

Thermal energy storage-enhanced self-leveling mortars made of pre-placed perlite aggregates filled with bio-based phase change materials[☆]

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ABSTRACT

This study investigates the development and performance of Self-Leveling Mortars (SLMs) enhanced with Phase Change Materials (PCMs) and expanded perlite aggregates to provide Thermal Energy Storage (TES) functionality for energy-efficient buildings. A cementitious encapsulation method was used to coat PCM-saturated perlite, forming durable macro-encapsulated aggregates. To preserve the structural integrity of these inclusions during casting, the pre-placed aggregate method was employed in combination with a highly flowable self-consolidating mortar matrix. Three mortar formulations were produced and analyzed: (a) reference SLM, (b) SLM with expanded perlite, and (c) SLM with PCM-filled perlite. Thermal performance was assessed through dynamic “DKK” tests and a custom ASTM C1363 Hot-Box apparatus. Tests reveal that PCM-enhanced mortars achieved the highest thermal buffering effect, exhibiting plateaus at 25–30 °C and reduced heat flux values. The apparent thermal conductivity, during the PCM phase change, dropped to 0.28 W/m × K, demonstrating effective latent heat absorption. The results clearly demonstrate enhanced passive thermal regulation with delayed heat transfer responses, supporting the use of PCM-perlite SLMs as energy-storage building materials. Complementary tests were then performed to verify material, mechanical and durability feasibility of the composites. Mechanical tests showed that the reference SLM achieved the highest compressive and flexural strengths, at 60.14 MPa and 5.79 MPa, respectively. The incorporation of expanded perlite reduced the compressive strength to 29.02 MPa, while PCM-enhanced aggregates further reduced it to 17.62 MPa. A slight improvement in flexural strength was observed for the PCM composite (i.e., 3.46 MPa) compared to the uncoated perlite (3.08 MPa), indicating potential reinforcement by the encapsulation shell. Water absorption tests revealed that PCM-enhanced perlite mortars exhibited low absorption within the first 180 min but experienced a sharp increase at 72 h (i.e., 3 days), surpassing all other mixes and suggesting long-term permeability issues. SEM-EDS analyses indicated high Ca/Si ratios (~7.75) in the shell, and TGA results showed minimal portlandite decomposition, both supporting the hypothesis of calcium hydroxide consumption by silica fume to form additional C–S–H gels. Overall, PCM-perlite SLM shows strong potential for TES-enabled building applications.

1. Introduction

The growing demand for energy-efficient and environmentally sustainable construction materials has led to significant innovations in the development of carbon-neutral cementitious composites. In the EU, the building sector is one of the most energy consumer, accounting for

nearly 40% of final energy consumption and responsible of around 36% of CO₂ emissions [1]. These numbers are largely driven by existing (often non-retrofitted) building stock, where typical residential energy consume typically ranges within 160–220 kWh/m²·year (i.e., heating + electricity) [2]. Such values remain far from Nearly-Zero Energy

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Buildings (NZEBS) targets and represent a clear call to action for current carbon-neutral EU directives [3].

In this context, expanded perlite emerged as a promising lightweight aggregate with remarkable potential in improving the thermal insulation properties of cement-based mortars [45]. Perlites is an amorphous volcanic aluminosilicate that expands when heated, creating an expanded cellular structure that provides low thermal conductivity and also contributes to enhanced acoustic insulation [6]. By incorporating perlite into cementitious materials, it is possible to reduce density and thermal conductivity, resulting in a more efficient material for energy-efficient buildings [7].

Despite these clear and well-known benefits of expanded perlite, one significant challenge in its application still lies in its highly porous nature, which leads to increased water demand in mortar mixes. This, in turn, can negatively affect the mechanical properties of the material (i.e., both compressive and tensile strengths). Recent studies have explored strategies to address this issue, including optimizing perlite granulometry and combining perlite via hybrid formulations with other sustainable constituents. These strategies aim to balance the thermal insulation benefits of perlite while meeting the mechanical and durability requirements of the designed composites [8].

Macro-encapsulation of Phase Change Materials (PCMs), as well as other thermal regulating/energy storing materials, can offer significant advantages in construction, particularly in reducing the need for active heating and cooling by providing passive thermal buffering [9]. These materials can absorb excess heat during the day and release it during cooler nighttime temperatures, thus (passively) maintaining more stable indoor climates. In the context of cementitious matrices, this capability is particularly beneficial for buildings in regions with high diurnal temperature variation [10]. In cement-based systems, macro-encapsulation of PCMs is crucial since it can help prevent the leakage of these latent-heat thermal materials over time, ensuring that the matrix continues to perform efficiently over long periods [11].

While the combined integration of expanded perlite and PCMs in cement-based composites has demonstrated promising results, there are still challenges that must be addressed. One of the primary concerns is the impact of macro-encapsulation on the mechanical properties of the matrix [12]. The inclusion of additional phases, particularly porous carriers and encapsulated inclusions, can alter the micro- and macro-structures of the cement matrix, potentially reducing its strength and durability. Therefore, achieving an optimal balance between thermal and mechanical performances still remains a critical aspect of ongoing research in this field [10].

From a technology perspective, the production of cementitious materials incorporating macro-encapsulated components must be also carefully managed. One of the critical challenges associated with the incorporation of rigid or brittle mineral aggregates, such as expanded perlite or commercially available macro-encapsulated phase change materials, into cementitious composites is their susceptibility to fracture during conventional mixing and casting. These fragile inclusions can break under mechanical stress, compromising both their intended functionality and the overall performance of the TES composite. In particular, the rupture of (either nano[13], micro [14] or macro [15]) PCM capsules can lead to leakage, reduced thermal efficiency, and adverse interactions with the surrounding cement matrix [16].

To mitigate these issues, advanced placement techniques that limit damage stresses on fragile aggregates are required. In this study, a self-consolidating technique [17] together with the pre-placed aggregate casting method [18] are employed to enable gravity-driven (highly-flowable) infiltration without vibration, thereby reducing mixing- and placement-induced damages. Self-consolidating concrete (SCC) is characterized by its high fluidity and ability to flow under its own weight, filling voids and surrounding the aggregates without inducing significant shear or compressive stresses [19]. This method helps to not only preserve the integrity of brittle aggregates but also enhances the homo-

geneity and surface finish of the final composite, making it a valuable approach for producing mortars with sensitive functional components [20].

This research aims to address these challenges by optimizing and integrating macro-encapsulated PCM into self-leveling cement-based mortars (SLMs) using the pre-placed aggregate method. By systematically evaluating the granulometry, encapsulation strategy, and thermal/mechanical responses of the resulting mortars, this study proposes a design methodology for advanced building materials capable of combining thermal energy storage with adequate structural performance. The work contributes to the development of energy-efficient construction materials aligned with sustainable building practices. Although porous carriers for PCM-cementitious composites have been investigated in several studies of literature, the development of latent-heat-enhanced self-leveling mortars produced via pre-placed casting remains quite limited for TES-oriented building applications. The proposed approach addresses two key research gaps: (i) preserving fragile PCM-filled porous aggregates due to mixing-induced damage, and (ii) enabling higher and unprecedented aggregate plus PCM content with no leakage, thus delivering an enhanced TES response under both steady-state and dynamic conditions.

2. Materials and methods

2.1. Materials

A Portland Cement from *Italcementi Tecnomem* 32.5 R (CEM II/B-LL 32.5 R) was used for both coating (cement-based encapsulation) and mortar production. This binder was selected for its chemical compatibility and quite similar hydration behavior in both matrix and the coating, allowing to improve the mechanical stability at the aggregate-matrix interface. Moreover, its early-age fresh performance and workability were adequate for making flowable self-leveling mortars (SLMs), as required for the pre-placed aggregate method.

High-purity fine silica sand (> 99%) and expanded perlite with a maximum size of 4.75 cm were used as a highly porous aggregates, employed as a carrier for PCMs. Its pore network allowed huge amounts of PCM uptake, increasing the latent heat storage of the final composite. The particle size distribution of the perlite is provided in Table 1, Table 2 summarizes the physical and mechanical properties of both sand and perlite, while their chemical compositions are detailed in Table 3. The information regarding granulometry, density, and absorption were obtained by conducting tests according to ASTM C136/C136M [21] and ASTM C29/C29M [22].

Silica fume (SF) with high purity (92.5% of the SiO₂) was used as a pozzolanic material. SF can tendentially enhance the microstructure of the matrix via formation of additional C-S-H gels and at the same time can improve the stability of the coating around the PCM-saturated perlite aggregates. To produce a self-flowable mortar, a powdered Polycarboxylate Ether (PCE) superplasticizer was used in this study, characterized by a white powder appearance, a bulk density of 610 kg/m³, and with a moisture content lower than 3.0%.

Table 1
Granulometric analysis of perlite aggregates.

Sieve Size (Code)	Sieve Size (mm)	Passing (%)
3/8"	9.5	100
#4	4.75	99.9
#8	2.36	51.8
#16	1.18	22
#30	0.6	13.6
#50	0.3	8.5
#100	0.15	3.6
#200	0.075	1.9

Table 2

Physical and mechanical analysis of perlite and silica sand aggregates.

Material	Particle Size (mm)	"BET" test (m ² /g)	Water Absorption (%)	Electrical Conductivity μ S/cm	Apparent Specific Gravity (g/cm ³)	Bulk density (kg/m ³)	Hardness (Mohs)	Thermal Conductivity (W/m $\times$ K)
Silica Sand	9×10^{-6} – 16×10^{-6}	< 3	21.50	< 5	2.18	845.00	6.00	~1.30
Perlite	0.075–4.75	–	166.42	> 500	468.17	116.90	5.50	0.05

Table 3

Chemical analysis of perlite and silica sand aggregates.

Component	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	K ₂ O	Na ₂ O ₃	Loss on Ignition (at 1000 °C)
Silica sand	> 99.70%	< 1700 ppm	< 350 ppm	< 250 ppm	< 120 ppm	< 50 ppm	< 0.50%
Perlite	69.99%	10.21%	1.48%	–	5.13%	4.79%	4.66%

In this study, PureTemp 25, a USDA-certified biobased [23] Phase Change Material (PCM), was used. It is a bio-based and biodegradable compound and belongs to the class of medium-temperature PCMs with a melting point of 25 °C. This outcome is also consistent with recent evidence showing that PCM-enhanced cementitious systems can provide tunable thermal energy storage and dynamic insulation performance, where the PCM transition temperature and content are key parameters governing the coupled energy-structural response. Particularly, its melting range is within the typical indoor comfort window to maximize the effectiveness of passive thermal regulation in building applications. Importantly, this choice was not subjective since it followed a previous screening of multiple candidate PCMs (spanning from 18 °C to 28 °C) within a previous multi-layer *meta*-building/*meta*-envelope design framework analyzed in previous works [24]. The optimal configurations showed that the best energy performance was achieved when high PCM fraction was concentrated in inner layers of buildings and walls, with melting temperatures predominantly in the 23–25 °C range, while minor contributions from adjacent transition temperatures depending on boundary conditions were registered.

The physical analysis of the PCM used is presented in Table 4, while its latent heat is evaluated by using the DSC tests.

To make encapsulated the PCM-enhanced perlite aggregates four steps were followed (see Table 5): i) the PCMs were initially heated in an oven at a controlled temperature of 40 °C until they reach a uniform liquid state; ii) the melted bio-based PCMs were poured into a glass container, added perlite, and placed under these conditions for 30 min; iii) the excess of bio-based PCM was removed from the surface by pouring the saturated aggregates into a colander and placing them in the refrigerator for an additional 30 min, resulting in 142.19% of weight variation due to PCMs absorption. In this step, the PCMs-enhanced perlite aggregates were thus saturated for approximately 5 s. Fig. 1 shows how perlite aggregates look before and after PCM impregnation. Then, iv) the PCM-aggregates were mixed (dry pre-coated method, see [25]) with cement type 2; v) this step was then repeated two more times, generating a total of 3 layers of cement, and in each instance, clean water was sprayed on the surface of the aggregate (using a spray bottle) until uniformly encapsulated aggregates were achieved. Cement-based shell weight was equal to 216% of the pure perlite in dry conditions.

The base mix design was calculated with 750.0 kg of cement per cubic meter, including 11% of silica fume by weight of cement, and utilizing 1237.4 kg of fine silica sand as aggregate (Table 6). The required

amount of water was calculated based on the volume of pozzolan using the Absolute Volume Equivalency, as mentioned in ACI 211.1–91 [26]. The water-to-powder ratio was 0.31, and the polycarboxylate ether superplasticizer accounts for 1% of the total cement-based materials. Tap water was used to produce all mixtures.

Three types of mortar were produced and studied: a) a reference mortar, being self-consolidating; b) pre-placed perlite mortar; c) pre-placed PCM-enhanced perlite mortar. In types 'b' and 'c', pre-placed perlite aggregate was put into the mold that has been capped with a heavy steel plate, leaving only a small area open for gravity filling of the fluid matrix (i.e., the reference SLM). After casting, this area is covered with a plate. All aggregates were maintained in the Saturated Surface Dry (SSD) condition prior to gravity filling using the preplaced method. The SSD condition was reached after an oven drying process at 90 °C and 55 °C, until mass constancy for raw perlite and PCM-aggregates, respectively.

2.2. Supporting tests: Mechanical and durability

2.2.1. Fresh properties

The flow properties of the developed mortars were evaluated according to ASTM C1437-20 using a standard flow table [27]. A critical issue in this study was related to the mechanical mixing process. Conventional casting procedures are characterized by rotational forces and collisions among aggregates or between them with the mixer blades, which often generate micro-cracking or rupture of the encapsulated PCM shells [28293031].

To mitigate the mentioned problem and to keep the integrity of the PCM carriers, a pre-placed aggregate concrete method was employed [323334]. In this approach, the PCM-perlite aggregates after saturation were filled into the frame, and then the self-consolidating mortar was poured into the partially capped mold using the gravity-assisted injection.

2.2.2. Water absorption test

The methodology for water absorption was based on ASTM C642-21. Due to the presence of PCMs in the mortar, which may be sensitive to high temperatures, the oven-drying step was modified from the classical procedure. Instead of drying at 110 °C as prescribed by the standard, the specimens were placed in an oven at 55 °C for 48 h to prevent potential PCM degradation. The drying process was complete when the

Table 4

PureTemp 25 (bio-based) PCM physical and chemical characterization.

Solid Density (g/cm ³)	Liquid Density (g/cm ³)	Latent Heat of Fusion (J/g)	Specific Heat (Solid) (J/g $\times$ °C)	Specific Heat (Liquid) (J/g $\times$ °C)	Thermal Conductivity (Solid) (W/m $\times$ K)	Thermal Conductivity (Liquid) (W/m $\times$ K)	Melting Point (°C)
0.95	0.86	187	1.99	2.29	0.25	0.15	25.2 $\pm$ 1.1

Table 5

PCM-enhanced perlite weight changes in different stages of production: weight variation of PCM-Enhanced Aggregate Compared to Raw Perlite.

Steps	Production Stage	Weight Variation (% of Raw Perlite)
1	Raw Perlite	–
2	PCMs Absorption	+ 142.19
3	Cement-Based Shell Encapsulation	+ 523.24
4	Final Encapsulated PCM-Enhanced Aggregate	+ 665.43
PCM Content in Final Aggregate = 18.58% of total weight		



Fig. 1. Perlite aggregates (left caption) and PCM-enhanced aggregates (right caption).

change in mass between two consecutive measurements was less than 5% [35].

The method allows to determine the apparent density and volume of permeable voids (i.e., open porosity) in the composite specimens. Given the use of a lightweight mortar, additional early-stage mass measurements were conducted to better assess the initial absorption kinetics and drying behavior. Mass recordings were performed at 30, 60, 120, and 180 min, in addition to the standard time points, following a procedure like EN 1015–18, which is commonly applied to fresh and hardened mortars. These intermediate measurements allowed for a more detailed understanding of the mass stabilization trend and moisture transport characteristics of the material.

2.2.3. Mechanical properties

The mechanical properties of the mortars were evaluated in terms of flexural and compressive strengths by considering the methodology of ISO 679: 40 × 40 × 160 mm prisms were cured for 28 days in lime-saturated water. The tests were then conducted with a Zwick/Roell Z050 universal testing machine using the three-point bending method. After flexural testing, the resulting half cuts (40 × 40 mm cross-section) were used to assess compressive strengths using a calibrated compression testing machine [36].

2.2.4. Thermogravimetric analysis (TGA), derivative Thermogravimetry (DTG), and Differential thermal analysis (DTA)

TGA, DTG and DTA tests and analyses were performed using the NETZSCH STA 409 instrument. Approximately 108.2 mg of the samples per each mixture was placed in an alumina (Al₂O₃) crucible, and the analysis was carried out under a static air atmosphere. The temperature

increased from room temperature up to the desired maximum/minimum value at a constant heating/cooling rate of 1 °C/min [37].

2.3. Thermal energy storage performance and heat-transfer assessment

2.3.1. Dynamische Kugel Kalorimetrie (DKK) tests

The *Dynamische Kugel Kalorimetrie* test (patented by Caggiano et al., 2018 DE102018125943A1) was conducted to evaluate the thermal energy storage behavior of the prepared composite samples. DKK, which stands for “Dynamic Spherical Calorimetry” is an experimental method designed to simulate realistic heating and cooling cycles on spherical specimens capturing their transient thermal response [38].

To conduct the DKK test, spherical specimens with 80 mm diameter, were cast with a K-type thermocouple at the barycenter of the sample (see Fig. 2). The test was conducted with samples at 28 days age. The sensors were then connected to a Picolog TC-08 datalogger. Samples were exposed to controlled thermal conditions: initially, each sample was placed in a climatic chamber at 10 °C, below the PCM solidification point. Afterward, by increasing the temperature to 50 °C (chamber temperature, without ventilation nor forced convection). Once the temperature stabilizes in the heating process, the chamber temperatures were again set to decrease to 10 °C (cooling process).

This procedure enabled the identification of thermal delays and plateaus indicative of latent heat absorption and release on the studied samples. The data from the DKK test has been recorded every 30 s. The heating rate in the chamber starts at 0.024 °C/s, and after reaching a steady state, it was maximally 0.003 °C/s. For the cooling phase, the starting rate is recorded at –0.014 °C/s, and in a steady state, it was –0.0002 °C/s.

2.3.2. Specific heat capacities (C_p) and thermal conductivity

The specific heat capacity (C_p) of the non-latent components (i.e., SLM mortar and perlite aggregates) was measured using a high-precision heat flux calorimeter (Setaram C80 Calvet).

The specific heat capacity C_p is calculated using the following equation:

$$C_p = \frac{Q}{m \cdot \Delta T} \quad (1)$$

where C_p is the specific heat capacity (in J/kg × K), Q is the heat absorbed (in Joules), m is the sample mass (in kg), and ΔT is the temperature rise (in Kelvin or °C). This measurement procedure follows the principles of ASTM E1269 [39], adapted for use with the Calvet 3D heat flux sensor.

For PCM-enhanced materials that exhibit dynamic and temperature-dependent behavior, their thermal characteristics, including specific heat capacity and thermal conductivity, were assessed using dynamic methods, as these properties fluctuate with temperature [40,41,42,43]. More specifically, the thermal behavior of PureTemp 25 was investigated using a heat-flux Differential Scanning Calorimetry (DSC) approach, strictly following the IEA SHC Task 42 / ECES Annex 29 (DSC 4229) standard [44]. The experiment was conducted using a DSC 300 Caliris® Classic from NETZSCH, which operates on the heat-flux principle. For evaluating enthalpy, ASTM E793 [45] is referenced, along with ASTM E794 [46] for measuring the melting point and solidification temperature. The tests were conducted at different heating and cooling rates of 0.125, 0.25, 0.5, 1, and 2 K/min, with the results at 0.25 K/min

Table 6

Base mix design used for (a) SLM | (b) SLM + Perlite | (c) SLM + Perlite + PCM mortars.

Mortar code	Cement (C)	Silica Fume (SF)	SP (PCE)*	Fine Sand**	w***/(C + P)	SP/(C + P****)	P/C
SLM	750.00 kg	82.50 kg	8.33 kg	1237.40 kg	0.31	1.00%	11.00%

* SP (PCE): Polycarbonate Ether Superplasticizer; ** Based on the size of the silica sand (mesh 2500), water absorption for the SSD condition was 21.50%; *** w = water; **** P: Pozzolan.

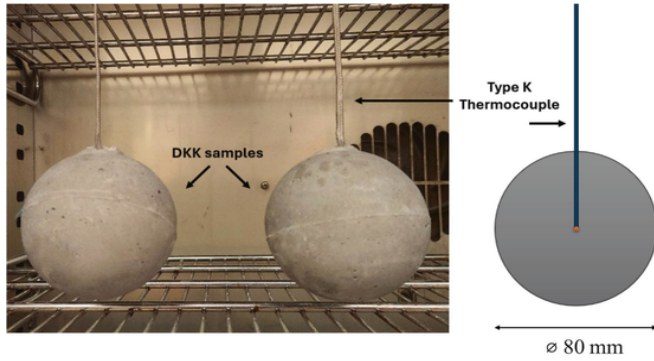


Fig. 2. Experimental setup for the DKK test: mortar sphere ($\varnothing$ 80 mm) suspended in an oven, equipped with a centrally embedded K-type thermocouple for internal temperature monitoring.

reflecting the enthalpy of the PCMs, as recommended for bio-based phase change materials, to ensure thermal equilibrium and minimize their influence on subcooling. The sample, weighing 12.21 mg, was placed in a pierced-lid aluminum crucible under nitrogen flow, and the complete melting and crystallization range was covered between 0 °C and 40 °C. Isothermal holds were included before and after each ramp to stabilize the thermal baseline. The high sensitivity of DSC enables accurate detection of enthalpy, phase transitions, and latent heat effects. Typically, only a small quantity of material (i.e., order of milligrams) is needed for testing. The specific heat capacity $C_p(T)$ at any given temperature (T) and in ($J/kg \times K$) can be derived from the heat flow signal using the following relationship:

$$C_p(T) = \frac{\dot{Q}}{\beta \cdot m} \quad (2)$$

where $\dot{Q}$ is the measured heat flow (W), β represents the heating rate (K/s), and m denotes the sample mass (kg). The actual $C_p(T)$ values were obtained by comparing the sample measured heat flow with that of a calibrated reference material, such as sapphire [454647].

The SLM mortar thermal conductivities were calculated using the approach of ASTM C1363-14 [48]. All tests were conducted with samples at 28 days age. The experimental investigation was carried out using a custom-designed Hot Box apparatus (see Fig. 3 and Table 7) built from two EPS thermal chambers, each with outer dimensions of 40 × 30 × 30 cm and an internal space of 34 × 24 × 24 cm. The box walls were 3 cm thick, providing sufficient thermal insulation to maintain stable boundary conditions during the test. To measure the heat flow uniformly, two axial fans (1700 PWM) produce a static pressure of 2.83 mm H₂O while maintaining a low noise level of 25.1 dB(A). Their high-pressure, balanced airflow made them suitable for controlled thermal convection within the enclosed environment. With adjustable rotating speeds, the fans were placed at the top and bottom before the test to ensure uniform airflow on both sides. Each fan was mounted 5 cm from the ceiling. After each fan, a separating sheet was placed in the middle of the polystyrene sheet, leaving a 5 cm gap on each side.

The test sample was a rectangular mortar sample (4 cm thickness and 4 × 16 cm surface area = 0.0064 m²). The lower box contains the heating system, while the upper box houses the cooling system. The heating system utilizes a series of heating elements, and the cooling system employs Peltier modules. Between these two EPS boxes, an additional EPS sheet was placed with a central cutout measuring 4x16 cm. Within this cutout, samples measuring with embedded K-type thermocouple sensors were placed. Eight Type-K thermocouples (accuracy ± 1.5 °C with range -200 °C to + 1350 °C) were employed to record surface and internal temperature and were placed in various sections. The thermocouples were positioned after the cooling or heating

elements, after the fans, and on the surface of the sample on both sides. This arrangement enables the measurement of temperature variations across all critical sections of the sample. In addition to the thermocouples placed inside the sample, heat flux sensors with a sensitivity of 3×10^{-6} V/(W/m²) with a range up to 1000 (W/m²), were positioned on the surface of the mortar sample. A Hioki data logger model LR-8432-20 was employed for this purpose, recording data every 10 s throughout the test duration.

The heat flux sensor measured both heat flow and temperature at the respective surface location. Care was taken to ensure that the entry and exit points for the numerous cables were sealed at the EPS boxes. Using the temperature difference between the hot and cold surfaces, the measured heat flux, and the physical dimensions of the specimen, the apparent thermal conductivity (λ) was calculated using the Fourier's law:

$$\lambda = \frac{Q \cdot L}{A \cdot (T_1 - T_2)} \quad (3)$$

where Q is the heat flux (W/m²), L is the specimen thickness (0.04 m), A is the surface area (0.0064 m²), and $T_1 - T_2$ is the temperature gradient across the specimen. This setup enabled a reliable and reproducible evaluation of the material thermal behavior under well-controlled and measurable conditions. Employing the ASTM 1363-14 method, which has been employed to calculate the relative thermal conductivities of the SLM, the thermal properties of the Perlite SLM mortar and PCM-enhanced Perlite SLM mortar were evaluated, and the Thermal Transmittance (i.e., $U - \text{value in } W/(m^2 \cdot K)$), Thermal Resistance ($R - \text{value in } m^2 \cdot K/W$), Apparent Thermal Conductivity ($\lambda - \text{value in } W/(m \cdot K)$), Heat Flow Rate ($Q - \text{value in } W$), Surface Resistances ($R_h, R_c - \text{value in } m^2 \cdot K/W$), Heat Transfer Coefficients ($h_h, h_c - \text{value in } W/(m^2 \cdot K)$) were extracted. The thermal performance of SLM mortars integrated with or without PCM-enhanced perlite aggregates were evaluated in their phase, before, during and after melting temperature.

2.3.3. Microstructural and chemical analysis using Scanning electron Microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDS)

The microstructure and elemental composition of cement-based samples were studied by Scanning Electron Microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS), respectively. SEM images as well as EDS spectra were collected by using a CeB6 source operated at 15 kV in ultra-high vacuum (UHV). Secondary electron detector (SED) was used to acquire SEM images at magnifications of 1000 × and 10,000 × to visualize the porous structure of the expanded perlite and its interaction with the cement paste matrix, including lamellae, cavities, and hydration products. EDS detector presents an accuracy better than 0.2 wt% (wt. %), energy resolution above 0.13 keV as well as atomic-number, absorption, and fluorescence (ZAF) correction. In all cases, EDS point analyses were performed to quantify elemental compositions, specifically calcium, silicon, aluminum, sulfur, and iron in localized portions of the cement-perlite interface. Samples were coated by thin conductive layers using a gold sputtering machine to collect high-quality images.

2.3.4. Analysis of crystalline phases and structural properties by X-ray diffraction (XRD)

Crystallographic properties of the cementitious samples in powders were evaluated by X-ray diffraction (XRD) at room temperature in air. PANalytical AERIS equipment was operated at 30 kV and 10 mA, with Bragg-Brentano geometry, Cu K α radiation, Ni filter and PIXcel1D detector. XRD patterns were collected with an angular range (2θ) of 15°-90° and step size of 0.022°. Profex software allowed indexing the compounds present in these complex samples [49]. Powder Diffraction File (PDF) cards with the best pattern quality possible (e.g., Star) were se-

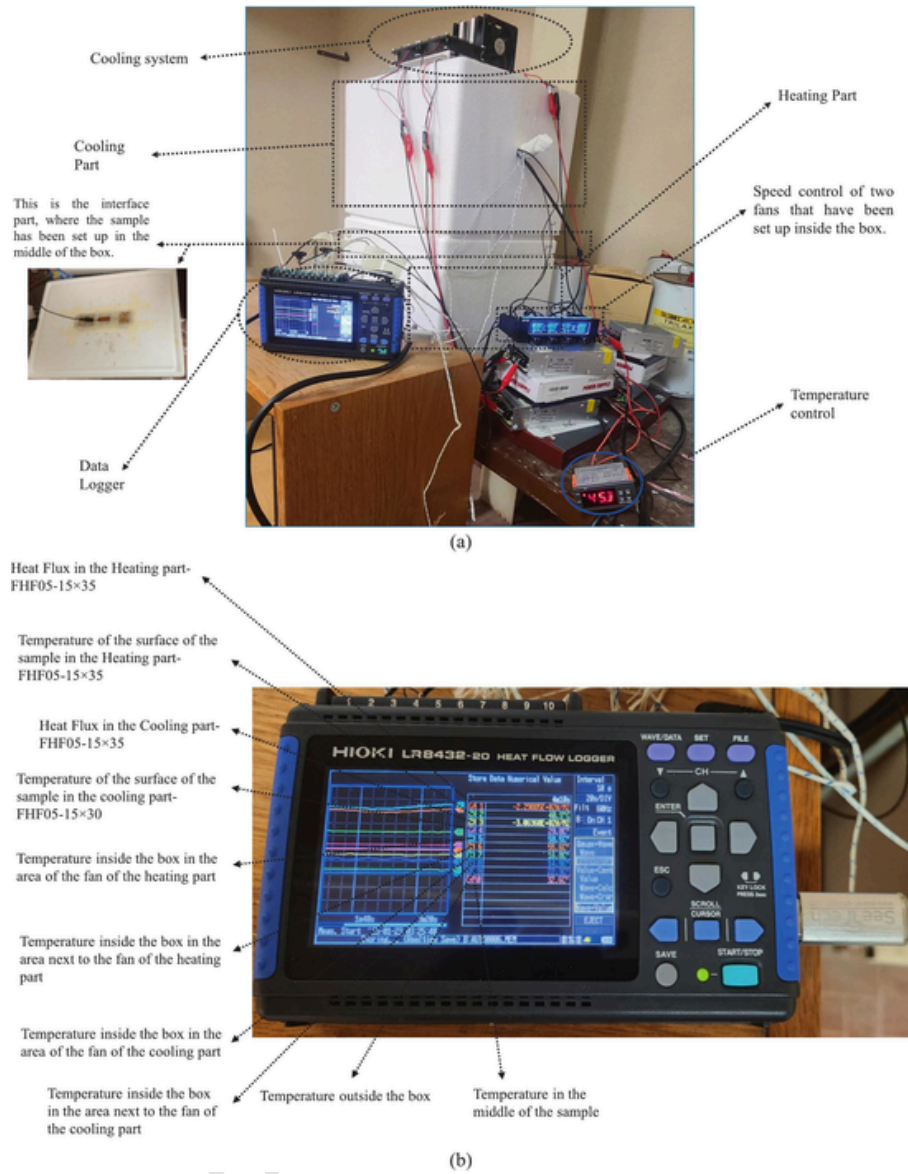


Fig. 3. (a) Customized Hot Box setup for thermal conductivity based on ASTM C1363-14. The system includes EPS thermal chambers with heating and cooling units, axial fans for uniform airflow, embedded sensors (K-type thermocouples and heat flux sensors) to monitor temperatures and heat flows, and (b) a heat flow datalogger.

lected [5051] and Fullprof suite software was used to estimate the content in wt. % of each phase by using the Rietveld Method [52].

2.4. Overview of the experimental plan

Table 8 summarizes the experimental campaign and methods employed to evaluate the fresh, mechanical, thermal, and microstructural properties of three mortars: i.e., the self-leveling mortar (SLM), the perlite-based SLM, and the PCM-enhanced perlite formulation. It shows the number of specimens or repetitions tested for each mortar type along with the code, standards or guidelines employed.

3. Results

3.1. Workability

The reference mortar exhibited good flowability, functioning effectively as a self-consolidating mortar without indication of bleeding or segregation. The flow table test was conducted in accordance with ASTM C1437-20 [27], using the standard flow mold (with a base diameter of 100 mm) as specified in ASTM C230/C230M-23 [53] (see Table 9). In both perlite-based SLM and PCM-enhanced ones, the preplaced aggregate method was utilized. Aggregates were initially packed into mini slump molds, and the self-consolidating mortar was cast around them within the testing frame. The procedure was further modified and the test carried out without performing the 25 drops of the flow table. This approach was designed to assess the natural fluidity of fresh mortar under its own weight.

Table 8

Base mix design used for (a) SLM | (b) SLM + Perlite | (c) SLM + Perlite + PCM.

Test Type	SLM	Perlite SLM	PCM-Perlite SLM
Flow test (ASTM C1437-20)	for each batch	for each batch	for each batch
Water absorption (ASTM C642-21, modified)	3 specimens	3 specimens	3 specimens
Flexural strength (ISO 679)	3 prisms (3-point bending)	3 prisms (3-point bending)	3 prisms (3-point bending)
Compressive strength (ISO 679)	3 halves from flexural	3 halves from flexural	3 halves from flexural
Dynamic Spherical Calorimetry (DKK)	2 spheres (Ø 80 mm)	2 spheres (Ø 80 mm)	2 spheres (Ø 80 mm)
Specific heat capacity (Heat Flux Calorimeter-HFC) & DSC)	1 sample via HFC (~2004 mg)	1 sample of perlite via HFC (~550 mg)	1 sample of raw PCM via DSC (~12.21 mg)
Thermal conductivity (ASTM C1363-14)	1 sample (4 × 16 × 4 cm)	1 sample (4 × 16 × 4 cm)	1 sample (4 × 16 × 4 cm)
TGA/DTG/DTA	1 sample (~108 mg)	–	–
SEM-EDS	–	1 sample of Perlite aggregate	1 sample of PCM-perlite aggregate
XRD + Rietveld analysis	1 powdered sample	–	–

Table 9

Flowability of mortars by ASTM C230/C230M-23.

Property / Test / Results	SLM (Reference Base Mortar)	SLM + Perlite	SLM + PCM-Perlite
Aggregate Placement Method	Fine sand during the mix	Preplaced aggregate	Preplaced aggregate
Casting Method	Poured directly	Mortar cast around packed aggregates	Mortar cast around packed PCM-aggregates
Average Flow Diameter (mm)	254	242	251
Calculated Flow (%)	154	142	151
Effect on Rheology / Flow	Natural flow observed	Slightly reduced flow due to internal friction	Improved flow due to reduced interparticle friction

All aggregates were preconditioned to a Saturated Surface-Dry (SSD) condition before casting. However, due to the high open porosity of the perlite aggregates, partial water absorption occurred during the test, which slightly affected the rheological behavior of the fresh mortar. Additionally, the irregular and porous surface texture of the perlite may have led to increased internal friction, negatively affecting the composite flow. In contrast, the PCM-enhanced aggregates, coated with a thin layer of cement powder, exhibited lower water absorption and smoother surface characteristics, which reduced interparticle friction and improved flowability compared to the conventional perlite mixes.

3.2. Water absorption

Water absorption tests are summarized in Fig. 4. At the 30-minute mark, the results show that both SLM Perlite and SLM PCM-Enhanced Perlite had higher initial water absorption compared to the base SLM. This indicates the impact of the intrinsic porosity of the perlite-based aggregates. Similar initial surface properties and capillary behavior can also be observed between the two perlite types of mixtures.

Table 7

Hot Box setup: list of components, sensors and measured quantities (see Fig. 3).

Element / Sensor	Location in Hot Box set-up	Quantity	Measured variable / Role
EPS thermal chambers	Top (cooling part) + bottom (heating part)	2	Insulated environment boxes for controlled boundary temperatures
Cooling Unit	Upper of the chamber	1 system	Maintains cold-side (boundary condition)
Heating unit (i.e., electrical heater)	Lower chamber	1 system	Maintains hot-side (boundary condition)
Axial fans (PWM)	One in each chamber	2	Ensures uniform airflow (i.e., convection boundary conditions)
Mortar specimen (4 × 4 × 16 cm)	Interface between chambers	1	Tested panel for heat transfer measurements
Heat flux sensor (hot side)	Specimen surface (heating side)	1	Measured heat flux at hot surface
Heat flux sensor (cold side)	Specimen surface (cooling side)	1	Measured heat flux at cold surface
Thermocouple – T ₁	Specimen surface (at hot side)	1	Hot-side surface temperature
Thermocouple – T ₂	Specimen surface (at cold side)	1	Cold-side surface temperature
Thermocouple – T _{mid}	Center of specimen	1	Internal temperature (mid-thickness)
Air thermocouples	Near fans / inside chambers	5	Air temperature monitoring and stability check of the test set-up
Data logger (Hioki LR8432-20)	External	1	Acquisition of heat flux + temperature channels

Over time (i.e., 60, 120, and 180 min), the absorption trend diverges: the SLM PCM-Enhanced Perlite mortar exhibits a remarkably stable absorption curve, maintaining a low and nearly constant level. This behavior contrasts sharply with the SLM Perlite, which shows a progressive and consistent increase of water uptake. The base SLM mortar also shows a gradual rise but consistently remains lower than the unmodified perlite mix.

At the 3-day mark, a trend shift occurs: SLM PCM-Enhanced Perlite displays a sharp increase in water absorption, surpassing both other formulations and becoming the highest value recorded. This significant late-stage uptake indicates a failure in the long-term effectiveness of the cementitious encapsulation. While the shell appears to temporarily block early-stage water ingress, its protective performance diminishes over time. The delayed water uptake likely results from aging and microstructural changes, such as the formation of interconnected capillaries and microcracks that eventually allow moisture to reach the highly porous PCM core.

The SLM Perlite also shows a pronounced increase in absorption after 3 days, though still below the PCM-enhanced version. In contrast, the base SLM maintains the lowest water absorption throughout the entire period.

This trend, especially the “L-shaped” time behavior as observed in Fig. 4 of the PCM-enhanced perlite, highlights an important material performance issue: despite initial success in reducing absorption, the long-term barrier effect of the cementitious shell is compromised. This may be linked to the lack of pozzolanic additives, the presence of unhydrated phases observed in EDS analysis, and the absence of superplasticizers that could improve matrix densification. Therefore, further optimization of the shell formulation is needed to improve long-term durability against water uptake.

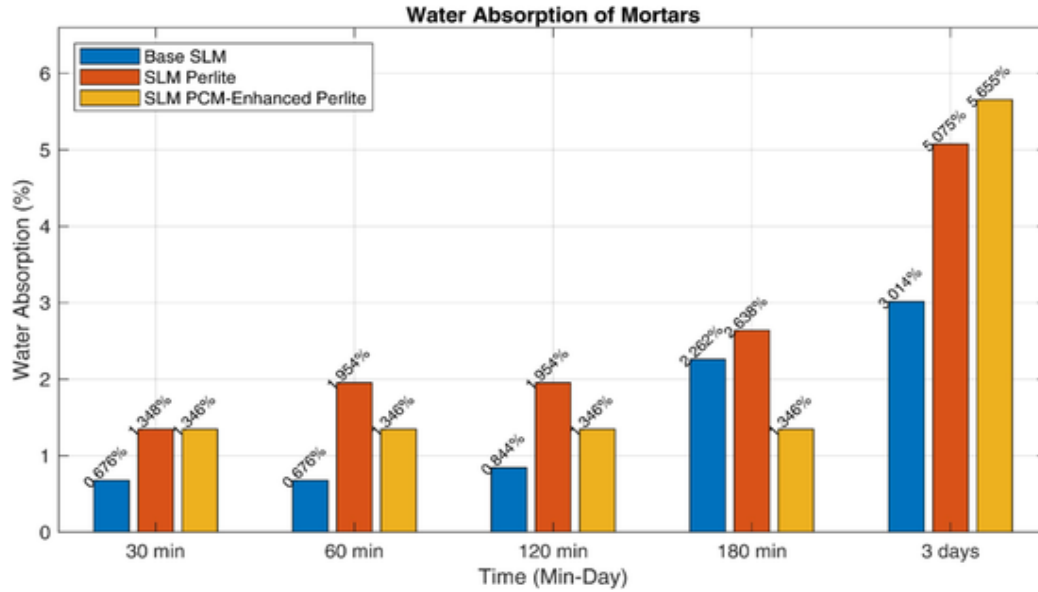


Fig. 4. Water absorption of mortars containing different types of lightweight aggregates (Base SLM, SLM Perlite, and SLM PCM-Enhanced Perlite) measured at different time intervals (30, 60, 120, and 180 min, and 3 days). Results highlight the influence of perlite and PCM encapsulation on water uptake behavior over time.

3.3. Mechanical properties

The reference mortar (SLM), with an average density of 1970.28 kg/m³, exhibited the best mechanical performance among all formulations (see Table 10) characterized by a mean flexural strength of 5.79 MPa, and compressive strength of 60.14 MPa. These values reflect the dense and cohesive matrix, typical of mid-to-high strength materials without lightweight aggregates. When 50% of the mortar volume was replaced with lightweight perlite aggregates, a sharp density reduction to 1464.84 kg/m³ was observed. This incorporation also negatively impacted the mechanical performance, reducing both flexural and compressive strengths to 3.08 MPa and 29.02 MPa, respectively. This can be attributed to the porous nature of perlite, which compromises the continuity and integrity of the matrix.

Interestingly, replacing standard perlite with PCM-enhanced perlite aggregates, pre-encapsulated in a cement-based shell, slightly raise the overall density to 1481.10 kg/m³. This modest increase is likely due to the denser shell structure limiting mortar penetration into the perlite pores. From a mechanical perspective, flexural strength increased to 3.46 MPa, suggesting a reinforcing effect of the encapsulation on bending resistance. However, compressive strength further declined to 17.62 MPa, likely due to the mechanical fragility of the PCM inclusions and potential weak interfacial bonding within the matrix. The mechanical performances of the investigated mortars are illustrated in Fig. 5, highlighting the comparison among flexural and compressive strengths of SLM, Perlite SLM, and PCM-Perlite SLM samples. These findings sug-

gest that the preplaced aggregate method combined with PCM-functionalized lightweight aggregates is promising for the development of lightweight mortars with energy storage functionality, though further optimization is needed to balance mechanical and TES performance.

3.4. Specific heat capacity C_p

The DSC results (Fig. 6) for PureTemp 25 demonstrated consistent and reliable thermal behavior across both slow and fast scan (heating/cooling) rates. At 0.25 K/min (the lowest rate), the heating curve displayed two overlapping endothermic peaks, with the dominant transition centered around 25.5 °C and a total melting enthalpy of 194.9 J/g. This double-peak pattern reflects the complex melting dynamics of bio-based PCMs when thermal equilibrium is maintained. The corresponding cooling cycle reveals a broader exothermic response, peaking at 22.0 °C, with a total enthalpy of -194.9 J/g, confirming the material stability and compliance with IEA Task 42 / DSC 4229 recommendations.

The results of Calorimetry of the non-latent components, i.e., SLM mortars and expanded perlite are presented in Fig. 7 and Table 11. The specific heat capacities (C_p) were evaluated across four temperature intervals of 28–30 °C, 30–32 °C, 32–34 °C, and 34–36 °C, using DSC-derived enthalpy data. Table 12. Table 13. Table 14.

For the SLM sample (mass = 2.004 g), the DSC analyses showed that the enthalpy changes, for each interval, increased constantly with temperature. The resulting C_p values ranged from 1.176 to 1.215 J/g × K, with a total average of 1.198 J/g × K for the examined temperature range. The progressive rise of SLM C_p implied a greater thermal reactivity of these mortars at higher temperatures, likely due to the presence of thermally active elements within the matrix. The steady evolution of C_p suggests stable sensible energy absorption properties.

Similarly, for the expanded perlite aggregates (mass = 0.550 g), the enthalpy changes (ΔH) showed consistent trends across the same temperature intervals, varying between 0.873 to 0.948 J. The corresponding C_p values ranged from 0.794 to 0.862 J/g × K. Its total average over the tested ranges was calculated to be 0.8262 J/g × K. Minor variations were observed across the intervals: this trend suggests a subtle decrease in C_p as temperature increases, indicating a relatively sta-

Table 10

Estimated and actual (measured) density values of the three mortars. The variation reflects the influence of matrix infiltration and aggregate porosity on final composite density.

Mortars type	SLM (% in volume)	Perlite (% in volume)	PCM Perlite (% in volume)	Designed density ρ_d (kg/m ³)	Actual density ρ_a (kg/m ³)	Variation ρ_d ρ_a densities (%)
SLM	50	50	0	1219.22	1464.84	20.15
Perlite						
SLM PCM	50	0	50	1415.73	1481.10	4.62
Perlite						

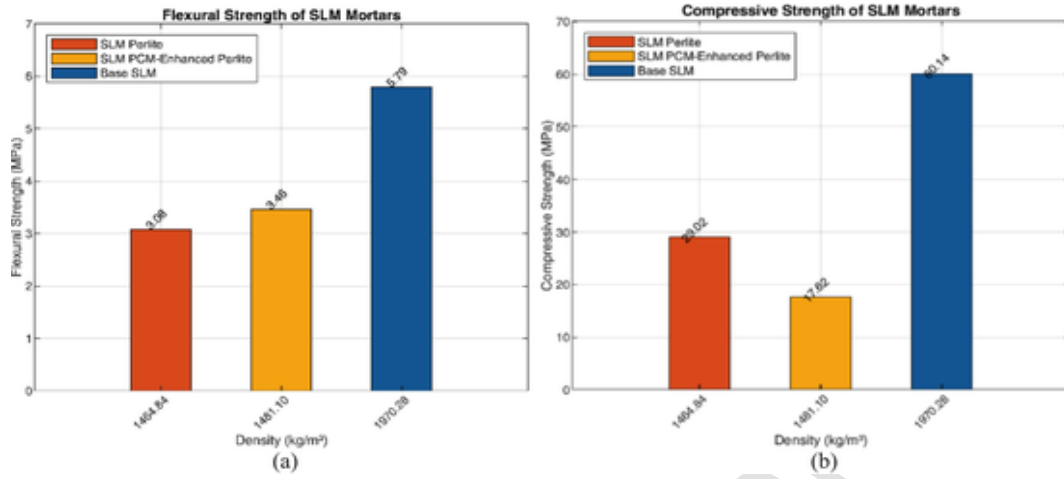


Fig. 5. Mechanical performance of SLM mortars: (a) flexural strength and (b) compressive strength of SLM, Perlite SLM, and PCM-Perlite SLM.

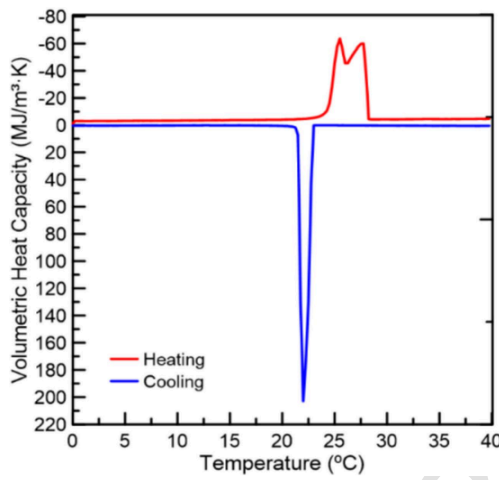


Fig. 6. DSC curve of PureTemp 25 at 0.25 K/min rates.

ble thermal response which reflects the insulating and inert thermal nature of expanded perlites.

3.5. The “Dynamische Kugel Kalorimetrie,, (DKK) tests

Fig. 8 illustrates the temperature evolution during cooling (left figures) and heating (right figures) phases for three types of mortar spheres: i.e., SLM (reference case), SLM perlite, and SLM PCM-enhanced perlite mortars. Results include two replications and the mean trend, where the scattering area indicates data dispersion.

All samples exhibit a typical negative exponential decay of temperature vs. time during cooling, as well as a similar continuous rise during heating. However, significant differences in thermal behavior are evident depending on the type of components and/or aggregate employed. During the cooling phase, the reference mortar (REF) presents a smooth and uninterrupted decline in temperature, indicative of a homogeneous thermal diffusion profile and absence of latent storage effects. Similarly, the SLM perlite mortars follow this trend but show a slightly slower cooling rate in the initial stage, consistent with the low thermal conductivity of expanded perlite, which acts as a thermal insulator which improves the overall composite insulation capacity.

In contrast, the SLM PCM-enhanced perlite mortars deviate markedly from the expected exponential decay, displaying a distinct

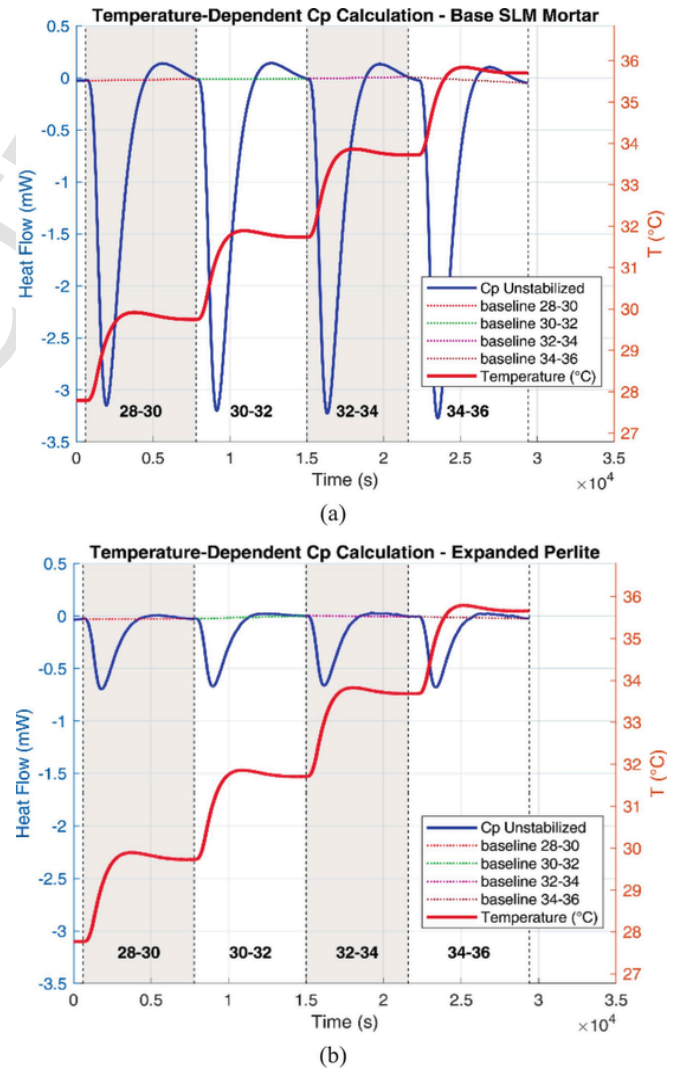


Fig. 7. Temperature-dependent specific heat capacity (C_p) for (a) SLM mortar and (b) expanded perlite using DSC data.

Table 11

Enthalpy variation (ΔH) and Specific Heat Capacity (C_p) of non-latent SLM mortar and expanded perlites across temperature ranges of 28–36 °C.

T-ranges (°C)	SLM Mortar ΔH (J)	SLM Mortar C_p (J/g × K)	Expanded Perlite ΔH (J)	Expanded Perlite C_p (J/g × K)
28–30	4.7127	1.1758	0.9483	0.8621
30–32	4.7765	1.1917	0.9003	0.8185
32–34	4.8517	1.2105	0.9136	0.8305
34–36	4.8713	1.2154	0.8731	0.7937
Total Average	–	1.1984	–	0.8262

thermal plateau between 25 and 30 °C. This plateau corresponds to the latent heat absorption during the phase change of the PCM embedded in the perlite structure, confirming its capacity to store thermal energy during cooling. Moreover, such behavior highlights the PCM role as a thermal buffer, providing potential for storing energy.

During heating cycles, a quite mirrored behavior can be observed. Both SLM and SLM perlite mortars show smooth and gradual temperature increases, reaffirming the limited thermal inertia of perlite alone. The PCM-enhanced mortar distinctly exhibits again a thermal plateau associated with the PCM melting transitions. It is the result from latent heat release, effectively retarding the temperature rise and serving as a passive thermal regulation mechanism. Additionally, the thermal hysteresis is classical observed between heating and cooling cycles further emphasizing the PCM functional contribution to energy storage and release within a narrow temperature range.

Fig. 9 reports the same curves as Fig. 8 by overlapping them. They make even clearer how perlite improves the insulating behavior of the mortars and how the incorporation of PCM-aggregates substantially enhances the system thermal regulation capacity by enabling thermal buffering and delayed heat transfer across heating and cooling cycles.

3.6. Custom-designed hot Box test

Fig. 10 presents the ASTM C1363 results for the reference SLM mortar panel ($4 \times 4 \times 16 \text{ cm}^3$) during the transient cooling/heating stages, along with the subsequent steady-state phase. The left-hand graphs illustrate the temperature profiles and heat flux curves, while the right-hand graphs report the calculated thermal parameters, including thermal conductivity (λ), overall thermal resistance (specimen + surface films) of the test assembly (R_{u}), and thermal transmit-

tance (U-value) over time. Each phase of the test was divided into segments with a duration of 500 s, and all parameters were calculated for each segment. Finally, the average total value of each parameter was calculated for the entire phase of each test.

In the cooling phase (Fig. 10a–b), the internal surface temperature (T_1 -Hot) decreases progressively from about 25.04 °C to 22.70 °C, while in the same period, the external surface temperature (T_2 -Cold) drops from 16.97 °C to 14.80 °C, before stabilizing. This creates a nearly constant surface temperature gradient of ~ 8.06 °C, which drives the heat flow. The mid-sample temperature decreases from about 23.99 °C to 20.52 °C, with the rate of change reducing from -0.197 °C/min to -0.075 °C/min, confirming the transition toward thermal equilibrium. The most pronounced variations occur in the earlier part of the phase, while later segments show smaller changes, marking the approach to steady-state conditions. The heat flux correspondingly rises from 36.8 W/m^2 to 92.1 W/m^2 . Over the same period, the U-value increases from 2.508 to $6.717 \text{ W/m}^2 \times \text{K}$, while the apparent thermal conductivity (λ) rises from 0.183 to $0.467 \text{ W/m} \times \text{K}$. Conversely, the overall thermal resistance (R_{u}) falls from 0.399 to $0.149 \text{ m}^2 \times \text{K/W}$, indicating the gradual stabilization of the specimen under cooling conditions.

During the heating steps (Fig. 10c–d), the hot-side temperature (T_1 -Hot) increases sharply from about 24.20 °C to 42.66 °C, while in the same period, the cold-side temperature (T_2 -Cold) rises more gradually from 14.47 °C to 19.85 °C, reflecting the mortar specimen thermal inertia. This produces an expanding surface temperature gradient, from ~ 9.73 °C to ~ 22.81 °C. The mid-sample temperature increases from 20.45 °C to 33.89 °C, with the rate of change starting at 0.369 °C/min and decreasing to nearly 0.004 °C/min at the end, confirming stabilization (steady state regime). The most pronounced variations occur in the early part of the phase, while later segments show smaller changes, marking the transition toward steady-state conditions. The heat flux initially rises rapidly from 173.1 W/m^2 to a peak of 491.5 W/m^2 , before gradually declining and stabilizing near 373.8 W/m^2 . Over the same period, the U-value reaches a maximum of $13.712 \text{ W/m}^2 \times \text{K}$ before settling to about $9.745 \text{ W/m}^2 \times \text{K}$, while the apparent thermal conductivity (λ) follows a similar pattern, increasing from $0.711 \text{ W/m} \times \text{K}$ to $0.938 \text{ W/m} \times \text{K}$ and then declining to $0.656 \text{ W/m} \times \text{K}$. Conversely, the overall thermal resistance (R_{u}) decreases from $0.099 \text{ m}^2 \times \text{K/W}$ to $0.066 \text{ m}^2 \times \text{K/W}$, indicating reduced resistance as the specimen approaches thermal equilibrium.

Once the system reaches a steady-state (Fig. 10e–f), both the surface temperatures and the heat flux remain essentially stable. The hot-side

Table 12

SLM Results for Cooling, Heating, and Steady-State Phases (ASTM C1363).

Phase	λ (W/m × K)	R ($\text{m}^2 \times \text{K/W}$)	$R_{\text{env_h}}$ ($\text{m}^2 \times \text{K/W}$)	$R_{\text{env_c}}$ ($\text{m}^2 \times \text{K/W}$)	R_{u} ($\text{m}^2 \times \text{K/W}$)	C (W/m ² × K)	U-value (W/m ² × K)
Cooling	0.340	0.133	0.019	0.086	0.238	8.502	4.829
Heating	0.752	0.054	0.020	0.017	0.091	18.795	11.154
Steady	0.644	0.062	0.019	0.023	0.104	16.092	9.575

Table 13

SLM Perlite Mortar Results for Cooling, Heating, and Steady-State Phases (ASTM C1363).

Phase	λ (W/m × K)	R ($\text{m}^2 \times \text{K/W}$)	$R_{\text{env_h}}$ ($\text{m}^2 \times \text{K/W}$)	$R_{\text{env_c}}$ ($\text{m}^2 \times \text{K/W}$)	R_{u} ($\text{m}^2 \times \text{K/W}$)	C (W/m ² × K)	U-value (W/m ² × K)
Cooling	0.357	0.113	0.036	0.062	0.211	8.936	4.765
Heating	0.481	0.088	0.024	0.022	0.133	12.029	7.735
Steady	0.307	0.130	0.023	0.042	0.196	7.687	5.105

Table 14

SLM PCM- Perlite Mortar Results for Cooling, Heating, and Steady-State Phases (ASTM C1363).

Phase	λ (W/m × K)	R ($\text{m}^2 \times \text{K/W}$)	$R_{\text{env_h}}$ ($\text{m}^2 \times \text{K/W}$)	$R_{\text{env_c}}$ ($\text{m}^2 \times \text{K/W}$)	R_{u} ($\text{m}^2 \times \text{K/W}$)	C (W/m ² × K)	U-value (W/m ² × K)
Cooling	0.296	0.135	0.066	0.074	0.274	7.44	3.63
Heating	0.337	0.120	0.061	0.050	0.233	8.436	4.403
Steady	0.280	0.143	0.057	0.064	0.263	7.011	3.804

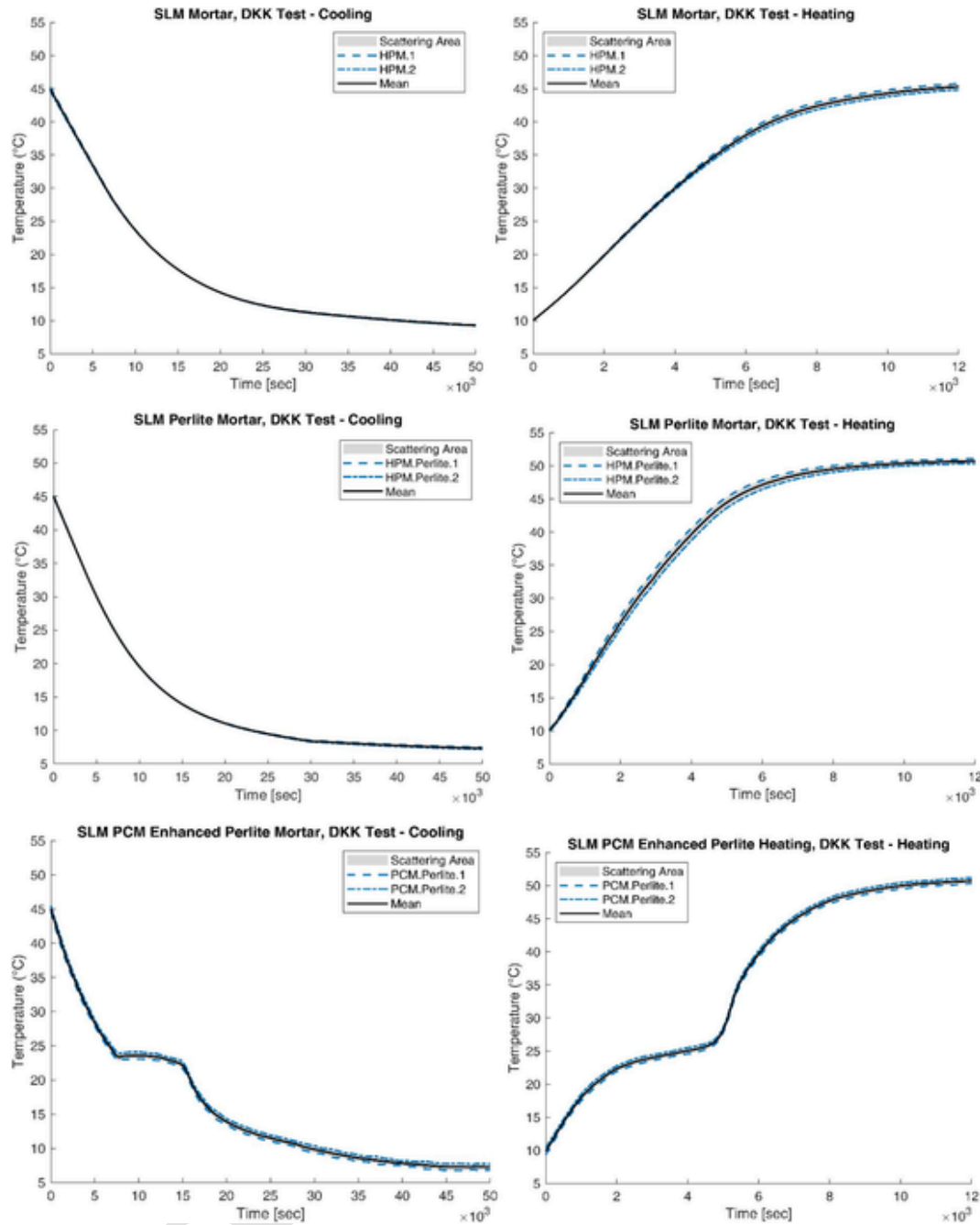


Fig. 8. Temperature-time curves for SLM, SLM Perlite, and SLM PCM-Enhanced Perlite mortars during cooling (left column) and heating (right column) cycles. Mean curve and Scattering behavior from the DKK thermal test.

temperature (T_1 -Hot) is nearly constant around 43.49 °C, while the cold-side temperature (T_2 -Cold) stabilizes near 22.00 °C, producing a constant surface temperature gradient of ~ 21.52 °C. The mid-sample temperature remains steady at ~ 35.21 °C, with the rate of change approaching zero, confirming full thermal equilibrium. The heat flux stabilizes in the range of 341.0–354.0 W/m². Over the same period, the U-value remains almost constant between 9.459–9.791 W/m² × K, while the apparent thermal conductivity (λ) stays stable between 0.635–0.658 W/m × K. The overall thermal resistance (R_u) stabilizes around 0.102–0.106 m² × K/W, reflecting steady thermal performance. Table 12 summarizes the thermal parameters obtained for the SLM mortar during the cooling, heating, and steady-state phases.

Fig. 11 shows the ASTM C1363 test results for the SLM mortar incorporating expanded perlite, considering cooling, heating, and steady-state phases. The left-hand plots display the temperature and heat flux trends, while the right-hand plots report the corresponding thermal parameters.

At the end of the cooling phase (Fig. 11a–b), the hot-side temperature (T_1) fluctuates slightly between 22.02 °C and 22.17 °C, while the cold-side temperature (T_2) ranges from 13.39 °C to 15.07 °C, producing a surface temperature gradient of ~ 7.10 –8.63 °C. The mid-sample temperature increases slightly from 17.66 °C to 17.94 °C, with the rate of change shifting from -0.039 °C/min to $+0.021$ °C/min, indicating a gradual stabilization. The heat flux remains moderate, varying between 62.6 and 69.5 W/m². Over the same period, the U-value rises from

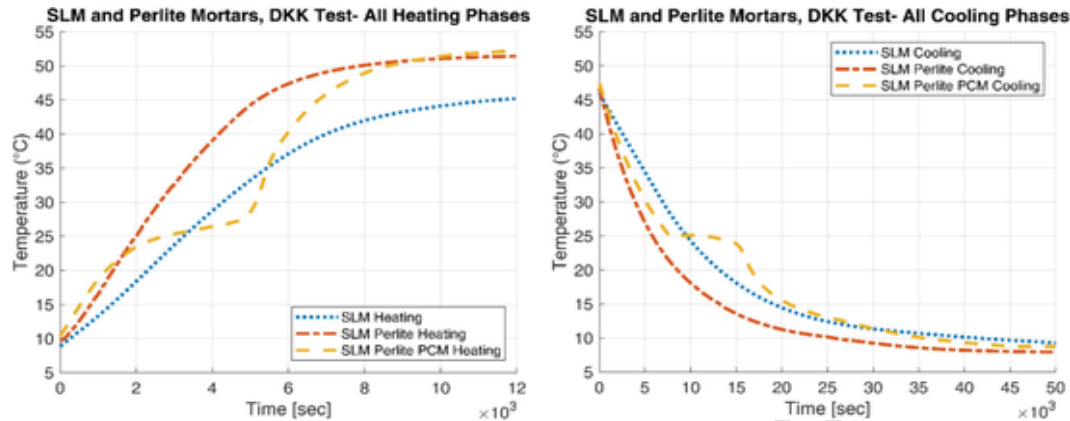


Fig. 9. Typical curves of heating and cooling phases of DKK test from the studied SLM mortars. Plateaus observed in PCM-enhanced composites indicate the latent heat absorption and release during phase transitions.

4.145 to $4.937 \text{ W/m}^2 \times \text{K}$, while the apparent thermal conductivity (λ) increases from 0.290 to $0.385 \text{ W/m} \times \text{K}$. The overall thermal resistance (R_u) remains relatively stable within 0.202 – $0.241 \text{ m}^2 \times \text{K/W}$, confirming consistent insulating performance. Compared to the reference SLM, the perlite mortar continues to exhibit slower heat transfer, in line with the low conductivity of lightweight aggregates.

In the heating phase (Fig. 11c–d), the hot-side temperature (T_1) increases sharply from about 25.82°C to 45.44°C , while the cold-side temperature (T_2) responds more slowly, rising from 15.09°C to 19.29°C , reflecting the thermal inertia introduced by perlite. This produces a widening surface temperature gradient, from $\sim 10.73^\circ\text{C}$ to $\sim 26.15^\circ\text{C}$. The mid-sample temperature increases from 18.04°C to 27.18°C , with the rate of change starting at 0.024°C/min and reaching a peak of 0.238°C/min before declining to 0.074°C/min , confirming the transition toward stabilization. The heat flux initially rises to a peak of 332.6 W/m^2 and then gradually decreases toward 231.4 W/m^2 at later stages. Over the same period, the U-value increases to a maximum of $9.756 \text{ W/m}^2 \times \text{K}$ before reducing to $5.953 \text{ W/m}^2 \times \text{K}$. The apparent thermal conductivity (λ) follows a similar pattern, decreasing from $0.649 \text{ W/m} \times \text{K}$ to $0.354 \text{ W/m} \times \text{K}$. Conversely, the overall thermal resistance (R_u) increases from 0.114 to $0.168 \text{ m}^2 \times \text{K/W}$, highlighting reduced heat transfer as the system approaches equilibrium.

During the steady-state phase (Fig. 11e–f), both surface temperatures remain stable, with the hot-side (T_1) at $\sim 46.20^\circ\text{C}$ and the cold-side (T_2) near 20.40°C , producing a constant surface gradient of $\sim 25.80^\circ\text{C}$. The mid-sample temperature stays steady at $\sim 29.00^\circ\text{C}$, with the rate of change approaching zero, confirming full thermal equilibrium. The heat flux stabilizes around 193.7 – 204.0 W/m^2 , significantly lower than the plain SLM. Over the same period, the U-value remains constant at 5.041 – $5.238 \text{ W/m}^2 \times \text{K}$, while the apparent thermal conductivity (λ) stabilizes between 0.301 – $0.316 \text{ W/m} \times \text{K}$. The overall resistance (R_u) averages $0.196 \text{ m}^2 \times \text{K/W}$, confirming the improved insulating performance due to the perlite addition.

Overall, when compared with the reference SLM, the addition of expanded perlite enhances the thermal resistance and reduces the transmittance of the mortar. The results demonstrate lower heat fluxes in steady-state and moderated transients during heating, confirming perlite beneficial role as a lightweight insulating aggregate. Table 13 summarizes the corresponding thermal parameters for the SLM Perlite mortars.

Following the same structure as before, Fig. 12 reports the thermal response of the SLM mortar containing expanded perlite and bio-PCM. Along with the cooling phase (Fig. 12a–b), the hot-side surface temperature (T_1) decreases slightly from 20.98°C to 20.24°C , while the cold-side temperature (T_2) drops from 14.56°C to 13.98°C , producing a con-

sistent surface gradient between 6.42°C and 6.27°C . The mid-sample temperature declines gradually from 16.63°C to 15.87°C , with the rate of change approaching zero, confirming the attainment of equilibrium. The heat flux remains stable, ranging from 47.0 to 46.1 W/m^2 across the phase. Over the same period, the U-value increases slightly from 3.554 to $3.712 \text{ W/m}^2 \times \text{K}$, while the apparent thermal conductivity (λ) varies marginally between 0.293 and $0.301 \text{ W/m} \times \text{K}$. The overall thermal resistance (R_u) remains nearly constant within 0.269 – $0.281 \text{ m}^2 \times \text{K/W}$. These results indicate that the PCM-perlite composite exhibits a strong buffering capacity during cooling, maintaining stable heat transfer while the PCM remains in the solid phase.

While the heating process is going on (Fig. 12c–d), the hot-side temperature (T_1) increases sharply from about 20.25°C to 47.05°C , while the cold-side temperature (T_2) responds more slowly, rising from 14.04°C to 27.77°C , reflecting the additional thermal inertia provided by the PCM. This produces a widening surface temperature gradient, expanding from approximately 6.20°C to 19.28°C . The mid-sample temperature increases progressively from 15.87°C to 33.41°C , with the rate of change starting very low (near zero), accelerating during the latent heat absorption to about 0.238°C/min , and then tapering back toward 0.009°C/min as the phase change completes, confirming the transition toward thermal stabilization. The heat flux rises rapidly through the phase-change window, from about 46.7 W/m^2 at the onset to a peak of nearly 225.6 W/m^2 , before gradually declining toward 140.1 W/m^2 in the later part of heating. Over the same period, the U-value shows a transient amplification: starting near $3.715 \text{ W/m}^2 \times \text{K}$, increasing to a maximum of about $10.734 \text{ W/m}^2 \times \text{K}$ during peak melting, and then reducing again to $3.923 \text{ W/m}^2 \times \text{K}$ as the system approaches equilibrium. The apparent thermal conductivity (λ) follows a similar evolution, beginning at around $0.301 \text{ W/m} \times \text{K}$, surging to $0.509 \text{ W/m} \times \text{K}$ during the latent transition, and settling to $0.291 \text{ W/m} \times \text{K}$ at the end of the heating block. Conversely, the overall thermal resistance (R_u) decreases sharply at the onset of melting, dropping to about $0.078 \text{ m}^2 \times \text{K/W}$, before recovering to $0.138 \text{ m}^2 \times \text{K/W}$ in the steady portion, highlighting the combined effect of sensible and latent storage in moderating heat transfer.

During the steady phase (Fig. 12e–f), the thermal response stabilizes, with nearly flat signals across all monitored channels. The hot-side surface temperature (T_1) remains steady at $\sim 47.30^\circ\text{C}$, while the cold-side (T_2) stabilized at around 27.96°C , sustaining a stable surface temperature difference of 19.24 – 19.27°C . The mid-sample temperature hovers near 33.69 – 34.04°C , with a rate of change of -0.001 to 0.005°C/min , confirming that quasi-equilibrium has been reached. The mean heat flux settles between 133.3 – 136.8 W/m^2 , yielding a consistent U-value of 3.786 – $3.836 \text{ W/m}^2 \times \text{K}$ and an overall resistance R_u of

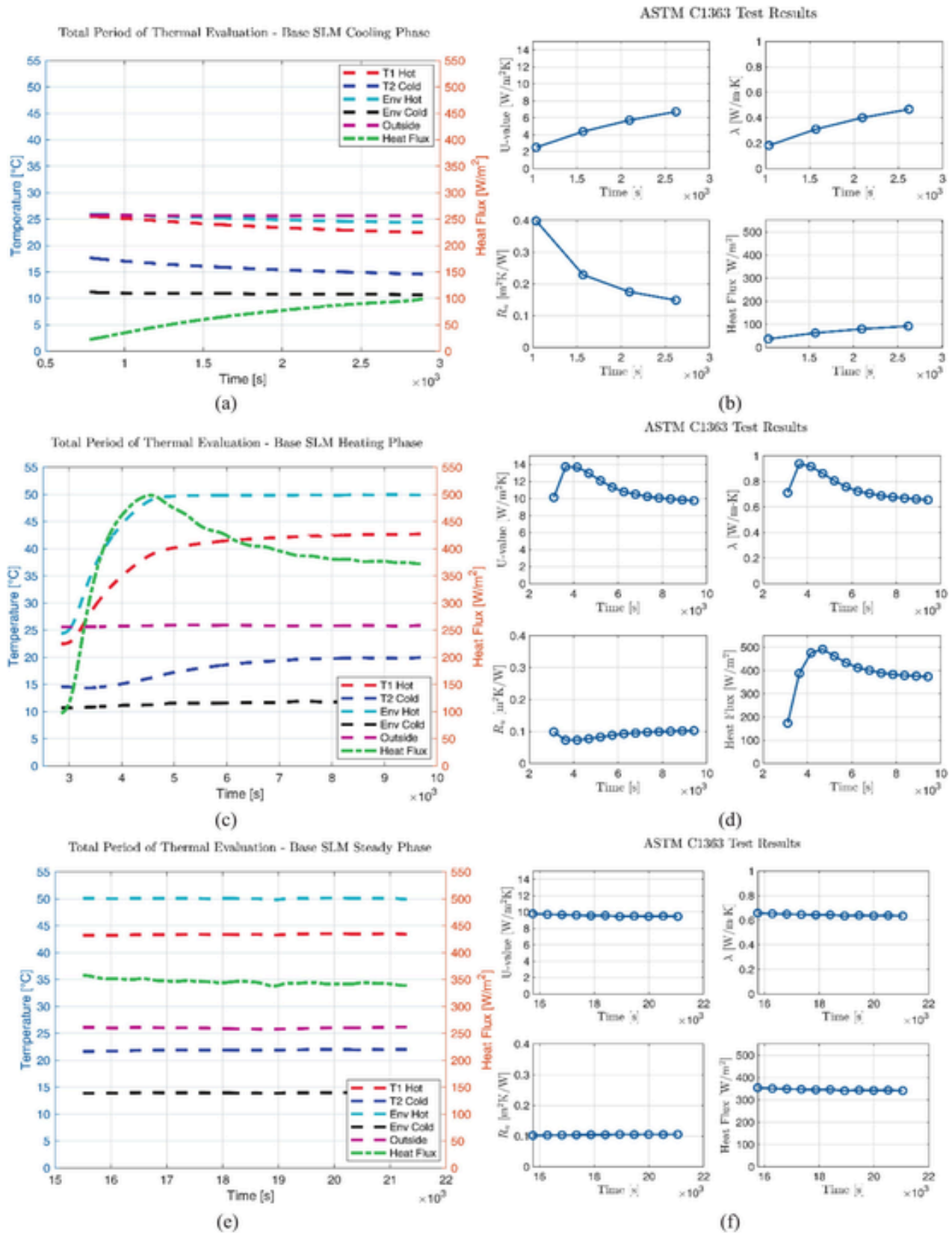


Fig. 10. Heat transfer characteristics of the SLM studied mortar in different thermal phases.

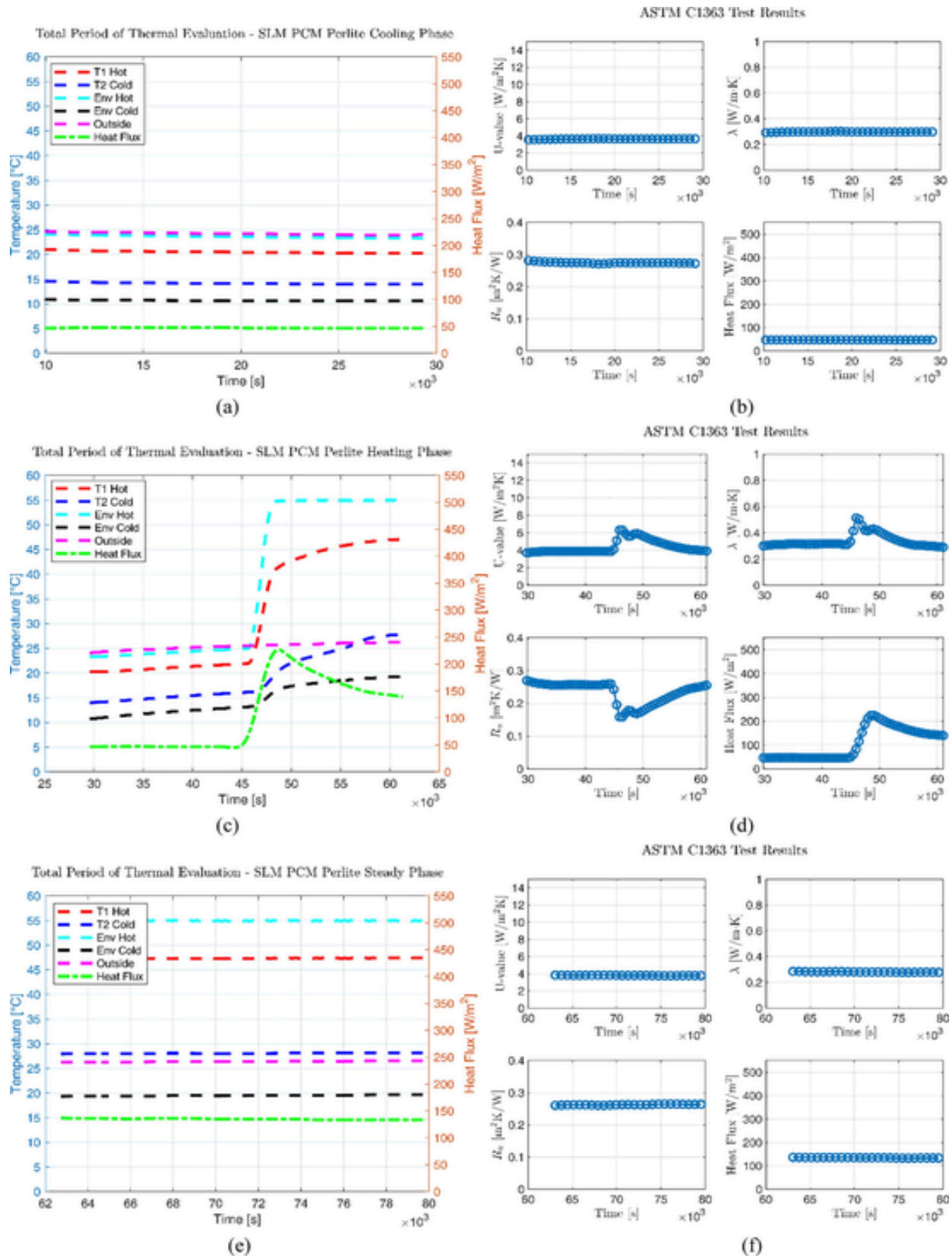


Fig. 11. Heat transfer characteristics of SLM Perlite Mortar in different thermal phases.

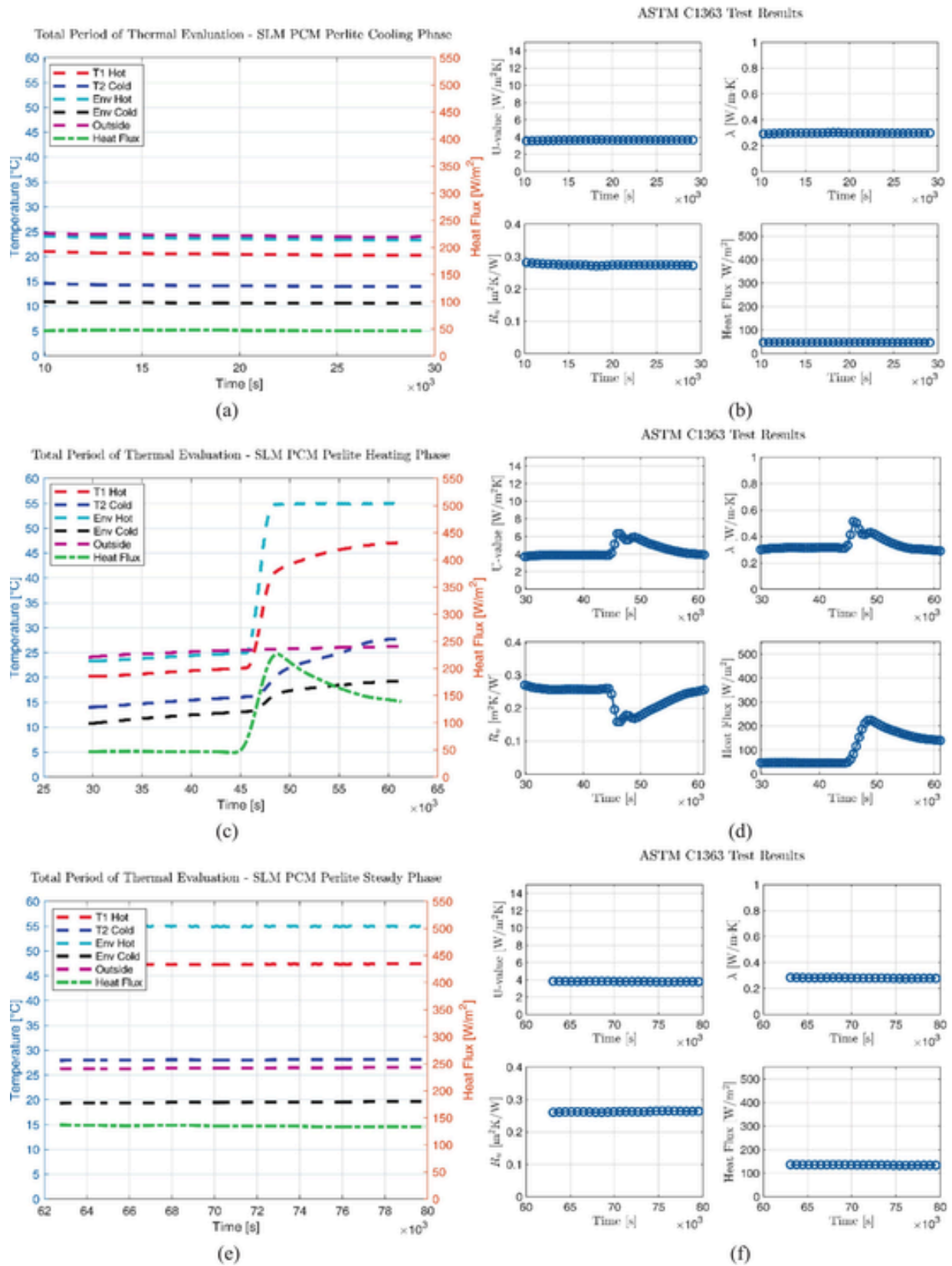


Fig. 12. Heat transfer characteristics of SLM PCM-Enhanced Perlite Mortar in different thermal phases.

0.261–0.265 $\text{m}^2 \times \text{K/W}$. The apparent thermal conductivity converges to $\lambda \approx 0.280 \text{ W/m} \times \text{K}$. Table 14 reports the thermal parameters obtained for the SLM PCM-Perlite mortar during cooling, heating, and steady-state conditions.

Table 15 presents an exhaustive analysis of the phase change processes. It can be observed that during the heating process, when the cooling chamber is working in parallel, the phase change in the composite is clearly detectable. PCM melting occurs when the material absorbs latent heat, leading to a stabilization in the mid-sample temperature (T_{mid}) rate, with a plateau-like behavior. In this stage, the additional input energy is primarily consumed by the phase transition rather than by raising the temperature.

Detailed interpretations of the phase transition processes for the SLM PCM-Enhanced Perlite Mortars can be stated as follows:

- **Segments 25–27:** the average T_{mid} rises only slightly, from 17.65 °C to 17.78 °C, while the T_{mid} Rate remains very low (~ 0.01 °C/min). The heat flux increases modestly from 45.8 W/m^2 to 73.3 W/m^2 , and the ΔT surface remains nearly constant at 5.83–6.05 °C. This indicates the onset of PCM activation, where the material starts absorbing heat but with minimal temperature rise.
- **Segments 28–29:** a pronounced jump occurs. The average T_{mid} increases from 17.96 °C to 18.63 °C, while the T_{mid} Rate rises to 0.03–0.10 °C/min. The heat flux increases significantly to 109.9–154.1 W/m^2 , and ΔT surface expands to 8.63–13.59 °C. These features confirm that the PCM is in the active melting zone, with latent heat absorption enabling substantial heat transfer without proportional temperature growth.
- **Segments 30–32:** this interval represents the peak of latent heat absorption. The heat flux reaches its maximum of 225.6 W/m^2 (segment 32), while the T_{mid} Rate peaks at ~ 0.17 °C/min (segment 31) before declining to 0.13 °C/min. ΔT surface also reaches its maximum of ~ 21.02 °C. Over this range, the average T_{mid} increases from 20.00 °C to 23.25 °C, reflecting the fully developed phase change process, where the PCM absorbs large amounts of heat while slowing the temperature rise.
- **Segments 33–41:** after the PCM is completely melted, sensible heating dominates. The heat flux gradually decreases from 220.2 W/m^2 to 169.5 W/m^2 , while the T_{mid} Rate stabilizes within 0.05–0.07 °C/min. ΔT surface levels of around 20.71–20.93 °C. Meanwhile, the average T_{mid} continues to increase from 24.35 °C to 29.46 °C, marking the end of the phase transition and the return to normal heat storage by sensible processes.

In summary, the most pronounced phase change effects are observed in segments 28–32, where both heat flux and T_{mid} Rate reach their maximum values, while the temperature rise shows a plateau-like moderation. This interval represents the dominant PCM melting period, during which latent heat storage strongly modifies the thermal response of the composite.

It can be concluded that the most evidenced phase change is occurring at segments 28–32, where the heat flux and T_{mid} Rate are the highest, while the temperature rise is reduced (i.e., plateau-like/mushy behavior in the T_{mid} Rate compared to subsequent segments).

Fig. 13 is displaying (a) the thermal conductivity (λ) vs. time and the same results (b) in terms thermal conductivity (λ) vs. Temperature (i.e., the Mean Mid T_{emp}) of the PCM-enhanced mortar sample. The graphs offer, even deeper, insights into the dynamic behavior of the SLM PCM-Enhanced Perlite Mortar by explaining the mechanisms driving the thermal performance during phase change and latent TES mechanisms. Particularly, it shows how prior to 18 °C, the thermal conductivity maintains a reasonably constant value, at about 0.297 $\text{W/m} \times \text{K}$. This initial phase corresponds to the pre-melting stage, where the PCM is fully in its solid state within the matrix, therefore exhibiting the thermal properties of the solid composite. As the temperature approaches the phase change zone (starting about 18 °C on the x-axis of Fig. 13b), the thermal conductivity shows a sharp increase, reaching a substantial peak (0.509 $\text{W/m} \times \text{K}$). This strong jump of λ is a direct consequence of PCM melting and energy absorption due to phase change mechanisms. PCM change from solid to liquid is accompanied by a consequent conductivity rise which allows the overall heat transfer mechanisms. Following this peak, the λ values progressively drop down at values of 0.280 $\text{W/m} \times \text{K}$, lower than the solid-state values. This post-peak behavior shows that once the majority of PCMs have melted, the material thermal conductivity settles into a new value representative of the composite with liquid PCM, where sensible heating once again dominates.

3.7. Thermal analysis (TGA/DTG/DTA) of SLM mortar

Fig. 14 shows thermogravimetric (TG) and derivative thermogravimetric (DTG) curves for the SLM sample. The TG curve (blue) shows the total mass loss of around 19% up to 900 °C, while the DTG curve (red) highlights critical breakdown peaks at around 96 °C and 791 °C.

More specifically, the TGA analysis of the reference mortar (SLM) highlights mainly two distinct mass-loss regions between 30 and 900 °C, as shown in Table 16. The first major mass loss (of about 47.91%) occurs in the 30–120 °C range, characterized by a prominent DTG peak at approximately 93–96 °C with a corresponding endothermic signal in the DTA curve around 100 °C. This thermal event is attrib-

Table 15
Changing the thermal properties of – SLM PCM-Enhanced Perlite Mortar during the phase change.

Segment	Time [s]	λ [$\text{W/m} \times \text{K}$]	Mean Heat Flux [W/m^2]	T_{mid} Rate [°C/min]	ΔT Surface [°C]	Start Mid T_{emp} [°C]	End Mid T_{emp} [°C]	Mean Mid T_{emp} [°C]
25	44,490	0.314	45.8	0.01	5.84	17.61	17.69	17.65
26	45,105	0.358	52.3	0.01	5.83	17.69	17.75	17.72
27	45,720	0.484	73.3	0.01	6.05	17.75	17.82	17.78
28	46,330	0.509	109.9	0.03	8.63	17.82	18.17	17.96
29	46,940	0.453	154.1	0.10	13.59	18.18	19.20	18.63
30	47,550	0.416	193.8	0.16	18.64	19.22	20.84	20.00
31	48,160	0.418	218.7	0.17	20.92	20.87	22.55	21.73
32	48,770	0.429	225.6	0.13	21.02	22.57	23.86	23.25
33	49,380	0.423	220.2	0.09	20.82	23.88	24.77	24.35
34	49,990	0.409	211.6	0.07	20.71	24.78	25.44	25.13
35	50,600	0.394	203.5	0.05	20.68	25.45	25.96	25.72
36	51,210	0.379	196.1	0.05	20.71	25.97	26.45	26.21
37	51,820	0.365	189.9	0.05	20.80	26.46	26.98	26.71
38	52,430	0.354	184.5	0.06	20.88	26.99	27.63	27.30
39	53,040	0.343	179.5	0.07	20.92	27.64	28.37	28.00
40	53,650	0.333	174.5	0.07	20.93	28.38	29.11	28.75
41	54,260	0.324	169.5	0.07	20.90	29.12	29.78	29.46

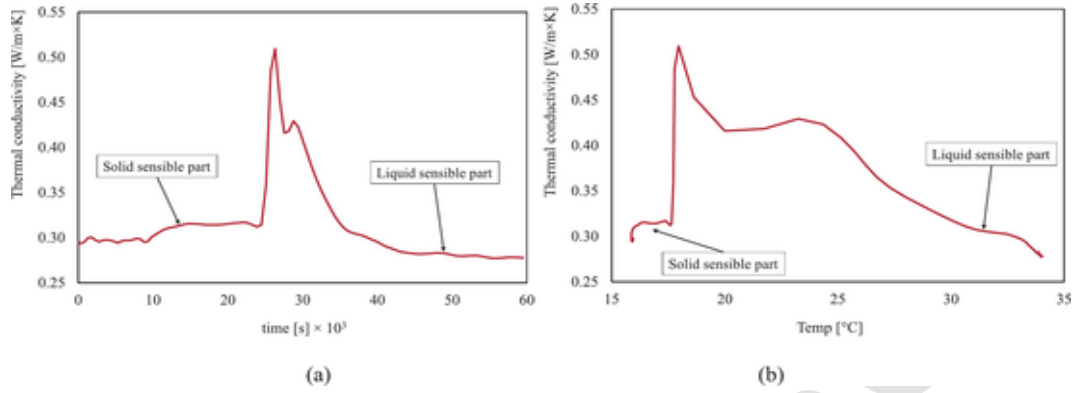


Fig. 13. Temperature-Dependent Thermal Conductivity and Surface Gradient of PCM-Enhanced Mortar.

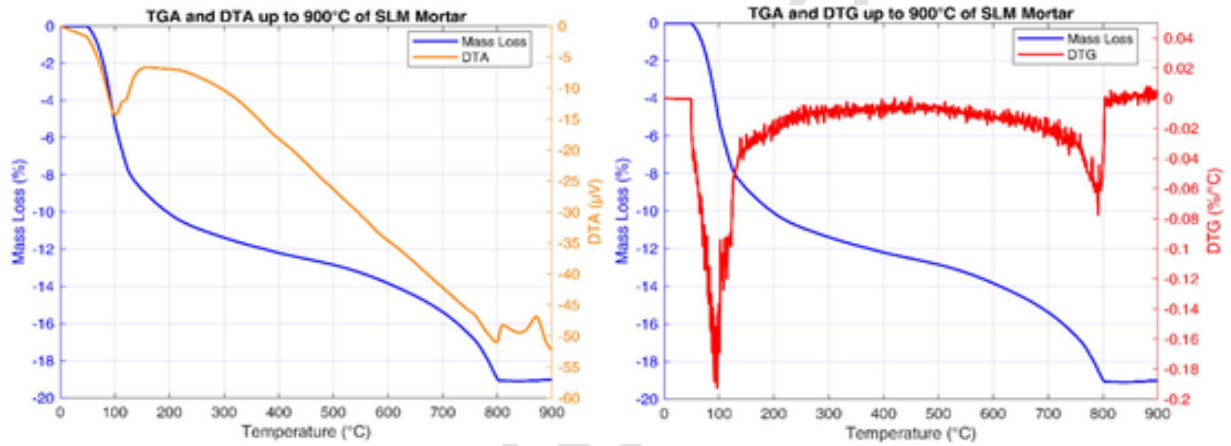


Fig. 14. Thermogravimetric (TGA – blue curve) and derivative thermogravimetric (DTG – red curve) analysis of SLM mortar up to 900 °C, showing mass-loss associated with dehydration and decarbonation of phases. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 16
Thermogravimetric mass loss events for SLM mortar (30–900 °C), with corresponding DTG peak positions and assigned phase transformations.

Temperature (°C)	Mass Loss (%)	DTG Peak (°C)	Assigned Phase/Transformation
30–120	~47.91	93 °C	Release of free water; dehydration of C–S–H gel and initial ettringite formation water
120–180	~14.60	~115 °C, 150 °C	Further loss of bound water from aluminate hydrates (ettringite continued decomposition, AFM dehydration)
400–500	~4.24	~450 °C	Dehydroxylation of portlandite ($\text{Ca(OH)}_2 \rightarrow \text{CaO} + \text{H}_2\text{O}$)
600–800	~33.25	~790 °C	Decarbonation of calcium carbonate ($\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$)

uted to the release of physically adsorbed water, due to dehydration of the calcium silicate hydrate (C–S–H) gel, and the initial water loss associated with ettringite formation. Between 120 and 180 °C, an additional 14.60% mass loss is observed, with DTG signals of around 115 °C and 150 °C. This stage typically corresponds to the release of bound water from hydrated aluminate phases, including the progressive decomposition of ettringite and dehydration of AFM (alumina-ferrite-monosulfate) phases.

A smaller mass loss (circa 4.24%) is detected in the 400–500 °C range, marked by a DTG peak at around 450 °C. This region is generally

linked to the dehydroxylation of portlandite ($\text{Ca(OH)}_2 \rightarrow \text{CaO} + \text{H}_2\text{O}$) and is indicative of low concentration or complete consumption of calcium hydroxide in the system due to the pozzolanic activity. Finally, in the 600–800 °C region, a broader DTG peak is observed at around 790–791 °C, corresponding to a 33.25% mass loss and a distinct DTA endotherm near 800 °C. This event is linked to the decarbonation of calcium carbonate ($\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$), reflecting the decomposition of carbonation products under the exposure of atmospheric CO_2 .

3.8. Scanning electron Microscopy (SEM)

Fig. 15a presents a SEM image of the raw expanded perlite and it shows the highly porous three-dimensional structure composed of thin, fragmented lamellae arranged in an interconnected network of cavities and channels. The observed topography is characteristic of materials subjected to rapid thermal expansion processes, during which residual water can be trapped within the vitreous particles and can also abruptly vaporize. The latter leads to the characteristic puffed-up appearance of the inert.

The irregular morphology and the presence of both open and closed pores, with micrometric dimensions ranging from 1 and 10 μm , confer to the expanded perlite properties such as low bulk density and high specific surface area. These characteristics make this material particularly attractive for applications in thermal and acoustic insulation, hydroponic growth media, and as a lightweight aggregate in the formula-

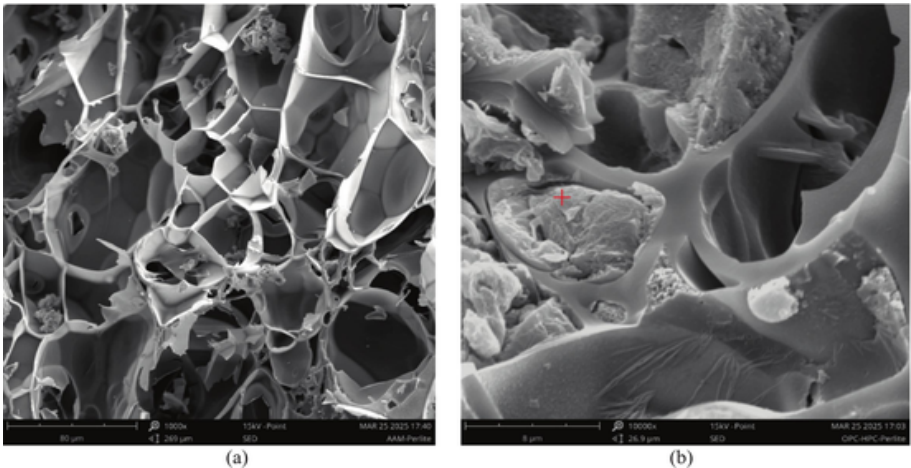


Fig. 15. A: sem image of perlite aggregates before coating and micrograph of perlite aggregate after cement-based coating (red cross is an indication of eds analysis area). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

tion of special concretes. Some regions show more collapsed structures, which may indicate mechanically weak zones induced by internal stresses during the expansion process.

Fig. 15b presents a SEM micrograph (with 10,000 × magnification) of the expanded perlite embedded within a cement paste matrix. In this image, it is evident that the porous perlite structure is partially infiltrated by the hydration products of the cement, resulting in intimate interfacial contact between the two phases. The cementitious matrix as a shell for the encapsulation appears to penetrate some of the open pores and/or surface irregularities, suggesting potential mechanical interlocking and chemical bonding at the interface. The morphology of the cement hydration products, surrounding the perlite, which is likely comprising the calcium silicate hydrate (C-S-H) gels and ettringite-like phases, demonstrates a relatively dense and cohesive structure, which may enhance the load transfer capability across the perlite-cement interface.

The investigation of the EDS (indicated at ‘+’ mark in Fig. 15b) indicates hydration products. The high calcium concentration (55.31 wt%, equivalent to 63.74 wt% CaO) and the relatively low silicon content (7.14 wt%, equivalent to 12.59 wt% SiO₂) yield a Ca/Si weight ratio of approximately 7.75. This value exceeds the typical range of 1.5–2.0 reported for calcium silicate hydrate (C-S-H), the primary hydration product in cementitious systems, suggesting the predominance of calcium-rich phases such as portlandite. Table 17 indicates the EDS composition of the analyzed area.

3.9. XRD

Fig. 16 shows the Rietveld refinement [54555657] of the SLM XRD pattern where at least 10 compounds were indexed. Each Crystallographic Information File (CIF) was used from Inorganic Crystal Structure Database (ICSD). Table 18 compares the quantities (% wt.) of the samples, where pristine and perlite adding powders evidenced same crystallographic composition. In all cases, quartz was the major

Table 17
EDS point analysis of perlite aggregates after cement-based coating.

Element	Weight (%)	Oxide	Wt. (%)
Ca	55.31	CaO	63.74
Si	7.14	SiO ₂	12.59
Al	2.98	Al ₂ O ₃	4.64
S	0.38	SO ₃	0.78
Fe	14.72	Fe ₂ O ₃	17.34

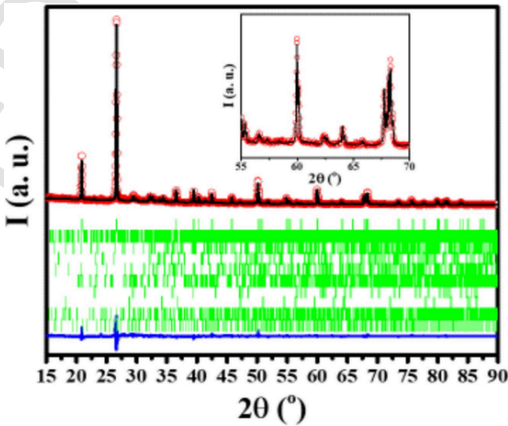


Fig. 16. Rietveld refinement for SLM sample powder. Intensity (I) is expressed in arbitrary units (a.u.). Observed (O), calculated (■) and difference between both (|), and Bragg positions (|) are indicated. Chi-square (χ^2) from fitting is 7.63%.

Table 18
Comparison of the overall composition (% wt.) obtained by Rietveld method. Errors are also indicated in ().

Phase	PDF file	Sample (% wt.)	
		SLM	SLM-Perlite
quartz	00-046-1045	74.89 (60)	73.12 (62)
alite	01-070-8632	9.73 (25)	12.84 (28)
belite	01-086-0398	2.77 (27)	3.16 (29)
ferrite	01-071-0667	2.34 (14)	2.31 (15)
gypsum	00-033-0311	2.26 (19)	2.23 (21)
hemihydrate	01-0183-0438	2.77 (19)	2.19 (20)
calcite	01-086-0174	1.65 (13)	1.33 (14)
portlandite	01-072-0156	0.51 (8)	0.55 (9)
ettringite	00-041-1451	2.50 (28)	1.53 (30)
aluminite	01-070-0839	0.58 (11)	0.73 (11)

component detected, which was mainly due to the use of pozzolanic material.

4. Conclusion

This work developed and evaluated self-leveling mortars incorporating PCM-enhanced perlite aggregates for thermal energy storage (TES) applications in buildings. The preplaced aggregate technique enabled the successful inclusion of fragile encapsulated aggregates without structural damage, resulting in workable and homogeneous mortar systems.

Thermal analyses demonstrated that the PCM-enhanced mortars provided significant benefits in terms of latent energy storage and dynamic thermal buffering. During the phase transition (25–30 °C), the apparent thermal conductivity decreased from 0.45 W/m × K (reference) to 0.28 W/m × K, while heat flux values were reduced by over 50%. In steady-state conditions, U-value dropped from 5.41 W/m² × K to 3.80 W/m² × K, confirming enhanced thermal insulation performance. In addition, the specific heat capacity increased by approximately 22%, reinforcing the composite ability to buffer thermal loads during TES performances and can lead to delay heat transfer under realistic temperature fluctuations.

Complementary mechanical and durability tests were performed to verify the feasibility of the composites for building applications. Mechanically, the reference SLM achieved compressive and flexural strengths of 60.14 MPa and 5.79 MPa, respectively. The inclusion of expanded perlite led to an expected 52% reduction in compressive strength, while PCM-enhanced aggregates reduced it by 70%, reaching 17.62 MPa. However, the encapsulation shell promoted a 12% increase in flexural strength compared to uncoated perlite, suggesting a structural contribution from the encapsulation layer. Water absorption remained low for the first 3 h but surpassed 3.5% after 72 h in PCM mortars, indicating a delayed but significant permeability increase.

The results confirm that PCM-enhanced lightweight aggregates can significantly improve the TES performance of mortars, particularly under dynamic conditions, making them strong candidates for passive energy control in NZEB buildings. Besides the material-scale investigation presented here, the proposed PCM-perlite SLM can be employed as a design thermal *meta*-variable to be employed within a general multi-layer building envelope (named as *meta*-envelope in previous works of the authors). In such a optimization-based framework, the building energy demand (based on heating, cooling and peak-load reduction) can be adopted as objective function of the optimization problem, while a key role is taken by the main design variables such as PCM melting temperature and type, PCM content/fraction in perlite, encapsulation shell thickness and thermal properties, and perlite fraction/distribution across layers. In this concept, future work will couple building-scale simulations (e.g., via EnergyPlus) with constrained optimization rules (including workability, mechanical stability or durability limits) to identify optimal multi-layer solutions for most effective energy-efficient envelope (inner vs. outer layers), thus to identify where the proposed PCM-perlite SLM (and PCM dosage) should be placed for maximizing TES benefits.

From an economic and environmental point of view, the aforementioned optimization strategy is crucial to quantify cost-effectiveness, since often PCM-related costs can clearly influence the overall construction price. A better and optimized allocation of PCM, across the full-scale building, can provide the highest energy benefit, minimize unnecessary PCM usage, and reduce the CO₂ footprint of the composite. In this sense, a complete LCA/LCC and techno-economic assessment will be performed in future work to quantify environmental outputs and costs/benefits versus energy savings achievements.

CRedit authorship contribution statement

Mahdi M.K. Zanjani: Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Saulo Rocha Ferreira:** Writing – review & editing, Validation, Data curation, Conceptualization. **Alberto Lagazzo:** Methodology, Data curation. **Juan F. Basbus:** . **Victor D. Fachinotti:** . **Antonio Caggiano:** Writing – review & editing, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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