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# Assessing the feasibility of impregnating phase change materials in lightweight aggregate for development of thermal energy storage systems



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#### HIGHLIGHTS

- Feasibility of PCMs incorporation/encasement in LWAs.
- Leakage of impregnated/encased PCMs in LWAs under freeze/thawing test.
- Leakage of impregnated/encased PCMs in LWAs under drying test.
- SEM microstructural assessment of waterproof bound in LWAs.

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#### ABSTRACT

This paper assesses the feasibility of impregnation/encasement of phase change materials (PCMs) in lightweight aggregates (LWAs). An impregnation process was adopted to carry out the encasement study of two different PCMs in four different LWAs. The leakage of the impregnated/encased PCMs was studied when they were submitted to freeze/thawing and oven drying tests, separately. The results confirmed that, the impregnation/encasement method is effective with respect to the large thermal energy storage density, and can be suitable for applications were PCMs cannot be incorporated directly such as asphalt road pavements.

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#### 1. Introduction

Phase change materials (PCMs) are used as thermal storage systems for assisting thermal control, as a consequence of their ability to store and release thermal energy during phase change processes (melting and freezing) [1]. During the process of solidification, PCMs release energy in the form of latent heat, and conversely, when PCMs melt, they absorb thermal energy from the surroundings. The magnitude of energy stored in a given PCM subject to certain environmental conditions strongly depends on its transition temperatures [2,3]. Many numerical and experimental investigations have been carried out in order to evaluate the incorporation of PCMs into building materials [1,4,5]. For instance, PCMs have been incorporated into plastering mortars [6,7], masonry walls [8], tiles [9], concrete [10], asphalt road layers [11] and plaster boards [12]. It is also relevant to highlight selected earlier research work that specifically focused on the incorporation of PCMs in

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asphalt road layers so as to reduce the number of freeze/thaw cycles experienced by such pavements [13–15]. In such cases, the effectiveness of PCMs for in delaying or preventing freezing was shown to be critically dependent on: ambient temperature, phase change temperature of the PCM, and thermal characteristics of the concrete or mortar in which it is incorporated. Previous studies [13,14] concluded that the use of PCMs to prevent freeze/thaw cycles is a promising solution, and identified two areas that require further investigation: (1) the method of encasement used to incorporate PCMs in mortar/concrete/asphalt materials, and (2) characterizing and improving the thermal performance of the final impregnated/encased PCMs composite.

In general, thermal energy storage composites incorporating PCMs are normally fabricated via the incorporation of encased/encapsulated PCMs into porous materials without incurring any leakage of the PCMs from the final composite material. When PCMs are introduced into porous materials without being encapsulated, it becomes necessary to introduce a covering layer to seal the impregnated porous materials, in order to prevent leakage of the PCMs [16-18]. Otherwise, when the ambient temperature exceeds the melting temperature of the PCMs, the PCM can leak from the porous material and consequently jeopardize the performance of the system. Previous research has primarily focused on the preparation of shape stable encased/encapsulated PCMs [19] and/or on characterization of the thermal and mechanical properties of the composites [20,21], whilst the issue concerning PCMs leakage from porous material has not been adequately addressed. It must be stressed at this point that, possible leakage of PCMs will reduce the heat storage capacity of the composite and consequently will decrease the functionality and effectiveness of the PCMs composites [22]. Additionally, the PCM is usually organic and the presence of leaked PCM in the matrix may chemically react with it and bring deleterious effects on durability performance [23].

In this investigation, a series of composites were manufactured with different impregnated LWA types. A number of LWAs were selected possessing distinct compositions and physical properties [13,15,24]. The composites (impregnated/encased PCMs in LWAs) were subsequently surface coated with a number of commercial waterproofing solutions.

With the aid of multiple laboratory freeze/thaw and oven drying test cycles, accompanied by repeated mass loss measurements, the feasibility of permanent and effective encasement of PCMs in LWAs was evaluated and verified.

The present research work thus addresses the leakage potential of the impregnated/encased PCMs composites.

#### 2. Experimental program

#### 2.1. Materials

#### 2.1.1. Phase change materials

Two types of organic PCM paraffins were considered: R3 Rubitherm RT series (melting temperature of 3 °C) and R5 Rubitherm RT series (melting temperature of 5 °C) [25]. The properties of the PCMs selected for this study were provided by the manufacturer [25], and are presented in Table 1. Selection of melting temperatures for the PCM's under study was based on the target application of reducing freeze—thaw cycles in pavements. Therefore, the melting temperatures were selected to be slightly above 0 °C as to attenuate the enduring of such temperature within the mortars into which the PCMs are to be applied. Furthermore, chemical compatibility with the porous materials [14] was taken into account, as well as the range of available PCMs products in the market.

### 2.1.2. Lightweight aggregates

Geometrical features of the pore structure (including porosity, pore diameter distribution, pore connectivity and pore shape) and chemical compatibility are some of the important factors to be considered when selecting porous materials for impregnation with organic PCM [26,27]. Four LWAs were chosen for this study, both inorganic and organic lightweight aggregates were adopted: expanded clay (IC) supplied by ARGEX – SA (Portugal) [28]; granulated expanded cork (GC)

**Table 1** Properties of PCMs [25].

Materials	Melting area (°C)	Density-liquid phase at 15 °C (kg/m³)	Latent heat capacity ± 7.5% (kJ/kg)
R3	2-5	770	198.0
R5	1-6	770	180.0

supplied by SOFALCA/ISOCOR Co., Ltd. [29]; expanded perlite (AP) and expanded vermiculite (EV) supplied by URBICULT Unipessoal Ltd [30]. Such materials have been considered suitable for impregnation in previous works [15,31–33]. The particle grain size distributions of LWAs was assessed with sieving method [34], and the results are presented in Fig. 1.

#### 2.1.3. Waterproofing materials

Bearing in mind the importance of adequately coating the impregnated LWAs to avoid possible leakages of the PCMs, four different coating solutions were trialled (Table 2), namely: Sikalastic-490T (a polyurethane, transparent waterproofing liquid membrane) [35], Weber Dry Latic (a liquid membrane used for waterproofing roofs) [36], Makote 3 (a waterproofing bituminous emulsion from MC-Bauchemie) [37] and ECM-2 from CEPSA (a cationic bituminous emulsion for cold asphalt mixtures) [38]. These waterproofing coating solutions need to have service temperature ranges adequate for the end applications aimed at in this research.

#### 2.2. Proposed procedures for encasement of PCMs and surface waterproofing

The absorption of different PCMs paraffin waxes into different LWAs was measured in accordance with EN 1097-6 [39]. The procedure for preparation of encased thermal energy storage LWAs regardless of PCM types are shown in Fig. 2. In the first stage, all lightweight aggregates were exposed to a jet of compressed air to remove dust and any loose superficial residue from the surfaces of the particles (see Fig. 2a). The lightweight aggregates were next dried in a ventilated oven until a constant weight was achieved. The duration of drying was a minimum of 24 h at temperatures of 110 °C, 80 °C, 80 °C and 65 °C for the IC, AP, EV and GC respectively. The temperature adopted to dry the granulated expanded cork (GC) was slightly lower than the rest so as to cause minimal damage to the cork internal structure. It should be noted that, the imposed drying temperatures on the LWAs were adequate since each type of LWAs was monitored using an electronic moisture meter (model KERN MLB\_N) and the materials was classified as dry only when the remaining moisture content was less than 1 mg of water per 0.01 kg of LWA (see Fig. 2b). The LWAs were subsequently cooled down to room temperature (approx. 22 °C) for 2 h. Following drying, a representative sample from each LWA was completely immersed in each PCM for 24 h (see Fig. 2c). Impregnated LWAs samples were next drained over filter paper to remove the excess of PCM for 2 h at room temperature, which incidentally was above the phase change temperature (see Fig. 2d). The surfaces of the impregnated LWAs were then dried with an absorbent sheet of paper.

The impregnated LWAs were subsequently soaked with different waterproof coating solutions until the surface of the particles was fully coated. This waterproof coating procedure was also performed at room temperature, i.e., above the phase change temperature of the PCMs. Afterwards, the soaked/impregnated/waterproofed LWAs were allowed to dry in the laboratory environment (room temperature/humidity), according to the necessary drying time of each waterproofing material type, as shown in Table 2. Then, the samples were then oven dried at 60 °C for 24 h and were next left to dry at laboratory environment for a further 7 days. Weight variations were monitored during the entire process, and it could be confirmed that all samples had weight variations of less than 0.1 percentage during the last three days of the process, thus indicating a complete hydrothermal equilibrium state. Therefore, the product obtained at the end could be considered as emulating an industrial process of encapsulation/encasement of PCM's.

#### 2.3. Research program and test procedures

In this investigation, the research program consists of three testing phases: (i) material testing; (ii) testing of PCM impregnated/encased LWAs and; (iii) performance of hardened mortar under thermal cycle loads.

#### 2.3.1. Material testing

2.3.1.1. LWAs testing. In the first phase of material testing, each type of non-impregnated LWAs (IC, AP, EV and GC) was submitted to the following tests in order to characterize their capacity for impregnation: (1) density analysis, (2) pore structure analysis and (3) absorption amount test.

With respect to particle density testing, a total of 12 LWA specimens were analyzed for saturated surface dry density and the results compared to the LWAs dry density values. Particle density testing consisted of 4 representative samples from each LWAs system (IC, AP, EV or GC) with 3 repeating specimens from each LWA. Particle density determination was carried in accordance with EN 1097-6 [39]: (i)

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