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Enhancing sustainability with waste hemp-shive and phase change material: Novel gypsum-based composites with advanced thermal energy storage properties

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ABSTRACT

This study addresses the rising demand for sustainable construction by introducing composite materials from natural and renewable resources: gypsum, hemp, and phase change materials (PCMs). These materials cater to the growing preference for eco-friendly building solutions. Incorporating hemp enhances sustainability while integrating PCMs into the porous hemp structure ensures adequate thermal energy storage and release without leakage. Firstly, the agricultural waste hemp shives and lauryl alcohol (LA) PCM were mixed to create shape-stabilized hemp/PCM composites. The highest PCM ratio was determined in shape-stabilized composites exhibiting non-leakage properties, which was 45 wt %. These composites were then incorporated into gypsum materials at loadings of 7.5 %, 15 %, 22.5 %, and 35 wt % to produce the final composites. Morphological, thermal, and chemical characteristics of shape-stabilized composites were examined using SEM, TGA, and DSC, while the solar thermoregulation tests assessed the gypsum matrix composites. The phase change temperature of PCM was determined as 20.24 °C with a melting enthalpy value of 224.4 J/g. The hemp/PCM shape-stabilized composites demonstrated an impressive melting enthalpy value of 100.2 J/g, with only a slight reduction to 99.5 J/g after 750 test cycles. When the ambient temperature exceeded 50 °C, the central temperature of the cabins containing PCM composites was found to be at least 4 °C cooler than those containing only gypsum. Conversely, when the ambient temperature dropped to around 20 °C, it was observed that the central temperature of the cabins with PCM composites was approximately 2 °C warmer than those with only gypsum. This study introduces a novel approach to creating environmentally friendly gypsum/hemp/PCM composites for thermal energy storage systems.

1. Introduction

Climate change has underscored the critical significance of natural thermal insulation and reducing the carbon footprint (Azarkamand et al., 2020; Owusu and Asumadu-Sarkodie, 2016; Walenta, 2020). Raw insulation materials, such as straw (Liu et al., 2019), hemp (Santoni et al., 2019), or wool (Ahmed et al., 2019), are climate-friendly options that effectively regulate indoor temperatures. Utilizing these renewable and low-impact resources can significantly reduce carbon emissions using alternative insulation materials (Kumar et al., 2020). This mitigates the environmental impact and contributes to energy efficiency, reducing energy demand for heating and cooling. Adopting these natural solutions is essential in our fight against climate change, promoting sustainability and environmental responsibility (Seddon et al., 2021; Wamsler et al., 2020).

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Abbreviations

PCM	Phase Change Material
LA	Lauryl Alcohol
TES	Thermal Energy Storage
DSC	Differential Scanning Calorimeter
TGA	Thermogravimetric analysis
SEM	Scanning Electron Microscopy
EDS	Energy Dispersive Spectroscopy
EPS	Expanded polystyrene
Ca	Calcium
O	Oxygen
S	Sulfur
R	Reference sample of gypsum
Mix1	92.5 wt % gypsum – 7.5 wt % hemp/PCM composite
Mix2	85 wt % gypsum – 15 wt % hemp/PCM composite
Mix3	77.5 wt % gypsum – 22.5 wt % hemp/PCM composite
Mix4	65 wt % gypsum – 35 wt % hemp/PCM composite

In building applications, there is a growing focus on using natural aggregates derived from waste materials in an environmentally friendly manner (Ghosn et al., 2020). Among these natural aggregates, hemp fiber-reinforced concrete exhibits strong scientific properties, such as high tensile strength, reduced shrinkage, and improved crack resistance. Incorporating hemp fibers enhances the strength and longevity of the composite materials (Bayraktar et al., 2023; Bhoopathi and Ramesh, 2019; Filazi et al., 2023). This is particularly important in construction, where materials must endure various stresses over time. These characteristics make it suitable for roofing, walls, and floors. Gypsum and hemp biocomposites are essential in building applications, particularly in stabilizing room humidity levels. These materials could absorb and release moisture as needed, helping to create a more comfortable and stable indoor environment (Charai et al., 2021; Pietruszka et al., 2019). Relative humidity (RH) levels are crucial for human health and building preservation, and materials like gypsum and hemp biocomposites contribute to maintaining an ideal RH (Ntimugura et al., 2020; Psomas et al., 2021). Trociński et al. (2023) found that including hemp fibers in gypsum composites led to a fourfold increase in setting time and enhanced flexural strength. Hemp fibers create a structural network within the composite material, augmenting its strength. Furthermore, the fibers impede the movement of water and gypsum particles during the setting process, resulting in an extended setting time. This dual influence of reinforcement and altered setting kinetics collectively contributes to the enhanced material properties. Adding hemp fibers, as proposed by Page et al. (2017), has been suggested as the most effective additive to reduce shrinkage during the concentration drying process. The mechanism behind the reduced shrinkage when adding hemp fibers is interpreted by the fibers' ability to create a reinforcing network within the concentrate material. These fibers help maintain the material's structural integrity as it dries, preventing excessive contraction and shrinkage. When Mutuk et al. (2023) examined the thermal properties of gypsum composites with hemp fiber reinforcement, they found that the thermal conductivity of unreinforced gypsum was higher than that of hemp fiber-reinforced counterparts. Hemp fibers are effective thermal insulators, impeding heat flow through the material. This effect is primarily due to the fibrous structure of hemp, which creates air pockets inside the material, acting as a barrier to heat transfer and thus improving insulation characteristics. Therefore, it is evident that with the improvement of these properties, it is possible to apply these materials in building applications. The primary purpose of this study is to improve the thermal storage properties of these composites, which are frequently used in building applications due to these advantages, by increasing their response capacity to temperature changes.

At this point, phase change materials (PCMs) are excellent candidates for enhancing thermal storage properties when added. At a constant temperature, PCMs can absorb or release significant thermal energy while undergoing phase transition, such as melting or solidifying. This ability to store and release at specific temperatures makes them valuable for improving the thermal performance of materials (Faraj et al., 2020; Jouhara et al., 2020; Wu et al., 2020). Some of the naturally occurring agricultural waste materials that can be augmented with gypsum (plaster) and PCMs for building applications include barley straws (Kehli et al., 2023), rice straws (Singh et al., 2023) and wood shavings (Mohammed et al., 2021). Barley straws can be enhanced with gypsum and PCM to reduce water absorption and increase tensile strength. This results in a more durable material that is less moisture-resistant, making it suitable for building applications (Majid et al., 2020). Rice straws can be processed with PCM to increase their thermal storage capacity. PCM-impregnated rice straws can help regulate building temperature by absorbing and releasing heat as needed (Rehman et al., 2021). Wood shavings increase thermal mass and storage capacity when processed with gypsum and PCM. A substantial reduction in heat gain is evident when incorporating PCMs alongside wood shavings into the mix. Throughout extended periods of elevated temperatures, there was an observed decrease of up to 5.8 °C in peak temperature, according to Mohammed et al. (2021).

Their shape-stabilized composites are achieved by impregnating the gypsum-agricultural waste fiber composite with PCMs. This is typically done by melting the PCM and infusing it into the porous structure of the composite. As the PCM cools and solidifies, it is trapped within the pores of the composite, giving it "shape stability" (Huang et al., 2019). Incorporating PCMs increases the composite's thermal mass, allowing it to store and release heat energy more effectively. This improves temperature regulation and reduces temperature fluctuations in building applications (Rathore and Shukla, 2021). Shape-stabilized composites reduce reliance on active heating and cooling systems, potentially diminishing energy expenses. By sustaining a consistent temperature, these materials contribute to a more comfortable environment, enhancing overall comfort without needing energy-intensive processes (Zhou et al., 2023). Using agricultural waste fibers and PCMs aligns with sustainability goals, as it repurposes waste materials and promotes energy-efficient construction (Ghani et al., 2021). Compared to other methods, shape-stabilized composites' critical difference and advantage lie in the effective encapsulation of PCMs within the composite. This immobilization of the PCM within the structure ensures that it remains in place and ready to absorb and release heat as needed. Other methods may involve loose PCM materials, which can be less reliable and practical in real-world applications. Shape-stabilized composites offer a more controlled and stable solution for thermal energy management in buildings (Mohaisen et al., 2022). Hekimoğlu et al. (2023) found that composites obtained by incorporating apricot kernel shell-derived activated carbon and PCMs into mortars exhibited compressive strengths of up to 14.7 MPa and a storage capacity of 30 J/g. The energy storage capacity of 30 J/g results from the activated carbon's ability to store energy due to its porous nature and high surface area.

Based on the information in the literature above, the addition of PCMs to hemp shives-gypsum composites can have a significant scientific impact on improving their thermal properties. Recently, in only one study as far as it was researched, Bumanis and Bajare (2022) surveyed PCM-modified gypsum hempcrete composites, demonstrating increased heat capacity utilizing microencapsulated PCM of paraffin wax and chopped hemp shives. However, with the appropriate selection of supporter material porosity and morphology, using non-encapsulated PCMs offers a more straightforward production process, reduces production costs, and, importantly, provides a higher heat energy storage capacity per unit mass. Therefore, in this study, non-capsulated LA PCMs doped into waste hemp sieve structures with high porosity were mixed with gypsum matrix to produce gypsum/hemp/PCM composites for the first time. Thus, the aim was to bridge the found in enhancing the thermal energy storage properties by hybridizing gypsum materials, indispensable for building applications in the literature, with hemp sieve/non-capsulated LA PCM composites. This streamlined production process translates to quantifiable benefits, such as a significant decrease in energy consumption during building heating and cooling applications. Additionally, eliminating microencapsulation processes leads to a notable decline in emissions, with an estimated reduction of CO_2 equivalents because of non-capsulated PCM's higher latent heat capacity. Furthermore, by utilizing hemp shives, agricultural waste products, as a supporting material, the production process contributes to waste reduction and promotes cleaner production practices. Incorporating hemp shives not only diverts waste from landfills but also enhances the sustainability of the composites by utilizing renewable and biodegradable resources. This approach aligns with sustainable principles by minimizing environmental impact and fostering a circular economy model, ultimately contributing to a cleaner and more sustainable future. Moreover, the importance of these materials for the building sector must be balanced. PCM-modified hemp/gypsum composites offer tremendous potential for enhancing buildings' energy efficiency and thermal performance. Their use can lead to the construction of more environmentally friendly, more comfortable, and cost-effective structures. Thus, adopting these innovative materials represents a significant advancement in sustainable building practices, with far-reaching implications for the construction industry. This study aimed to advance sustainable construction practices by introducing innovative gypsum/hemp/PCM composite materials that offer improved energy efficiency and indoor comfort. This study envisions these composites being utilized in various construction applications, such as insulation, wall panels, and roofing systems, where their superior thermal properties can positively impact building energy performance. Through further research and development, the aim is to explore the full extent of their potential and facilitate their integration into mainstream construction practices.

2. Materials and methods

This section includes a list of materials used and an explanation of the experimental methods. It describes the materials and their purposes and provides detailed descriptions of testing procedures, experimental design, measurement methods, and analyses.

2.1. Gypsum

This study utilized commercially available β-hemihydrate gypsum from Dalsan Gypsum Co. Ltd. in Ankara, Turkey. Designed explicitly for crafting decorative elements like cornices, this β-gypsum powder offers several advantages. Its extended working time makes the application process user-friendly and minimizes material wastage. Additionally, it boasts superior strength and surface hardness, enhancing the durability of the elements. The refined grain size of the β-gypsum powder ensures a smooth cast surface, improving the aesthetic appeal. Table 1 provides comprehensive details on the physical attributes of the gypsum powder and the mechanical properties of gypsum plaster formulated with a precise 0.6 water/binder ratio. Gypsum was used as a primary matrix for fabricating gypsum-hemp-PCM final composites in this study.

Table 1
The β-Gypsum powder's physical attributes and the mechanical characteristics of gypsum plaster are elucidated in this study.

Properties	β-Gypsum
Water/Plaster ratio	Molding 7–7.5 lt water to 10 kg Bonding 6–6.5 lt water to 10 kg
Setting time starts	>8 min
Final setting time	30 min
Compressive strength (minimum)	100 kgf/cm^2 (4 × 4 block)
Flexural strength (minimum)	45 kgf/cm^2 (4 × 4 × 16 block)
Pass 200-μm sieve (minimum)	99.5%
Pass 100-μm sieve (minimum)	95%
Bulk density (powder)	750–800 kg/m^3
Dry density	1050–1100 kg/m^3
Reaction to fire	A1

2.2. PCM

The PCM of lauryl alcohol ($CH_3(CH_2)_{11}OH$) that has a purity of ≥98.0% and melting temperature of 22–26 °C was procured from Sigma Aldrich Co. This PCM is used for fabricating the preparation of shape-stabilized hemp-PCM composites, followed by the hemp-PCM composites, which were to be reinforced to gypsum matrix to enhance thermal energy storage/thermal regulation purposes of gypsum-hemp-PCM final composites.

2.3. Hemp

AUBIOSE brand hemp (shiv) obtained from the Champagne region in France was sourced from the MarsLab company. During cultivation, no agricultural pesticides were used, ensuring it contains no foreign substances and is 100% natural. It does not contain fibers. The producer produces the shiv by grinding separate hemp stems. The shiv (broken hemp stem) was dried in an oven at 100 °C for 24 h and then ground in a grinder. The resulting hemp powders were separated based on particle size through sieve analysis carried out with a sieve, as given in Fig. 1. Sieving processes were carried out to obtain particles below 180 μm. Although the size range discussed is broad, particles smaller than 180 μm and those near this size offer several advantages for PCM-infused shape-stabilized composites. These benefits include enhanced surface area for PCM dispersion, improved interfacial adhesion between PCM and hemp particles, increased thermal conductivity due to homogeneous PCM distribution, and efficient PCM encapsulation with reduced risk of leakage. Furthermore, the comprehensive size range, encompassing larger particles, enhances mechanical strength and structural integrity, forming a porous network aiding PCM impregnation, preventing

Fig. 1. Sieving analysis machine used in the study.

agglomeration, and settling for uniformity. However, larger hemp particles present challenges such as potential weak points in the composite and hindered PCM distribution, leading to lower thermal conductivity. Additionally, there's an increased risk of particle settling during manufacturing. Thus, a particle size of <180 μm was chosen for hemp shives in this study to optimize composite performance and stability. Hemp shives were used as supporters for preparing shape-stabilized hemp-PCM composites, which were to be reinforced into the gypsum matrix to fabricate final gypsum-hemp-PCM composites.

2.4. Preparation of shape-stabilized composite Hemp/PCM

A Hemp/LA PCM composite was created using the direct mixing method. This approach combined dried hemp shives with a specified quantity of molten LA within 40 wt %, 45 wt %, and 50 wt %. This process yielded various hemp/LA PCM mixtures, allowing the ideal PCM mass fraction in the hemp/LA PCM composite to be evaluated. As illustrated in Fig. 2, a leakage issue was observed in the Hemp composite structure doped with 50 wt % PCM in this context. Therefore, hemp composites reinforced with 45 wt % PCM, where this issue was not observed, was utilized to produce gypsum matrix composites in the following fabrication process. This choice of PCM reinforcement content enhances the overall efficiency and functionality of the hemp composites within gypsum matrices, showcasing a well-founded scientific approach to material composition for improved outcomes.

2.5. Fabrication of the gypsum matrix composites

The shape-stabilized Hemp/PCM composite was replaced with the gypsum by weight, as given in Table 2. A dual-component system was established to formulate composite gypsums within the experimental framework. The initial component featured gypsum, while the second component comprised shape-stabilized hemp/PCM composites characterized by an optimal non-leakage ratio of 45 wt % PCM. The compositional details governing the production of these two-component gypsums are meticulously outlined in Table 2. Five distinct composition ratios were systematically employed to synthesize reference samples and composite gypsums. The initial specimen, designated as R in Table 2 and Fig. 3, exclusively incorporated 100 wt % gypsum matrix. Subsequently, Mix1 integrated a blend of 92.5 wt % gypsum and 7.5 wt % of the leakage-resistant hemp/PCM composite, which, notably, contained 45 wt % PCM. The other formulations denoted as Mix2, Mix3, and Mix4, encompassed compositions of 85% gypsum (alongside 15 wt % hemp/PCM45 wt % composite), 77.5 wt % gypsum (paired with 22.5 wt % hemp/PCM45 wt % composite), and 65 wt % gypsum (integrated with 35 wt % hemp/PCM45 wt % composite) respectively, as visually represented in Fig. 3. This methodical approach not only ensures the systematic exploration of various composition ratios but also aligns with the scientific rigor inherent in the study, offering extensive considering of impact of different components on the resulting composite gypsums.

Table 2
Mixture contents of shape-stabilized Hemp/PCM composites with gypsum matrix.

Code	Gypsum (g)	Shape-stabilized Hemp/PCM composite (g)	Replacement ratio (wt. %)	Water (g)
R	300	0	0	154
Mix1	277.5	22.5	7.5	166
Mix2	255	45	15	184
Mix3	232.5	67.5	22.5	248
Mix4	195	105	35	280

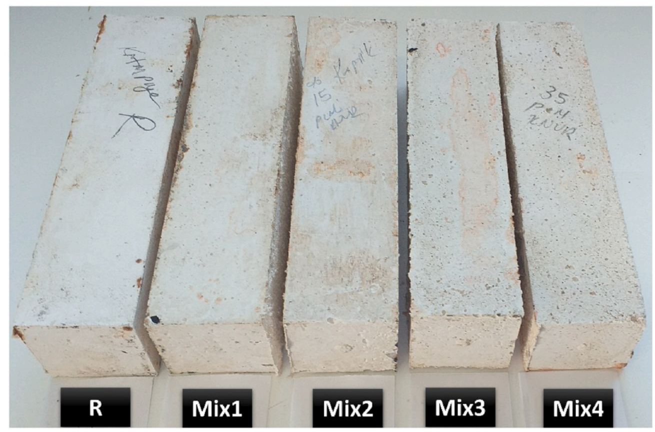


Fig. 3. The fabricated samples with different Hemp/PCM composites contents in gypsum matrix.

2.6. Characterizations

The mechanical properties of Hemp/PCM/gypsum composites were evaluated through compressive strength tests. After seven days, the ASTM C349 standard ASTM C349-08 was followed for mechanical testing of samples with 40 × 40 × 160 mm. For thermal conductivity measurements, the TCi Thermal Conductivity Analyzer, which has a range of 0–500 W/mK, by ASTM D7984, was employed. This method involved applying a momentary heat pulse to the sample surface, and thermal effusivity was determined based on the temperature increase over time.

The phase change heats, TES abilities of pure LA, and the enhanced lightweight functionalized PCM were determined using a Differential Scanning Calorimeter (DSC, Hitachi) in a controlled nitrogen gas environment. Further, to evaluate the effect of thermal cycling on the reliability of latent heat properties, the Hemp/PCM composite was subjected to consecutive cycles of heating and cooling using a thermal cycler (BIOER TC-25/H model). Each cycle involved heating the composite to 60 °C and maintaining this temperature for 3 min, followed by rapid cooling to 0 °C and sustaining this temperature for the same

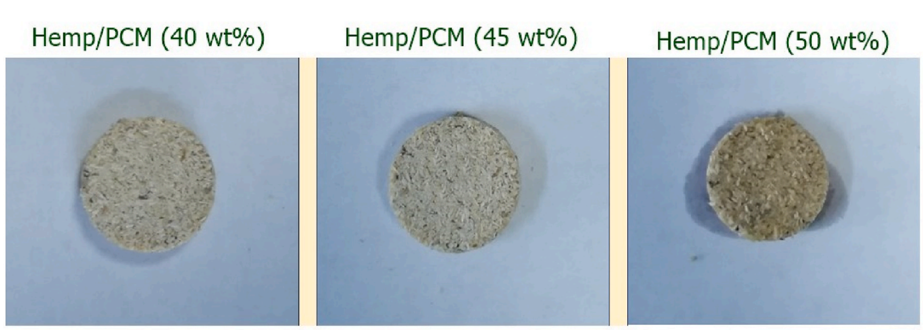


Fig. 2. Fabricated shape-stabilized Hemp/PCM composites in different content of PCMs.

duration. This process was repeated for 750 cycles. Subsequently, DSC analysis was conducted on the cycled Hemp/PCM composite to evaluate any alterations in its latent heat properties. Thermogravimetric analysis (TGA) was performed using the Linseis TGA instrument under an argon atmosphere. The heating rate was 20 °C/min from 25 to 500 °C. Scanning Electron Microscopy (SEM) combined with Energy Dispersive Spectroscopy (EDS) was employed to examine internal and micro-structural variations in pure hemp shives and different mix ratios of Hemp/PCM composites.

To assess the efficacy of R and Mix1-Mix4 wallboards in regulating solar temperature under authentic atmospheric conditions, two identical test cabins of 200 × 200 × 20 mm were constructed for on-site experimentation. Each cabin featured a double-glazed rooftop window (140 × 140 × 2 mm) designed to permit a solar radiation transmission coefficient of 0.77. The inner walls, the room floor, and the perimeters of the window were enveloped with a 5 cm layer of expanded polystyrene foam (EPS). The experiment took place on a rooftop situated at coordinates 41° 38′ 8.99″ N and 32° 20′ 15″ E in the Bartın province of Turkey, spanning from August 10 to August 14, 2023, on a day characterized by a mix of sunshine and clouds. Temperature readings were meticulously recorded using a data logger, while the globe solar irradiance was gauged through a pyranometer. K-type thermocouples were employed to determine ambient and room temperatures precisely. The configuration of the experimental setup and placement of thermocouples are delineated in Fig. 4 for reference.

3. Results and discussion

This section presents and analyzes the findings obtained from the experiments. Therefore, this section includes the study's results,

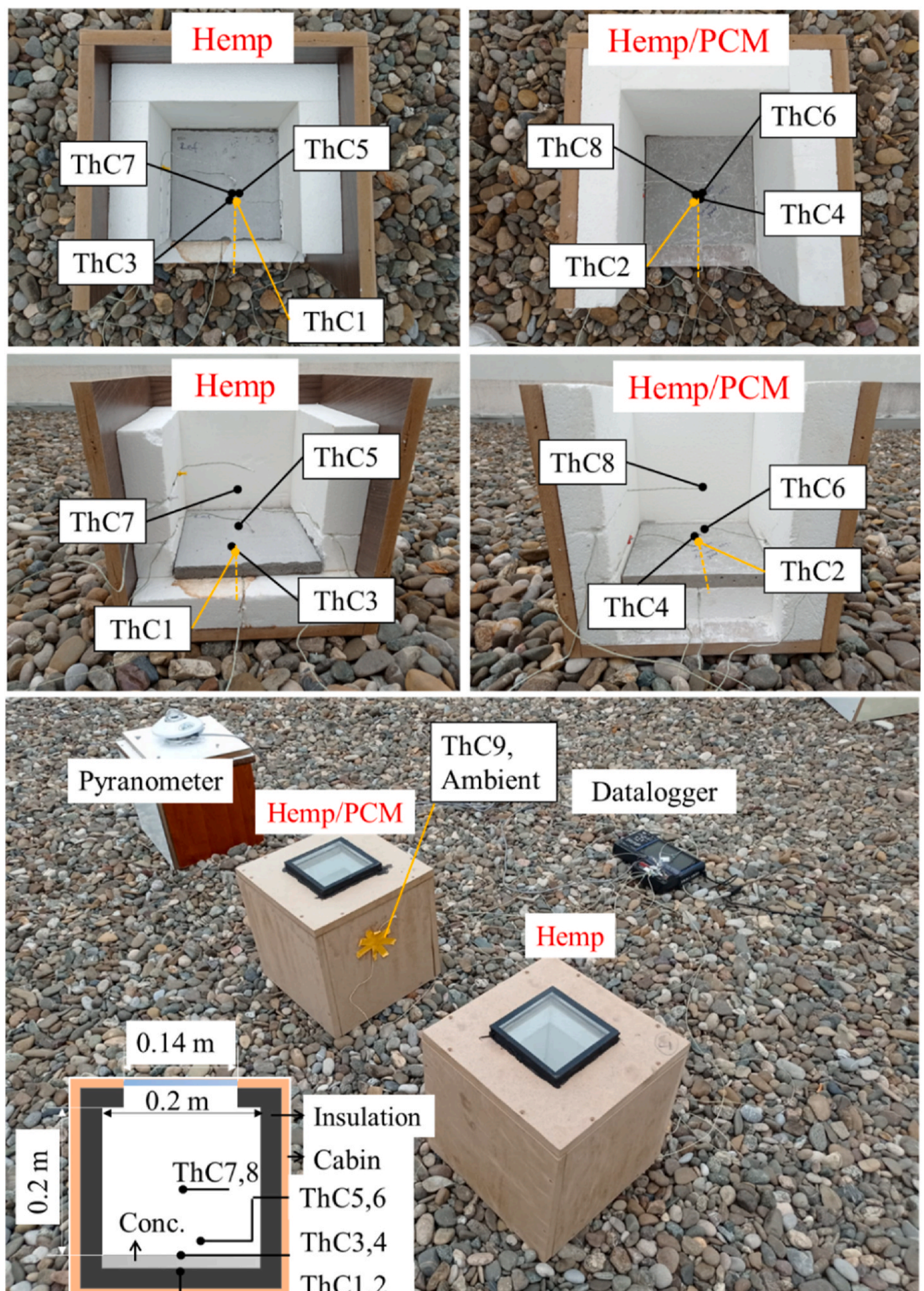


Fig. 4. Configuration of thermoregulation experiments and thermocouple placements within the test modules.

interpretation of the results, comparison with previous research, and discussion regarding the implications of the findings.

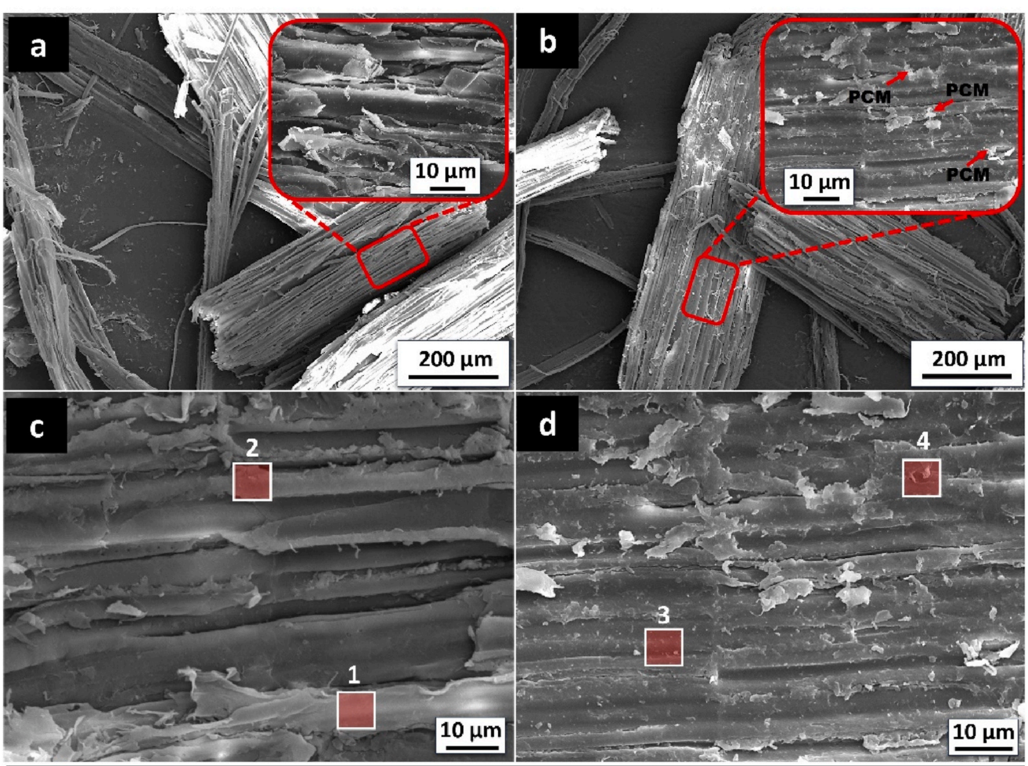
3.1. Morphological evaluation of Hemp/PCM composites

Morphological examination and elemental distribution ratios of pure hemp and hemp composites impregnated with 45% PCM produced in the study are presented in Fig. 5. As evident in Fig. 5a, the structure of hemp shives displays numerous pores. Observing abundant pores within the hemp shives suggests a favorable microstructure for PCM impregnation. The presence of these pores creates an ideal environment for accommodating the PCM, ultimately contributing to the composite material’s enhanced thermal properties and performance. Hemp shives’ porous structure makes it a promising option for TES due to efficient heat storage and release. In connection to this, the morphological change resulting from PCM impregnation into hemp shives is evident from Fig. 5b. It is apparent that the pores present in the structure of pure hemp are filled with PCM, leading to a notable reduction in the pore volume. This morphological transformation underscores the successful impregnation of PCM within the hemp shives. The significant decrease in the pore volume of the composite material compared to pure hemp indicates the effective integration of PCM. This alteration in the microstructure of the composite suggests an improved capacity for energy storage and controlled release. It is a promising development for applications with an essential need for efficient thermal regulation and energy storage.

Fig. 5c and d, respectively, illustrate the points where EDS elemental analyses were conducted on pure hemp shives and hemp/PCM composites. It is clear that in the analyses performed on the pure hemp structure (Spectrum 1 and Spectrum 2), the carbon content ranges between 94% and 97%. However, in the analyses obtained from the hemp/PCM structure (Spectrum 3 and Spectrum 4), the carbon content remains above 99%, and the presence of calcium (Ca) elements, as seen in the pure hemp structure, is not detected at these points. This indicates that the PCM additive is well-distributed within the hemp matrix. These results confirm the effective distribution of PCM within the hemp shive’s structure. In contrast to the reduction observed in pure hemp, maintaining high carbon content in the hemp/PCM composites suggests that the PCM is integrated without significantly altering the carbon composition. The absence of detectable calcium elements in the hemp/PCM composites further supports the successful incorporation of PCM. This distribution of PCM within the hemp shives is a positive outcome, as it enhances the composite’s thermal storage capabilities, making it a promising option for various TES applications.

3.2. Microstructural evaluation of Hemp/PCM reinforced gypsum matrix composites

Fig. 6 presents SEM images illustrating the microstructural development of hemp/PCM composites incorporated into gypsum matrices at different ratios. The SEM analysis of pure gypsum material, denoted as “R" in Fig. 6a, exhibits a uniform and entirely needle-like microstructure. When examining the internal structure of Mix1 composites (Fig. 6b), it is evident that hemp shives are dispersed within the gypsum matrix. As the Hemp/PCM ratio increases, the amount of porosity in the structure increases, as observed in Mix2 (Fig. 6c), and is further pronounced in the internal structures of Mix3 (Fig. 6d) and Mix4 (Fig. 6e). This indicates that the interface between hemp shives and the matrix



e

Spectrum	C	Ca	K
1	94.3	3.1	2.6
2	97.5	1.0	1.5
3	100.0	–	–
4	99.1	–	0.9

Fig. 5. SEM investigation of the (a, c) Hemp and (b, d) Hemp/PCM45% composites, and (e) elemental results of EDS.

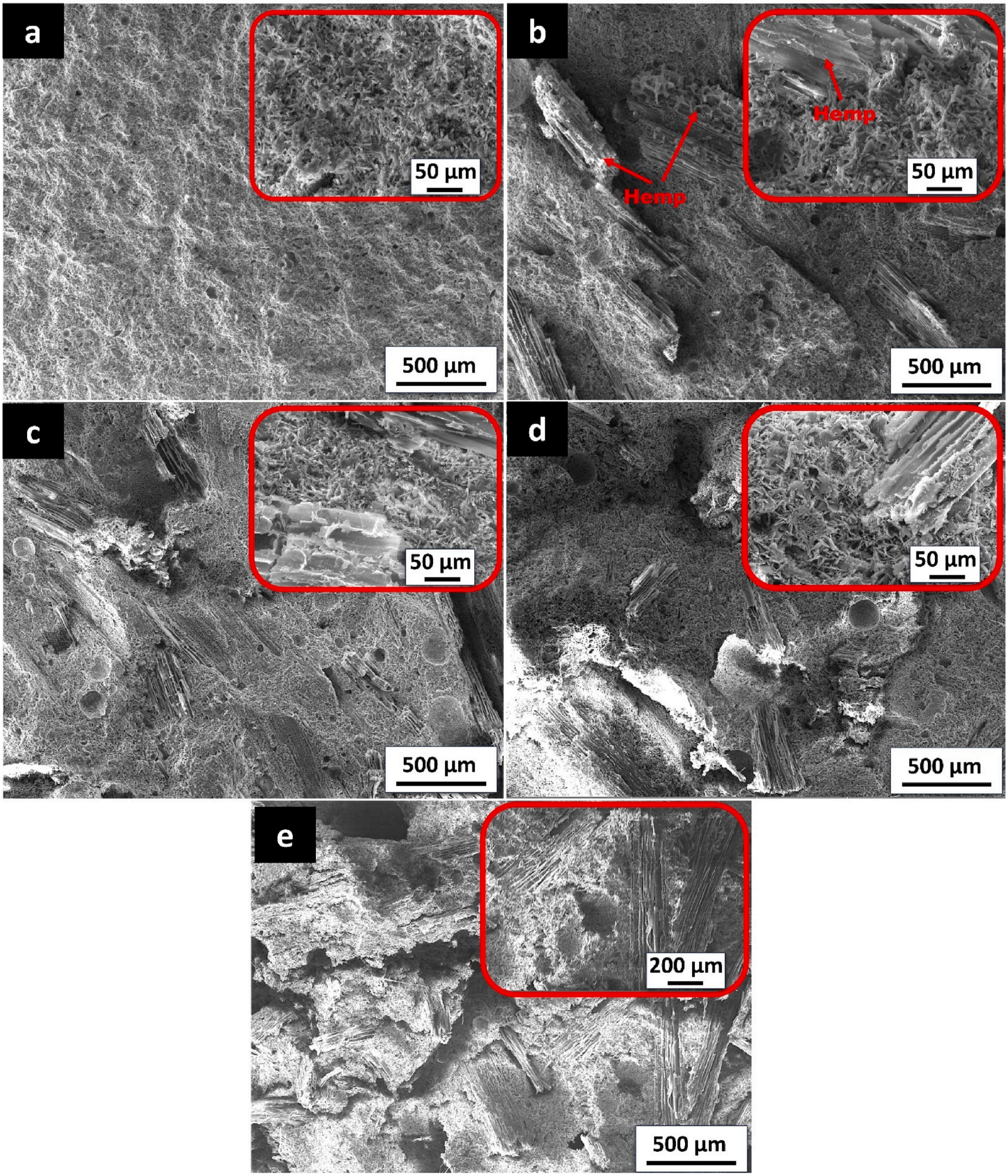


Fig. 6. Microstructural investigation of the (a) R, (b) Mix1, (c) Mix2, (d) Mix3, and (e) Mix4 composite samples.

gypsum does not form a strong bond, with Mix4 exhibiting the lowest compressive strength. However, considering the added PCM content and the improved thermal properties, the decrease in compressive strength can be deemed negligible. These observations suggest that the microstructure of the composites becomes more porous and less uniform as the Hemp/PCM ratio increases. This indicates that the strong interfacial bonding between hemp shives and the gypsum matrix is compromised, resulting in decreased compressive strength. Nonetheless, given the improved thermal properties associated with the increased PCM content, the reduction in compressive strength is of minimal concern. The critical point to note is that the trade-off between mechanical strength and enhanced thermal properties should be considered when using these composites for specific applications, as the potential for improved thermal performance might outweigh the decrease in compressive strength, depending on the application's requirements.

In Fig. 7, EDS mapping analysis results are presented, showing the distribution of elements within the structures of materials R and Mix3. In the structure of material R, naturally occurring elements such as calcium (Ca), sulfur (S), and oxygen (O) are uniformly distributed (Fig. 7a). However, in the structure of Mix3 composites, the presence of hemp and PCM has led to an area where carbon (C) element density is observed within the gypsum, indicating the inclusion of hemp/PCM composites into the gypsum matrix (Fig. 7b). The EDS mapping results emphasize the distinct elemental distribution between material R and Mix3 composites. This reflects the successful inclusion of hemp and PCM within the gypsum structure. The observed elemental distribution is consistent with the expectations of the composite's composition, providing evidence of the integration of hemp and PCM into the gypsum matrix. This distribution supports the potential for improved thermal properties and reinforces the suitability of these composites for thermal energy storage applications.

3.3. Physico-mechanical properties and thermal conductivity

Table 3 presents the dry unit weight, compressive strength, and thermal conductivity values of Mix1-Mix4 composites obtained by incorporating Hemp/PCM composites into the gypsum matrix at different ratios. The dry unit weight of pure gypsum materials (R), without Hemp/PCM, was calculated as 1.43 g/cm^3. As the proportion of Hemp/PCM composites increased, this value decreased to as low as 0.95 g/cm^3. The "dry unit weight" refers to the density of a material when it is in a dry state and is typically measured in grams per cubic centimeter (g/cm^3). It is a crucial factor in assessing the compactness and weight of a material, which can have implications for its load-bearing capacity and thermal properties. In this context, the decrease in dry unit weight as the ratio of Hemp/PCM composites increased indicates that introducing these composites into the gypsum matrix resulted in a lighter and less dense material. This density drop is related to the lower density of Hemp/PCM composites compared to pure gypsum. The change in dry unit weight is a crucial factor in the construction and engineering field, as it influences the overall performance and behavior of the composite material, including its load-bearing capabilities and thermal conductivity. The lower dry unit weight suggests that the composite may be lighter and potentially more thermally insulating, which could be advantageous depending on the specific application and design considerations. In this context, gypsum matrix composites' compressive strength is 35 wt % Hemp/PCM decreased from 20.7 to 1.2 MPa. It is evident that the addition of shape-stabilized hemp/PCM composites to the gypsum matrix results in a dramatic decrease in the compressive strength of the gypsum/hemp/PCM composites. This decline in compressive strength is attributed to the introduction of voids or weak points in the composite structure due to the incorporation of the hemp/PCM composites. Additionally, the difference in mechanical properties between the hemp/PCM composites and the gypsum matrix leads to uneven stress distribution within the composite, further compromising its compressive strength. Furthermore, the decreases in mechanical properties due to the prevention of hydration in the gypsum structure by filling the pores in the gypsum with PCM composites have also been reported in the literature (Fei et al., 2021). According to the NP EN 998-1 standard (Portuguese Institute for Quality, 2010), it has been reported that gypsum mortars used in construction applications should have compressive strength values starting from 0.4 MPa (Cunha et al., 2023). Since PCM-modified gypsum mortars are not intended for structural applications, it should be emphasized that gypsum matrix mortars with compressive strength values above this threshold are suitable for building applications for thermal regulation and heat storage. Additionally, research on similar compressive strength values for modified gypsum materials is frequently reported in the literature (Bumanis and Bajare, 2022; Powała et al., 2022). Therefore, the compressive strength of 1.2 MPa obtained in Mix4 composites in this study was considered acceptable for construction applications.

On the other hand, the thermal conductivity exhibited a similar trend, dropping from 0.622 to 0.181 W/mK, as calculated in Table 3. While the compressive strength of gypsum/hemp/PCM composites may diminish to 1.2 MPa, and also the thermal conductivity may drop to 0.181 W/mK, these materials still possess acceptable properties for energy efficiency and thermal regulation (cooling in hot weather and thermal insulation in cold weather) applications. The low thermal conductivity value (0.181 W/mK) indicates the material's potential as an effective thermal insulator. Low thermal conductivity restricts the transfer of external influences into indoor spaces, leading to increased energy efficiency. Therefore, buildings can save on heating and cooling expenses by implementing this solution. Including PCM can aid in the absorption and storage of thermal energy. This means the material can absorb heat in hot weather and release it in cold weather, contributing to the regulation of indoor temperatures. Storing and releasing heat helps maintain indoor temperatures that are more constant and comfortable. This can assist in cooling indoor spaces during hot weather and providing warmth during cold weather. Adding hemp, known for its lightweight and environmentally friendly properties, can contribute to sustainability goals in building construction.

3.4. DSC analysis results

DSC (Differential Scanning Calorimetry) analyses were conducted on pure PCM and Hemp/PCM45%wt—specimens to evaluate their TES characteristics. Fig. 8 displays the DSC thermograms that were obtained. Relying on DSC outcomes, it was found that temperatures at which melting and solidification began were determined for pure PCM as 20.24 °C and 18.82 °C, respectively. In contrast, for Hemp/PCM45 (45 wt %). Composites, these temperatures were slightly lower, measuring 19.87 °C and 20.64 °C for melting and solidification, respectively. The corresponding heat storage capacities were 224.4 and 223.2 J/g at melting and solidification phase transitions for pure PCM (LA). However, for Hemp/PCM45. Composites, these values were reduced to 100.2 J/g and 99.3 J/g. The Hemp/PCM45. Composite specimen underwent extended thermal cycling stability testing to assess its prolonged usability through 750 cycles. Fig. 3 and Table 4 show the DSC thermograms of the Hemp/PCM45. Composite were juxtaposed at the initial (1st) and final (750th) cycles. Remarkably, minimal disparity was observed in endothermic-exothermic peaks between the 1st and 750th cycles. This suggests that the hemp/PCM45. The composite sample consistently maintained its latent heat characteristics even after undergoing 750 cycles of melting and solidification. Consequently, the melting and solidification enthalpy values for hemp/PCM45. Composite samples, as determined after 750 cycles, were observed as 99.5 and 98.5 J/g, respectively. These findings demonstrate the suitability of hemp/PCM45 composites for thermal energy storage applications in building construction. Slightly lower phase transition temperatures of the composites compared to pure PCM indicate their potential to provide effective thermal regulation. Moreover, the ability of these composites to maintain their latent heat properties after 750 cycles suggests their long-term usability and durability. Therefore, these materials can be recommended for use in building applications to provide temperature control by storing and releasing thermal energy as needed.

In our study, the latent heat-absorbing values obtained from hemp/LA composites were compared with those obtained from composites produced by impregnating similar fibers with PCM, as shown in Table 5. It is evident that latent heat storage values exhibited by shape-stabilized PCM composites, made using similar materials, have been enhanced compared to the composites developed in this study. Scientifically, this implies that hemp shives/LA in the composite material formulation has led to improved latent heat storage capabilities compared to similar composites incorporating PCM. The observed enhancement suggests

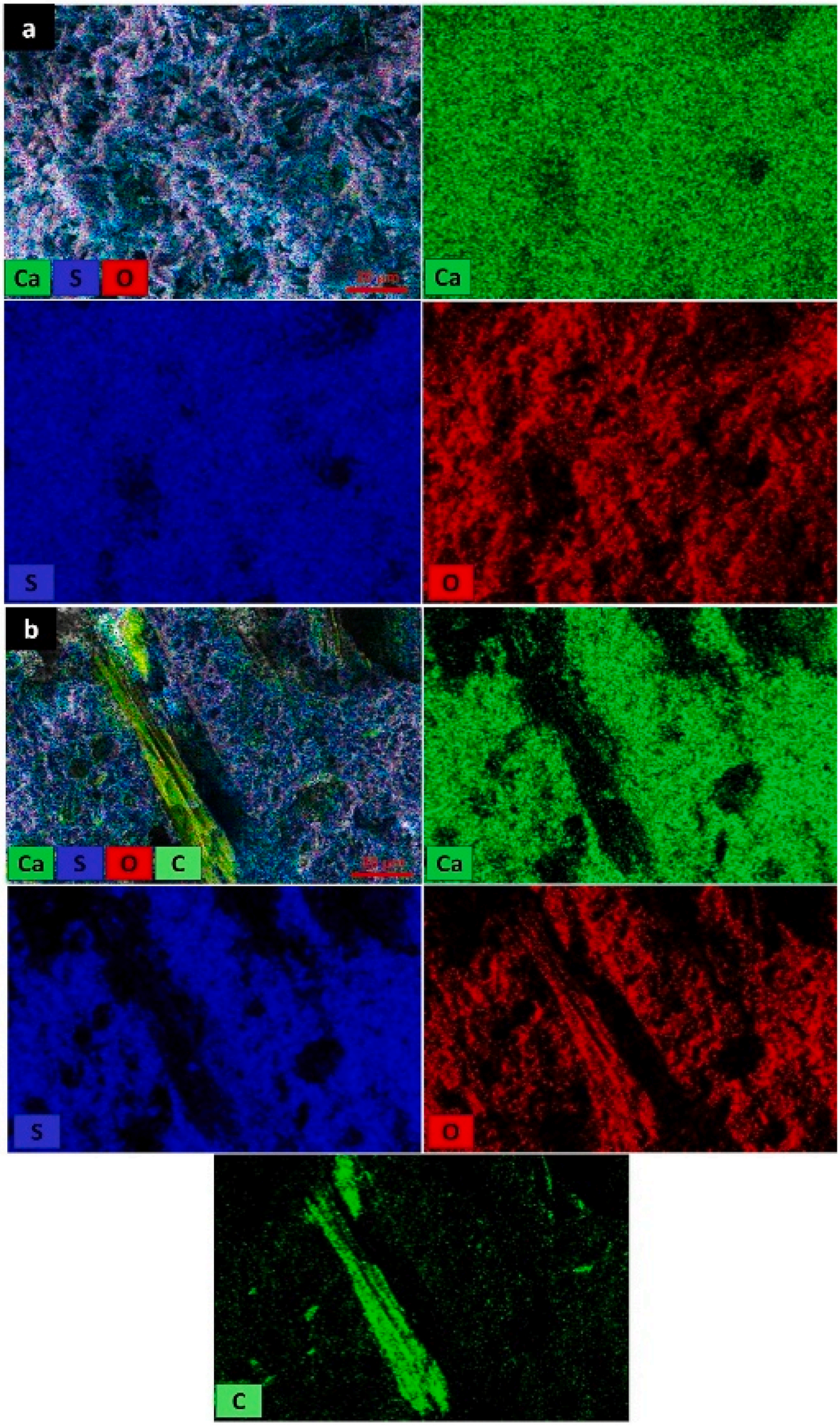


Fig. 7. Elemental investigation of the (a) R and (b) Mix3 composite samples.

Table 3
Physico-mechanical and thermal properties of the samples.

Code	Dry unit weight (g/cm^3)	Compressive strength (MPa)	Thermal conductivity (w/mK)
R	1.43	20.7	0.622
Mix1	1.34	9.2	0.406
Mix2	1.23	6.3	0.319
Mix3	1.09	3.5	0.218
Mix4	0.95	1.2	0.181

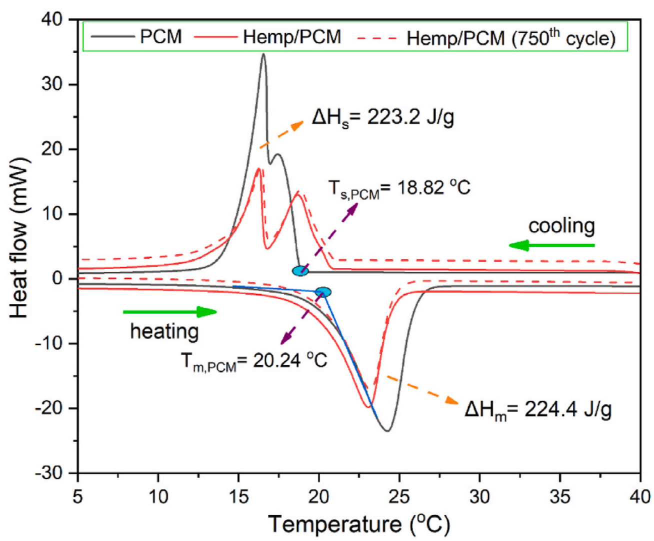


Fig. 8. DSC peaks of the pure PCM, Hemp/PCM composites and with 750th cycle of tests.

Table 4
The values show the storage characteristics of hemp/PCM composites.

Sample	Melting Point (°C)	Melting Enthalpy (J/g)	Solidification Point (°C)	Solidification Enthalpy (J/g)
PCM	20.24	224.4	18.82	223.2
Hemp/PCM	19.87	100.2	20.64	99.3
Hemp/PCM (750th cycle)	19.93	99.5	20.66	98.5

that the developed composites hold promise for applications where efficient thermal energy storage is critical. The comprehensive comparison presented in Table 5 underscores the significance of the research findings and the meticulous work conducted in this study.

Moreover, Sawadogo et al. (2023) obtained melting enthalpy and solidification enthalpy values of 79.3 J/g and 70.4 J/g, respectively, for macro-sized hemp shives matrix and 50 wt % LA-reinforced composites, as presented in Table 4. It is evident in this study that, despite having a 45 wt % LA content, our composite exhibits enhanced thermal properties. The micron-sized hemp shives used in our study may be critical to this improvement. The improved thermal properties observed in our study despite a 45 wt % LA content can be scientifically explained through the impact of the micron-sized hemp shives used in the composite. Reduction in shive size increases the surface area available for interaction with the surrounding matrix. As the size of the hemp shives decreases, there is a significant improvement in the bonding at the interface between the shives and the matrix material. The supercooling value expresses the tendency of a liquid to remain in a liquid state below its standard freezing point. Supercooling typically occurs when a liquid can be cooled to a specific temperature before reaching its freezing point. Therefore, a controllable supercooling level in a system or process is often preferable. According to the comparative results in Table 4 from the literature, the supercooling values (difference among melting and solidification temperatures) were the lowest in this study, approximately 1 °C. A low supercooling value indicates that a liquid tends to solidify near its standard freezing point, allowing for a more predictable solidification process. Maintaining a lower supercooling level in thermal energy storage systems is advantageous because it ensures a more efficient and reliable process when releasing stored thermal energy. Reduced supercooling minimizes uncertainties in the timing and characteristics of the phase transition, contributing to the overall performance and effectiveness of the storage system.

3.5. Thermal stability results

The peaks representing the results of TGA experiments conducted to assess the thermal stability of Hemp/PCM composites are provided in Fig. 9. As observed, pure PCM undergoes complete degradation without reaching a temperature as high as 300 °C and leaves behind only 0.32 wt % residue after heating to 600 °C. In contrast, Hemp material, due to the evaporation of moisture content present in it, starts exhibiting weight loss at around 120 °C, losing approximately 3% of its weight by the time it reaches 251 °C. However, the primary degradation of Hemp material occurs after 251 °C, with the complete degradation process extending until approximately 400 °C. At the end of heating to 600 °C, about 19 wt % residue was detected. When examining the peaks representing the thermal stability of composites obtained by incorporating 45% PCM into Hemp materials, it is observed that these composites exhibit their primary degradation event between 215 °C and 333 °C. As a result of the presence of both PCM and hemp in their structures, they yield approximately 8% residue at the end of thermal analysis. This analysis reveals that hemp materials possess relatively high thermal stability. This suggests that the combination of hemp and PCM can improve the thermal stability of composites, making them appropriate for various usages that require resistance to elevated temperatures and thermal degradation.

3.6. Solar thermo-regulative performance

The thermoregulation tests conducted for 96 h between 10 and 14.08.2023 aimed to scientifically assess and understand the impact of solar radiation on temperature regulation. These tests aimed to

Table 5
Comparing the latent heat storage characteristics obtained from DSC results with those derived from analogous materials in the existing literature.

Material type	Melting Point (°C)	Melting Enthalpy (J/g)	Solidification Point (°C)	Solidification Enthalpy (J/g)	Reference
Hemp shives/LA-50%	37.80	79.30	33.80	70.40	Sawadogo et al. (2023)
Wood fiber/CA-SA-45%	24.06	79.90	22.09	79.30	Sarı et al. (2020)
Bamboo-fiber/PEG4000-50%	58.42	70.30	34.72	~60.00	Zheng et al. (2022)
Silk fiber/n-Octadecane-14.2%	22.95	37.58	4.630	34.98	Zhao et al. (2017)
Carbon fiber/CNT/Paraffin-70%	40.01	81.94	44.69	80.74	Cheng et al. (2023)
Yarn fiber/Paraffin	58.03	60.967	57.85	63.47	Miao et al. (2023)
Kevlar fiber/Paraffin-50%	61.30	72.50	57.80	66.30	Lv et al. (2023)
Hemp/PCM-45%	19.87	100.20	20.64	99.30	This study

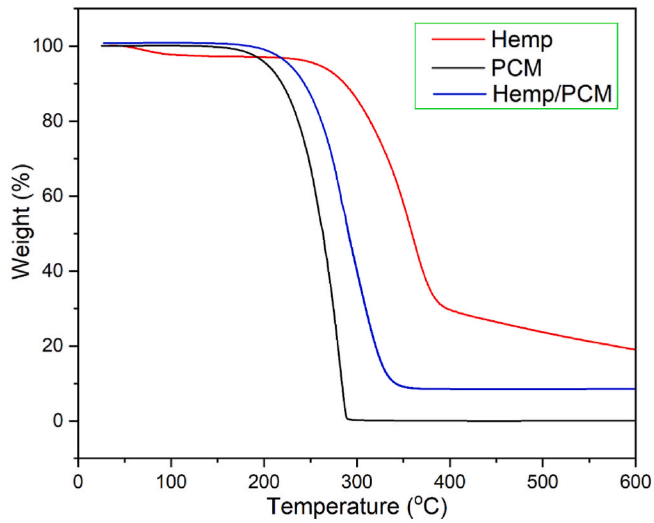


Fig. 9. TGA peaks of the samples.

investigate how different surfaces respond to solar radiation over an extended period. The surfaces tested included a room with hemp/PCM and reference rooms with hemp. Solar radiation represents the primary source of external heat for buildings. Understanding the variation in solar radiation over time allows us to quantify the energy a system receives, which is fundamental for assessing its thermal performance. In this regard, the global, direct, and diffuse solar radiation curves were obtained during the thermoregulation tests conducted for 96 h between 10 and 14.08.2023, as seen in Fig. 10. The solar radiation measurements commenced around the 16.5th hour on the first day. (August 10, 2023). It is evident from the tests conducted throughout the 2nd, 3rd, and 4th days that, while the weather was partly cloudy, there was a continuous cloud cover on the 2nd and 3rd days. In contrast, on the 4th day, stable weather prevailed with almost no cloud cover. This observation is supported by the significant fluctuations in the solar radiation curves on the 2nd and 3rd days, as depicted in Fig. 10, and the near absence of fluctuations on the 4th day. The dynamic solar radiation patterns on the 2nd and 3rd days correspond to the continuous cloud transitions, while the stability in solar radiation on the 4th day aligns with the minimal cloud cover. This information underscores the influence of cloud cover on solar radiation and highlights the distinct weather conditions experienced on each of the mentioned days. The highest solar radiation value among the test days, above 1000 W/m^2, was recorded around 3:15 p.m. on August 11, 2023. Similarly, on August 12, 2023, the measurement indicated 1000 W/m^2 around 14:00, and on August 13, 2023, it was measured at 857 W/m^2 around 13:00. These values and timings signify the peak solar radiation levels during the respective afternoons of the mentioned dates.

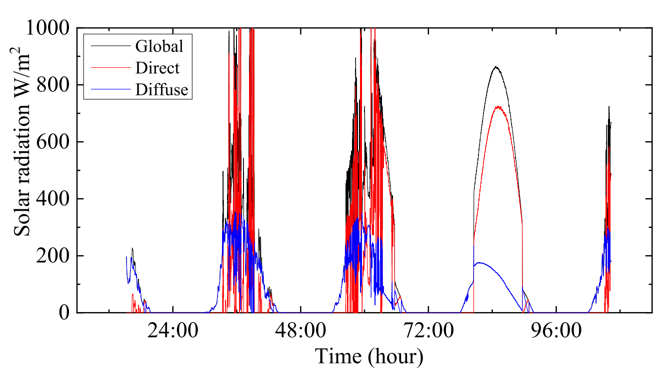


Fig. 10. The global, direct, and diffuse solar radiation curves obtained during the thermoregulation tests conducted for 96 h between 10 and 14.08.2023.

Monitoring temperature variations at different surfaces, including lower, upper, and near surfaces and the room center, allows for a comprehensive thermal comfort assessment within the space. This information is crucial for evaluating whether the occupants would experience conditions that meet comfort standards. Within this scope, Fig. 11 shows temperature variations on lower, upper, and near surfaces with room center for test room (Hemp/PCM) and reference rooms (Hemp) on 10–14.08.2023. Temperature variations provide insights into how materials, particularly the hemp/PCM, respond to changes in environmental conditions. Therefore, lower surface temperatures of the test sample being lower than the reference sample of hemp indicate the effectiveness of the hemp/PCM in maintaining lower temperatures, possibly through phase change processes as seen in Fig. 11a. On August 11th, 12th, and 13th, 2023, the ambient temperatures reached maximum values of 36.58 °C, 43.5 °C, and 53.9 °C, respectively. At an ambient temperature of 53.9 °C, the surface temperature within the test room was lower than the reference room. The implication here is that the response of the lower surface temperature in the test room is subject to the influence of fluctuations in the ambient temperature. The lower surface temperature of the test room surpasses that of the reference room at lower ambient temperatures, which may indicate the effectiveness of the test room's features in maintaining a comparatively higher temperature. Conversely, the lower surface temperature of the test room falling below that of the reference room at higher ambient temperatures might indicate the test room's efficiency in mitigating heat. A similar development has been observed in upper surface temperatures, as can be understood in Fig. 11b. However, when examining the times when temperature changes peaked, it is understood that due to the temperature fluctuations on August 11 and 12, 2023, the surface temperatures of rooms containing PCM remained more stable. This situation can be interpreted as PCM's ability to absorb energy. On August 13, 2023, when the ambient temperature reached its peak, it was observed that, based on the changes in near-surface and room-center temperatures (Fig. 11c and d, respectively), the temperature values in the room containing the reference sample remained higher compared to the one containing PCM. In summary, during high ambient temperatures throughout the day, temperatures in rooms containing PCM-containing materials are relatively lower compared to rooms without PCM. Conversely, during low ambient temperatures at night, temperatures in rooms containing PCM-containing materials have been somewhat higher than in rooms without PCM. This phenomenon can be attributed to the absorption of latent heat during daytime temperatures exceeding the melting point of PCM, leading to a more relaxed environment. Conversely, latent heat releases as temperatures drop at night, reaching the solidification temperature of PCM and resulting in a milder environment.

The temperature differences on boards displaying the surfaces and room center temperatures of reference and test samples can be examined to gain a more detailed understanding and draw conclusions regarding these temperature variations. This analysis explores the temperature variations on different surfaces and the central area of the rooms. In this context, the temperature difference curves obtained between the test (Hemp/PCM) board and reference (Hemp) board at the lower and upper surfaces (a) and near-surface and room center during the thermoregulation tests conducted for 96 h between 10 and 14.08.2023 were given in Fig. 12. First, at warmer ambient temperatures, temperature differences were obtained between the test (Hemp/PCM) board and reference (Hemp) board at the lower and upper surfaces are lower than that of near-surface and room center as seen in Fig. 12. In hotter ambient temperatures, the PCM within the test board might be activated, absorbing, and storing heat. This activation can lead to a more moderate temperature difference at the lower and upper surfaces than near-surface and room center. Moreover, the thermal inertia and PCM response time may delay its full activation, initially causing the lower and upper surfaces to exhibit more minor temperature differences. Near-surface and room center, being closer to the PCM, might experience

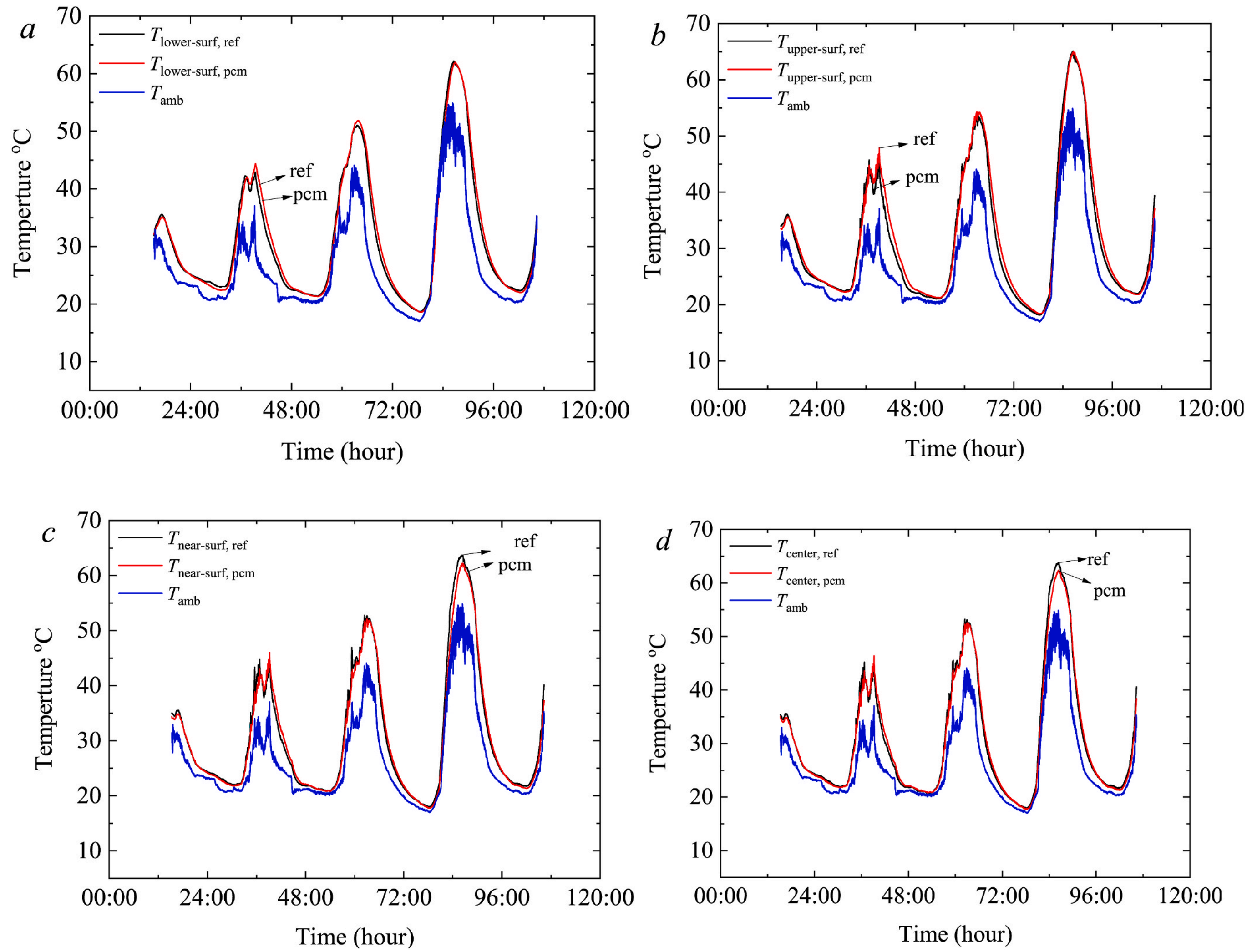


Fig. 11. The temperature change curves obtained for the test room (Hemp/PCM) and reference rooms (Hemp) at lower surface (a), upper surface (b), near-surface (c), and room center during the thermoregulation tests conducted for 96 h between 10 and 14.08.2023.

more immediate effects. Also, ventilation and airflow patterns within the test board could contribute to differences in temperature distribution. Near-surface and room-center areas might be more exposed to convective heat transfer, resulting in more considerable temperature differences. Conversely, temperature difference values at the near surface and room center are lower than those of other surfaces at lower ambient temperatures. The thermal inertia and response time of the PCM play a role. During lower temperatures, the PCM might take longer to release stored heat, causing a delay in temperature changes at the near surface and room center compared to other surfaces. Based on this information, on August 13, 2023, when more significant temperature differences were observed, at the times when the ambient temperature reached its maximum, the temperature difference values between the test board and reference board at the lower and upper surfaces were measured at approximately 3.12 °C (Fig. 12a). Meanwhile, during the same period, temperature differences between the test board and reference board, based on near-surface and room-center temperatures, were determined to be 6 °C for near-surface and 4.39 °C for the room center (Fig. 12b). On the night of August 11, 2023, when colder weather conditions prevailed, the temperature difference values for the lower and upper surfaces were calculated as −3 °C and −4 °C (Fig. 12a), respectively. Correspondingly, these values for near-surface and room center were approximately 2.54 °C (Fig. 12b). These findings highlight the dynamic response of the test and reference boards to varying ambient temperatures, with notable temperature differences at different surfaces and locations. The data suggests that the performance of the boards, particularly in terms of temperature differentials, is influenced by the specific thermal conditions during these periods.

3.7. Heat management ability of composite phase change materials

To assess the heat management ability of hemp/gypsum/PCM composites of Mix4, infrared thermal camera measurements were conducted to track surface temperature fluctuations during cooling processes for reference samples of hemp/gypsum and test samples of Mix4. Corresponding thermal images captured via the infrared camera are presented in Fig. 13. When comparing the temperature values obtained from the surfaces of both samples heated to a specific temperature simultaneously (at the 0^{th} minute of the cooling process), it is observed that the Mix4 composite has lower surface temperatures. In this case, since Mix4 contains PCM while R does not, the Mix4 sample has a higher heat storage capacity than the R sample. This higher heat storage capacity allows Mix4 composite to absorb more heat when heated, reducing its surface temperature. Therefore, the lower surface temperatures of the Mix4 sample can be attributed to the effective heat storage and distribution capabilities of the PCM it contains. This result confirms the thermoregulation tests conducted in previous sections.

During the cooling process, the PCM within Mix4 composites

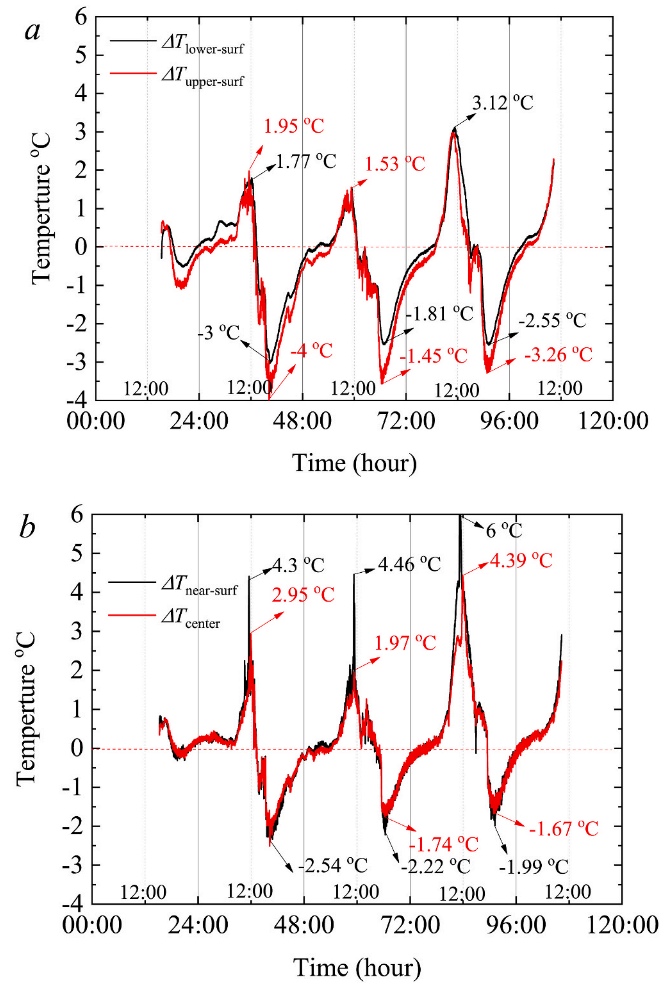


Fig. 12. The temperature difference curves obtained between the test (Hemp/PCM) board and reference (Hemp) board at the lower and upper surfaces (a) and near-surface and room center during the thermoregulation tests conducted for 96 h between 10 and 14.08.2023.

effectively reduces the required solidification time from the liquid phase. It transitions through heating to reach thermal equilibrium. Therefore, within the initial 80 min, the cooling rate of Mix4 containing PCM is faster than that of the R material and reverses until thermal equilibrium is reached in subsequent periods. The solidification temperature of the PCM used in this study was around 18 °C, as given in the section where the DSC results were provided. Approximately 80 min into the cooling process, this temperature range is reached. The PCM within the Mix4 composites has begun to undergo phase change during this time interval and started releasing heat by attempting to maintain the temperature within the phase change temperature range (Fig. 13). This phenomenon is related to intricate thermal mechanisms governed by PCM. PCM in the composite material undergoes a phase change near its solidification temperature of 18 °C, absorbing substantial latent heat during the transition from solid to liquid. This latent heat absorption allows the Mix4 material to draw heat more effectively from its surroundings during the cooling process, contributing to a lower temperature reduction rate. In other words, PCM transitions to a liquid phase as the temperature rises to around 40 °C during heating. In comparison, it crystallizes as the temperature drops below 18 °C, allowing stored heat to be released during the cooling process. In this context, beyond the 100th minute of the cooling process, the temperature falls below the solidification threshold of the PCM. Consequently, the cooling rate of Mix4 composites decelerates while temperatures remain higher than those of the R material. In this context, until the 80th min of cooling, the temperature values determined on the R materials' surface were higher than those of the Mix4s. In contrast, from the 80th minute to the end of the test, the surface temperatures of the Mix4 material were recorded to be higher. Finally, it should be emphasized that the slower cooling of PCM-containing gypsum structures when the ambient temperature falls below the melting point of PCM is important for maintaining a more stable indoor temperature than PCM-free gypsum materials.

4. Conclusions

This research delves into a novel exploration of hybrid composites, introducing in the literature the incorporation of hemp/PCM composites with 45% PCM content into the gypsum matrix at varying ratios. The study meticulously examines the physico-mechanical, microstructural, thermal, and solar thermoregulation properties of the resulting hybrid composites, capitalizing on the unique attributes of hemp shive particles, characterized by their high porosity and exceptional PCM absorption capacity. The derived critical insights are outlined in the following points:

1. The hemp shive shape-stabilized composites with 45 wt % LA-PCM exhibited leakage-proof characteristics. This can be attributed to their inherent porosity, emphasizing the role of porosity in achieving the observed leakage-proof property.
2. Microstructural examinations, based on SEM and EDS, revealed the distribution of 45 wt % LA-PCM within hemp shive particles, which inherently contain sufficient pores, occurs homogeneously.
3. While the melting temperature and enthalpy of pure PCM were determined to be 20.24 °C and 224.4 J/g, respectively, these values were found to be 19.87 °C and 100.2 J/g for Hemp/PCM45 shape-stabilized composites. Even after 750 melting-solidification cycles, almost no change was observed in the enthalpy values. This stability can be attributed to the material's robustness under repeated phase transition processes.
4. The hemp-PCM composite synthesis ensured that the thermal degradation temperature of hemp/PCM composites (about 250 °C) falls within the range of the degradation temperatures of hemp (after 300 °C) and PCM (about 200 °C).
5. The results of solar thermoregulation analysis demonstrated that temperature differences in the near surfaces of rooms containing hemp and hemp/PCM gypsum composites reached up to 6 °C. This highlights the impactful role of PCM in effectively modulating temperatures within these spaces.
6. During the cooling process, the ability to achieve a slower cooling rate after reaching the phase change temperature of PCM with the developed hemp/PCM/gypsum material represents a significant advantage in energy efficiency and effective heat management.
7. The successful incorporation and homogeneous distribution of 45 wt % LA-PCM within hemp shive particles reinforced gypsum boards further contribute to the stability and effectiveness of these composites in modulating temperatures, showcasing their viability for sustainable and energy-efficient building materials.

The fact that thermoregulation tests were conducted only on specific days during the summer season and that only one type of phase change material was used in the study may lead to restrictive conclusions. It is evident that in real-life conditions, different weather conditions occur throughout the year, which can impact these limitations. Therefore, using phase change materials with lower or higher melting points based on other weather conditions should be considered a subject for future research in gypsum-based composites. Additionally, the impact of using the mentioned PCM composites on cost-saving and CO_2 emission reduction values should be investigated to verify the results obtained better.

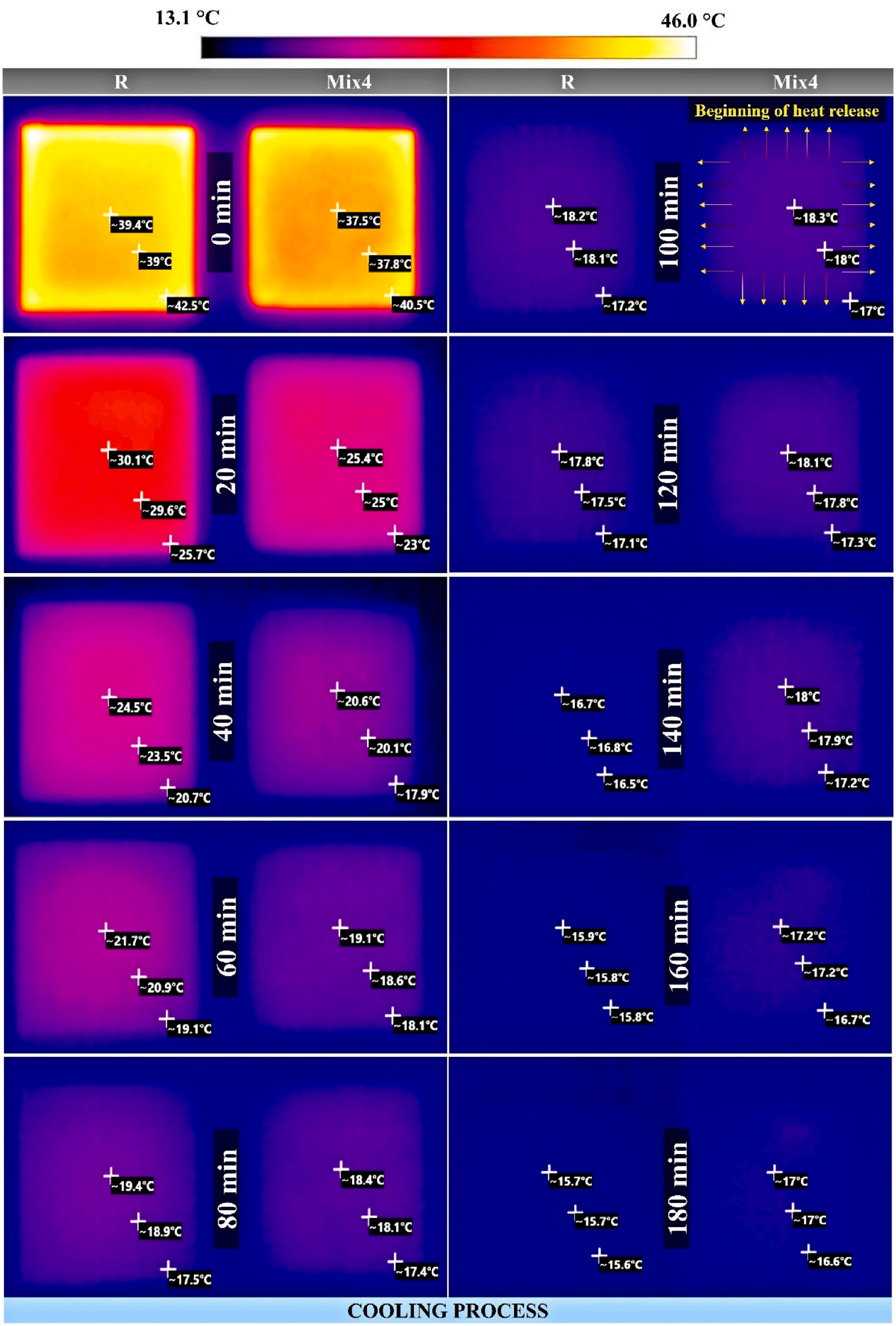


Fig. 13. Temperature profiles at various time intervals of R and Mix4 samples during the cooling process.

CRediT authorship contribution statement

Osman Gencel: Writing – review & editing, Writing – original draft, Visualization, Supervision, Methodology, Investigation, Formal analysis, Conceptualization. **Onur Güler:** Writing – review & editing, Writing – original draft, Visualization, Investigation. **Abid Ustaoğlu:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis. **Ertuğrul Erdoğmuş:** Investigation, Formal analysis. **Ahmet Sarı:** Writing – review & editing, Writing – original draft, Methodology, Formal analysis, Conceptualization. **Gökhan Hekimoğlu:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis. **Yalçın Boztoprak:** Methodology, Investigation, Formal analysis. **Serkan Subaşı:** Investigation, Formal analysis.

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.

Data availability

Data will be made available on request.

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Not applicable.

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