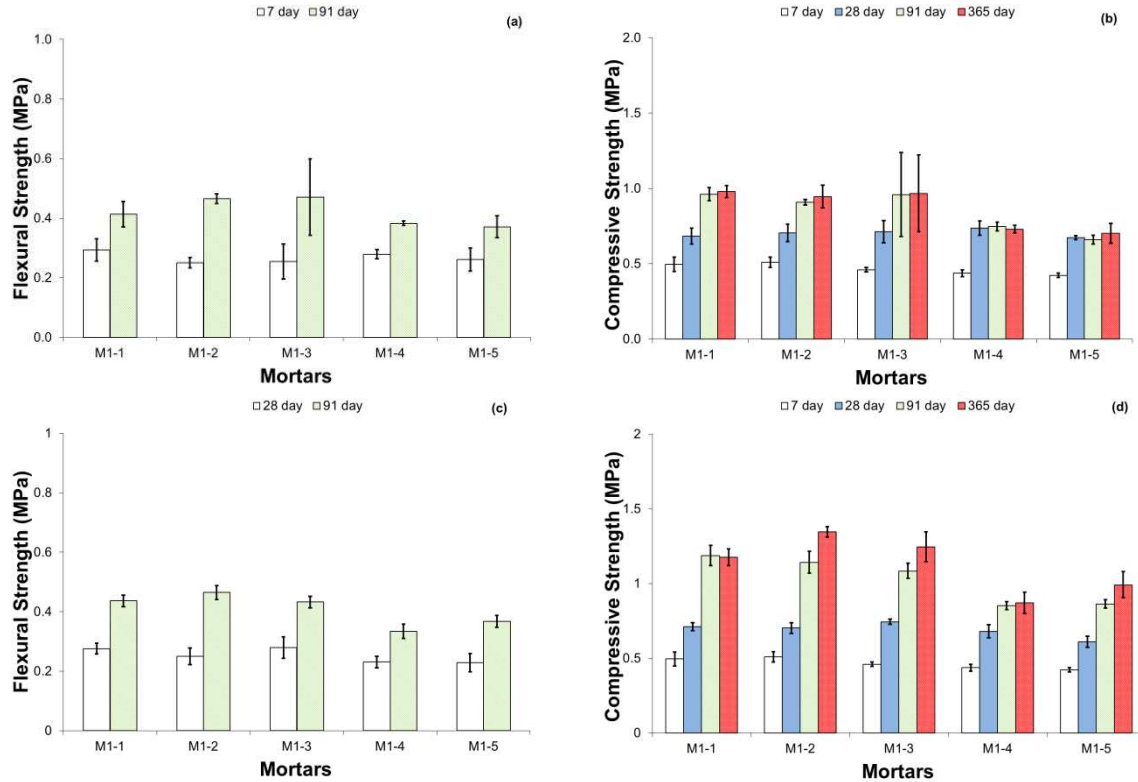
275 3.2 Compressive and flexural strength

276

277 3.2.1 M1 mortar formulations

278

279 The data in Figures 3a and 3b show the evolution of flexural and compressive strength
 280 of the M1 mortars subjected to air curing. Under flexural load, mixes M1-1 to M1-5 yield
 281 similar strengths at 7 days. All mortars show a considerable and similar gain in flexural
 282 strength up to 91 days (all significance testing used the student t-test conducted at 95%
 283 confidence). Under compression, mortars M1-1 to M1-5 display essentially similar initial
 284 strength development up to 28 days. Thereafter, mortars M1-1, M1-2 and M1-3 show a
 285 further strength increase up to 91 days with no further change between 91 and 365
 286 days. In contrast, mortars M1-4 and M1-5 show no gain in compressive strength after 28
 287 days and, consequently, these materials are significantly weaker at the later ages.



288 Fig 3: Strengths of M1 mortars – (a) flexural strength air curing, (b) compressive
 289 strength air curing, (c) flexural strength water curing, (d) compressive strength water
 290 curing. Error bars represent ± 1 Standard Deviation.

291

292 Figures 3c and 3d display the influence of water curing on the strength development of
 293 the mortars M1-1 to M1-5. In flexure, mortars M1-1, M1-2 and M1-3 exhibit similar
 294 strengths at 91 days with mortars M1-4 and M1-5 also being similar but weaker than the
 295 other mortars. With the exception of M1-4, the strength of all mortars is similar at 91
 296 days for both curing conditions. Up to an age of 28 days, all mortars show similar
 297 compressive strengths and there is little difference in the compressive strengths of
 298 samples subjected to the different curing conditions. However, at ages of 91 and 365
 299 days water curing generally generates higher strength (typically 20 – 40%) reflecting the
 300 extra potential for hydraulic reactions in the lime-ggbs system. The exception is mortar
 301 M1-3 which exhibited a high variability of strength under air curing at these ages, so

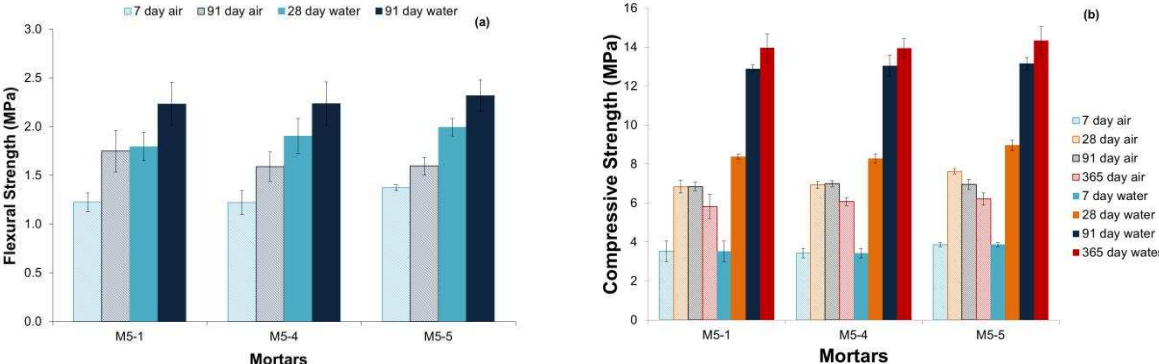
302 influencing the statistical comparison; it is not believed that the apparent difference
303 exhibited by this mortar formulation is genuine.

304

305 **3.2.2 M5 mortar formulations**

306

307 The data in Figures 4a and 4b show the evolution of flexural and compressive strength
308 of the M5 mortars. There is little influence of lime type in the M5 mortars. As expected
309 the highest strengths are registered by water cured samples such that by an age of 1
310 year a strength enhancement in the range 130 – 140% is observed. Whilst the latter
311 curing yields continuous strength increase up to an age of 1 year, air cured samples
312 indicate a maximum strength at either 28 or 91 days with a subsequent reduction at an
313 age of 1 year; this was not observed in the M1 mortars.



314 Fig 4: Strengths of M5 mortars – (a) flexural strength, (b) compressive strength. Error
315 bars represent ±1 Standard Deviation.

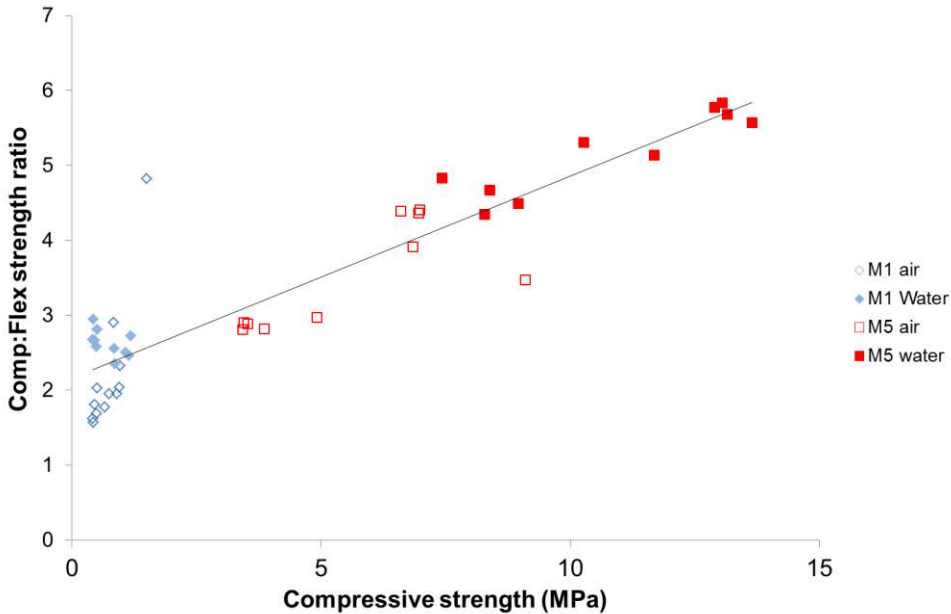
316

317 The influence of curing regime on long term development of alkali-activated slag
318 concrete (sodium silicate and calcium hydroxide activation) [14] and of calcium
319 hydroxide activated slag mortars [15] has been previously observed. The strength
320 reduction was explained by the high shrinkage and micro-cracking associated with this
321 binder system. It may be observed that the absence of strength reduction is seen in the

322 lime-rich M1 mortars and, whilst any correlation is purely speculative, the strength
323 reduction in the M5 and calcium hydroxide activated slag mortars [15] occurs in mortars
324 with a larger hydraulic component and less lime.

325

326 Both compressive and flexural strengths have been measured for all mortars and curing
327 conditions at 2 ages. The ratio of compressive:flexural strength as a function of
328 compressive strength is shown in Figure 5. As expected the ratio increases as the
329 compressive strength increases reflecting a more brittle material. There is a certain
330 degree of scatter, particularly at lower strengths; a correlation coefficient of 0.872 is
331 achieved.



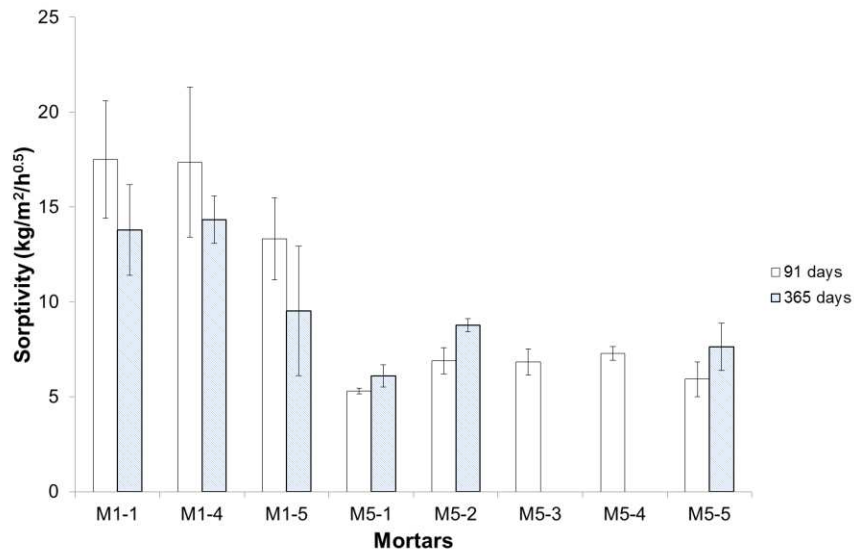
339 **3.3 Moisture transport**

340

341 The specification of mortars is dependent upon their end-use. In the case of restoration
342 works on historic buildings their breathability is an important property. However, this is a
343 topic with differing philosophical approaches adopted by different restorers. One
344 approach, as codified in BS EN 998-1:2010 [16], may be summarized as one in which
345 the movement of water is to be minimised where-as the movement of water vapour is to
346 be encouraged. This approach frequently requires the use of hydrophobic treatments
347 within the mortar system. Another viewpoint is that this approach only transfers the
348 original problems to other areas of the structure which have not been treated with such a
349 mortar [e.g. 17, 18]. It is argued that since water transport is much quicker than vapour
350 transport both parameters are important.

351

352 In the current work, breathability was assessed by sorptivity and water vapour
353 permeability (WVP) measurements following air curing and the results are presented in
354 Figures 6 and 7 respectively.



355

356

Fig 6: Sorptivity of M1 and M5 mortars.

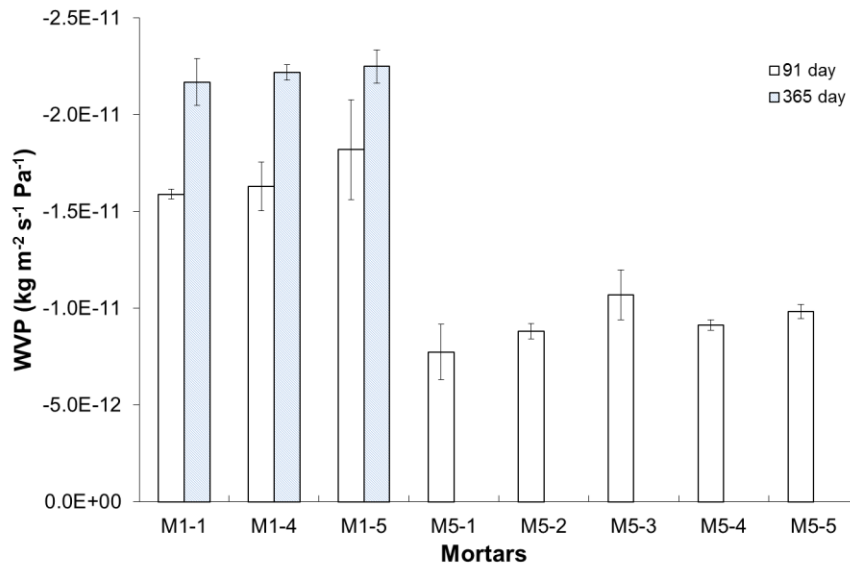


Fig 7: WVP of M1 and M5 mortars.

357

358

359

360 It is apparent that the sorptivity and WVP of the M5 mortars is less than that of the M1
 361 mortars reflecting their finer pore structure. Although the average values suggest that all
 362 mortars show a variation in sorptivity with age, the variability within each data set is such
 363 that there is no statistical difference between mortars. By way of contrast, the WVP of
 364 the M1 mortars generally increases with age although that of the M1-5 mortar only
 365 marginally fails the significance test. There is no influence of lime source on the
 366 sorptivity or WVP of the M1 mortars at ages either 91 or 365 days unlike that observed
 367 for compressive strength, nor on the sorptivity or WVP of the M5 mortars at 91 days
 368 which is the only test age with data for all of this group of mortars.

369

370 Both M1 and M5 mortars exceed a commonly accepted value of 4 Kg/m²/hr^{0.5} for the
 371 sorptivity, or Water Absorption Coefficient, for restoration mortars for renders [4].
 372 Obviously, neither mortar type complies with the low water absorption criterion of BS EN
 373 998-1 although the M1 mortars do meet the requirement for WVP ($\mu > 15$).

374

375 The lower “permeability” of the M5 mortars is reflected in their reduced rate of
376 carbonation as measured by depths of complete carbonation using the phenolphthalein
377 test (Table 4).

Mortar	28d	91d	365d
M1	2 - 3	9 - 10	Complete
M5	1 - 3	6 - 7	13 - 15

378

Table 4: Carbonation depths (mm) of M1 and M5 mortars

379

380 **3.4 Discussion**

381

382 An influence of lime type has been observed in strength performance but not in moisture
383 transport. It is possible that this may be a function of the greater variability within the
384 latter determinations and had a larger number of samples been tested at each stage a
385 significant difference might have been obtained. The following discussion will focus on
386 the strength data.

387

388 The similar performance of the M1-1, M1-2 and M1-3 mortars is to be expected as the
389 materials are of essentially identical composition with the key parameters of lime source
390 and ggbs/CH & w/b ratios being common. The data provides confirmation that
391 manipulation of the lime drying process (quicklime stoichiometric ratio and mixing time)
392 allows the production of identical mortars from quarried aggregates containing different
393 amounts of free water. Likewise, the performance of mortars M1-4 and M1-5 may be
394 similarly explained; despite M1-4 being a lime-dried mortar and M1-5 comprising a
395 commercial CL90, the source of quicklime in both cases is the same.

396

397 It was previously shown that lime E yielded higher long term strengths than lime C1 in
398 the M1 mortars but not in the M5 mortars (sections 3.2.1 and 3.2.2). The differences in
399 BET surface area (Table 1) do not account for the different performances; however, the
400 greater strength of the mortars produced with lime E may reflect the higher CaCO_3
401 content of the slaked lime. In this case, the lime-rich M1 mortars contain a larger quantity
402 of CaCO_3 than do the M5 mortars. Studies of the use of CaCO_3 as an addition to
403 Portland cement [19] show that fine CaCO_3 can be both a reactive component as well as
404 a filler. Additional hydration within the AF_m phases is observed at low carbonate
405 additions and is accompanied by reduced porosity. A study of the inclusion of metakaolin
406 and limestone in Portland cement mortars has shown that the carbonate reacts with the
407 metakaolin to produce AF_m phases [20]. The identification of AF_m in alkali-activated
408 cements is uncertain and may be depend upon detection technique and the particular
409 alkali utilized [e.g. 21, 22]. Further, whilst the addition of 1-3% fine calcite has been
410 found to yield a small increase in the early age strength of an alkali-activated cement, no
411 or even a negative impact occurred at later ages [19]. Little modern research has been
412 undertaken on the lime-ggbs system and certainly not in the low slag contents currently
413 being considered and further fundamental research is required to examine the role of
414 CaCO_3 within this system.

415

416 In contrast to the M1 mortars, the strength development of M5-1, M5-4 and M5-5
417 mortars appears unaffected by the different limes used in their manufacture. The
418 reasons for this difference are unclear but the discrepancy indicates that the properties
419 of the lime are of greater significance in mortars with higher lime content. It seems
420 reasonable to assume that at low ggbs/lime ratios, the development of the mortar
421 microstructure would be more significantly influenced by changes in lime properties than

422 in mortars where the lime content is much reduced. In cement-lime-sand mortar
423 systems, Sebaibi *et al.* [24] stated that 'it is necessary to have a high lime substitution
424 percentage to influence the microstructure of the mortar'; however, the PC/lime ratios
425 used in their mortar formulations were far higher than the ggbs/lime ratios employed in
426 the current work.

427

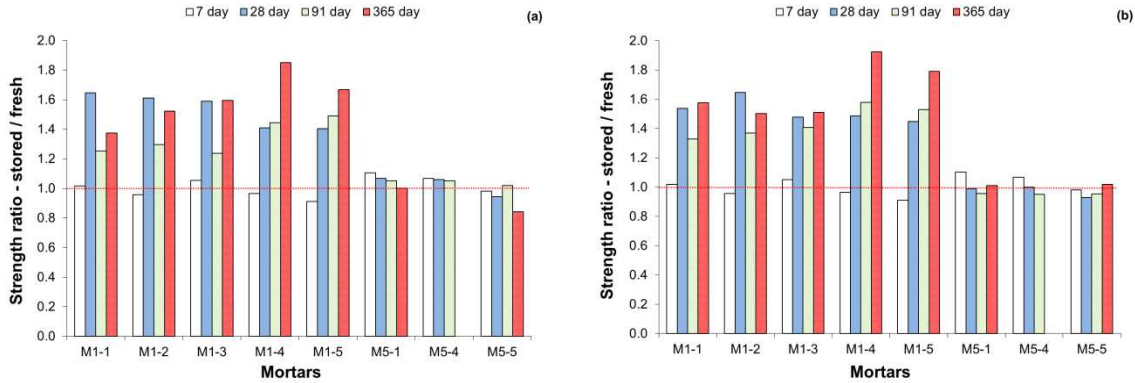
428 **4 The effect of storage on lime-dried mortar mixes**

429

430 **4.1 Optimally dried mortars**

431

432 The data shows that the performance of lime-dried mortars is very similar to those
433 produced using commercial CL90 hydrated lime. However, one of the essential
434 requirements of the lime-drying process is the ability to produce mortars that can be
435 stored in silos or bags without significant degradation prior to use. The lime-drying
436 process is unlikely to remove water absorbed within the pores of the aggregate unless
437 excessive heat is generated during the slaking process. As a pre-cursor to work on lime-
438 dried mortars a range of mortars was produced using NHL 3.5 as the binder phase in
439 which the sand was pre-prepared at various moisture states between oven dry and
440 saturated surface dry. They were blended in the volume ratio 2.25:1 and stored for 8
441 weeks prior to mixing into mortars of common flow. There was no statistical difference
442 (95% confidence limits) between mortar strengths at ages up to 2 years indicating no
443 influence of absorbed water on mortar performance. Hence, a series of lime-dried
444 mortars was produced following a 10-week storage (see section 2.2.2) in which the
445 expectation was that any influence of storage would be a function of free water rather
446 than absorbed water.



447 Fig 8: Relative strengths of stored and fresh mortars; (a) air cured, (b) water cured.

448

449 Figure 8 shows the relative compressive strength of the M1 and M5 series of “stored”
 450 mortars (both water and air-cured) in comparison to the “freshly” prepared materials
 451 described in section 3.2. It is apparent that there is a difference in performance between
 452 the M1 and M5 mortars. The M5 “stored” mortars are generally similar to their “fresh”
 453 counterparts at all ages except 365 days where the air-cured “stored” mortars lose
 454 strength at a more rapid rate than the “fresh” mortars (see Fig 8a). At 365 days the
 455 “stored” mortars are some 77% of the 91 day strengths in contrast to 87% for the “fresh”
 456 mortars. In contrast, the strengths of the M1 “stored” mortars are considerably greater
 457 than those of the fresh materials at ages beyond 7 days under both air and water curing.
 458 The strength increases are accompanied by an increase in bulk density and a decrease
 459 in the porosity in the stored mixes (Table 5).

Mortar	Open porosity (%)		Density (kg/m ³)	
	Fresh	Stored	Fresh	Stored
M1-1	31.6	30.6	1781	1809
M1-2	31.4	30.6	1794	1808
M1-3	31.4	30.2	1795	1818
M1-4	32.8	30.9	1749	1803
M1-5	34.2	32.6	1686	1766

Table 5: Influence of storage on M1 mortars

460

461

462 Only a very limited study was undertaken on M1 mortars (M1-1, M1-4 & M1-5) to
 463 examine the influence of storage upon the moisture transport properties at an age of 91
 464 days curing in air. Whilst strength was affected by storage there is no significant
 465 influence on moisture transport.

466

467 The precise reason for the observed differences between the M5 and M1 mortar series
 468 is unclear but appears to be related to changes in the water demand of the plastic
 469 mortars following storage. Table 6 shows the w/b ratios required to achieve the specified
 470 flow values during mortar manufacture. For the M1 series, the data show a marked
 471 reduction in water demand after storage whereas comparatively little change is observed
 472 for the M5 series. It seems reasonable to assume that the reduction in water demand is
 473 a result of changes in the properties of the calcium hydroxide during storage. These
 474 changes were discussed in detail elsewhere [1] and include decreases in surface area
 475 and possibly a small increase in the quantity of calcium carbonate within the lime; see
 476 Tables 8 and 9 of ref [1]. For a given lime, it seems likely that a reduction in surface area
 477 would lead to a lower water demand in the plastic mortars and, as discussed previously,
 478 the presence of calcium carbonate leads to enhanced strength in lime mortars.

479 Furthermore, the high proportion of lime in the M1 mixes suggests that these materials
 480 would be more sensitive to changes in lime composition than the slag-rich M5 series.
 481 Despite the increasing use of pre-mixed lime mortars in UK construction practice, there
 482 is no evidence within the literature of research to determine potential changes in the
 483 properties of these materials following extended periods of silo storage. In the current
 484 paper, changes were not confined to mortars prepared with the lime-drying technique
 485 (see mortar M1-5) and it seems reasonable to assume that other lime rich mortars may
 486 also be subject to the same phenomenon. Hence, further research is recommended in
 487 this area.

Mortar	w/b ratio		Change (%)
	“Fresh”	“Stored”	
M1-1	1.35	1.30	3.7
M1-2	1.36	1.31	3.7
M1-3	1.36	1.29	5.1
M1-4	1.37	1.31	4.4
M1-5	1.41	1.35	4.3
M5-1	1.03	1.01	1.9
M5-2	1.01	1.00	1.0
M5-3	0.99	0.98	1.0

488 Table 6: Influence of storage on w/b ratio to achieve common flow.
 489

490 **4.2 Non-optimally dried mortars**

491

492 Three regions of drying have been previously defined [1]. Optimal drying is considered to
 493 occur when the measured evaporation during the drying process equals the balance of

494 the original free moisture and that removed by chemical combination after accounting for
495 the stoichiometric ratio of quicklime used and its purity. In contrast, sub-optimal drying
496 does not remove sufficient water and super-optimal drying removes too much water
497 leaving a residual amount of free lime - see ref [1] for a fuller discussion. The
498 assumption was that sub-optimal drying would lead to degradation during storage whilst
499 super-optimal drying would yield an unsound binder. The latter has previously been
500 confirmed for storage periods of up to 4 weeks [1].

501

502 A series of 10 mortars was produced based upon the optimal processing criteria for the
503 M1-1 mortar. By reducing the stoichiometric ratio of quicklime sub-optimal mortars were
504 produced and by increasing it super-optimal mortars resulted. After lime-drying half the
505 mortar was used to produce “fresh” mortars whilst the remainder was stored in sealed
506 boxes for 10 weeks to produce “stored” mortars. All mortars were produced to a
507 common flow of 170 mm and subject to both air and water curing. Table 7 shows the key
508 data of stoichiometric ratio and the stoichiometric variation from optimal conditions for
509 each mortar together with the w/b ratio to achieve the desired flow. It can be seen that
510 the optimally dried mortar has the highest water demand of all mortars; the reason for
511 which is not immediately apparent.

Stoichiometric ratio (%)	Stoichiometric variation (%)	w/b	
		“Fresh”	“Stored”
50.0	-9	1.18	1.17
54.9	-4	1.21	1.25
55.9	-3	1.21	1.26
56.9	-2	1.24	1.24
57.9	-1	1.28	1.23
58.9	Optimal	1.35	1.31
59.9	+1	1.26	1.22
60.9	+2	1.28	1.22
61.9	+3	1.28	1.20
67.0	+8	1.26	1.18

Table 7: Sub-optimal, optimal and super-optimal mortars

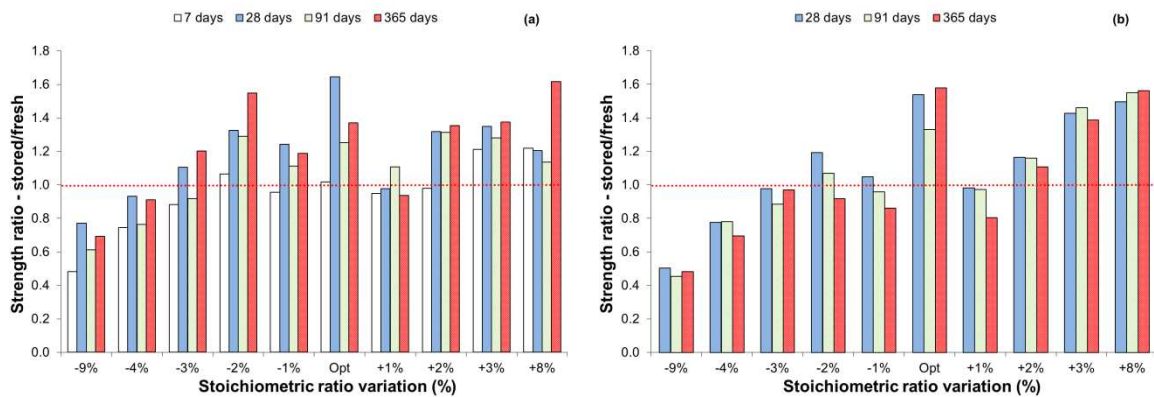
512
513

514 The water demand of the “stored” optimal and super-optimal mortars is less than their
515 “fresh” counterparts as previously observed in section 4.1. This might be explained by
516 the presence of unslaked lime in the “fresh” mortars placing a demand for water to slake
517 the quicklime during mortar production whilst this was air-slaked during the storage
518 period. In contrast, the trend for the sub-optimal mortars is less clear but, in general,
519 appears to be either unaffected or shows an increase in water demand which may be a
520 reflection of the generation of increased surface area by hydration of ggbs during
521 storage.

522

523 It is clear from the raw data that the use of super-optimal drying has not been
524 detrimental to strength development under either storage period or curing condition. This

525 would support the supposition that any free lime remaining after lime-drying has been
 526 converted to slaked lime during the storage period or the mortar mixing process. The
 527 relative performance of each mortar, expressed as the ratio of the compressive strength
 528 of the “stored” / “fresh” mortars at ages between 7 – 365 days is shown in Figure 9.
 529 Under both curing conditions both the optimal and super-optimal mortars display a
 530 strength enhancement following the 10 week storage period; the super-optimal +1%
 531 mortar may be an anomaly. Whilst the sub-optimal -1% and -2% mortars subject to air
 532 curing also show strength enhancement at all ages, the water cured samples register a
 533 reduction in relative strength with age suggesting that the longer term hydration of the
 534 ggbs may have been compromised during storage. The sub-optimal -3% mortar appears
 535 to present a threshold in behaviour in that whilst storage has not affected the strength
 536 neither is strength enhancement observed. As the stored mortars become wetter (-4%
 537 and -9% variation) it is clear that they have degraded to the extent that the “stored”
 538 mortars are substantially weaker at all ages, more so following water curing than air
 539 curing.



540 Fig 9: Influence of varying the quicklime addition on relative strength of “fresh” and
 541 “stored” mortars subject to (a) air curing and (b) water curing.

542

543 This data suggest that in the practical implementation of the lime-drying process there is
544 a narrow range of quicklime addition in which the quality of mortar is maintained whilst
545 also minimizing the financial cost of using excessive quicklime.

546

547

Part B – Roman cement mortars

548

5 Materials and Methods

550

551 A Roman cement developed during the EU funded ROCARE project from marls sourced
552 in Gartenau, Austria, and a 0 - 4 mm carbonate sand were used. The cement was
553 manufactured by The Institute of Ceramics and Building Materials (MBM) in Krakow,
554 Poland; details may be found elsewhere [5]. Two mixes have been de-activated with 7%
555 de-activation water and 30 min storage prior to the formulation of the final mortar. The
556 deactivation water, expressed as a percentage of the cement weight, is first added to the
557 oven dry sand and mixed for 2 min at 62 rpm (a Hobart mixer was used for all mortar
558 production). Subsequently, the cement was added to the wet sand and the whole mixed
559 for a further 2 minutes at 62 rpm before being stored in an airtight box. At the end of the
560 30 minute de-activation storage time, the first mix was oven dried for 24 hours at 105°C
561 in an atmosphere circulated over silica gel and soda lime while the second mix was lime-
562 dried by means of the addition of a pre-determined amount of quicklime. The moisture
563 content of the DARC mortar was determined as 0.63%. Based on previous experience
564 the lime content was specified as 76% stoichiometric ratio with a 5 minute mixing time.
565 Both mixes were then stored in airtight boxes for 4 weeks prior to formation into mortars.
566 A third mix (control mix) was prepared and stored for the same period of time with no
567 drying carried out.

568

569 At the end of the 4 week storage period, mortars were manufactured at a sand:cement
570 ratio of 2.5:1 by volume and constant w/c ratio of 0.81. The mortars were produced by
571 mixing the products of the previous drying processes in a Hobart mixer with water for 30
572 s at 62 rpm. At this time the mixer was stopped for 30 s during which the mortar
573 adhering to the wall and bottom part of the bowl was removed by means of a rubber
574 scraper and placed in the middle of the bowl. The mixing was then continued at 125 rpm
575 for 8 minutes.

576

577 Flow and workable life were measured according to BS EN 1015-3:1999 [7] and BS EN
578 1015-9:1999 [25] respectively. Samples for strength determination were cast in 40 × 40
579 × 160 mm steel moulds in two layers and vibration compacted before being covered with
580 a polythene sheet. Mortar beams were de-moulded after 24 h and cured in water at 20°C
581 prior to testing at 28 days.

582

583 Differential Thermo-gravimetric (DTG) tests (a heating rate of 5°C per minute to 150°C
584 under a nitrogen atmosphere) were also performed on the oven dried mix and on the
585 control mix immediately after the end of the 30 minute storage period.

586

587 **6 Mortar properties**

588

Mortar	Flow (mm)	Work Life (min)	Comp Strength (MPa)	Flex Strength (MPa)
Control	170	>141	9.84	2.98
Oven Dried	155	~50	11.8	3.51
Lime Dried	140	~47	13.2	3.92

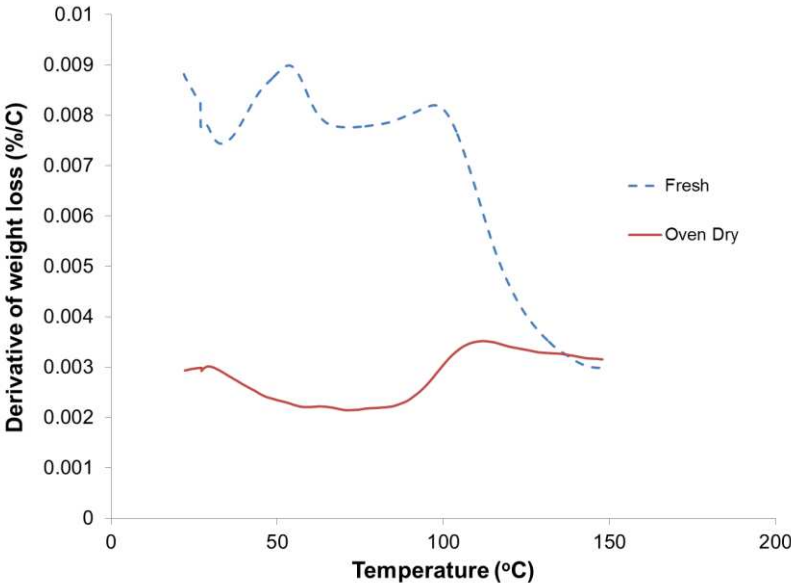
589

Table 8: Properties of Roman cement mortars

590

591 In Table 8 it is shown that both the oven dried and the lime-dried mortars exhibit a lower
592 fluidity than that of the control mortar, with the lowest flow being observed for the lime-
593 dried mortar. This means that these mortars will require higher amounts of water to
594 achieve the desired workability. Whilst the higher water demand of the lime dried mortar
595 can be explained by the presence in this mix of calcium hydroxide formed in the reaction
596 of quicklime with de-activation water, it was not expected that the oven dried mix would
597 have a higher water demand than that of the control mix. In fact, in a previous study [5] it
598 was shown that during storage of a DARC mortar (i.e. the control mortar) the water
599 demand for the subsequent wet mortar was increased as the storage period was
600 prolonged. Whilst all 3 “dry” mortars in the current study were stored for 4 weeks, the
601 oven-dried mortar was conditioned to a lower free moisture content such it might have
602 been expected to perform similarly to a DARC mortar that had been subject to a 30 min
603 only storage period, i.e. a lower water demand or higher fluidity than that of the control
604 mortar. A possible explanation of this data is that the oven-drying causes the de-
605 hydroxylation of the AF_m phases. In order to verify this hypothesis, DTG tests were
606 undertaken on the control mix (fresh) and on the oven-dried (OD) mix and results are

607 shown in Figure 10. It is apparent that the use of oven drying has removed water from
608 the system which may be accounted for by both the loss of evaporable water [26] and
609 partial de-hydroxylation of the AF_m phases [27]. In this case, the reverse process (re-
610 hydroxylation) might take place during the early life of the constituted mortar and
611 account for the increased demand for mixing water.



612
613 Fig 10: DTG curves of control (fresh) mix and oven dried (OD) mix (5°C per minute to
614 150°C).

615
616 Table 8 also shows that the workable life of the mortar produced with the control mix is
617 at least 3 times longer than that produced with the oven dried and with the lime-dried
618 mix. This can be in part attributed to the higher flow of the control mix mortar and in part
619 to the longer storage under conditions of free moisture experienced by this mix, which
620 has been shown to increase workable life [5]. Further study including the production and
621 testing of mortars at constant flow is necessary to better understand the effect of the
622 drying processes on workable life. Both compressive and flexural strength (28 days) are

623 higher for the dried mix mortars than for the control mix mortar, with the highest strength
624 observed for the lime-dried mix mortar.

625

626 It is not possible to undertake a strict comparison between the degradation observed in
627 the stored DARC mortars (see Figs. 13 & 14, ref 5) and the comparative performance of
628 the various mortars reported here-in since the sand:cement and w/c ratios are different
629 in the two studies. However, the conditioning of the current control mix equates to that of
630 the DARC mortar stored for 4 weeks in [5]. Using these mortars as the basis for
631 comparison the strength of the lime-dried mortar is 134% of the control mortar whilst the
632 strength of the 30 minute stored DARC mortar is 152% of that of the 4 week stored
633 DARC mortar [5]. Thus, the lime-drying has largely mitigated the degradation attributed
634 to the effects of free moisture present during prolonged storage of the DARC mortars; all
635 free moisture would not have been immediately removed by the addition of quicklime so
636 some residual degradation would be expected.

637

638 Experience suggests that once the water content of the lime-dried mortar is adjusted to
639 yield the desired flow, the strength would be similar to that of the control mix and the
640 workable life extended into the target range of 1 – 2 hours; additional work is required to
641 prove this assumption.

642

643 **7 Conclusions**

644

645 From the results described in Parts A and B of this paper it can be concluded that:

646

- 647 • The lime-drying process has been successfully employed to produce mortars of M1
648 and M5 classification using ggbs as the hydraulic component.

- 649 • By careful manipulation of the amount of quicklime added (stoichiometric ratio) and
650 the length of the mixing time during the lime-drying process it is possible to produce
651 identical mortars from sands with differing water contents.
- 652 • The composition of different quicklimes may have a significant influence on the
653 strength of hardened mortars prepared from the lime drying process, particularly in
654 lime-rich blends with low ggbs/CH.
- 655 • The strength of the lime-slag mortars produced by the lime-drying process are very
656 similar to those of mortars produced using equivalent commercial hydrated lime.
- 657 • The lime dried materials may be silo-stored prior to use without changes to the
658 mortar classification.
- 659 • The work has highlighted a requirement for further research to examine the effects of
660 silo storage on the subsequent water demand and strength of lime mortars
661 containing a high proportion of lime.
- 662 • Lime-drying overcomes strength degradation previously observed in Roman cement
663 mortars retarded by a pre-hydration technique and subsequently stored.

664

665 **Acknowledgments**

666

667 The authors are grateful for the funding provided by the Engineering and Physical
668 Sciences Research Council (EP/D025036/1), Technology Strategy Board (Prog. No:
669 876) and Lime Technology Ltd, the EU under the 7th Framework ROCARE project (Call
670 identifier FP7-ENV-2008-1, Project number 226898) and Castle Cement Ltd, Civil and
671 Marine Ltd and Lhoist UK Ltd for the supply of materials. Thanks are also due to Dr.
672 Mike Lawrence at the University of Bath for his assistance with the mercury porosimetry

673 analyses and to Dr Grzegorz Adamski of The Institute of Ceramics and Building
674 Materials, Krakow, Poland for the manufacture and supply of the Gartenau cement.

675

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