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1 **Title:**

2 Developing a PCM-enhanced mortar for thermally active precast walls

3

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22

23 **Abstract**

24 This work presents an experimental research on the thermal properties of novel cementitious
25 mortars incorporating microencapsulated Phase Change Materials intended to be used as the
26 innermost layer in a precast radiant building component actively controlled by an integrated
27 hydronic system.

28 The characterization was developed in two steps: the first one focused on the effects of different
29 fine aggregates and admixtures for a fixed PCM content and the second one on the effect of
30 changing the amount of PCM. Results show that using silica aggregates and antifoaming

31 admixture outperform the other options, producing mortars with statistically significant higher
32 thermal conductivities, diffusivities and effusivities. Besides, increasing the amount of PCM
33 significantly reduces conductivity and diffusivity, but the effusivity is practically invariant. This
34 suggests that the mortar design has to be defined by predominantly focusing on diffusivity, in
35 order to achieve appropriate heat penetration rates and activation times for an efficient system
36 operation.

37

38 **Keywords:** Phase Change Material, Thermal energy storage, Thermal diffusivity, Thermal
39 effusivity, Cementitious mortar.

40

41 **1. Introduction**

42 The total final energy demand of the European Union member states was approximately 1084
43 Mtoe (Million Tonnes of Oil Equivalent) in 2015, according to the latest data published by
44 Eurostat [1]. Building-related sectors such as residential and services were the most energy-
45 intensive demanding sectors producing together a demand of 422 Mtoe corresponding to 39%
46 of the total, with about two-thirds corresponding to the residential sector [1,2]. Despite the
47 increasing interest in promoting energy-efficient buildings and the current energy policies aimed
48 to reduce the energy impact of the construction sector [3–5], the weight of building-related
49 sectors on the European final energy balance has slightly increased from 35% in 1990 to 39%
50 in 2015. Looking at the energy end-uses in buildings, space heating is the most energy
51 consuming one in the residential sector, representing 71% of the total consumption of
52 households, whereas cooling represents almost 10% of the total energy consumption in
53 Southern countries mainly due to a rapid penetration of air conditioning systems [2,6].

54

55 In this context, strategies focused on achieving thermal comfort in buildings, while reducing
56 heating and cooling energy demand, such as Thermal Energy Storage (TES) systems and
57 Thermally Activated Building Systems (TABS), have gained interest proving high energy
58 savings potential [7]. Building integration of TES technologies improves the building energy
59 efficiency by reducing peak loads, uncoupling the energy demand from its availability, allowing
60 the integration of renewable energy sources and providing an efficient management of thermal

61 energy [8–10]. TABS consist of hydronic pipes embedded in building elements and actively
62 used to transfer heat and/or cold to building components such as walls, ceiling or floors [10].
63 The thermal mass activation of building components has a big potential for energy use
64 reduction in buildings mainly due to its low temperature operation (low temperature heating and
65 high temperature cooling, as a result of the large heat exchange surfaces) and the high thermal
66 inertia that enables shaving peak loads and shifting energy consumption to low energy cost
67 periods [11,12]. Furthermore, radiative heat exchange of TABS provides comfort conditions,
68 avoiding typical draught problems of convective systems [13–15].

69 Thermal characteristics of conventional cement-based mixtures without PCM have been
70 analysed by several authors. Although the values vary considerably depending on the specific
71 composition of the mixtures, typical values are in the range from 1.5 to 3.4 W m⁻¹ K⁻¹ for thermal
72 conductivity [16–19], from 0.38 to 0.90 mm² s⁻¹ for thermal diffusivity [17,19,20] and from 323 to
73 1800 W·s^{0.5}·m⁻²·K⁻¹ for thermal effusivity [18,20].

74 Available literature on building materials incorporating Phase Change Materials (PCM) is
75 extensive since a wide range of different materials and building products, such as gypsum
76 plaster, gypsum boards, concrete, panels, bricks, membranes and insulating materials, have
77 been considered as matrix materials to include PCM. The main objective of these studies was
78 to improve the thermal behaviour of passive construction systems by increasing the thermal
79 storage capacity of the building envelope [17,21–27].

80

81 However, research on the use of latent heat storage materials in active building components is
82 not very extensive, likely because TABS and PCM in most cases have been investigated as
83 individual technologies.

84

85 In spite of this, TABS in combination with PCM might result in an improvement of the thermal
86 comfort in buildings and at the same time in a reduction of the HVAC energy consumption,
87 mainly due to (i) the increased energy storage density of latent heat storage systems that
88 contributes to the peak loads shifting, including in the case of lightweight buildings, (ii) the
89 flexible operation of the hydronic pipes that provide active charging and discharging of the PCM
90 depending on the actual heating or cooling needs, and (iii) the possibility to be efficiently

91 coupled with heat pumps and solar-assisted systems, being a low-temperature heating and
92 high-temperature cooling system [28–30].

93

94 Regarding the hydronic thermal activation of building components incorporating PCM, a first
95 study was proposed by Koschenz and Lehmann [31]. They designed a ceiling panel
96 incorporating a 5 cm thick layer made of gypsum and microencapsulated PCM (25% by weight)
97 with embedded capillary tubes for the night cooling of lightweight buildings. One of the findings
98 of the development process was the significant thermal conductivity reduction of gypsum after
99 the PCM addition and the need of incorporate aluminium fins to compensate for the decrease of
100 the gypsum thermal conductivity from the expected value of $0.8\text{-}1.0 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ to $0.2 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$
101 caused by PCM addition.

102

103 Another study on the thermal mass activation of a concrete slab including PCM by means of
104 embedded hydronic pipes was proposed by Jin and Zhang [32]. The proposed system consists
105 of a radiant floor with two layers of PCM with different melting temperatures to cover both
106 heating and cooling periods. The objective of the paper was to define the optimal melting
107 temperatures of the layers in order to store heat or cold energy in off-peak period and release
108 the energy in peak period. Authors found by numerical analysis that the fluctuations of the floor
109 surface temperature and heat flux were reduced by using PCM, not only for the direct effect of
110 latent heat capacity but also as a consequence of the low thermal conductivity of the PCM
111 layers. Moreover, they found that the optimal melting temperatures were 38°C and 18°C for
112 heating and cooling respectively, and that the energy released by the floor with PCM in peak
113 period was increased by 41% for heating and 38% for cooling in comparison with the radiant
114 floor without PCM.

115

116 A similar study on the development of radiant floor panels incorporating granulated PCM and
117 activated by water pipes was presented by Arsuini et al. [33]. Results showed that the
118 integrated PCM layer improved the thermal performance of the floor during summer cooling
119 regimes by storing the internal gains without temperature increase. However, the heating
120 performance was not as good as expected mainly due to the inefficient heat exchange between

121 the water and the environment produced by the increased thermal resistance of the melted
122 granular PCM. In order to lower the contact resistance between the pipes and the PCM, a steel
123 matrix was inserted in the PCM layer providing the necessary ~~conductibility~~conductivity to
124 bypass the parasitic resistances introduced by the PCM.

125

126 Regarding the thermal properties of self-compacting concrete including different amounts of
127 microencapsulated PCM, a study was performed by Hunger et al. [16]. They found out that
128 increasing the amount of PCM significantly increases the specific heat capacity as expected.
129 However, the thermal mass presents an upper limit, corresponding to 4-5% PCM in weight, due
130 to the decreasing concrete density. Furthermore, they found out that the addition of PCM results
131 in a substantial reduction of thermal conductivity (of about 40% for the concrete with 5% of PCM
132 by mass in comparison with the reference mix) that improves the concrete insulation properties,
133 but makes the PCM thermal activation more difficult. In the same research line, Pomianowski et
134 al. [34] presented an experimental method to determine the specific heat capacity of concrete
135 materials incorporating PCM as a function of the temperature for the melting process. In this
136 work, the authors emphasise that an efficient application of PCM-enhanced concretes in
137 buildings is strongly limited by their low thermal conductivity that hinders the PCM thermal
138 activation, as also indicated in [18].

139

140 In light of these results, it seems clear that the thermal properties of PCM-added materials have
141 a fundamental role in the development of efficient TABS solutions. In fact, the very high latent
142 heat storage capacity of the PCM can be unexploited if the thermal activation of the material
143 cannot be reached due to the low thermal conduction in the composite. Likewise, the low
144 thermal conductivity and density of cementitious material including PCM can decrease the
145 building element thermal inertia and thus the performance of TABS [18,31]. In fact, in order to
146 fully exploit the improved thermal storage capacity of the PCM-based material in comparison
147 with the conventional one, it is crucial that the addition of the PCM does not produce a
148 substantial drop in density and thermal conductivity, as has been highlighted by several studies
149 [16,18,31,34]. Bearing in mind these premises, the general objective of this work is the
150 formulation of a PCM-enhanced mortar to be used as the inside layer in precast thermally

151 activated façade components. More specifically, this work presents the experimental research
152 carried out to assess the thermal properties of different cementitious mortars added with
153 microencapsulated PCM in order to point out the optimum solution in terms of mix components
154 for this specific application.

155

156 **2. Methodology**

157 **2.1 General approach**

158 This study analyses five properties of different cementitious mortars containing
159 microencapsulated PCM; density, air content, thermal conductivity, thermal diffusivity, and
160 thermal effusivity have been experimentally studied as the first step in the definition of the
161 optimum formulation of a PCM-added mortar for precast thermally activated building
162 components. In fact, for this application, besides having good workability and proper self-
163 compacting properties, the mortar is required to have specific thermal characteristics to achieve
164 a good compromise between the energy storage capacity and the charging and discharging
165 rates of the element. In particular, in this study three thermal characteristics were analysed:

- 166 a) Thermal conductivity λ [$\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$]
- 167 b) Thermal diffusivity α [$\text{m}^2\cdot\text{s}^{-1}$]
- 168 c) Thermal effusivity e [$\text{W}\cdot\text{s}^{0.5}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$]

169

170 Thermal conductivity is the property of a material to conduct heat and describes the transport of
171 energy through the mass. In spite of the importance of this fundamental property to represent
172 how well a material conducts heat, it is not adequate to predict the behaviour of a TES system
173 in which the heat transfer process depends on transient heat flows. In fact, the heat flow
174 through building components rarely reaches a steady state, so for an accurate estimation of the
175 thermal behaviour, it is necessary to take into account the heat storage capacity of the element.
176 To this aim, derived properties incorporating the material heat storage capability, such as
177 thermal diffusivity and effusivity, are much more useful dynamic performance indicators from
178 which the behaviour of the system may be deduced.

179 Thermal diffusivity α is [calculated according to Eq. 1:](#)

180 $\alpha [m^2s^{-1}] = \frac{\lambda [Wm^{-1}K^{-1}]}{\rho [kg\ m^{-3}] c_p [J\ kg^{-1}K^{-1}]}$ Eq. 1

181

182 ~~Where λ is the defined as the~~ thermal conductivity, ~~ρ divided the by~~ density and ~~c_p the~~ specific
183 heat (i.e. the heat storage capacity of a material per unit volume).

184 Thermal effusivity e (also called specific admittance, heat penetration coefficient or thermal
185 inertia) is ~~defined~~ calculated according to the Eq. 2 [18,35,36]:

186 $e [Ws^{0.5}m^{-2}K^{-1}] = \sqrt{(\lambda [Wm^{-1}K^{-1}] \rho [kg\ m^{-3}] c_p [J\ kg^{-1}K^{-1}])}$ Eq. 2

187

188 ~~as the square root of the product of thermal conductivity, density and specific heat [32–34].~~

189 These two derived properties, despite containing the same variables, describe different
190 characteristics of the materials regarding their thermal behaviour in dynamic conditions. In
191 particular, diffusivity defines how fast the material temperature adapts to the surrounding
192 temperature, whilst effusivity assesses the material ability to exchange thermal energy with its
193 surroundings [20]. Briefly, materials with high diffusivity values transmit boundary heat flux
194 fluctuations more quickly than materials with low values do, while materials with high effusivity
195 values will more readily absorb a surface heat flux. For this specific system, consisting of a
196 radiant wall incorporating on the inner side a PCM-added mortar activated by means of water
197 pipes, thermal diffusivity mainly limits the capacity of activating the PCM in deeper layers and
198 consequently the charging times, as long as thermal effusivity would dominate the surface heat
199 flux and the passive heat exchanges with the indoor ambient. Therefore, a good balance
200 between both properties is required in order to achieve a material that can store high amounts
201 of thermal energy under fast heat transfer process, allowing short charging and discharging
202 cycles and thus being adequate to be used in PCM-enhanced thermally activated building
203 elements.

204

205 The analysis presented in this study was developed in two phases: in the first step the study
206 focused on the effect of fine aggregates and antifoaming admixture on specific characteristics of
207 the mortar for a constant amount of PCM; in the second one, the effect of the PCM content on
208 the properties of the mortar was assessed.

209

210 **2.2 Step 1 - Aggregate and antifoaming effects - 2×2 factorial design**

211 The first step the study focused on pointing out the effect of ~~two factors~~: (i) the fine aggregates
212 and (ii) the antifoaming admixture (AF) on several properties of the mortar, such as density, air
213 entrapped, and thermal ~~properties, i.e.~~ conductivity, diffusivity and effusivity ~~of the mortar~~. To
214 conduct this study, a full factorial experiment with the aforementioned two factors, each one
215 taking two levels (two different fine aggregates, silica and barite 0-4 sands and using or not the
216 AF) was carried out. The advantage of this study is the capability of examining not only the
217 effect of each factor on the response variable, but also the effect of the interaction between the
218 factors on the response variables.

219 The interest of using heavy aggregates in the formulation of mortars with PCM can be explained
220 taking into account that when a portion of the aggregates is replaced by microencapsulated
221 PCM, a significant reduction in density is produced [16,37]. Although the density reduction may
222 not be critical in terms of thermal mass reduction, since it is counterbalanced by the additional
223 latent heat capacity provided by the phase change material [27], on the other hand, it could lead
224 to a significant reduction in thermal effusivity, delaying the thermal activation of the building
225 component. So, heavy aggregates could be a solution to obtain mixtures with acceptable
226 densities when high amounts of PCM are incorporated. Furthermore, the authors have
227 observed in previous pilot tests that the density reduction produced by the PCM addition is not
228 only due to the direct effect of replacing heavier components by lighter ones, but also to the
229 trapped air introduced into the mixture by the PCM [38]. In fact, for higher PCM contents in the
230 mixture, a larger amount of entrapped air was observed, probably due to the coating of the
231 microcapsules that acts as an air-entrainment agent. The consequent increase in porosity leads
232 to lower density and especially lower thermal conductivity, which implies lower thermal
233 diffusivity and effusivity. Therefore, the effectiveness of using an antifoaming admixture in order
234 to reduce the entrapped air was analysed.

235

236 It is worth highlighting that in this first part of the study the PCM content was maintained
237 constant for all the mixtures (28% in volume), in order to take into account only the effects
238 produced by the aggregates and the AF admixture.

239

240 In order to perform the analysis of the data with a statistical power higher than 80% and a
241 confidence level of 95%, three replications of each aggregate-admixture combination were
242 executed, following a fully randomised sequence for both mixing process and test performing.
243 Next, a two-way Analysis of Variance (ANOVA) statistical test was carried out in order to
244 investigate the effects of the independent variables and the interaction between them on the
245 dependent variable [39]. In short, this analysis was performed to establish if the aggregate, the
246 admixture and the interaction between them significantly affect the density, the air entrapped,
247 and finally the thermal performance of the mortars.

248

249 **2.3 Step 2 - PCM content effect – regression**

250 In the second step, the effect of the PCM content on the properties of the mortar was assessed.
251 With this aim, the best performing mixture in terms of thermal properties found out in the first
252 phase of the research was taken as the reference dosage for defining other mixtures with
253 different PCM content. Starting from the mortar with 28% PCM in volume selected from Step 1,
254 four mixes with a PCM content ranging between 20% and 32% in volume were defined,
255 reducing the content of fine aggregates accordingly. For all the mixtures, all the others
256 components, i.e. cement, water, superplasticizer and antifoaming admixture, were kept constant
257 in order to account for the PCM effect only. It has to be emphasized that a wider range of
258 mixtures in terms of PCM content could not be analysed because the PCM content was limited
259 by the workability of the mixtures. In fact, for PCM contents higher than 32% the mixture
260 exhibited a very high viscosity, in accordance with the direct relationship between the PCM
261 content and the viscosity of the mixture observed by other authors [16,37,40]. On the contrary,
262 for a PCM content lower than 19% in volume the mixture was too fluid causing the segregation
263 of the aggregates. This aspect can be explained considering that the reference composition of
264 the mortar containing 28% PCM had already been optimized in terms of workability for using the
265 compound in the fabrication of precast elements, and then if cement and water amounts are
266 kept constant whereas PCM dosage is reduced, the viscosity of the mix also decreases. In
267 other words, to analyse mixtures with a lower content of PCM, a reformulation of the mortar
268 would have been necessary, but in this case a comparative analysis would not have made
269 sense, being the mixtures completely different.

270 Regarding the analysis of the data, four samples of each mixture were prepared for a total of 16
 271 samples, obtaining a statistical power higher than 80% at a confidence level of 95%. Both
 272 sample preparation and testing were performed following a randomised sequence to minimise
 273 the influence of systematic errors. Next, to determine if the differences among the mean values
 274 were significant, and thus if the PCM content affects the thermal performance of the mortar, a
 275 one-way ANOVA was carried out [41]. The goal of this analysis is to investigate if the between-
 276 sample variance is much larger when compared to the within-sample variance, in other words, if
 277 the variations of the response variables (i.e. density, air content and thermal properties) are
 278 largely caused by the PCM quantity, rather than chance variation.

279

280 **3. Materials and equipment**

281 **3.1 Mortar mix preparation**

282 3.1.1 Mortar mix preparation for step 1

283 The cementitious mortars analysed in this study consists of cement CEM I 52.5 R, silica or
 284 barite 0-4 sand as fine aggregates, superplasticizer agent, antifoaming admixture and
 285 microencapsulated PCM Micronal DS 5038X, commercially available from BASF. The general
 286 properties of the PCM are shown in Table 1.

287

288 Table 1. Properties of the PCM used in the research (manufacturer data)

Material	Product type	Melting point [°C]	Total storage capacity [kJ kg ⁻¹]	Latent heat capacity [kJ kg ⁻¹]	Apparent density [kg m ⁻³]
BASF					
Micronal® DS 5038X	Powder	26	145	110	250-350

289

290 To prepare the specimens, the following assumptions have been made:

- 291 – The liquid phase (consisting of water, superplasticizer and AF admixtures) to cement
 292 ratio was kept constant for all the mixtures. For the formulations without AF, water
 293 content was increased by the same quantity of the AF used in the mixtures with AF.

294 | – The fine aggregate-to-cement ratio ~~at in-volume (2.4)~~ as well as the PCM content by
 295 | volume ~~at (28%)~~, were kept constant for all the mixtures, in order to point out the effect
 296 | of fine aggregates and AF only. This means that the theoretical volume is constant for
 297 | all the mixtures, but the total mass varies depending on the fine aggregate used, being
 298 | the densities of silica and barite sand substantially different.

299 | As regards the superplasticizer (SP) content, it was established performing a set of preliminary
 300 | tests using the Marsh funnel and varying its content from 0.6% to 1.3% with a fixed liquid to
 301 | cement ratio. In this way, the SP saturation point was determined and used in further
 302 | experimentations. All the formulations analysed in the first phase of the study are shown in
 303 | Table 2.

304

305 | Table 2. Mix compositions in the first phase of the study

	I [Silica w/o AF]		II [Silica w/ AF]		III [Barite w/o AF]		IV [Barite w/ AF]	
	Mass [kg]	Vol [dm ³]	Mass [kg]	Vol [dm ³]	Mass [kg]	Vol [dm ³]	Mass [kg]	Vol [dm ³]
Total water	0.600	0.600	0.597	0.597	0.600	0.600	0.597	0.597
Cement	0.600	0.190	0.600	0.190	0.600	0.190	0.600	0.190
Superplasticizer	0.008	0.007	0.008	0.007	0.008	0.007	0.008	0.007
0/4 Silica sand	1.200	0.448	1.200	0.448	-	-	-	-
0/4 Barite sand	-	-	-	-	2.015	0.448	2.015	0.448
PCM	0.482	0.482	0.482	0.482	0.482	0.482	0.482	0.482
Antifoaming	0.000	0.000	0.003	0.003	0.000	0.000	0.003	0.003
Theo. vol. [dm ³]		1.728		1.728		1.728		1.728
Total mass [kg]		2.890		2.890		3.705		3.705
Theo. density [kg dm ⁻³]		1.673		1.672		2.144		2.144
Water-to-cement ratio		1.0		1.0		1.0		1.0
Agg. to cem. ratio in vol.		2.4		2.4		2.4		2.4
SP/Cem [wt.%]		1.3		1.3		1.3		1.3
AF/Tot [vol.%]		0.0		0.2		0.0		0.2
PCM/Tot [wt.%]		17		17		13		13
PCM/Tot [vol.%]		28		28		28		28

306

307 Regarding the mixing procedure, it started with the dry mixing of cement, fine aggregate and
 308 PCM for 30 seconds to obtain a homogeneous mixture. Next, the superplasticizer was dissolved
 309 into the water and gradually added to the mixture during mixing for 60 seconds. At this point the
 310 AF admixture was incorporated and mixing continued for another two minutes to ensure a
 311 proper amalgamation of all the components. For the mixtures without AF, the same procedure
 312 was followed with the difference that the mixing continued for 3 minutes without interruptions
 313 after the first 30 seconds of dry mixing.

314 3.1.2 Mortar mix preparation for step 2

315 To formulate the specimens for the second stage of the study, the following assumptions were
 316 made:

- 317 – Constant amounts of water, SP and AF admixtures as well as of cement.
- 318 – Increasing aggregate-to-cement ratio in volume from 2.0 for mixture A (32% PCM in
 319 volume) to 3.1 for mixture D (19% PCM in volume).
- 320 – Decreasing PCM content from 32% in volume (mixture A) to 19% in volume (mixture D).

321 Also in this case, the theoretical volume is constant for all samples, but the total mass varies
 322 due to the different densities of silica sand and PCM. Mortars analysed in the second phase of
 323 the research are shown in Table 3.

324

325 Table 3. Mixture compositions in the second phase of the study

	A (32 vol.% PCM)		B (28 vol.% PCM)		C (24 vol.% PCM)		D (19 vol.% PCM)	
	Mass [kg]	Vol [dm ³]	Mass [kg]	Vol [dm ³]	Mass [kg]	Vol [dm ³]	Mass [kg]	Vol [dm ³]
Total water	0.597	0.597	0.597	0.597	0.597	0.597	0.597	0.597
Cement	0.600	0.190	0.600	0.190	0.600	0.190	0.600	0.190
Superplasticizer	0.008	0.007	0.008	0.007	0.008	0.007	0.008	0.007
0/4 Silica sand	1.023	0.382	1.200	0.448	1.390	0.519	1.594	0.595
PCM	0.548	0.548	0.482	0.482	0.411	0.411	0.335	0.335
Antifoaming	0.003	0.003	0.003	0.003	0.003	0.003	0.003	0.003
Theo. vol. [dm ³]		1.728		1.728		1.728		1.728
Total mass [kg]		2.779		2.890		3.009		3.137
Theo. density [kg dm ⁻³]		1.608		1.672		1.741		1.815

Water-to-cement ratio		1.0		1.0		1.0		1.0
Agg. to cem. ratio in vol.		2.0		2.4		2.7		3.1
SP/Cem [wt.%]		1.3		1.3		1.3		1.3
AF/Tot [vol.%]		0.2		0.2		0.2		0.2
PCM/Tot [wt.%]		20		17		14		11
PCM/Tot [vol.%]		32		28		24		20

326

327 **3.2 Analysis**

328 3.2.1 Workability

329 Considering that workability of the paste is a fundamental property that constrains the feasibility
330 of using the compound in the fabrication of precast elements, other mixtures that did not meet
331 the required rheological characteristics were discarded from the study in a previous step.

332 Further characterizations were only performed for the usable mixtures reported in Table 2 and
333 Table 3. To assess the mortar composition influence on workability, the mini-slump flow test as
334 an indirect measure of the yield stress and fluidity was conducted.

335

336 3.2.2 Density

337 To calculate the density of mortars, the volume and mass of specimens casted in plastic cups
338 were measured. Volume was measured by completely filling the cup with water and checking
339 the total filling by positioning a piece of planar transparent glass on the top of the plastic cup. In
340 this way, it was ensured that no air bubbles remained inside the cup and so the specimen
341 volume was determined by weighing the water mass, after taring the scale with the empty cup
342 and the piece of glass. In the unit conversion from kilogrammes to cubic meters a water density
343 of 1000 kg m^{-3} was assumed. To measure the mass of the water, as well as the mass of
344 mortars and slurries specimens, a scale Mettler Toledo SB32001 DeltaRange was used.

345

346 3.2.3 Air content

347 To calculate the air content of the mortars, the gravimetric method described in the ASTM C138
348 standard was used [42]. First, the fresh densities of the mixtures were calculated following the
349 same method described in the previous section. Next, the theoretical density was calculated by

350 dividing the total mass by the theoretical volume of the mixtures (see Table 2 and Table 3). With
351 these data, the air content was calculated following Eq. 34.

$$352 \quad AC [\%] = \frac{\rho_{\text{theo}} [kg/dm^3] - \rho_{\text{fr}} [kg/dm^3]}{\rho_{\text{theo}} [kg/dm^3]} \cdot 100 \quad \text{Eq. 34}$$

353 being AC the air content, ρ_{theo} the theoretical density, and ρ_{fr} , the fresh density.

354

355 3.2.4 Mechanical strength

356 Despite the mortars investigated will not be used for structural purposes, the flexural and
357 compressive strength of the samples have been measured according to Standard EN 1015-11
358 [43], with the aim of assessing the order of magnitude of these properties and the applicability of
359 the mortars for practical uses.

360

361 **3.3 Thermal properties**

362 To assess the thermal properties of the samples, a Decagon Devices KD2 PRO instrument
363 equipped with two different probes (TR-1 and SH-1) was used [44]. This apparatus uses
364 transient line heat source methods to measure thermal properties, as described in the following
365 sections. A critical issue for accurate measurements by means of this method is to ensure a
366 good thermal contact between the sensor and the material, in order to minimise the thermal
367 contact resistance. With this aim, pilot holes were bored in fresh mixes by using pilot pins
368 having exactly the same diameter as the measuring needles. Pilot pins were firstly coated with a
369 thin film of release-agents and then were installed in the centre of fresh mortar samples as
370 shown in Figure 1. Once hardened, pins were removed, sensors were coated with thermal
371 grease, inserted into the cast hole and readings started to be taken. During the measurement,
372 the temperature of the samples was kept as constant as possible and 15 minutes between each
373 measurement were allowed for temperatures to equilibrate. The measurement time was set to
374 10 minutes for most accurate results according to the manufacturer instructions, minimising
375 errors caused by the large diameter needle and the contact resistance between the sensor and
376 the sample.

377

379 3.3.1 Thermal conductivity

387 To assess the thermal conductivity of the samples, the Decagon Devices KD2 PRO was
388 equipped with the single needle TR-1 sensor [44]. This probe consists of a needle with a heater
389 and a temperature sensor inside. A constant current passes through the heater and the system
390 monitors the temperature of the sensor over time by using a high-resolution temperature
391 sensor. Specific algorithms are applied to analyse measurements made during heating and
392 cooling intervals and to compute thermal conductivity [44]. According to the manufacturer
393 specifications, the accuracy of the single needle TR-1 sensor (2.4 mm diameter x 10 cm long) in
394 the conductivity range from 0.2 to 4.0 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ is $\pm 10\%$.
388



390 Figure 1. Mortar samples curing with the pilot pins inserted inside (left) and thermal conductivity
391 measurement on hardened samples with the Decagon Devices KD2 PRO (right).

391

392 3.3.2 Thermal diffusivity

393 To measure the thermal diffusivity of the mortars, Decagon Devices KD2 PRO instrument was
394 equipped with the dual needle SH-1 probe. The operation of this sensor is similar to the single
395 needle, but in this case the heater and temperature sensors are placed in separate needles.
396 Heat is applied to the heated needle for a set heating time and the temperature is measured in
397 the monitoring needle 6 mm distant during the heating and the following cooling period.
398 Readings are then processed taking into account the ambient temperature registered at the
399 beginning of the test and the heat per unit length provided by the heating needle. Different
400 equations are applied during the heating and cooling periods to fit three constants and compute
401 the thermal diffusivity [44]. According to the manufacturer specifications, the accuracy of the
402 dual needle SH-1 sensor (1.3 mm diameter x 3 cm long, 6 mm spacing) in measuring diffusivity
403 of materials with conductivity higher than $0.1 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ is $\pm 10\%$.

403

404 Another output data of the measurements performed using SH-1 sensor is the volumetric heat
405 capacity, that was also measured in order to calculate the thermal effusivity as explained in the
406 following section. The accuracy of the probe in the determination of the volumetric heat capacity
407 of materials with conductivity higher than $0.1 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ is $\pm 10\%$.

408

409 3.3.3 Thermal effusivity

410 Thermal effusivity was calculated as the square root of the product of thermal conductivity and
411 volumetric heat capacity. Thermal conductivity was assessed using probe TR-1 as explained in
412 Section 3.3.1 and volumetric heat capacity was measured using probe SH-1. Volumetric heat
413 capacity was also used to calculate the specific heat capacity, as the ratio between volumetric
414 heat capacity and density.

415

416 4. Results

417 4.1 Step 1 - Aggregate and antifoaming effects - 2x2 factorial design

418 4.1.1 Density

419 A relevant aspect to be considered is how the fine aggregates and AF agent affect the density
420 of the mixture, being density an important parameter for the thermal properties of the material.
421 Table 4(a) shows the density values of the different mixtures, with three replications for each
422 aggregate-AF combination. It can be seen that the higher values are obtained with barite
423 aggregates and AF admixture ($1.93\text{-}1.94 \text{ kg dm}^{-3}$), whereas when using silica without AF the
424 density ranges between 1.31 kg dm^{-3} and 1.34 kg dm^{-3} . For both aggregates, using AF agent
425 produces an increase in the density of the mixture.

426

427 Table 4. Density values of the samples analysed (a) and summary output from the two-way ANOVA (b). It
428 consists of the Sum of Squares (SS), the Degree of Freedom (DF), the Mean Squares (MS), the F-value
429 (F), the associated p-value and the F critical.

Density [kg dm^{-3}]		Two-way ANOVA						
(a)		(b)						
	Fine aggregates	Source of variation	SS	DF	MS	F	p-value	F crit.

	Silica	Barite	Antifoaming	0.088	1	0.088	838.8	2.19E-09	5.32
W/ Antifoaming	1.55	1.93	Aggregates	0.584	1	0.584	5568.1	1.16E-12	5.32
	1.53	1.93	Interaction	0.006	1	0.006	58.7	5.95E-05	5.32
	1.54	1.94	Within	0.001	8	0.000			
W/O Antifoaming	1.34	1.81							
	1.31	1.80	Total	0.679	11				
	1.31	1.82							

430

431 Two-way ANOVA statistical test reported in Table 4(b) allows concluding that the AF, the
432 aggregates and the interaction between them significantly affect the density of the mortars. In
433 fact, all the p-values are lower than the significance level (0.05), so the null hypotheses is
434 rejected (there are not statistically significant differences in density) and it is concluded with
435 95% confidence that the use of AF, different aggregates, and interaction between them
436 produces mortars statistically different in density.

437

438 4.1.2 Air content

439 Air content of the mixtures is shown in Table 5(a). It can be clearly noticed the effect of the AF
440 admixture, that provides a substantial reduction in the air content of the mixtures. In fact, the air
441 content is reduced from about 20% to 6% and from 15% to 9% when silica or barite aggregates
442 are respectively used. In this sense, it can be observed that using common silica aggregates in
443 the formulation of a mortar with PCM without AF admixtures produces very high air contents
444 (about 20%), with all the limitations that this implies in terms of high porosity and low
445 conductivity as it will be discussed in the next section. When barite is used, the same amount of
446 PCM produces lower air contents (about 14%), however the effectiveness of the AF seems to
447 be higher when silica aggregates are used in comparison with mixes based on barite.

448

449 Table 5. Air content values of the samples analysed (a) and summary output from the two-way ANOVA (b)

Air content [%]			Two-way ANOVA						
(a)			(b)						
	Fine aggregates		Source of variation	SS	DF	MS	F	p-value	F crit.
	Silica	Barite	Antifoaming	268.8	1	268.8	786.20	2.83E-09	5.32
W/ Antifoaming	5.8	8.7	Aggregates	6.1	1	6.1	17.71	2.93E-03	5.32

	6.8	8.6	Interaction	38.9	1	38.9	113.71	5.24E-06	5.32
	6.6	8.4	Within	2.7	8	0.3			
W/O Antifoaming	18.5	14.3							
	19.9	15.1	Total	316.5	11				
	20.1	14.0							

450

451 Two-way ANOVA shows that also in this case AF, aggregates, and interaction between them
452 produce mortars with statistically significant differences in the air content, being the p-values
453 lower than 0.05 in all the cases.

454

455 4.1.3 Thermal conductivity

456 Higher thermal conductivity values are obtained using silica aggregates with AF admixture as
457 shown in Table 6(a). In general, it can be observed that the use of silica aggregates seems to
458 be more favourable in maximizing this parameter, probably because this type of aggregate itself
459 has higher thermal conductivity than the barite. In fact, thermal conductivity values for silica-
460 based mortars with high air content (about 20%, without AF) are still higher than those of barite
461 based mixtures with lower air content (about 8%, with AF).

462

463 From a statistical point of view (Table 6(b)), the two independent variables (aggregates, AF)
464 produce a statistically significant difference in the thermal conductivity, being the p-values of the
465 main factors AF and aggregates lower than 0.05 in both cases. However, in this case the
466 interaction term has a p-value higher than the significance level ($p\text{-value}=0.32 > 0.05=\alpha$), so the
467 null hypothesis cannot be rejected, hence there are no significant differences in the interaction
468 between aggregates and AF agent. This means that the effect of the AF admixture on the
469 thermal conductivity is the same for both using silica and barite aggregates (and vice versa).

470

471 Table 6. Thermal conductivity values of the samples analysed (a) and summary output from the two-way
472 ANOVA (b)

Thermal conductivity [$\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$]		Two-way ANOVA						
(a)		(b)						
	Fine aggregates	Source of variation	SS	DF	MS	F	p-value	F crit.

	Silica	Barite	Antifoaming	0.095	1	0.095	49.68	1.07E-04	5.32
W/ Antifoaming	1.09	0.79	Aggregates	0.170	1	0.170	89.45	1.28E-05	5.32
	1.03	0.83	Interaction	0.002	1	0.002	1.14	3.18E-01	5.32
	1.08	0.77	Within	0.015	8	0.002			
W/O Antifoaming	0.88	0.65							
	0.92	0.67	Total	0.282	11				
	0.78	0.62							

473

474

475

476 4.1.4 Thermal diffusivity

477 Thermal diffusivity values range between 0.20 to 0.23 mm² s⁻¹ for the mortar with silica

478 aggregates and AF, and between 0.14 and 0.15 mm²s⁻¹ for the mortar with barite without AF

479 agent, as shown in Table 7(a). This means that the first one allows a 30% faster heat

480 penetration than the second one, reducing the time required to activate the element. Two-way

481 ANOVA shows that the independent variables cause a statistically significant difference in the

482 thermal diffusivity; however the interaction term has a p-value higher than the significance level

483 (p-value=0.80 > 0.05=α), meaning that the effect of the admixture on the thermal conductivity is

484 the same for both types of fine aggregates (Table 7(b)).

485

486 Table 7. Thermal diffusivity values of the samples analysed (a) and summary output from the two-way

487 ANOVA (b)

Thermal diffusivity [mm ² ·s ⁻¹]			Two-way ANOVA						
(a)			(b)						
	Fine aggregates		Source of variation	SS	DF	MS	F	p-value	F crit.
	Silica	Barite	Antifoaming	0.005	1	0.005	15.46	4.34E-03	5.32
W/ Antifoaming	0.23	0.19	Aggregates	0.004	1	0.004	11.64	9.20E-03	5.32
	0.22	0.19	Interaction	0.000	1	0.000	0.07	8.01E-01	5.32
	0.20	0.17	Within	0.003	8	0.000			
W/O Antifoaming	0.21	0.14							
	0.17	0.15	Total	0.011	11				
	0.15	0.14							

488

489 4.1.5 Thermal effusivity

490 Also in terms of thermal effusivity, the better performance mortar is obtained using silica
 491 aggregates with AF admixture as shown in Table 8(a). In particular, this combination in average
 492 outperforms the other solutions by 25% (barite w/o AF), 19% (barite w/ AF), and 11% (silica w/o
 493 AF). This result can be explained taking into account that thermal effusivity combines thermal
 494 conductivity with density and specific heat capacity. Mixtures based on silica have relatively
 495 high thermal conductivity and low density, whereas mixtures based on barite present low
 496 conductivity and high density, as shown in Table 4 and Table 6. If the mixtures w/ AF are
 497 analysed, first, the product of the average conductivity by the average density is almost
 498 constant being $1639 \text{ W}\cdot\text{kg}\cdot\text{m}^{-4}\cdot\text{K}$ for silica aggregates and $1547 \text{ W}\cdot\text{kg}\cdot\text{m}^{-4}\cdot\text{K}$ for barite
 499 aggregates. However, the specific heat capacity of the mortar is considerably much higher
 500 when silica instead of barite is used, with average values of $3194 \text{ J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ for the first one and
 501 $2235 \text{ J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ for the second one. The same occurs with mixes w/o AF: the product of
 502 conductivity by density is constant, with values of $1138 \text{ W}\cdot\text{kg}\cdot\text{m}^{-4}\cdot\text{K}$ and $1174 \text{ W}\cdot\text{kg}\cdot\text{m}^{-4}\cdot\text{K}$ for
 503 silica and barite, respectively, but the specific heat capacity is about $3671 \text{ J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ for silica
 504 and only $2528 \text{ J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ for mortar with barite. The overall result is that mortars based on silica
 505 outperform the others option in terms of thermal effusivity, regardless the use of AF admixture.

506
 507 Table 8. Thermal effusivity values of the samples analysed (a) and summary output from the two-way
 508 ANOVA (b)

Thermal effusivity [$\text{W}\cdot\text{s}^{0.5}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$]			Two-way ANOVA						
(a)			(b)						
	Fine aggregates		Source of variation	SS	DF	MS	F	p-value	F crit.
	Silica	Barite	Antifoaming	1.1E+05	1	1.1E+05	10.58	1.1E-02	5.32
W/ Antifoaming	2289	1824	Aggregates	4.1E+05	1	4.1E+05	39.35	2.4E-04	5.32
	2172	1903	Interaction	9.4E+03	1	9.4E+03	0.89	3.7E-01	5.32
	2400	1850	Within	8.4E+04	8	1.1E+04			
	1904	1755							
W/O Antifoaming	2203	1766	Total	6.2E+05	11				
	2008	1647							

509

510 A two-way ANOVA examined the effect of antifoaming and aggregates on thermal effusivity,
 511 showing at 95% confidence that both AF and aggregates result in statistically significant
 512 difference, with the p- values of the main factors lower than 0.05. The interaction, however,
 513 does not have a statistically significant effect on the effusivity, meaning that the effect of the
 514 aggregates on this parameter is the same for either using or not the AF admixture (and vice
 515 versa).

516
 517
 518

519 **4.2 Step 2 - PCM content effect – One-way ANOVA**

520 4.2.1 Density

521 As stated in the methodology section, the second step of the study focused on the effects
 522 produced by different amounts of PCM on the properties of the mortars. The first parameter
 523 analysed was the density of the mortars in the hardened state, as it can be seen in Table 9(a).
 524 As expected, for increasing PCM contents density decreases, ranging between 1.72 and 1.75
 525 kg dm⁻³ for mixtures with 20% PCM and 1.44-1.46 kg dm⁻³ for mortars with 32% PCM. In order
 526 to establish whether there is a statistically significant difference between the group means, a
 527 one-way ANOVA analysis was conducted as shown in Table 9(b). The significance value (i.e.
 528 the p-value = 1.8E-13) is below 0.05 and therefore it can be concluded that there is a
 529 statistically significant difference in the mean density between the different mortars. However,
 530 ANOVA does not provide any information about pairwise differences between groups but only
 531 demonstrates that there are statistically significant differences between the groups as a whole.
 532 So, a Tukey-Kramer multiple comparisons post-hoc test was performed to show which mortars
 533 differed from each other in terms of density [45]. The output of Tukey-Kramer test revealed that
 534 density is statistically significantly between all the mixtures (Table 10).

535

536 Table 9. Density values of the samples analysed (a) and summary output from the one-way ANOVA (b).

Density [kg·dm ⁻³]				One-way ANOVA						
(a)				(b)						
PCM	PCM	PCM	PCM	Source of Variation	SS	DF	MS	F	p-value	F crit.

20%	24%	28%	32%							
1.75	1.65	1.55	1.45	Between Groups	0.175	3	5.84E-02	633.8	1.78E-13	3.49
1.72	1.64	1.56	1.44	Within Groups	0.001	12	9.22E-05			
1.72	1.64	1.55	1.46	Total	0.176	15				
1.73	1.66	1.55	1.45							

537

538

539

540

541

542 Table 10. Tukey–Kramer statistical test applied to find mixtures that have significantly different densities
543 from each other.

Tukey-Kramer Multiple Comparisons			
Comparison	Absolute difference	Critical range	Result
20% to 24%	0.082	0.020	Different
20% to 28%	0.180	0.020	Different
20% to 32%	0.279	0.020	Different
24% to 28%	0.097	0.020	Different
24% to 32%	0.197	0.020	Different
28% to 32%	0.100	0.020	Different

544

545 4.2.2 Air content

546 Table 11(a) shows the air-content values of the mortars. As it can be seen, the air content
547 increases when higher amounts of PCM are added into the mix. In fact, mortars with 20% PCM
548 have values in the range of 2%-3%, whereas in the case of 32% PCM the air content raises up
549 to 8%-9%, despite the same quantity of AF admixture has been used for all the mixtures. Also
550 for this parameter, the ANOVA analysis shows that the probability that the differences of the
551 mean air content values are due to chance is less than the statistical significance at 0.05 (p-
552 value = 1.4E-07), so the null hypothesis that all the mixes have the same air content can be
553 rejected and the alternative hypothesis that they have not (Table 11(b)) can be accepted. A
554 Tukey-Kramer post hoc test revealed that the air content was statistically significantly different
555 between all the mortars with the exception of mortars D (20% PCM) and C (24% PCM), being in
556 this case the absolute difference lower than the critical range (Table 12).

557 Table 11. Air content values of the samples analysed (a) and summary output from the one-way ANOVA
 558 (b).

Air content [%] (a)				One-way ANOVA (b)						
PCM 20%	PCM 24%	PCM 28%	PCM 32%	Source of Variation	SS	DF	MS	F	p-value	F crit.
1.6	3.3	5.6	8.1	Between Groups	69.18	3	23.06	62.31	1.38E-07	3.49
3.5	4.2	5.3	9.0	Within Groups	4.44	12	0.37			
3.3	4.1	5.8	7.9	Total	73.62	15				
3.3	3.2	6.1	8.2							

559
 560 Table 12. Tukey–Kramer statistical test used to find mixtures that have significantly different air contents
 561 from each other.

Tukey-Kramer Multiple Comparisons			
Comparison	Absolute difference	Critical range	Result
20% to 24%	0.791	1.278	Not Different
20% to 28%	2.791	1.278	Different
20% to 32%	5.383	1.278	Different
24% to 28%	1.999	1.278	Different
24% to 32%	4.591	1.278	Different
28% to 32%	2.592	1.278	Different

562
 563
 564 4.2.3 Mechanical strength
 565 The flexural strength of the mortars ranges between 3.4 MPa and 4.2 MPa decreasing as the
 566 PCM content increases. Compressive strength follows the same pattern ranging from 11.5 MPa
 567 to 13.6 MPa. These values are in accordance with no structural mortars, suggesting that the
 568 PCM-added mortars are suitable for practical uses.

569
 570 4.2.3 4.2.4 Thermal conductivity

571 Since density decreases and air-content increases for increasing amounts of PCM, it is
 572 expected that the thermal conductivity will also decrease. Table 13(a) shows that effectively
 573 there is an inverse relationship between PCM content and conductivity, being the conductivity
 574 for mortars with 20% PCM in the range of 1.17-1.27 W·m⁻¹·K⁻¹ and for mixtures with 32% PCM

575 between $0.93 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ and $1.11 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$. ANOVA shows that the within-sample variance is
 576 lower than the between-sample variance (Table 13(b)), meaning that the thermal conductivity
 577 variation among mortars is largely caused by actual different thermal behaviours, rather than
 578 chance variation ($p\text{-value} = 0.016 < 0.05 = \alpha$).

579

580 However, it is worth mentioning that in this case, even though the p-value is still lower than the
 581 level of significance, it is not as low as in the previous cases. This result is highlighted by the
 582 Tukey-Kramer post hoc test that shows that the thermal conductivity is statistically significantly
 583 different only between mortar D (20% PCM) and A (32% PCM), and between mortar C (24%
 584 PCM) and A (32% PCM), as shown in Table 14. This means that according to the experiment
 585 performed and equipment used in the characterization, using 32% PCM in the mortar mixture
 586 provides statistically different thermal conductivities with respect to using 20% or 24% PCM,
 587 while all the other combinations of mixtures statistically have the same conductivity.

588

589 Table 13. Thermal conductivity at 20°C (a) and summary output from the one-way ANOVA (b).

Thermal conductivity at 20°C [$\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$]				One-way ANOVA						
(a)				(b)						
PCM 20%	PCM 24%	PCM 28%	PCM 32%	Source of Variation	SS	DF	MS	F	p-value	F crit.
1.20	1.14	1.20	1.02	Between Groups	0.107	3	0.036	5.12	0.016	3.49
1.27	1.21	1.01	1.01	Within Groups	0.084	12	0.007			
1.17	1.20	0.96	1.11	Total	0.191	15				
1.24	1.26	1.23	0.93							

590

591 Table 14. Tukey–Kramer statistical test used to find mixtures with significantly different thermal
 592 conductivity values.

Tukey-Kramer Multiple Comparisons			
Comparison	Absolute difference	Critical range	Result
20% to 24%	0.015	0.175	Not Different
20% to 28%	0.119	0.175	Not Different
20% to 32%	0.201	0.175	Different
24% to 28%	0.105	0.175	Not Different
24% to 32%	0.187	0.175	Different

28% to 32%	0.082	0.175	Not Different
------------	-------	-------	---------------

593

594 | 4.2.44.2.5 Thermal diffusivity

595 Thermal diffusivity decreases for increasing PCM contents, as shown in Table 16(a). Values
596 range between 0.243 and 0.292 mm²·s⁻¹ for mortars with 20% PCM and between 0.158 to 0.198
597 mm²·s⁻¹ for mortars with 32% PCM. Taking into account that for increasing amounts of PCM
598 both the average thermal conductivity and the average density of the mortars decrease linearly
599 and approximately with the same slope, it can therefore be concluded that the ratio between
600 thermal conductivity and density is almost constant. Thus, the decreasing thermal diffusivity
601 means that, as expected, the thermal capacity of the mixture increases with the PCM content.
602 ANOVA shows that the differences between the mortars in terms of thermal diffusivity are in
603 general statistically significant (p-value =0.002, Table 16(b)), and the Tukey–Kramer test points
604 out that introducing 32% PCM in the mortar causes a statistically different thermal diffusivity
605 from mixes incorporating 20% or 24% PCM.

606

607 Table 15. Thermal diffusivity at 20°C values (a) and summary output from the one-way ANOVA (b).

Thermal diffusivity at 20°C [mm ² ·s ⁻¹]				One-way ANOVA						
(a)				(b)						
PCM 20%	PCM 24%	PCM 28%	PCM 32%	Source of Variation	SS	DF	MS	F	p-value	F crit.
0.292	0.242	0.247	0.198	Between Groups	0.017	3	5.50E-03	8.85	0.002	3.49
0.273	0.263	0.206	0.198	Within Groups	0.007	12	6.22E-04			
0.276	0.243	0.179	0.195	Total	0.024	15				
0.243	0.274	0.262	0.158							

608

609 Table 16. Tukey–Kramer statistical test performed to find mixtures with significantly different thermal
610 diffusivity values.

Tukey–Kramer Multiple Comparisons			
Comparison	Absolute difference	Critical range	Result
20% to 24%	0.016	0.052	Not Different
20% to 28%	0.048	0.052	Not Different
20% to 32%	0.084	0.052	Different
24% to 28%	0.032	0.052	Not Different

24% to 32%	0.068	0.052	Different
28% to 32%	0.036	0.052	Not Different

611

612 | 4.2.54.2.6 Thermal effusivity

613 Thermal effusivity ranges between 2200 and 2500 $W \cdot s^{0.5} \cdot m^{-2} \cdot K^{-1}$ and it does not vary
614 substantially with the PCM content (Table 18). For this parameter, the ANOVA analysis shows
615 that the p-value is higher than the significance level, so there is no statistically significant
616 evidence to conclude that there is a difference in the thermal effusivity for the mortars
617 considered. In fact, the thermal effusivity is practically constant for all mortars, with an average
618 value of 2347 $W \cdot s^{0.5} \cdot m^{-2} \cdot K^{-1}$ and a very low standard deviation of 23 $W \cdot s^{0.5} \cdot m^{-2} \cdot K^{-1}$.

619

620 This result can be explained taking into account that the thermal effusivity is defined as the root
621 square of the product between density, conductivity, and specific heat. In this case, for
622 increasing amounts of PCM, the reduction in density and conductivity is compensated by the
623 increase in the specific heat of the mixture.

624

625 Table 17. Thermal effusivity at 20°C values (a) and summary output from the one-way ANOVA
626 (b).

Thermal effusivity at 20°C [$W \cdot s^{0.5} \cdot m^{-2} \cdot K^{-1}$]				One-way ANOVA						
(a)				(b)						
PCM 20%	PCM 24%	PCM 28%	PCM 32%	Source of Variation	SS	DF	MS	F	p- value	F crit.
2214	2316	2413	2287	Between Groups	6648	3	2216	0.190	0.901	3.49
2424	2361	2219	2262	Within Groups	140086	12	11674			
2221	2430	2258	2512	Total	146734	15				
2512	2402	2392	2335							

627

628

629 5. Conclusions

630 The presented study describes the experimental campaign performed to characterise the
631 thermal properties of novel mortars incorporating microencapsulated PCM designed to be used
632 as the internal layer in thermally active precast walls. For this specific application, the layer is

633 activated by a water pipe in order to store and/or exchange heat with the indoor environment
634 and the HVAC system depending on the operating conditions. Therefore, the PCM-added
635 mortar is required to have a good balance between thermal diffusivity, that measures how
636 quickly the heat can penetrate the material under unsteady conditions (and consequently the
637 heat penetration depth at a given time) and thermal effusivity, that describes the rate of thermal
638 energy transfer on the material surface and thus the material ability to exchange thermal energy
639 with its surroundings.

640

641 The characterization has been developed in two steps: in the first one, a full factorial experiment
642 focused on the effects of using different fine aggregates (silica and barite) and antifoaming
643 admixture on the characteristics of the mortars, keeping the PCM content constant. In the
644 second step, the effect of the PCM amount introduced in the mixtures (from 20% to 32% in the
645 total volume of the mixture) was assessed, reducing proportionally the fine aggregates and
646 keeping constant all the others components (i.e. cement, water, superplasticizer, and
647 antifoaming admixture).

648

649 Results show that the combination consisting in using silica fine aggregate and an antifoaming
650 admixture outperform the other options in terms of thermal properties, producing mortars with
651 statistically significant higher values of conductivity, diffusivity and effusivity. The use of heavy
652 aggregates like barite produces a significant increase in the mortar density (of about 25% and
653 37% in the case of using or not the antifoaming admixture, respectively); however this potential
654 advantage does not result in better thermal properties, since the decrease in conductivity
655 produced by this aggregate exceeds the increase in density, in comparison with silica-based
656 mortars.

657

658 Increasing amounts of microencapsulated PCM produce a statistically significant density
659 reduction not only due to the direct effect of replacing heavier mix component by lighter one, but
660 also because the trapped air increases when increasing amounts of PCM are introduced. This
661 makes that density falls from about 1.7 kg dm^{-3} for mortars with 20% PCM (3% trapped air) to
662 about 1.4 kg dm^{-3} for mortars with 32% PCM (8% trapped air). Concerning thermal properties,

663 conductivity and diffusivity decreases when increasing PCM content. In particular, moving from
664 20% to 32% PCM reduces the thermal conductivity from $1.2 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ to $1.0 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ (17%
665 reduction) and the diffusivity from $0.27 \text{ mm}^2\cdot\text{s}^{-1}$ to $0.19 \text{ mm}^2\cdot\text{s}^{-1}$ (31% reduction), meaning that
666 the heat will penetrate 30% faster in the 20% PCM mortar in comparison with the mortar
667 incorporating 32% PCM. Other than that, differences between mortars with similar PCM content
668 are not very clear without performing a detailed statistical analysis. In this case, one-way
669 ANOVA and Tukey-Kramer post hoc test have led to the conclusion that both thermal
670 conductivity and diffusivity are statistically significantly different only between the mortar with the
671 higher PCM content (32%) and mortars with 24% and 20% PCM. For all the other combinations,
672 the results obtained do not allow to conclude that the differences are statistically significant.
673 Finally, the study of the thermal effusivity has revealed that for increasing amounts of PCM,
674 density and conductivity reduction is counterbalanced by the increase of the specific heat
675 capacity, concluding that effusivity is practically invariant to the PCM content, with an average
676 value of about $2300 \text{ W}\cdot\text{s}^{0.5}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$ and a coefficient of variation lower than 1%. This result
677 implies that when increasing the amount of PCM the energy storage potential is enhanced, but
678 the thermal inertia presents an upper limit, due to the relevant decrease of both thermal
679 conductivity and density. Moreover, a constant effusivity means that the internal surface of the
680 wall in contact with the indoor air will allow the same heat flux for all the mortars. This suggests
681 that the choice of the most suitable material for this application has to be done by predominantly
682 focusing on diffusivity, in order to define the most appropriate heat penetration rates and
683 activation times for an efficient operation of the precast radiant system.

684

685 Therefore, further experimental studies on small and full-scale models should be conducted in
686 order to assess the charging and discharging rates and the energy storage potential of the
687 latent heat storage system under dynamic operating conditions. Moreover, the same
688 methodology could be applied to different finishing mortar mixtures without PCM including
689 aggregates with different properties.

690

691

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699

700 **7. References**

- 701 [1] Eurostat, Energy balance sheets. 2015 Data. 2017 Edition, 2017. doi:10.2785/52802.
- 702 [2] M. Santamouris, Innovating to zero the building sector in Europe: Minimising the energy
703 consumption, eradication of the energy poverty and mitigating the local climate change,
704 Sol. Energy. 128 (2016) 61–94. doi:10.1016/j.solener.2016.01.021.
- 705 [3] European Commission, Directive 2010/31/EU of the European Parliament and of the
706 Council of 19 May 2010 on the energy performance of buildings, Off. J. Eur. Union,
707 L153. (2010) 13–35.
- 708 [4] European Commission, Directive 2012/27/EU of the European Parliament and of the
709 Council of 25 October 2012 on energy efficiency. Off J Eur Union 2012, L315: 1-56.,
710 (2012) 1–56.
- 711 [5] CTE, Código Técnico de la Edificación. Boletín Oficial del Estado, BOE 12/09/13,
712 Madrid, Spain; 2013, (2013). <http://www.codigotecnico.org> (accessed October 11, 2017).
- 713 [6] Energy efficiency trends and policies in the household and tertiary sectors. An analysis
714 based on the ODYSSEE and MURE databases, 2015. [http://www.odyssee-](http://www.odyssee-mure.eu/publications/br/Buildings-brochure-2012.pdf)
715 [mure.eu/publications/br/Buildings-brochure-2012.pdf](http://www.odyssee-mure.eu/publications/br/Buildings-brochure-2012.pdf).
- 716 [7] P. Arce, M. Medrano, A. Gil, E. Oró, L.F. Cabeza, Overview of thermal energy storage
717 (TES) potential energy savings and climate change mitigation in Spain and Europe,
718 Appl. Energy. 88 (2011) 2764–2774. doi:10.1016/j.apenergy.2011.01.067.
- 719 [8] A. de Gracia, L.F. Cabeza, Phase change materials and thermal energy storage for
720 buildings, Energy Build. 103 (2015) 414–419. doi:10.1016/j.enbuild.2015.06.007.
- 721 [9] J. Heier, C. Bales, V. Martin, Combining thermal energy storage with buildings – a

- 722 review, *Renew. Sustain. Energy Rev.* 42 (2015) 1305–1325.
723 doi:10.1016/j.rser.2014.11.031.
- 724 [10] L. Navarro, A. de Gracia, S. Colclough, M. Browne, S.J. McCormack, P. Griffiths, L.F.
725 Cabeza, Thermal energy storage in building integrated thermal systems: A review. Part
726 1. active storage systems, *Renew. Energy.* 88 (2016) 526–547.
727 doi:10.1016/j.renene.2015.11.040.
- 728 [11] D. Saelens, W. Parys, R. Baetens, Energy and comfort performance of thermally
729 activated building systems including occupant behavior, *Build. Environ.* 46 (2011) 835–
730 848. doi:10.1016/j.buildenv.2010.10.012.
- 731 [12] J. Romaní, A. de Gracia, L.F. Cabeza, Simulation and control of Thermally Activated
732 Building Systems (TABS), *Energy Build.* 127 (2016) 22–42.
733 doi:10.1016/j.enbuild.2016.05.057.
- 734 [13] S.P. Corngati, A. Kindinis, Thermal mass activation by hollow core slab coupled with
735 night ventilation to reduce summer cooling loads, *Build. Environ.* 42 (2007) 3285–3297.
736 doi:10.1016/j.buildenv.2006.08.018.
- 737 [14] G.P. Henze, C. Felsmann, D.E. Kalz, S. Herkel, Primary energy and comfort
738 performance of ventilation assisted thermo-active building systems in continental
739 climates, *Energy Build.* 40 (2008) 99–111. doi:10.1016/j.enbuild.2007.01.014.
- 740 [15] G. Salvalai, J. Pfafferott, M.M. Sesana, Assessing energy and thermal comfort of
741 different low-energy cooling concepts for non-residential buildings, *Energy Convers.*
742 *Manag.* 76 (2013) 332–341. doi:10.1016/j.enconman.2013.07.064.
- 743 [16] M. Hunger, A.G. Entrop, I. Mandilaras, H.J.H. Brouwers, M. Founti, The behavior of self-
744 compacting concrete containing micro-encapsulated Phase Change Materials, *Cem.*
745 *Concr. Compos.* 31 (2009) 731–743. doi:10.1016/j.cemconcomp.2009.08.002.
- 746 [17] A. Jayalath, R. San Nicolas, M. Sofi, R. Shanks, T. Ngo, L. Aye, P. Mendis, Properties of
747 cementitious mortar and concrete containing micro-encapsulated phase change
748 materials, *Constr. Build. Mater.* 120 (2016) 408–417.
749 doi:10.1016/j.conbuildmat.2016.05.116.
- 750 [18] M. Pomianowski, P. Heiselberg, R.L. Jensen, Dynamic heat storage and cooling
751 capacity of a concrete deck with PCM and thermally activated building system, *Energy*

- 752 Build. 53 (2012) 96–107. doi:10.1016/j.enbuild.2012.07.007.
- 753 [19] J. Fořt, A. Trník, L. Scheinherrová, Z. Pavlík, M. Pavlíková, R. Černý, Thermal
754 Properties of Cement Based Composites with Municipal Solid Waste Incinerator Fly Ash
755 Accessed by Two Different Transient Methods, 22 (2016) 132–137.
756 doi:http://dx.doi.org/10.5755/j01.ms.22.1.7203.
- 757 [20] J.A. Clarke, *Energy Simulation in Building Design*, Butterworth-Heinemann, 2001.
- 758 [21] J. Košny, PCM-Enhanced Building Components, in: *PCM-Enhanced Build. Components*,
759 2015: p. 281. doi:10.1007/978-3-319-14286-9.
- 760 [22] V. V. Tyagi, S.C. Kaushik, S.K. Tyagi, T. Akiyama, Development of phase change
761 materials based microencapsulated technology for buildings: A review, *Renew. Sustain.*
762 *Energy Rev.* 15 (2011) 1373–1391. doi:10.1016/j.rser.2010.10.006.
- 763 [23] Y. Konuklu, M. Ostry, H.O. Paksoy, P. Charvat, Review on using microencapsulated
764 phase change materials (PCM) in building applications, *Energy Build.* 106 (2015) 134–
765 155. doi:10.1016/j.enbuild.2015.07.019.
- 766 [24] P. Meshgin, Y. Xi, Effect of Phase-Change Materials on Properties of Concrete, *ACI*
767 *Mater. J.* 109 (2012). doi:10.14359/51683572.
- 768 [25] P.K. Dehdezi, M.R. Hall, A.R. Dawson, S.P. Casey, Thermal, mechanical and
769 microstructural analysis of concrete containing microencapsulated phase change
770 materials, *Int. J. Pavement Eng.* 14 (2013) 449–462.
771 doi:10.1080/10298436.2012.716837.
- 772 [26] Z. Pavlík, A. Trník, J. Ondruška, M. Keppert, M. Pavlíková, P. Volfová, V. Kaulich, R.
773 Černý, Apparent Thermal Properties of Phase-Change Materials: An Analysis Using
774 Differential Scanning Calorimetry and Impulse Method, *Int. J. Thermophys.* (2012) 851–
775 864. doi:10.1007/s10765-012-1169-1.
- 776 [27] L. Navarro, A. de Gracia, D. Niall, A. Castell, M. Browne, S.J. McCormack, P. Griffiths,
777 L.F. Cabeza, Thermal energy storage in building integrated thermal systems: A review.
778 Part 2. Integration as passive system, *Renew. Energy.* 85 (2016) 1334–1356.
779 doi:10.1016/j.renene.2015.06.064.
- 780 [28] D. Kalz, J. Pfaffert, P. Schossig, S. Herkel, Thermally Activated Building Systems
781 Using Phase-Change-Materials, *Proc. of EUROSUN.* (2006) 1–6.

- 782 http://www.celsius.co.kr/phase_change_materials/download/energy/Thermally_Activated
783 [_Building_Systems_Using_PCMS.pdf](#).
- 784 [29] G.K. Pavlov, B.W. Olesen, M. Skrupskelis, O.B. Kazanci, G.K. Pavlov, B.W. Olesen, M.
785 Skrupskelis, O.B. Kazanci, Ground source heat pump combined with thermo-active
786 building system with incorporated PCM for low-energy residential house, Proc. Innostock
787 2012 12th Int. Conf. Energy Storage. (2012).
- 788 [30] L. Hyun-Hwa, K. Yoon-Ji, L. Jae-Han, S. Kim, S. Seung-Yeong, Evaluation of the
789 thermal performance of a Thermally Activated Building System (TABS) using Phase-
790 Change Materials, in: ASim2014 Proc., IBPSA Journal of Building Performance
791 Simulation, Nagoya, Japan, 2014: pp. 411–418. http://www.ibpsa.org/?page_id=564.
- 792 [31] M. Koschenz, B. Lehmann, Development of a thermally activated ceiling panel with PCM
793 for application in lightweight and retrofitted buildings, Energy Build. 36 (2004) 567–578.
794 doi:10.1016/j.enbuild.2004.01.029.
- 795 [32] X. Jin, X. Zhang, Thermal analysis of a double layer phase change material floor, Appl.
796 Therm. Eng. 31 (2011) 1576–1581. doi:10.1016/j.applthermaleng.2011.01.023.
- 797 [33] R. Ansuini, R. Larghetti, A. Giretti, M. Lemma, Radiant floors integrated with PCM for
798 indoor temperature control, Energy Build. 43 (2011) 3019–3026.
799 doi:10.1016/j.enbuild.2011.07.018.
- 800 [34] M. Pomianowski, P. Heiselberg, R.L. Jensen, R. Cheng, Y. Zhang, A new experimental
801 method to determine specific heat capacity of inhomogeneous concrete material with
802 incorporated microencapsulated-PCM, Cem. Concr. Res. 55 (2014) 22–34.
803 doi:10.1016/j.cemconres.2013.09.012.
- 804 [35] J. Van Der Maas, E. Maldonado, A new thermal inertia model based on effusivity, Int. J.
805 Sol. Energy. 19 (1997) 131–160. doi:10.1080/01425919708914334.
- 806 [36] S.V.. Szokolay, Introduction to Architectural Science, 2004.
807 <http://www.ncbi.nlm.nih.gov/pubmed/23342180>.
- 808 [37] M. Fenollera, J. Míguez, I. Goicoechea, J. Lorenzo, M. Ángel Álvarez, The Influence of
809 Phase Change Materials on the Properties of Self-Compacting Concrete, Materials
810 (Basel). 6 (2013) 3530–3546. doi:10.3390/ma6083530.
- 811 [38] L. Olivieri, J.A. Tenorio, D. Revuelta, C. Bartolomé, J. Sanchez, S. Alvarez, L. Navarro,

- 812 L. Cabeza, J.L. Cano, Development of PCM-enhanced mortars for thermally activated
813 building components, in: Advanced Building Skins GmbH (Ed.), Conf. Proc. 12th Conf.
814 Adv. Build. Ski., Bern, Switzerland, 2017: pp. 561–571. <https://abs.green/home/>.
- 815 [39] T.W. MacFarland, Two-way analysis of variance : statistical tests and graphics using R,
816 Springer, 2012.
- 817 [40] M.Z. Pomianowski, P. Heiselberg, R.L. Jensen, Experimental investigation of thermal
818 conductivity of concrete containing micro-encapsulated phase change materials, in: 7th
819 Int. Symp. Heating, Vent. Air Cond. - Proc. ISHVAC 2011, 2011: pp. 185–191.
- 820 [41] N. Draper, H. Smith, Applied regression analysis, 2nd ed., Wiley, 1981.
- 821 [42] ASTM International, ASTM C138/C138M-17a, Standard Test Method for Density (Unit
822 Weight), Yield, and Air Content (Gravimetric) of Concrete, (2017).
823 doi:10.1520/C0138_C0138M-17A.
- 824 [43] CEN, EN 1015-11: Methods of test for mortars for masonry. Part 11: Determination of
825 flexural and compressive strength of hardened mortar, (1999) 16.
- 826 [44] Decagon Devices, KD2 Pro Thermal Properties Analyzer, (2016) 67.
827 http://manuals.decagon.com/Manuals/13351_KD2_Pro_Web.pdf.
- 828 [45] D. LeBlanc, Statistics: Concepts and Applications for science, Jones and Bartlett, 2004.
829