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Use of sustainable Phase Change Material (PCM) in mortars for building energy efficiency

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Abstract. Academic and industrial research are moving towards the development of innovative solutions and materials able to limit energy consumption for the thermoregulation of a building. One solution is the use of phase change materials (PCMs) that can absorb, store, and release energy according to their physical state that changes when the ambient temperatures changes. In this work, new sustainable PCMs were developed through the “form-stable” method according to the principles of Circular Economy. The new PCM materials consisted, in fact, of an inert matrix (obtained as byproduct of stone processing) impregnated by low toxic, low flammable polymer, namely polyethylene glycol (PEG). The PEG/stone composite materials were used to replace the fine aggregates in mortars based on different binders providing the mortars with thermoregulation performance. A comprehensive characterization was performed on the new PCMs by evaluating their thermal stability and thermal efficiency. The main properties (in fresh and hardened states) of the mortars with or without PCMs were analyzed. The mortars containing PCMs were also subjected to further investigations to evaluate their thermal behavior in response to external climatic conditions. Encouraging results were obtained, confirming the effectiveness of the mortars containing the new PCMs in the thermoregulation of indoor environments.

1. Introduction

Energy efficiency in buildings is becoming one of the key steps to avoid energy waste. The building sector is a major contributor to massive energy use. Most of this is, in fact, used to power cooling and heating devices [1]: the more energy we consume, the greater the negative impact on the environment.

Climate change is now a reality and avoiding waste, decreasing use, and being more energy independent is possible with the use of alternative sources [2]. The possibility of storing energy to use it in times of need is becoming a very current line of research and it is on this path that this work fits. For several years, the behavior of some materials using the Latent Heat Thermal Energy Storage (LHTES) approach has been studied [3]. These materials, known with the name of Phase Change Materials (PCMs), can store energy by absorbing it from the environment while changing their physical state. Specifically, a PCM can become liquid from its solid state if the external temperature increases. During this transition of state, it absorbs thermal energy, which is stored and then released when the PCM returns to the solid state as the temperature decreases [4,5]. External temperatures are on average high during the day and decrease at night. During this cycle, the PCM can undergo state changes, absorbing, storing, and releasing energy. Of all the possible applications of a PCM, its inclusion in building materials is certainly the most interesting. Over the years, several PCMs have been incorporated into wallboards [6,7], floors [8,9], ceilings [10], bricks [11,12] and even in windows [13,14]. However,



the most common solution involves embedding PCM within mortars because they can cover a very large surface area and thus provide a superior heat transfer benefit [15]. In recent years, many studies along these lines are also focusing on the use of geopolymer-based mortars that include PCMs [16]. Several are methods to add a PCM in a building materials, as reported in the literature [15,17]. In this paper, the idea was to integrate the developed PCMs through its inclusion into a mortar. This solution presents several advantages: first, a mortar covers most of the surfaces of a building, so the action of the PCM is extended over a large area. Furthermore, a mortar can be molded into any shape, and the quality analyses can be carried out easily [18,19]. Of the possible ways to integrate a PCM into a mortar, the form-stable method is the simplest and most feasible. Moreover, with form-stable method: i) it is possible to prepare a composite material using very simple and inexpensive equipment; ii) it is possible to select the raw materials according to the final properties to be obtained; iii) it is possible to exploit as a matrix a porous material (for instance, a natural stone as in this study) derived from the waste of its processing, thus lowering the cost of raw materials; and iv) it is possible to create a composite material that completely replace the normal aggregates that are used in the mortar [20,21]. In the present study, a local stone, i.e. Lecce stone, was selected as inert matrix for the PCMs. The preparation of these materials and their characterization (chemical, physical, thermal and morphological) was deeply studied and reported elsewhere [20–23]. Following these studies, it was possible to state that the PCM composite material is appropriate for the intended purpose: thus, it can be used as an aggregate for mortars.

Two composite PCMs were developed, based on Poly-Ethylene Glycol 1000 (PEG1000) and Poly-Ethylene Glycol 800 (PEG800). PEG polymer was selected as the active phase of the PCMs for its thermal properties and also for its low toxic and low flammability nature [24]. In general, a PCM has characteristic melting and crystallization temperatures which correspond to the temperatures at which it undergoes the changes of state. Keeping in mind the objective of this work, the PEG1000 was selected because it has melting/crystallization temperatures in a range corresponding to a warm climate, such as the Mediterranean one [20]; while the PEG800 undergoes the change of state at lower temperatures, it may be, therefore, more appropriate for colder climates, such as continental ones [22]. The two PEGs were, then, included in flakes of waste Lecce stone, to produce form-stable PCMs. The latter were incorporated as aggregate in different mortars to provide thermal energy storage capacity in buildings.

The investigation on the thermal characteristics of a PCM consisting of a mix of both PEGs (i.e., 50%wt of LS/PEG1000 and 50%wt of LS/PEG800) highlighted the possibility to extend the range of temperatures in which the resulting PCM would be efficient, allowing it to be used in different geographical areas. In the present work, the preparation of different mortar compositions based on different binders (i.e. aerial and hydraulic lime, gypsum, and cement) is described. The mortars can be divided into 3 groups according to the type of PCM they contain: i) those including PEG1000; ii) those that contain PEG800; iii) finally, those that contain the mixed PCM. The main purpose was to study the effect of the presence of the different PCMs on some physical properties of the mortar, such as workability and compressive and flexural strength, as well as on their thermal properties.

2. Materials

2.1. Preparation of Lecce Stone/PEG composite PCMs

Two form-stable PCMs, incorporating PEG800 and PEG1000 as the polymeric active component and Lecce Stone (LS) flakes as the supporting material, were developed using a vacuum impregnation setup, as described in our previous work [20,22].

LS is a natural stone (composed mainly of CaCO_3) with a high open porosity, resulting in an excellent matrix for the PCM. The stone pieces were obtained as waste material from a local quarry. The stone flakes, that should have been disposed, were reused following the principles of Circular Economy. They were ground and sieved achieving a proper granulometry (between 1.6 mm and 2.0 mm), as it can be seen in Fig. 1a.

Poly-Ethylene Glycol is a low-cost, low toxic, low flammable polymer, available on the market in different molecular weights which correspond to different melting and crystallization temperatures. The PEGs selected in the study were PEG800 and PEG1000. The former, provided by Wuhan Fortuna

Chemical Co. (Wuhan, China), has a melting temperature of 12.7 °C and a crystallization temperature of 9.3 °C; the latter, produced by Sigma-Aldrich Company (Darmstadt, Germany), has a melting temperature of 39.3 °C and a crystallization temperature of 19.4 °C. The reported characteristic temperatures were considered appropriate for the aim of this research, i.e., develop mortars to apply in buildings located in different geographical areas, i.e., the Mediterranean area with higher average temperatures and the Continental area characterized by colder climate.

In a first stage of the work, PEG1000 was selected to produce the PCM: when it was observed that its energy storage and release capacity was hardly activated at low temperatures, PEG800 was added in the study to extend the use of the new PCMs at lower temperatures, i.e., in the colder season of the Mediterranean climate and in a Continental climate. Two form-stable PCMs were, then, prepared by including each PEG, at a temperature just above their respective melting points, in Lecce stone granules of appropriate granulometry, obtaining LS/PEG800 and LS/PEG1000 PCMs, respectively. The result is reported, respectively, in Fig. 1b and 1c.

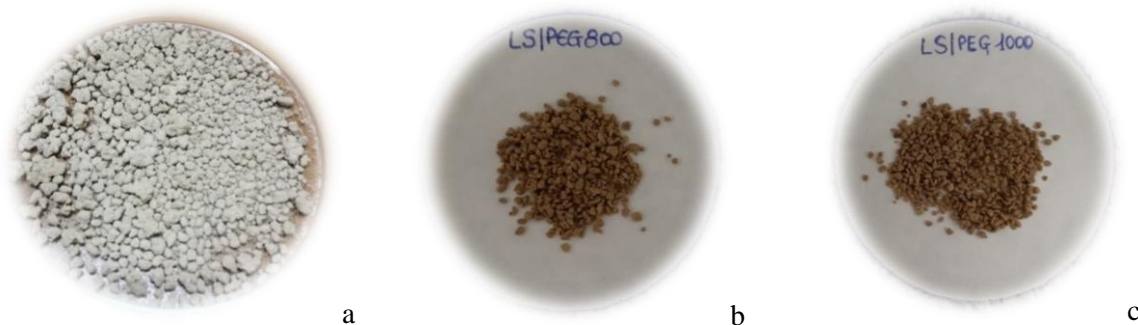


Figure 1. Materials used: a) Lecce Stone (LS); b) Lecce Stone impregnated with PEG800 (LS/PEG800); c) Lecce Stone impregnated with PEG1000 (LS/PEG1000).

Through the thermogravimetric analysis (TGA) carried out on PCM composites it was possible to measure the amount of PEG absorbed by the LS substrate. This value was found to be approximately around 23% for both and PEG1000 [20] and PEG800 [22]. The same analysis was useful to demonstrate that both PCM composite materials have a thermal resistance and a thermal stability suitable for the intended purpose, as reported in literature [25,26]. The procedure employed to analyze the leakage of PEGs is reported in [22], following what was reported in other similar studies [27,28]. The results of this test showed that no significant PEGs leakage occurred, even when the temperatures were higher than the melting point of each PEG. This means that the inert LS support can absorb and retain the PEGs contributing to their thermal stability.

2.2. Preparation of LS/PEG-incorporated into different mortar formulations

To evaluate the effect of the prepared LS/PEGs on the mechanical and thermal properties of the mortars based on different binders, several mixtures were manufactured. The mortar compositions were basically designed to be used as interior coatings.

Different binders were employed, i.e., aerial lime, hydraulic lime, gypsum, and cement. These binders were supplied by different Portuguese companies: aerial lime (AL) with a density of 2450 kg/m³ was supplied by Lhoist (Alcanade, Portugal); a natural hydraulic lime (HL) with a density of 2700 kg/m³, was supplied by Cimpor (Lisbon, Portugal); a conventional gypsum (G) with high fineness and density of 2960 kg/m³, was provided by Sival (Souto da Carpalhosa, Leira, Portugal); finally, a CEM I 42.5 R cement (C), with a density of 3030 kg/m³, was supplied by SECIL (Lisbon, Portugal).

A superplasticizer (SP), i.e., a polyacrylate (MasterGlenium SKY 627), supplied by BASF, with a density of 1050 kg/m³, was also used in some mortars to reduce the amount of water required for the mixing, to increase the final mechanical properties. For the same reason, different amounts of binder were used.

The first formulations were made using 500 kg/m³ of binder, and then gradually this amount was increased up to 1000 kg/m³. To develop ecological mortars, the binder content was initially kept low. However, the need to obtain mortars with good mechanical properties led to an increase in the binder as well as in the superplasticizer contents. In Table 1 all the mortar mixes realized according to the European Norm EN 998-1 [29] are reported.

Table 1. Mortar formulations (reported as kg/m³).

| Mortar formulations | Binder content | Aggregates | | | SP | Water Saturation | Water | Water/Binder |
|--|----------------|------------|----------------|-----------------|----|------------------|-------|--------------|
| | | LS | PEG800 content | PEG1000 content | | | | |
| AL ₅₀₀ _LS | 500 | 1500 | 0 | 0 | 0 | 378 | 289 | 0.60 |
| AL ₅₀₀ _LS/PEG1000 | | 1500 | 0 | 345 | 0 | 0 | 395 | 0.80 |
| AL ₈₀₀ _LS | 800 | 175 | 0 | 0 | 15 | 44 | 600 | 0.75 |
| AL ₈₀₀ _LS/PEG1000 | | 220 | 0 | 51 | 15 | 0 | 600 | 0.75 |
| AL ₁₀₀₀ _LS | 1000 | 668 | 0 | 0 | 20 | 168 | 347 | 0.35 |
| AL ₁₀₀₀ _LS/PEG800 | | 979 | 225 | 0 | 20 | 0 | 310 | 0.31 |
| AL ₁₀₀₀ _LS/PEG800_LS/PEG1000 | | 979 | 113 | 113 | 20 | 0 | 310 | 0.31 |
| HL ₅₀₀ _LS | | 1480 | 0 | 0 | 15 | 370 | 300 | 0.60 |
| HL ₅₀₀ _LS/PEG1000 | 500 | 1678 | 0 | 386 | 15 | 0 | 350 | 0.70 |
| HL ₈₀₀ _LS | | 1092 | 0 | 0 | 15 | 275 | 320 | 0.40 |
| HL ₈₀₀ _LS/PEG1000 | 800 | 1729 | 0 | 398 | 15 | 0 | 375 | 0.47 |
| HL ₁₀₀₀ _LS | | 682 | 0 | 0 | 20 | 171 | 380 | 0.38 |
| HL ₁₀₀₀ _LS/PEG800 | 1000 | 1082 | 249 | 0 | 20 | 0 | 320 | 0.32 |
| HL ₁₀₀₀ _LS/PEG800_LS/PEG1000 | | 1082 | 124 | 124 | 20 | 0 | 320 | 0.32 |
| G ₅₀₀ _LS | 500 | 1454 | 0 | 0 | 15 | 366 | 325 | 0.65 |
| G ₅₀₀ _LS/PEG1000 | | 1645 | 0 | 378 | 15 | 0 | 375 | 0.75 |
| G ₈₀₀ _LS | 800 | 1169 | 0 | 0 | 15 | 294 | 329 | 0.40 |
| G ₈₀₀ _LS/PEG1000 | | 1472 | 0 | 339 | 15 | 0 | 340 | 0.43 |
| G ₁₀₀₀ _LS | 1000 | 763 | 0 | 0 | 20 | 192 | 385 | 0.39 |
| G ₁₀₀₀ _LS/PEG800 | | 1129 | 260 | 0 | 20 | 0 | 336 | 0.34 |
| G ₁₀₀₀ _LS/PEG800_LS/PEG1000 | | 1129 | 130 | 130 | 20 | 0 | 340 | 0.34 |
| C ₅₀₀ _LS | 500 | 1392 | 0 | 0 | 15 | 350 | 340 | 0.68 |
| C ₅₀₀ _LS/PEG1000 | | 1790 | 0 | 412 | 15 | 0 | 350 | 0.70 |
| C ₈₀₀ _LS | 800 | 1070 | 0 | 0 | 15 | 269 | 296 | 0.37 |
| C ₈₀₀ _LS/PEG1000 | | 1347 | 0 | 310 | 15 | 0 | 360 | 0.45 |
| C ₁₀₀₀ _LS | 1000 | 772 | 0 | 0 | 20 | 194 | 390 | 0.39 |
| C ₁₀₀₀ _LS/PEG800 | | 1307 | 301 | 0 | 20 | 0 | 300 | 0.30 |
| C ₁₀₀₀ _LS/PEG800_LS/PEG1000 | | 1307 | 150 | 150 | 20 | 0 | 300 | 0.30 |

A total of 28 mortar formulations were manufactured: 12 of them were reference mortars, i.e., they did not contain any PCM; 8 of them included only LS/PEG1000 as PCM; 4 of them included only LS/PEG800 as PCM; finally, 4 of them contained a mixed PCM, composed by 50% wt of LS/PEG800 and 50% wt of LS/PEG1000 [30–32]. The label “water saturation” in Table 1 is referred to the amount of water used to saturate the LS aggregates before the mixing. In fact, to avoid the absorption of water inside LS aggregates, it is necessary to saturate them. This is not necessary when the aggregates contained the PEGs, since they are already saturated by the polymer.

3. Methods

The workability of the different mortar formulations, listed in Table 1, was evaluated through the flow table method, according to the European standard EN 1015-3 [33]. The workability test, or flow table test, is a method used to determine the consistency of fresh mortar. Through this method, it is therefore possible to assess the fluidity of the mortar when it is in its fresh state. (Fig. 2a).

The mechanical characteristics of the prepared mortars, evaluated in flexural and compressive mode, were determined according to the European standard EN 1015-11 [34] using a Lloyd dynamometer instrument. For each mortar composition, 3 prismatic specimens (40x40x160 mm³) were prepared by casting the fresh mortars in iron molds. After a first curing stage of 2 days, the specimens were demolded and left for additional 26 days in a storing room at standard temperature (25 °C) and humidity level (50%). The flexural tests were, then, performed at a speed of 6 µm/s, while the compressive tests at a speed of 12 µm/s. (Fig. 2b)

The thermal properties were measured with a Differential Scanning Calorimeter (DSC, Star^c System, Mettler Toledo) instrument. (Fig. 2c) The thermal characteristics were evaluated on: i) each single pure PEG (i.e., PEG800 and PEG1000); ii) the PEGs blend (i.e., 50%wt. of PEG800 and 50%wt. of PEG1000); iii) the produced LS/PEG systems (i.e., LS/PEG800 and LS/PEG1000); iv) some selected mortar formulations containing the PCM aggregates. To assess the thermal properties of the materials, each sample was subjected to sequential thermal cycles, between -10 °C to 75 °C on heating, and from 75 °C to -10 °C on cooling. The heating and cooling rates used during the DSC analyses were 10 °C/min; the tests were performed under nitrogen atmosphere (flow rate: 60 ml min⁻¹). The amount of each analyzed sample was between 10 and 20 mg; aluminum crucibles were used. Three samples were analyzed for each material, and the results averaged.

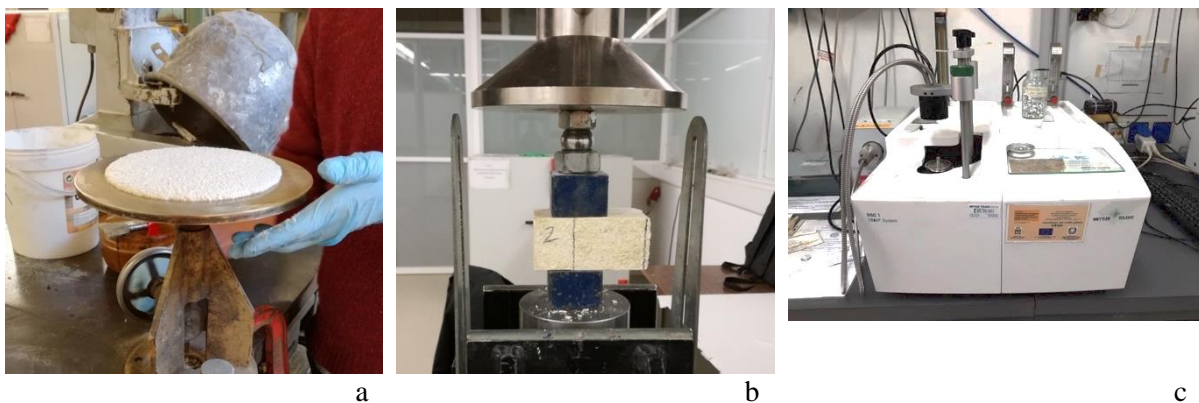


Figure 2. Equipment used for the characterization of the mortar formulations: a) flow table test; b) mechanical properties (compressive mode); c) Differential Scanning Calorimeter (DSC).

4. Results and discussion

The physical properties of the all the mortars reported in Table 1 were analyzed in both fresh and solid states. The flow table test was first performed on the produced mortars, to evaluate their workability and, consequentially, their application adequacy. In our work the workability values of mortar formulations must be comprised in the range 160-180 mm. As it can be observed in Table 2, all the produced mortars showed an adequate workability value, confirming the suitability of the selected formulations. Table 2 also reports the values of flexural and compressive strengths recorded on each mortar cured for 28 days, with the indication of the classification according to the standard EN 998-1 [29]. Each test was repeated at least 3 times on different mortar samples, and the results were averaged and presented with the corresponding experimental error.

Table 2. Physical properties of the mortar compositions: workability and mechanical performance in flexural and compressive mode.

| Sample | Workability (mm) | Flexural Strength (N/mm ²) | Compressive Strength (N/mm ²) | Classification EN 998-1:2010 |
|-------------------------------|------------------|--|---|------------------------------|
| AL ₅₀₀ _LS | 161 ± 2.0 | 0.5 ± 0.1 | 0.5 ± 0.0 | n/a |
| AL ₅₀₀ _LS/PEG1000 | 170 ± 3.0 | 0.2 ± 0.1 | 0.2 ± 0.1 | n/a |

| | | | | |
|--|-----------|------------|------------|------------|
| AL ₈₀₀ _LS | 175 ± 3.0 | 0.9 ± 0.0 | 1.5 ± 0.1 | CSI |
| AL ₈₀₀ _LS/PEG1000 | 180 ± 2.0 | 0.5 ± 0.1 | 1.2 ± 0.1 | n/a |
| AL ₁₀₀₀ _LS | 178 ± 2.0 | 0.6 ± 0.2 | 1.5 ± 0.2 | CSI |
| AL ₁₀₀₀ _LS/PEG800 | 160 ± 3.0 | 0.3 ± 0.1 | 0.4 ± 0.0 | n/a |
| AL ₁₀₀₀ _LS/PEG800_LS/PEG1000 | 175 ± 2.0 | 0.4 ± 0.0 | 0.6 ± 0.1 | n/a |
| HL ₅₀₀ _LS | 170 ± 2.0 | 1.1 ± 0.3 | 2.8 ± 0.8 | CSII |
| HL ₅₀₀ _LS/PEG1000 | 177 ± 3.0 | 0.1 ± 0.1 | 0.4 ± 0.1 | CSI |
| HL ₈₀₀ _LS | 165 ± 2.0 | 2.8 ± 0.5 | 17.0 ± 0.2 | CSIV |
| HL ₈₀₀ _LS/PEG1000 | 175 ± 2.0 | 0.4 ± 0.1 | 1.5 ± 0.1 | CSI - CSII |
| HL ₁₀₀₀ _LS | 175 ± 1.0 | 5.2 ± 1.5 | 11.7 ± 0.5 | CSIV |
| HL ₁₀₀₀ _LS/PEG800 | 170 ± 3.0 | 2.3 ± 0.4 | 3.5 ± 0.2 | CSII-CSIII |
| HL ₁₀₀₀ _LS/PEG800_LS/PEG1000 | 163 ± 2.0 | 2.1 ± 0.5 | 3.6 ± 0.5 | CSII-CSIII |
| G ₅₀₀ _LS | 160 ± 1.0 | 3.2 ± 0.2 | 4.8 ± 0.2 | CSII |
| G ₅₀₀ _LS/PEG1000 | 180 ± 3.0 | 0.5 ± 0.1 | 0.4 ± 0.2 | CSI |
| G ₈₀₀ _LS | 160 ± 1.0 | 4.1 ± 0.2 | 16.4 ± 0.6 | CSIV |
| G ₈₀₀ _LS/PEG1000 | 160 ± 1.0 | 1.6 ± 0.2 | 3.3 ± 0.3 | CSII |
| G ₁₀₀₀ _LS | 170 ± 4.0 | 9.3 ± 1.3 | 22.3 ± 0.2 | CSIV |
| G ₁₀₀₀ _LS/PEG800 | 165 ± 3.0 | 1.7 ± 0.0 | 4.3 ± 1.7 | CSII-CSIII |
| G ₁₀₀₀ _LS/PEG800_LS/PEG1000 | 163 ± 1.0 | 2.3 ± 0.0 | 6.2 ± 0.5 | CSII-CSIII |
| C ₅₀₀ _LS | 160 ± 1.0 | 5.8 ± 0.3 | 20.5 ± 0.4 | CSIV |
| C ₅₀₀ _LS/PEG1000 | 160 ± 1.0 | 1.0 ± 0.2 | 1.1 ± 0.2 | CSI |
| C ₈₀₀ _LS | 160 ± 1.0 | 9.2 ± 0.9 | 26.3 ± 0.4 | CSIV |
| C ₈₀₀ _LS/PEG1000 | 178 ± 3.0 | 1.9 ± 0.3 | 3.4 ± 0.8 | CSII |
| C ₁₀₀₀ _LS | 180 ± 0.5 | 11.8 ± 1.1 | 65.6 ± 6.1 | CSIV |
| C ₁₀₀₀ _LS/PEG800 | 170 ± 1.0 | 2.1 ± 0.1 | 3.9 ± 1.2 | CSII-CSIII |
| C ₁₀₀₀ _LS/PEG800_LS/PEG1000 | 170 ± 4.0 | 2.0 ± 0.2 | 4.4 ± 0.7 | CSII-CSIII |

It is well known that an adequate water amount for the mix design of a mortar is necessary to ensure a good workability. However, if the amount of water exceeds a certain limit, a more porous mortar is obtained, with poor mechanical properties. Therefore, superplasticizers (SP) are commonly used as they improve the workability of the mortars even using limited amounts of water. As it can be seen from data reported in Table 1, after the first two mortar compositions (i.e., AL₅₀₀_LS and AL₅₀₀_LS/PEG1000), a fixed amount of SP (i.e., 15 kg/m³) was used. By adding the SP, a slight increase in mechanical properties was in fact, noted: this observation suggested to use the SP in all the mortars. The amount of SP was even increased (from 15 to 20 kg/m³) in the mortars containing 1000kg/m³ of binder with the aim to further their mechanical properties.

According to the results reported in Table 2, the addition of the PEG-based PCM caused a reduction of compressive and flexural strength values in all the mortar compositions. This trend is well known, and reported in literature [35,36]. The mortars developed in the study, however, were designed as coatings and, for such applications, lower mechanical strength values are sufficient. According to the classification given in the standard [34], the minimum value that is necessary to achieve is 1.5 MPa. Therefore, only the mortars containing an amount of binder between 800 and 1000 kg/m³ were considered appropriate for our purposes.

The DSC thermograms obtained from calorimetric analysis performed on the pure PEGs and on their blend is shown in Figure 3.

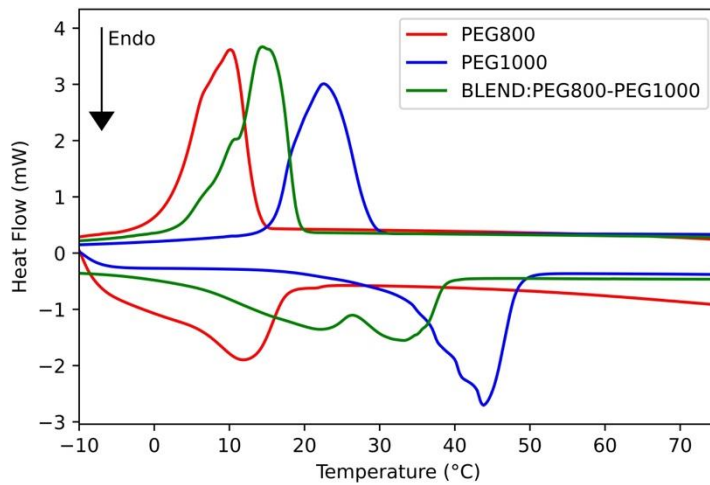


Figure 3. DSC curves recorded on pure PEGs (PEG800, PEG1000) and on the blend composed by 50%wt of PEG800 and 50%wt of PEG1000.

As previously reported [20], PEG1000 polymer exhibits an endothermic (melting) peak approximately at 43 °C during the heating stage and an exothermic (crystallization) peak around 23 °C when the temperature is reduced. Melting/crystallization enthalpy of about 129 J/g was calculated from DSC measurements, in accordance with the results of different studies performed on the same material [24].

PEG800 shows two endothermic peaks: one centered at around 18 °C and a smaller one at 25 °C. Similarly, in the cooling stage two exothermic peaks were detected, one at about 13 °C and the other at 9 °C. Melting/crystallization enthalpy of about 150 J/g was calculated for PEG800.

The physical blend made by the two PEGs presents a wider temperature range due to the combination of the two PEGs, with two endothermic peaks, one at 22 °C and the other at 33 °C, and an exothermic peak at around 16 °C. The melting/crystallization enthalpy of the blend is about 155 J/g.

The DSC test confirmed that the thermal characteristics displayed by PEG1000 and PEG800 were appropriate for developing form-stable PCMs to be used as TES material. The blend, on the other hand, was not considered to produce a PCM due to the difficulty to include in the stone flakes polymeric materials characterized by quite different melting point. Nevertheless, a mixed PCM was obtained upon the addition in mortars of the same amount of LS/PEG800 and LS/PEG1000, obtaining a PCM indicated as LS/PEG800_LS/PEG1000.

The thermal characteristics of the two single PCMs (i.e., LS/PEG800 and LS/PEG1000) were also analyzed. Figure 4 reports the thermograms of the two PCMs and in Table 3 are summarized their characteristic temperatures.

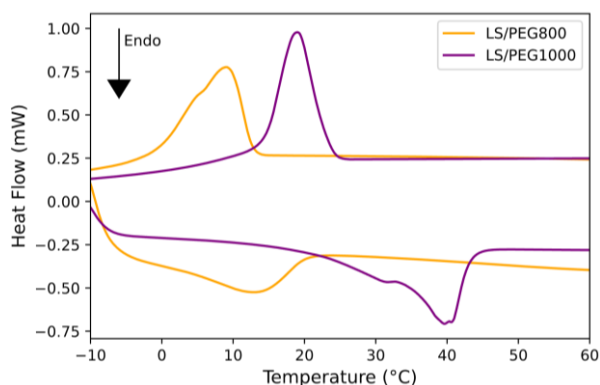


Figure 4. DSC curves recorded on LS/PEG800 and LS/PEG1000 form-stable PCMs.

| | LS/PEG800 | LS/PEG1000 |
|-------------------------------|------------|------------|
| T_m (°C)* | 12.7 ± 1.4 | 39.3 ± 0.7 |
| ΔH_m (J/g)** | 28.3 ± 3.4 | 27.7 ± 0.9 |
| T_c (°C)* | 9.3 ± 0.9 | 19.4 ± 0.9 |
| ΔH_c (J/g)** | 28.1 ± 0.9 | 26.2 ± 1.1 |

*T_m and T_c: melting and crystallization peak temperatures, respectively.

** ΔH_m and ΔH_c: enthalpy measured during heating and cooling stages, respectively.

Table 3. LHTES properties measured with DSC on LS/PEG800 and LS/PEG1000.

The results from DSC analysis confirmed that the selection of the different PEGs is appropriate even when they are included in a LS matrix.

The investigation proceeded further with the aim of evaluate the LHTES of all the mortar formulations containing the form-stable PCMs under investigation in order to assess if the two PEGs were suitable, in terms of melting/crystallization temperature ranges, even when included in the mortars.

Figure 5a shows the DSC curves for the mortars containing LS/PEG1000 aggregates while Figure 5b reports the curves relative to the mortars containing LS/PEG800 aggregates; finally, in Figure 5c, the thermograms relative to the mortars containing the mixed PCM, i.e., 50%wt of LS/PEG800 and 50%wt of LS/PEG1000 aggregates, are illustrated.

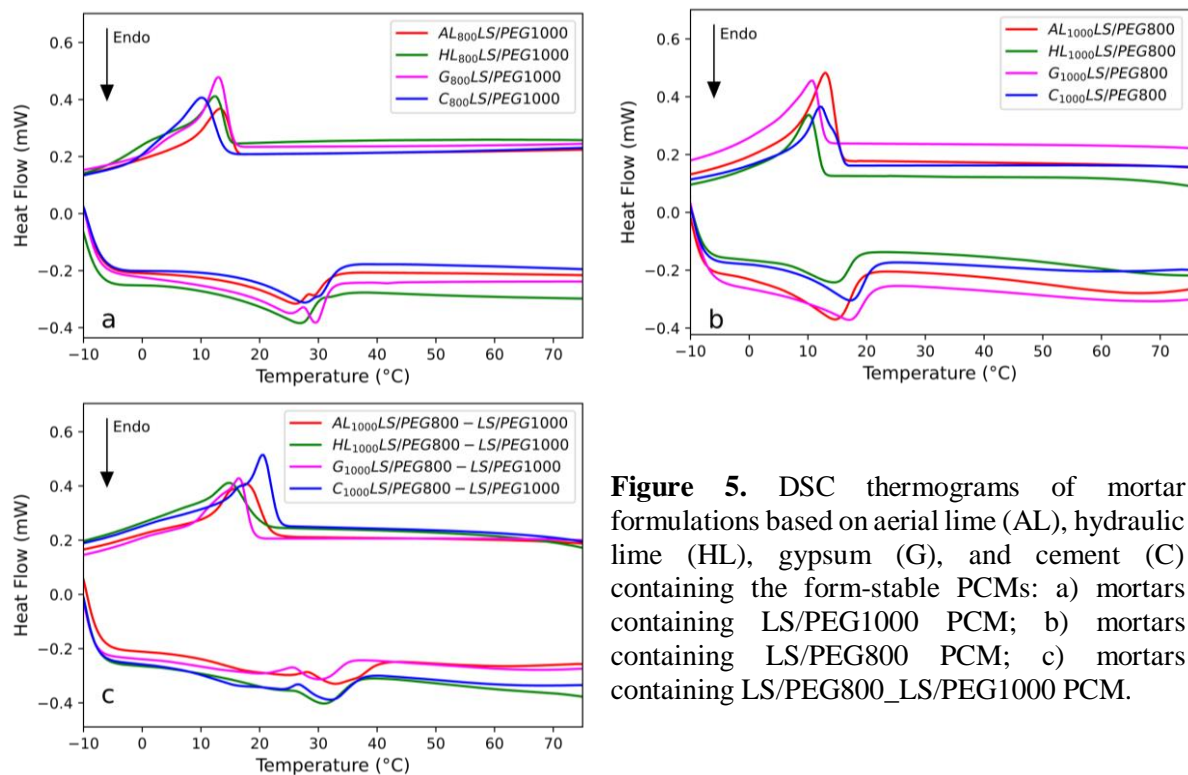


Figure 5. DSC thermograms of mortar formulations based on aerial lime (AL), hydraulic lime (HL), gypsum (G), and cement (C) containing the form-stable PCMs: a) mortars containing LS/PEG1000 PCM; b) mortars containing LS/PEG800 PCM; c) mortars containing LS/PEG800_LS/PEG1000 PCM.

In each graph, the peaks related to the melting of the polymeric component of any PCM in the heating stage and its crystallization in the cooling stage are always present; the peak temperatures for both melting and crystallization processes clearly depend on the specific PCM, and in turn on the specific PEG, added in each mortar. In Figure 3a, the characteristic melting/crystallization temperatures of the mortars resume those of the PEG contained in them, i.e., PEG1000. In fact, the characteristic temperatures in the melting phase range from a minimum of 2 °C to a maximum of 40 °C, with peaks settling around 30 °C. The thermal window in the cooling phase ranges from about 20 °C to 0 °C, with peaks around 12 °C. These temperatures are considered appropriate for warm climates, typical of the Mediterranean area. In Figure 3b, DSC curves of mortars containing the composite LS/PEG800 are presented. In this case, the characteristic temperatures recorded in the heating stage range from a minimum of -2 °C to a maximum of 25 °C with peaks around 16-17 °C. The characteristic temperatures recorded in the cooling stage range from a minimum of -6 °C to a maximum of 19 °C with peaks around 13 °C. In this case, it is evident that the phase change temperatures were shifted to lower values. This validates the idea that the use of PEG 800 is effective in colder geographic areas, i.e. those typical of continental climates. Finally, Figure 3c reports the DSC curves of mortars containing both a 50/50 mix of LS/PEG800 and LS/PEG1000: it is clear that in both melting and cooling phases the DSC curve are

wider than those relative to the mortars containing a single PEG. This is due to the thermal contribution of both PCMs. Consequently, the characteristic temperatures of these mortars are extended and the thermal window within which they are thermally active is wider. This mixed PCM could be, therefore, suitable for buildings located in different geographical areas or where a large temperature excursion is expected.

The characteristic temperatures and the thermal properties analyzed are summarized in Table 4. The test was repeated 3 times for each different mortar samples; the results were averaged, and they are presented with the corresponding experimental error. Through the thermal assessment was demonstrated that the latent heats of these mortars were sufficient to influence the internal temperature of an environment.

Table 4. Initial (T_i), peak, final temperatures (T_f) and enthalpy measured through DSC during heating (melting) and cooling (crystallization) stages on mortars based on different binders and containing different form-stable PCMs.

| H E A T I N G S T A G E | Sample | T_i (°C) | Peak _{melt} (°C) | T_f (°C) | ΔH_{melt} (J/g) |
|--|--|-------------------------------|---------------------------|-------------------------|-------------------------|
| | | AL ₈₀₀ _LS/PEG1000 | 2.8 ± 0.9 | 27.9 ± 0.5 | 34.5 ± 2.1 |
| | HL ₈₀₀ _LS/PEG1000 | 2.2 ± 1.0 | 26.0 ± 0.8 | 41.8 ± 1.1 | 7.9 ± 0.9 |
| | G ₈₀₀ _LS/PEG1000 | 2.7 ± 0.5 | 28.9 ± 1.0 | 35.9 ± 0.7 | 7.8 ± 1.2 |
| | C ₈₀₀ _LS/PEG1000 | 3.4 ± 0.1 | 30.0 ± 0.3 | 36.3 ± 0.1 | 7.7 ± 0.2 |
| | AL ₁₀₀₀ _LS/PEG800 | 3.3 ± 1.9 | 15.0 ± 1.0 | 24.1 ± 1.2 | 11.8 ± 0.4 |
| | HL ₁₀₀₀ _LS/PEG800 | -2.3 ± 0.8 | 14.0 ± 0.8 | 21.2 ± 0.5 | 9.1 ± 0.9 |
| | G ₁₀₀₀ _LS/PEG800 | 2.5 ± 1.1 | 16.4 ± 0.8 | 24.6 ± 2.0 | 7.8 ± 0.6 |
| | C ₁₀₀₀ _LS/PEG800 | -2.0 ± 0.8 | 17.3 ± 0.2 | 25.8 ± 1.8 | 9.5 ± 0.5 |
| | AL ₁₀₀₀ _LS/PEG800_LS/PEG1000 | 4.3 ± 0.9 | 32.3 ± 0.8 | 45.2 ± 0.5 | 9.7 ± 2.1 |
| | HL ₁₀₀₀ _LS/PEG800_LS/PEG1000 | 6.9 ± 3.1 | 32.4 ± 2.6 | 38.4 ± 0.8 | 9.1 ± 1.2 |
| | G ₁₀₀₀ _LS/PEG800_LS/PEG1000 | 3.2 ± 0.9 | 30.8 ± 1.6 | 38.9 ± 2.7 | 8.1 ± 0.4 |
| | C ₁₀₀₀ _LS/PEG800_LS/PEG1000 | 3.5 ± 1.8 | 33.5 ± 0.2 | 42.3 ± 0.4 | 9.7 ± 0.9 |
| C O O L I N G S T A G E | T_i (°C) | Peak _{crys} (°C) | T_f (°C) | ΔH_{crys} (J/g) | |
| | AL_LS ₈₀₀ /PEG1000 | 19.5 ± 2.1 | 14.6 ± 1.1 | 0.2 ± 1.3 | 8.8 ± 1.2 |
| | HL_LS ₈₀₀ /PEG1000 | 18.9 ± 0.8 | 13.5 ± 0.2 | 1.6 ± 0.4 | 6.0 ± 0.7 |
| | G_LS ₈₀₀ /PEG1000 | 16.9 ± 1.0 | 10.8 ± 0.7 | 0.6 ± 0.9 | 7.5 ± 1.2 |
| | C_LS ₈₀₀ /PEG1000 | 16.7 ± 0.2 | 10.8 ± 0.3 | -1.1 ± 0.2 | 8.7 ± 0.4 |
| | AL ₁₀₀₀ _LS/PEG800 | 17.7 ± 1.5 | 13.1 ± 1.1 | -6.4 ± 0.9 | 12.5 ± 1.0 |
| | HL ₁₀₀₀ _LS/PEG800 | 14.6 ± 1.4 | 12.4 ± 3.0 | -3.3 ± 0.6 | 10.3 ± 1.2 |
| | G ₁₀₀₀ _LS/PEG800 | 15.8 ± 1.9 | 11.5 ± 0.3 | -1.5 ± 0.9 | 9.2 ± 1.1 |
| | C ₁₀₀₀ _LS/PEG800 | 19.1 ± 0.6 | 13.0 ± 1.3 | -0.4 ± 0.2 | 10.5 ± 1.0 |
| | AL ₁₀₀₀ _LS/PEG800_LS/PEG1000 | 29.0 ± 0.9 | 18.6 ± 1.6 | -0.6 ± 1.2 | 10.8 ± 1.4 |
| | HL ₁₀₀₀ _LS/PEG800_LS/PEG1000 | 25.1 ± 0.7 | 15.2 ± 0.5 | -1.4 ± 2.2 | 9.2 ± 3.5 |
| | G ₁₀₀₀ _LS/PEG800_LS/PEG1000 | 25.7 ± 1.9 | 17.5 ± 2.3 | -1.8 ± 3.1 | 9.2 ± 1.8 |
| | C ₁₀₀₀ _LS/PEG800_LS/PEG1000 | 25.9 ± 0.5 | 19.5 ± 2.4 | 0.3 ± 0.2 | 11.3 ± 3.4 |

Experiments are currently underway to evaluate the storage/release properties of some of the mortars presented so far. In fact, an experimental setup has been set up to understand the thermal behavior of mortars containing LS/PEG800 and LS/PEG1000 when subjected to a controlled temperature program.

5. Conclusions

In this work, the main results of the workability test, mechanical and thermal analyses conducted on different mortar formulations containing three form-stable PCMs are presented. The PCMs were realized by impregnating (very porous) Lecce Stone granules with Polyethylene Glycol polymers

possessing different molecular weights, i.e., PEG 800 and PEG 1000. The obtained PCMs, LS/PEG800, LS/PEG1000 were included as aggregates in aerial lime, hydraulic lime, gypsum, and cement mortar mixtures. A mix of the two PCMs (LS/PEG800_LS/PEG1000 50/50 %wt) was added to the mortars based on the same binders. The workability of the fresh mortar mixes and the mechanical and thermal properties of the cured mortars were evaluated as a function of the PCM added, to establish the suitability of the mortars containing a PEG-based PCM. Although the addition in mortars of the PEG-based PCMs led to modifications in their characteristics, in both fresh and solid states, with a proper selection of the composition it was possible to obtain mixtures possessing suitable workability and mortars displaying adequate mechanical properties. Referring to the LHTES properties, the selection of a PEG characterized by higher melting/crystallization temperatures (i.e., PEG1000) allows to obtain mortars with phase change temperatures suitable for Mediterranean climates; on the other hand, the selection of a PEG characterized by lower melting/crystallization temperatures (i.e., PEG800) allows to obtain mortars with phase change temperatures suitable for continental climates. Furthermore, the mortars containing the mixed PCM showed a wider interval of melting/crystallization temperatures, suggesting that this mortar could be suitable in both warm and cold climates.

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