6th Historic Mortars Conference

21st to 23rd September 2022
Ljubljana, Slovenia

Conference Proceedings

Proceedings of the 6th Historic Mortars Conference - HMC 2022
21-23 September 2022, University of Ljubljana, Ljubljana, Slovenia

Edited by
Violeta Bokan Bosiljkov
Andreja Padovnik
Tilen Turk
Petra Štukovnik

CIP - Kataložni zapis o publikaciji
Narodna in univerzitetna knjižnica, Ljubljana

691.53(082)(0.034.2)

HISTORIC Mortars Conference (6 ; 2022 ; Ljubljana)
6th Historic Mortars Conference [Elektronski vir] : 21st to 23rd September 2022, Ljubljana, Slovenia : conference proceedings / [edited by Violeta Bokan Bosiljkov ... et al.]. - Ljubljana : Faculty of Civil and Geodetic Engineering, 2022

ISBN 978-961-6884-77-8
COBISS.SI-ID 121348355

An investigation of the salt weathering resistance of heritage repair mortar mixes 525
Anupama V.A., Divya Rani S., Swathy Manohar and Manu Santhanam

Design rationale and field testing of a gypsum-based grout for wall painting stabilization in the Chapel of Niketas the Stylite, Cappadocia, Turkey 534
Jennifer Herrick Porter, Yoko Taniguchi and Hatice Temur Yildiz

Comparative evaluation of properties of laboratory test specimens for masonry mortars prepared using different compaction methods 550
Vadim Grigorjev, Miguel Azenha and Nele De Belie

The challenge on development of the repair mortars for historical buildings in severe marine environment: Paimogo Fort, a case study 563
Maria do Rosário Veiga and Ana Rita Santos

Practical test for pozzolanic properties by A. D. Cowper: implementation and innovation 578
Marlene Sámano Chong, Alberto Muciño Vélez, Ivonne Rosales Chávez and Luis Fernando Guerrero Baca

Determination of the salt distribution in the lime-based mortar samples using XRF and SEM – EDX characterisation 595
Marina Aškrabić, Dimitrije Zakić, Aleksandar Savić, Ljiljana Miličić, Ivana Delić-Nikolić and Martin Vyšvařil

Developing a lime-based injection grout with no additives for very thin delamination: the role of aggregates and particle size/morphology 607
Chiara Pasian, Jennifer H. Porter, Mariia Gorodetska and Stephanie Parisi

Enhancement of latent heat storage capacity of lime rendering mortars 620
Andrea Rubio-Aguinaga, José María Fernández, Íñigo Navarro-Blasco and José Ignacio Álvarez

Obtaining of repair lime renders with microencapsulated phase change materials: optimization of the composition, application, mechanical and microstructural studies 634
Andrea Rubio-Aguinaga, José María Fernández, Íñigo Navarro-Blasco and José Ignacio Álvarez

Time-dependent deformations of lime-based mortars and masonry specimens prepared with them 649
Ioanna Papayianni and Emmanuella Berberidou

Influence of methyl cellulose in injection grout on mould growth on mural paintings - preliminary results 657
Andreja Padovnik, Violeta Bokan Bosiljkov, Polonca Ropret and Janez Kosel

OBTAINING OF REPAIR LIME RENDERS WITH MICROENCAPSULATED PHASE CHANGE MATERIALS: OPTIMIZATION OF THE COMPOSITION, APPLICATION, MECHANICAL AND MICROSTRUCTURAL STUDIES

Andrea Rubio-Aguinaga [1], José María Fernández[1], Íñigo Navarro-Blasco [1] and José Ignacio Álvarez [1]

(1) University of Navarra, Pamplona, Spain

Abstract: Different batches of repair lime rendering mortars were designed by mixing microencapsulated Phase Change Materials (PCMs) and other additives. The final aim of these renders is to improve the thermal efficiency of the envelope of the Built Heritage, while allowing the practitioners to apply a render with positive final performance. The combinations of the PCMs in different weight percentages, a superplasticiser (to increase the fluidity of the render keeping constant the mixing water), an adhesion improver and a pozzolanic additive were studied. The adhesion of these renders onto bricks and limestone specimens and the shrinkage and cracking of the mortars were studied in detail. X-ray diffraction technique was used to study the composition and evolution of the carbonation process. Compressive strength measurements were studied in hardened specimens. In addition, the porous structure of the rendering mortars was studied by mercury intrusion porosimetry to assess the effect of the PCMs' addition. Results have shown that these thermally enhanced mortars are feasible materials for real-life application in the context of architectural heritage restoration and conservation.

1 Introduction

A widely recognized good practice in rehabilitation is the use of lime-based mortars for restoration works of the Architectural Heritage. It has positive environmental benefits compared to cement, due to their lower energy consumption in the production phase, lower ingredient contributions and potential global warming processes. Furthermore, their atmospheric CO_2 absorption properties are also useful minimizing the carbon footprint [1]. Lime mortars have been widely used both in the interior and exterior of buildings, some of its most frequent applications are: renders and plasters, masonry bedding mortars, ornamental pieces, finishing mortars, flooring mortars... [1]

However, traditional residential buildings, ancient monuments and historic buildings commonly show low levels of thermal efficiency. Therefore, when repair works are addressed, the enhancement of the thermal performance of the building is required. There are many thermal consumption reduction strategies; nevertheless, most envelope upgrades available cannot be applied to cultural heritage repair work. This is because their application, their high price, and their incompatibility with some architectural heritage structures do not meet the general requirements of recognition of the tangible and intangible values of Cultural Heritage as well as the performance standards for protection and restoration, such as minimal modification of the aesthetic and history of the property, minimal intervention, highest compatibility of the materials to be introduced and use of new materials and methods for restoration and conservation.

In this context of reduction of the thermal demand, phase change materials are becoming increasingly important. Phase Change Materials, also known as PCMs, are systems capable of storing latent thermal energy through phase changes. Usually, these phase changes are solidification and melting. Thus, during the melting process, the heat gain is stored in the form of latent heat of fusion and, during the solidification process, this latent heat is released [2]. Therefore, depending on the external temperature of the system, heat is released or absorbed regulating moderately the temperature of the medium [3].

This work focuses on the "in bulk" incorporation of microencapsulated PCMs in lime rendering mortars as an interesting option for improving the thermal properties of historic buildings and monuments. Specifically, three different solid-liquid microencapsulated PCMs were directly incorporated during the mixing process to fresh air lime mortars. Solid-liquid PCMs were selected because of the higher enthalpy associated with the phase change. The PCMs were also microencapsulated with the aim of maintaining the macroscopic shape of the PCM, increasing the heat transfer area and avoiding undesired movements in the matrix of the mortar [4].

The aim of this work is the optimization of the composition of air lime-based renders containing PCMs, studying their physical-mechanical performance after their application. Since the incorporation of PCMs in bulk would lead to alterations of the fresh and hardened mortar's properties, this research work addresses the use of different additives to overcome practical problems. Therefore, different percentages of an adhesion booster were added along with different proportions of a superplasticizer to adjust the fluidity of the fresh renders avoiding an excess of mixing water.

In addition, metakaolin was added as a pozzolanic agent. Metakaolin is generally processed by calcining high purity clay and it contains active forms of silica and alumina which react with portlandite ($Ca(OH)_2$) yielding hydrated calcium silicate phases (C-S-H). The formation of new hydrated phases provides the enhancement of several properties of the lime-based mortars such as high values of mechanical strength and durability, low water permeability and good cohesion between binders and aggregates [5,6].

In this way, several batches of mortars were prepared in order to optimize and obtain the best composition of PCM-bearing rendering mortar, according to the final application of the material.

2 Materials and methods

2.1 Materials

Calcitic air lime mortars were prepared by mixing calcitic air lime supplied by Cal Industrial S.A. (Calinsa Navarra), classified as CL-90 by European regulations and calcitic sand (supplied by CTH Navarra). Percentages of binder/aggregate weight ratio were 21.7/78.3, whereas the percentage of mixing water was fixed at 25 wt. % of the total weight of the mortar.

Different additives and mineral admixtures were also used to optimize the mix composition of the renders. As an adhesion booster, different percentages of a starch derivative (Casaplast) were added along with various percentages of a polycarboxylated ether derivative (MasterCast GT 205) as a superplasticizer to adjust the fluidity of the fresh renders avoiding an excess of mixing water.

Metakaolin (MK, supplied by METAVER) was added in some of the mortars (20 wt. % with respect to the weight of lime, bwol) in order to increase the final strength and durability of the rendering mortars.

Three different solid-liquid microencapsulated PCMs supplied by Microtek, were used: two paraffin-based PCMs with melamine microcapsule with melting points of 18°C and 24°C (denoted as 18PCM and 24PCM) and a bio-based microencapsulated PCM with melting temperature of 29°C (29PCM). Percentages bwol of 5%, 10% and 20% of PCM were directly added to fresh air lime mortars during the mixing process.

As control group two PCM-free mortars (CTRL-1, MK-free, and CTRL-2, with 20 wt.% of MK) were prepared in order to compare the PCM performance (Table 1 and Table 2 gather the composition of the mixes).

2.2 Experimental methods

For the preparation of the fresh grouts, air lime, sand, metakaolin, adhesion booster, the corresponding PCM and an initial percentage of superplasticizer (0.25% bwol) were blended for 5 min using a solid additives mixer BL-8-CA (Lleal, S.A., Granollers, Spain) to achieve a homogenous mix.

A fixed percentage of mixing water (25 wt. %) was then added at low speed for 270 s in a Proeti ETI 26.0072 (Proeti, Madrid, Spain) mixer. Accumulative additions of 0.25% bwol of superplasticizer were added until adequate fluidity (as measured by the flow table test) and adhesion on a brick surface for render application were achieved at the discretion of a technician specialized in the production of mortars' mixtures and their application in the form of a single layer.

Regarding the hardened state study, fresh mixtures were moulded into cylindrical specimens with dimensions of 33 mm diameter and 39 mm height, and then cured at lab conditions (20°C ± 0.5 °C and 45% ± 5% RH).

2.3 Characterization methods

2.3.1 Fresh state tests

Several fresh state tests were carried out in order to characterize the fresh mortars. The fluidity of the fresh mortars was measured using the flow table test (UNE-EN 1015-3 standard [7]). The density of the paste and the percentage of entrained air were determined according to the UNE-EN 1015-6 standard [8] and UNE-EN 1015-7 standard [9] respectively. In addition, the water retentivity was measured using the UNE-EN 83-816-93 [10]. Lastly, the setting time was measured according to the UNE-EN 1015-9 standard [11].

2.3.2 Single coat mortars: Adhesion, shrinkage, cracks formation

As these mortars are intended to be used as renders, the performance of a single coat applied on a water-saturated brick was studied. Renders of 0.5 cm thickness of the different mortars were layered on saturated bricks. In order to evaluate the degree of adhesion and the formation or not of either cracks or fissures, a qualitative evaluation based on visual appearance after 1 month of the application was carried out. The criterion was the following: degree 0 (complete adhesion to the substrate and no evidence of cracks), degree 1 (complete adhesion and presence of very shallow and few cracks), degree 2 (complete adhesion and presence of numerous and shallow cracks) and degree 3 (poor adhesion and presence of numerous and deep cracks).

2.3.3 XRD

X-ray diffraction technique was used to study the composition and evolution of the carbonation process. X-Ray diffraction measurements were carried out in a Bruker D8 Advance diffractometer with Cu Kα1 radiation, from 5° to 80° (2θ), 1 s per step and a step size of 0.03°. The evaluation of the data was executed with DIFFRACplusEVA ® from Bruker.

2.3.4 Compressive strength

The determination of the compressive strength of the hardened mortars at the ages of 28 days and 91 days was carried out with a Frank/Controls 81565 press with a Proeti ETI 26.0052 compressive breaking device and a breaking speed of 20-50 N/s with a time interval between 30 and 90 s. Three specimens of 33 mm diameter and 39 mm height were used for each case in order to achieve representative values.

2.3.5 Mercury intrusion porosimetry

Mercury intrusion porosimetry was used to study the porous structure of the rendering mortars with the aim of assessing the effect of the PCMs' addition. Mercury intrusion porosimetry was carried out in a Micromeritics AutoPore IV 9500 apparatus. Measurements of cubic fragments of the mortars of ca. 1 cm edge were performed with a pressure range between 0.0015 and 207 MPa.

3 Results and discussion

3.1 Optimization of the mix composition: assessment of the adherence

Firstly, some mortars were prepared without the use of an adhesion booster (Figure 1), which showed a poor performance. It was observed that the fresh mixture needed an additive capable, on one hand, of improving adhesion and, on the other hand, of avoiding/minimizing the crack formation. A viscosity enhancer with a sticky nature such as the starch and the starch derivatives might be useful to this aim. Starch derivatives have been shown to be able to increase the adherence of a plain lime mortar and to reduce the number of cracks [12]. Their performance as water retainers prevents a quick drying off when the renders were applied onto absorbent substrates. For this reason, percentages by weight (with respect to the weight of lime) of 0.25% and 0.50% of a starch derivative as an adhesion booster were added to the admixture.

(a) (b)

Figure 1. Rendering on saturated brick of mortar: (a) containing 10% PCM, 20% MK and 0.45% SP; (b) containing 20% MK and 0.35% SP.

As it is shown in the comparison between Figures 2 (a -b), with 0.25 wt. % of the additive, and Fig. 3 (a-b), 0.50 wt.% of the additive, the low dosage (0.25%) was not optimal (no really good adherence, some cracks). However, 0.50% (Fig. 3 (a-b)) significantly improved adhesion and prevented crack formation. For this reason, this ratio was selected for the preparation of the mortars.

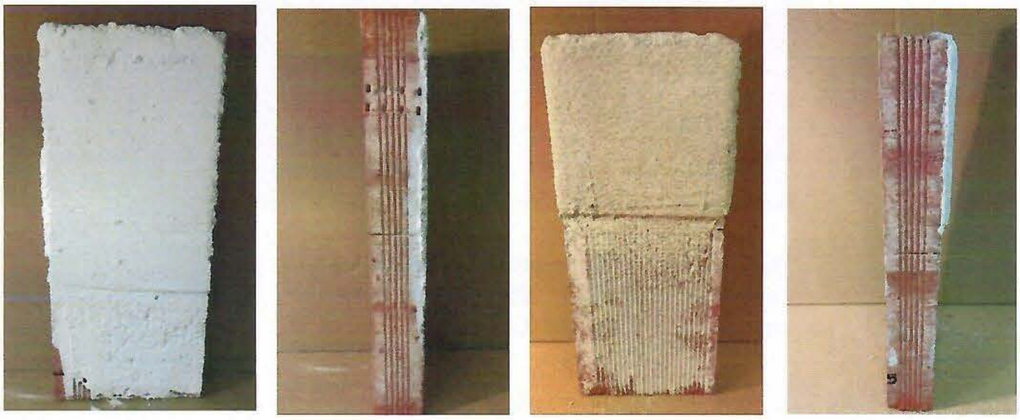
(a) (b) (c)

Figure 2. Renderings on saturated brick of mortar containing: (a) 0.25% starch and 0.40% SP (frontal); (b) 0.25% starch and 0.40% SP (side face); (c) 1.25% bwol of SP.

Simultaneously, for each render composition, the percentages of the superplasticizing additive were adjusted in order to achieve a workable consistency. Percentages from 0.1% to 1.25% bwol were tested, obtaining at the respective ends either unworkable dry renders (yielding a non-applicable render) or, on the other hand, highly fluid and slippery renders, which came unstuck from the brick, dropping off (Figure 2 (c)).

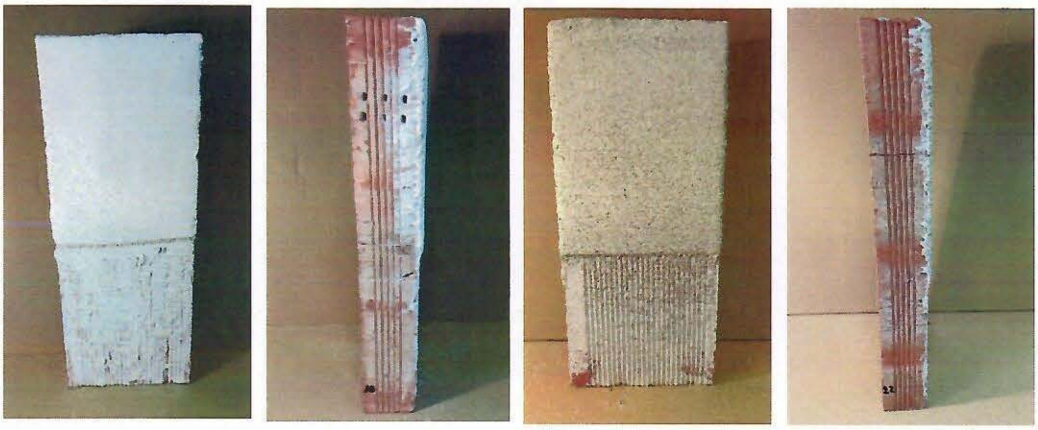
The effectiveness of the polycarboxylate-based SP for lime mortars had been pointed out in the literature [13] and in the current work was seen to be really useful to mitigate the impact in fluidity ascribed to the MK and PCM addition. Low dosages of the tested SP were effective in providing suitable consistency to the renders without extra mixing water requirements.

In this way, controlling fluidity, adhesion and prevention of cracks formation optimal percentages of adhesion booster and superplasticizer were selected for each render composition with PCMs (Table 1). Some examples of the optimal single coat mortars are included in Figures 3-4.



(a) (b) (c) (d)

Figure 3. Rendering on saturated brick of mortar containing (a) 0.50% starch and 0.60% SP (frontal); (b) 0.50% starch and 0.60% SP (side face); (c) 0.50% starch, 0.75% SP and 5% 24PCM (frontal); (d) 0.50% starch, 0.75% SP and 5% 24PCM (side face).



(a) (b) (c) (d)

Figure 4. Rendering on saturated brick of mortar containing (a) 0.50% starch, 0.75% SP, 20% MK and 10% 18PCM (frontal); (b) 0.50% starch, 0.75% SP, 20% MK and 10% 18PCM (side face); (c) 0.50% starch, 0.75% SP, 20% MK and 20% 29PCM (frontal); (d) 0.50% starch, 0.75% SP, 20% MK and 20% 29PCM (side face).

3.2 Fresh state tests

Once the percentages of adhesion booster and superplasticiser had been adjusted for each render, fresh state tests were carried out on all the batches of mortar. Final compositions of the mortars along with their fluidity, as measured by the slump values (Figure 5), and their qualitative evaluation on the degree of adhesion and the formation or not of cracks and fissures after 1 month are included in Tables 1-3.

Table 1. Compositions for PCM-free and 24PCM-bearing renders and their fluidity and adhesion test.

	PCM-free		**24PCM**					
Render	**CTRL-1**	**CTRL-2**	**24PCM-1**	**24PCM-2**	**24PCM-3**	**24PCM-4**	**24PCM-5**	**24PCM-6**
PCM (wt. %)	0	0	5	5	10	10	20	20
SP (wt. %)	0.6	0.75	0.75	0.75	0.75	0.75	0.75	0.75
MK (wt. %)	0	20	0	20	0	20	0	20
Starch (wt. %)	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
Air lime (wt. %)	21.7	21.7	21.7	21.7	21.7	21.7	21.7	21.7
Calcitic sand (wt. %)	78.3	78.3	78.3	78.3	78.3	78.3	78.3	78.3
Water (wt. %)	25	25	25	25	25	25	25	25
Slump (mm)	182	180	177	177	175	171	161	164
Qualitative evaluation of rendering (0-3)	0	0	0	0	0	0	0	1

Table 2. Compositions for 18PCM-bearing renders and their fluidity and adhesion test.

	18PCM					
Render	**18PCM-1**	**18PCM-2**	**18PCM-3**	**18PCM-4**	**18PCM-5**	**18PCM-6**
PCM (wt. %)	5	5	10	10	20	20
SP (wt. %)	0.75	0.75	0.75	0.75	0.75	0.75
MK (wt. %)	0	20	0	20	0	20
Starch (wt. %)	0.50	0.50	0.50	0.50	0.50	0.50
Air lime (wt. %)	21.7	21.7	21.7	21.7	21.7	21.7
Calcitic sand (wt. %)	78.3	78.3	78.3	78.3	78.3	78.3
Water (wt. %)	25	25	25	25	25	25
Slump (mm)	178	173	189	175	183	195
Qualitative evaluation of rendering (0-3)	0	0	0	0	1	1

Table 3. Compositions for 29PCM-bearing renders and their fluidity and adhesion test.

	29PCM					
Render	**29PCM-1**	**29PCM-2**	**29PCM-3**	**29PCM-4**	**29PCM-5**	**29PCM-6**
PCM (wt. %)	5	5	10	10	20	20
SP (wt. %)	0.75	0.75	0.75	0.75	0.75	0.75
MK (wt. %)	0	20	0	20	0	20
Starch (wt. %)	0.50	0.50	0.50	0.50	0.50	0.50
Air lime (wt. %)	21.7	21.7	21.7	21.7	21.7	21.7
Calcitic sand (wt. %)	78.3	78.3	78.3	78.3	78.3	78.3
Water (wt. %)	25	25	25	25	25	25
Slump (mm)	179	195	190	211	214	194
Qualitative evaluation of rendering (0-3)	0	0	0	0	0	0

All mortars showed complete adhesion to the substrate and no (or at most minimal) cracks were formed. In addition, all consistencies were suitable for their application as a render (no slippage).

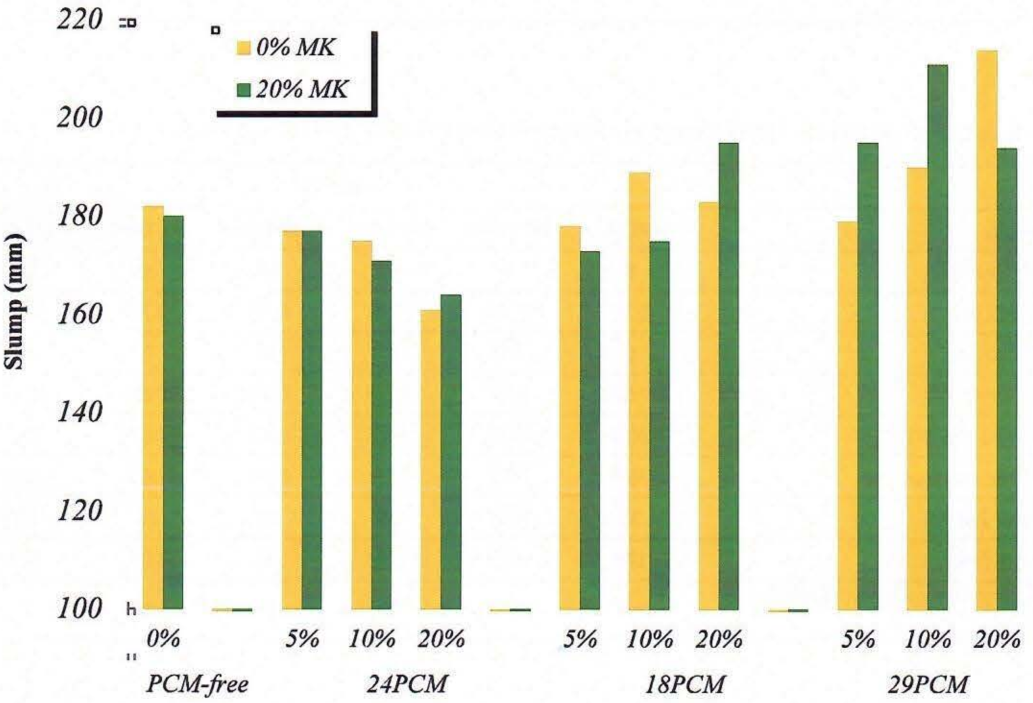


Figure 5. Fluidity values (slump measured in the flow table test) of the different renders.

As it is shown in Figure 5, the bio-based 29PCM increases in general the fluidity of the render, even with the highest percentages assayed (20%). This could be due to the PCM particle size, spherical shape and a subsequent ball-bearing effect.

Fresh state tests results are included in Tables 4-5.

Table 4. Fresh state tests results of PCM-free renders and renders containing 24PCM.

	PCM-free		24PCM					
Render	**CTRL-1**	**CTRL-2**	**24PCM-1**	**24PCM-2**	**24PCM-3**	**24PCM-4**	**24PCM-5**	**24PCM-8**
Stiffening time (min)	1157	1305	1211	1168	1392	1540	1432	1295
Paste density (kg/L)	1.94	1.94	1.83	1.83	1.85	1.83	1.79	1.78
Entrained air (%)	4.3	2.4	6.3	7.2	7.0	6.4	7.0	5.6
Water retentivity (%)	95.9	95.6	95.2	93.1	93.1	93.7	92.2	94.8

Table 5. Fresh state tests results of mortars containing 18PCM and 29PCM.

	18PCM						29PCM					
Render	**18PCM-1**	**18PCM-2**	**18PCM-3**	**18PCM-4**	**18PCM-5**	**18PCM-6**	**29PCM-1**	**29PCM-2**	**29PCM-3**	**29PCM-4**	**29PCM-5**	**29PCM-6**
Stiffening time (min)	1119	1306	1580	1341	1769	1377	1397	1179	1047	1212	1277	1185
Paste density (kg/L)	1.95	1.93	1.93	1.94	1.89	1.89	1.90	1.89	1.89	1.91	1.87	1.85
Entrained air (%)	2.2	2.8	1.7	1.7	1.1	3.0	4.3	6.0	5.2	4.5	6.0	5.6
Water retentivity (%)	93.5	94.3	92.3	95.0	93.1	93.4	96.9	93.7	93.7	94.9	92.7	94.3

Generally, as it is shown in Tables 4-5, due to the use of different additives, stiffening time, entrained air, paste density and water retentivity are not dramatically affected by the addition of any of the PCMs in any percentage.

3.3 Mineralogical characterization (XRD)

X-ray diffraction patterns of the PCM-free renders at 28 and at 91 days of curing are shown in Figure 6. The progress of the carbonation process is observed since the intensities of the characteristic peaks of portlandite (PDF 44-1481) decay while the intensities of the characteristic peaks of calcite (PDF 05-0586) increase. These variations are subtle since the curing days are not far apart. In addition, it is possible to observe quartz (PDF 33-1161) coming from the pozzolanic agent. The C-S-H phases resulting from the pozzolanic reaction are hardly distinguished due to their amorphous nature [6].

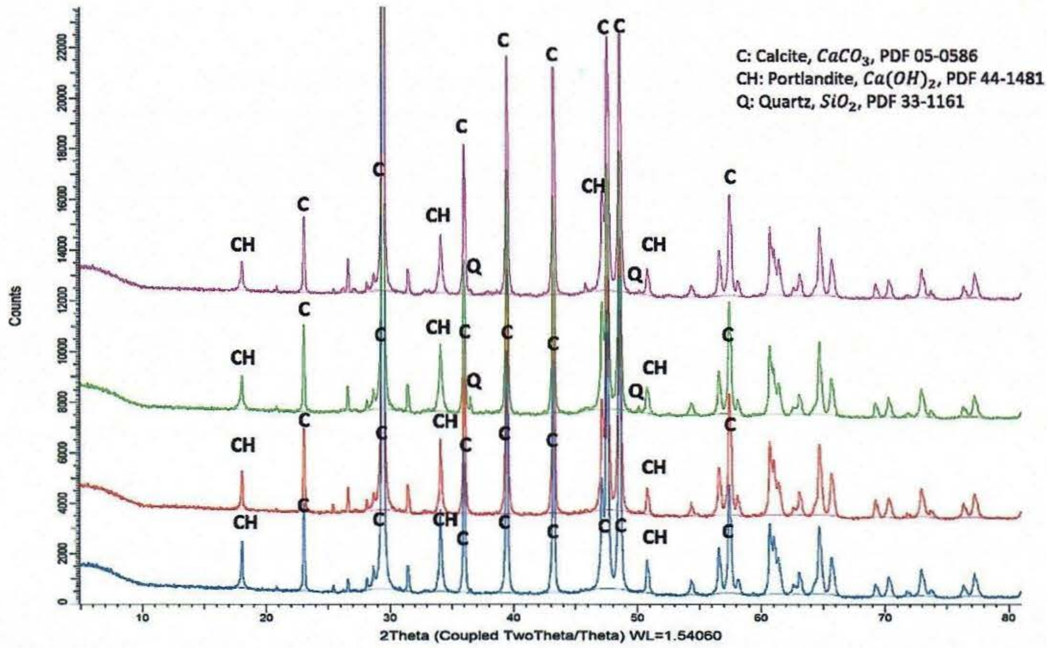


Figure 6. XRD patterns of the PCM-free renders.

The XRD patterns of the Phase Change Materials are displayed in Figure 7. The halo between 10° and 30° 2Θ show in 18PCM' diffractogram is due to its amorphous condition. The sharp diffraction peaks shown in 24PCM pattern correspond to a paraffin wax (PDF 53-1532) and to the melamine-based microcapsule (PDF 02-0164). 29PCM datasheet does not include composition information. However, bio-based PCMs are usually organic fatty acid esters made from underutilized and renewable raw materials, mainly animal fats and vegetable oils [14].

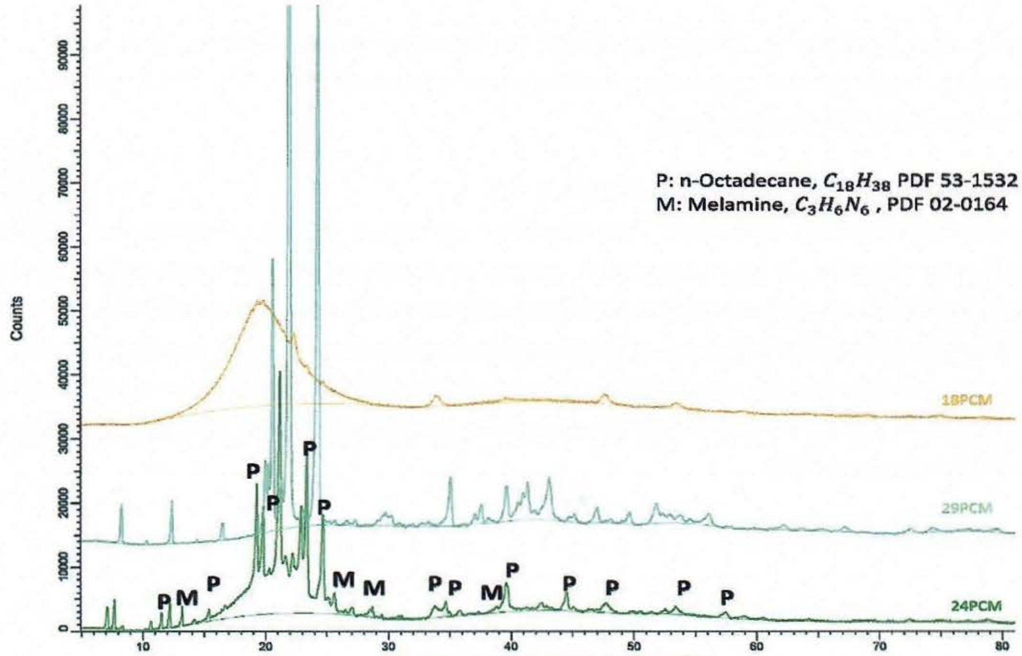


Figure 7. XRD diffractograms of the PCMs.

Percentages of portlandite ($Ca(OH)_2$, PDF 44-1481) present for each render at each curing day are included in Table 6. It is observed that the amount of portlandite decreases with the age of curing, evidencing the progress of carbonation.

Table 6. Percentages of portlandite of the different renders calculated with XRD.

Render	Curing days	$Ca(OH)_2$ (%)	Render	Curing days	$Ca(OH)_2$ (%)	Render	Curing days	$Ca(OH)_2$ (%)
CTRL-1	28	3.0	24PCM-6	28	3.9	29PCM-1	28	4.9
	91	2.7		91	3.3		91	3.5
CTRL-2	28	2.6	18PCM-1	28	3.0	29PCM-2	28	4.5
	91	2.5		91	2.6		91	3.3
24PCM-1	28	8.6	18PCM-2	28	4.4	29PCM-3	28	6.3
	91	4.6		91	3.4		91	4.8
24PCM-2	28	2.4	18PCM-3	28	3.3	29PCM-4	28	3.2
	91	2.3		91	2.1		91	2.4
24PCM-3	28	4.7	18PCM-4	28	3.2	29PCM-5	28	4.3
	91	3.1		91	2.2		91	2.5
24PCM-4	28	2.6	18PCM-5	28	3.8	29PCM-6	28	4.2
	91	2.3		91	3.5		91	3.1
24PCM-5	28	5.7						
	91	5.0						

3.4 Compressive strength

Compressive strength values of the mortars at 28 and 91 curing days are included in Figure 8. In general, the addition of the pozzolanic agent increases the compressive strength of the material. It is observed that the PCM addition does not severely affect the mechanical strength of the mortars. This is particularly true for the lowest percentage of PCM (5%). The 18PCM yielded, on average, the highest compressive strength values. For MK-free renders, the values were in general similar, or higher, than that of the control mortar. However, the presence of PCM was detrimental for the strength of MK-bearing renders compared to control mortar.

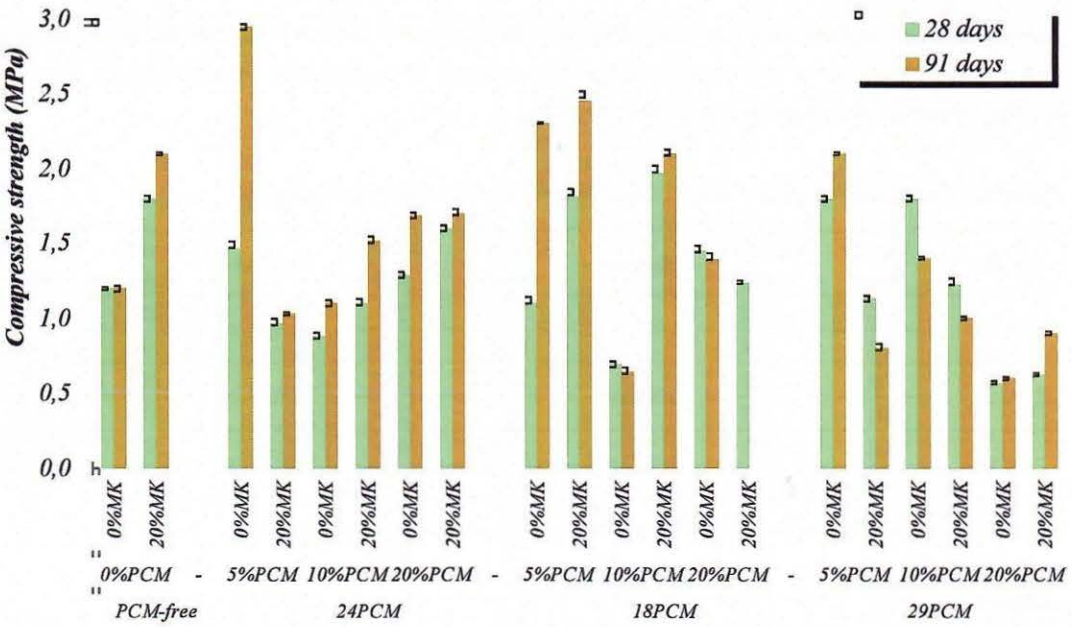


Figure 8. Compressive strength of renders at different curing times.

3.5 Pore size distribution

The effect of the PCM addition on the pore size distribution has been studied (Table 7). PCM-free renders have shown a reduction in porosity and average pore size as the curing process progresses. This porosity reduction was sharper for MK-free control, in which the absence of pozzolanic reaction leads to increased carbonation.

For PCM-bearing renders, an increase in the total porosity and in the average pore size as the percentage of PCM increases was generally observed. This increase could be due to the inherent porosity of the PCM and/or the possible generation of discontinuity with the conglomerate matrix (to be checked with SEM studies).

In addition, the filling effect of metakaolin has been verified by comparing the PCM-free renders with and without 20% MK CTRL-1 and CTRL-2 [6]. This pattern was also observed with some mortars containing PCM, for example, 24PCM-3 (MK-free) and 24PCM-4 (20% MK) and 24PCM-5 (MK-free) and 24PCM-6 (20% MK).

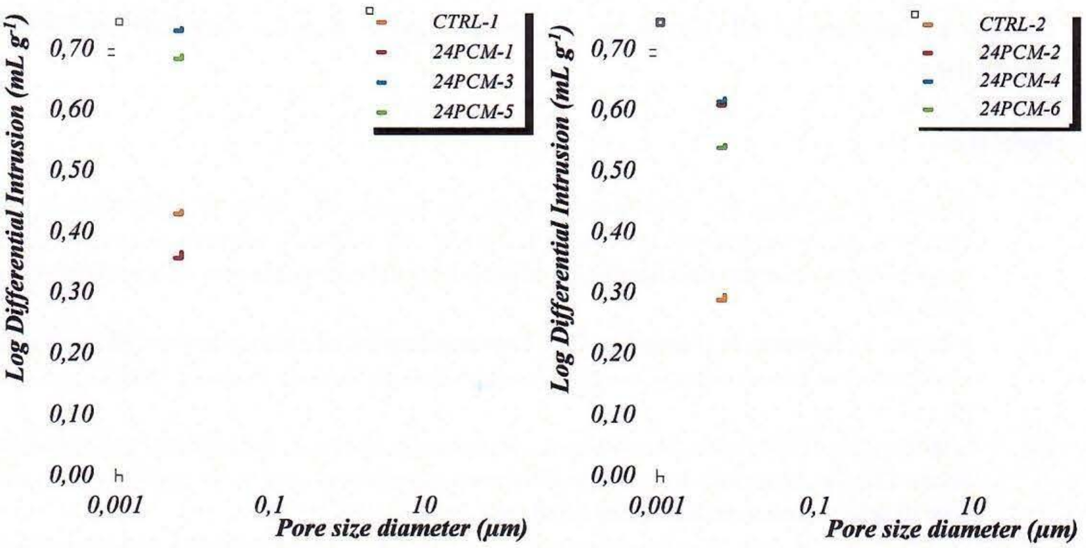
Table 7. Porosity percentages and average pore diameter.

Render	PCM (%)	Curing days	Porosity (%)	Average pore diameter (μm)
CTRL-1	0	28	36.7	0.3543
	0	91	26.0	0.3335
CTRL-2	0	28	36.4	0.3222
	0	91	35.0	0.3043
24PCM-1	5	28	41.6	0.3276
	5	91	39.0	0.4176
24PCM-2	5	28	40.3	0.4530
	5	91	41.5	0.5434
24PCM-3	10	28	42.0	0.7033
	10	91	40.1	0.6936

24PCM-4	10	28	39.7	0.5687
	10	91	38.6	0.5352
24PCM-5	20	28	44.1	0.9492
	20	91	39.5	0.7840
24PCM-6	20	28	35.9	0.5742
	20	91	38.9	0.5882
18PCM-5	20	28	36.4	0.4400
	20	91	34.1	0.5229

Comparison of pore size distributions are included in Figure 10. It is shown how the unimodal distribution is maintained after the addition of the PCMs in every percentage. However, a shift of the main peak of the distribution together with changes in the area under the curve corresponding to the increase in mean pore size and total porosity are observed.

In general, the variations in porosity are consistent with the compressive strength results (please, see 3.4 section). However, in some cases, an increase in mechanical strength is observed along with higher porosity (e.g. 24PCM-1). Further work will be necessary to study the microstructural interaction between the different rendering mortar's components by SEM. The increase in porosity might be related to the inherent porosity of the PCM, while at the same time increasing by filling effect the compactness of the binding matrix, thus explaining the higher compressive strength.



(a) (b)

Figure 10. Comparison of pore size distribution of (a) MK-free renders containing 24PCM at 91 curing days; (b) Renders containing 24PCM and 20% MK at 91 curing days.

4 Conclusions

Microencapsulated PCMs have been successfully incorporated in bulk into air lime-based rendering mortars. Furthermore, the composition of these mortars has been optimized considering their final application as building envelopes. Different chemical additives have been used to obtain workable, easily spreadable mortars with good adhesion and low cracking. Different quantities of a polycarboxylated-based superplasticizer have been adjusted to control the flowability of the paste. An adhesion booster (starch derivative) has been added to improve adhesiveness and to reduce crack formation. Metakaolin as pozzolanic agent has also been included to improve the compressive strength performance.

The fresh state results have shown that the addition of these additives can mainly affect the fluidity of the mortar, while the water retention, setting time, entrained air and paste density have not been greatly affected. The compressive strengths of the mortars have not been dramatically modified for the lowest dosage of PCM. The 18PCM yielded on average the highest strengths. Finally, the addition of PCMs resulted in an increase in porosity and in the average pore diameter compared to PCM-free renders.

After the optimization of the composition of the mixes and due to the use of chemical additives, the incorporation of PCMs has been feasible and not detrimental to the properties of the render. Specific mix compositions have been ascertained for each one the renders as a function of the microencapsulated PCM type and its percentage. These renders have been demonstrated to be easily applicable.

In a further work, the assessment of the thermal efficiency has been carried out to study the thermal behaviour of the PCMs. The use of models that imitate building envelopes might be also suggested as future achievement.

Acknowledgements

Funded by Ministerio de Ciencia e Innovación, grant number PID2020-119975RB-I00 LIMORTHER.

References

[1] Alvarez, J.I.; Veiga, R.; Martínez-Ramírez, S.; Secco, M.; Faria, P.; Maravelaki, P.N.; Ramesh, M.; Papayianni, I.; Válek, J. RILEM TC 277-LHS report: a review on the mechanisms of setting and hardening of lime-based binding systems. Mater Struct Constr 2021, 54.

[2] Himran, S.; Suwono, A.; Mansoor, G.A. Characterization of alkanes and paraffin waxes for application as phase change energy storage medium. Energy Sources 1994,16, pp. 117–28.

[3] Frigione, M.; Lettieri, M.; Sarcinella, A.; Barroso de Aguiar, J. Sustainable polymer-based Phase Change Materials for energy efficiency in buildings and their application in aerial lime mortars. Constr Build Mater 2020, 231.

[4] Liu, H.; Wang, X.; Wu, D. Innovative design of microencapsulated phase change materials for thermal energy storage and versatile applications: A review. Sustain Energy Fuels 2019, 3, pp. 1091–149.

[5] Arizzi, A.; Cultrone, G. Aerial lime-based mortars blended with a pozzolanic additive and different admixtures: A mineralogical, textural and physical-mechanical study. Constr Build Mater 2012, 31, pp. 135–43.

[6] Duran, A.; González-Sánchez, J.F.; Fernández, J.M.; Sirera, R.; Navarro-Blasco, I.; Alvarez, J.I. Influence of two polymer-based superplasticizers (poly-naphthalene sulfonate, PNS, and lignosulfonate, LS) on compressive and flexural strength, freeze-thaw, and sulphate attack resistance of lime-metakaolin grouts. Polymers 2018, 10.

[7] European Committee for Standarization. UNE-EN 1015-3 Methods of Test for Mortar for

Masonry. Part 3: Determination of consistence of fresh mortar (by flow table). EN 2006.
[8] European Committee for Standarization. UNE-EN 1015-6 Métodos de ensayo de los morteros para albañilería. Parte 6: Determinación de la densidad aparente del mortero fresco. EN 1999.
[9] European Committee for Standarization. UNE-EN 1015-7:1999 Methods of Test for Mortar for Masonry. Part 7: Determination of Air Content of Fresh Mortar. EN 1999.
[10] S European Committee for Standarization. UNE-EN 83-816-93 Test methods. Fresh Mortars. Determination of water retentivity. EN 1993.
[11] European Committee for Standarization. UNE-EN 1015-9:2000 Methods of Test for Mortar for Masonry. Part 9: Determination of Workable Life and correction Time of Fresh Mortar. EN 2000.
[12] Izaguirre, A.; Lanas, J.; Álvarez, J.I. Behaviour of a starch as a viscosity modifier for aerial lime-based mortars. Carbohydr Polym 2010, 80, pp. 222–8.
[13] Fernández, J.M.; Duran, A.; Navarro-Blasco, I.; Lanas, J.; Sirera, R.; Alvarez, J.I. Influence of nanosilica and a polycarboxylate ether superplasticizer on the performance of lime mortars. Cem Concr Res 2013, 43, pp. 12–24.
[14] Naresh, R.; Parameshwaran, R.; Vinayaka, V. Bio-based phase-change materials. Bio-Based Materials and Biotechnologies for Eco-Efficient Construction. LTD 2020. pp. 203–242.