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Glass fiber reinforced gypsum composites with microencapsulated PCM as novel building thermal energy storage material

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ABSTRACT

A comprehensive study involving the fabrication and characterization of gypsum plasterboards combined with microencapsulated phase change material (mPCM) is introduced to evaluate their benefits regarding thermo-regulation management and energy saving performance in buildings. For this purpose, the produced new type of gypsum plasterboard was subjected to the detailed chemical, morphological, mechanical, physical and thermal tests. The gypsum plasterboard incorporated with mPCM (7.5 wt%) and reinforced by glass fiber has latent heat capacities as high as 16.7 and 16.6 J/g at onset melting and solidification temperature of 11.90 °C and 12.09 °C, respectively. TGA analyzes revealed high thermal stability of gypsum plasterboard up to about 140 °C. Outcomes exhibited that mPCM-gypsum plasterboard can substantially diminish cooling load of a building during the daytime even at the high room temperature and offer decline in heat necessity of a house during night-time. During peak room temperature hours, the mPCM-gypsum plasterboard achieved 3 °C lower temperatures than the reference room, and it provided a cooler room temperature for about 7 h during the daytime while a warmer temperature of 0.3 °C was achieved at the cold weather. As a result, the produced gypsum-based composites can be considered as an energy-saving and indoor temperature regulating material in buildings.

1. Introduction

Thermal Energy Storage (TES) is among the research topics that attract attention in the field of energy and it has significant effects on growing share of renewable energy in energy utilization and providing energy efficiency by reducing the heating and cooling loads of buildings. The TES methods hold the energy in three ways. First one is sensible heat stored in solid/liquid/gas media. Second one is latent heat stored as status variation between liquid–solid, between liquid–gas, and between solid–solid. The third one is the heat generated by thermochemical reaction [1,2]. Among these methods, latent heat storage is regarded as the best method for incorporation in civil engineering materials like mortar, concrete and gypsum derived products [3,4]. PCMs have ability to store and release the latent heat for the duration of the phase change cycle and thereby providing thermal comfort in buildings and reducing energy consumption [5].

For avoiding the leakage problem of PCM the duration of phase transition period from solid-to liquid phase and before incorporating them into the building materials, some techniques such as nano-encapsulation, microencapsulation, macroencapsulation, form-stabilization, direct incorporation and immersion has been developed [6–11]. The main disadvantage of direct incorporation and immersion methods is the PCM leakage issue encountered after many thermal cycles that may affect the physical, thermal, mechanical and durability properties. Even though PCM leakage is prevented in form-stabilization technique, thermal conductivity of form-stabilized PCMs is low which

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Table 1
Characteristics of α-gypsum used in this work.

Property	Value
Appearance	Powder, dust granular
Colour	Light pink
pH value	6.5
Soaking time	30 s
Beginning of set	10–16 min
End of set	20–24 min
Hardening time	40 min
Compressive strength (dry)	43 MPa
Bending strength (dry)	9 MPa
Surface hardness	87 shore D
Fire classification	A1 (TS EN 13279–1)
Particle size	0.5% on 100 μm, 6% 62 μm, 14% 42 μm

Table 2
Mixture proportions (wt%).

Code	Gypsum	Fiber	mPCM	Polymer additive	Water
Mix1	62.4	3.8	0	2.6	31.2
Mix2	61.4	3.8	1.5	2.6	30.7
Mix3	60.5	3.8	3.0	2.6	30.1
Mix4	59.3	3.8	4.5	2.6	29.8

Table 3
Experimental information.

Experiment	Standard No	Size (mm)	Time
Flow diameter	ASTM C 230 [31]	–	Fresh gypsum
Apparent porosity and unit weight	ASTM C948 [32]	50 × 50 × 50	28 days
Flexural strength	ASTM C 348 [33]	40 × 40 × 160	28 days
Thermal conductivity	ASTM D7984 [34]	300 × 300 × 50	28 days
Compressive strength	ASTM C 109 [35]	50 × 50 × 50	28 days
Thermoregulation performance	–	300 × 300 × 20	28 days

limits its applications. To compensate it, supplements having high thermal conductivity like carbon nanotube, nanofiber, silver nanowire, titanium dioxide etc. are employed [12–14]. Nano materials have, however, some disadvantages such as very high cost, dispersion problem, application problem etc. Besides, there are some parameters such as climate, inertness, density, non-toxic, the aimed TES, thermal stability, physical compatibility, applicability etc., which influence the choice of PCM. Zhu et al. [15] reported that the PCMs fit to civil engineering usages.

Gypsum as one of the oldest building interior material is used as binder since ancient times, 6000 years BCE [16]. It is still one of important building material due to low cost, fire resistance, thermal and sound insulations. Usage of gypsum based materials such as binder, plaster, block element, wall panel, ceiling panel, raised floor covering panel etc. in the buildings is increasing. Pore-makers such as expanded vermiculite, diatomite and expanded granules are used for the improvement of the thermal and sound isolation while different types of fibers are used to increase mechanical properties of gypsum based products [17]. The above mentioned advantages of gypsum makes it an ideal candidate for the creation of new type of TES green building materials to be used for thermal management applications in the building envelopes [18,19]. Within this framework, there are some studies focusing on application of PCMs to the gypsum. Sarı [20] produced composites made up of polyethylene glycol with gypsum and obtained 24.18 ± 1.0 J/g latent heat. The impregnation ratio of PEG was 18 wt% in the form stable Gypsum/PEG composite, and the melting temperature of this composite was 10.55 °C. Thermal performance experiment was also applied to reveal thermal management performance of the developed gypsum/PEG wallboard and it was found that the inner center temperature of the room made of this composite was lower averagely 2.08 °C compared to reference room during the heating process. Jeong et al. [19] impregnated n-octadecane and beeswax in liquid state into commercially available gypsum cement boards under vacuum condition. They observed that boards having n-octadecane and beeswax had 48.03 J/g and 34.08 J/g latent heat, respectively. Park et al. [21] investigated thermal and moisture performance of gypsum board containing expanded vermiculite and perlite, nano carbon as porous materials and n-octadecane as PCM. The composite PCM showed improved thermal and energy storage performance when compared with traditional gypsum board. Serrano et al. [22] produced gypsum board containing PCM as polymeric sheet and pellet forms. The authors found that gypsum board having 25% PCM was much thermal insolation. The boards containing PCM-sheet and pellets-PCM showed 41% and 33% higher latent heat effect than ordinary gypsum board. Karaipekli and Sarı [23] produced pumice/organic PCM/gypsum plasters. Latent heats of these products were observed as 55.40 J/g and 72.38 J/g respectively. Bake et al. [24] produced gypsum plaster board containing up to 15% by volume of microencapsulated PCM (50–300 μm size and 95 J/g latent heat). The plasterboard with 15% PCM remained at a temperature of approximately 1.5 °C higher than ordinary gypsum plasterboard during the cooling period. Borreguero et al. [25] used microencapsulated PCM up to 15% by weight to produce gypsum board. They observed 4.5 kWh of energy in each cycle for 1 m^3 room coated by gypsum containing 15% PCM. That value corresponds to energy which is used by 3 light bulbs with 60-W and saves 1.395 kg CO_2 emission. Yahay and Ahmad [26] immersed the gypsum boards into the eutectic mixes of lauric-stearic acid and evaluated their performance when incorporated in ceiling panels of buildings. They stated that the use of ceiling panels integrated with PCM can significantly increase energy efficiency through active cooling systems. Lachheb et al. [27] added significant heat storage properties to gypsum plaster by integrating it with a microencapsulated PCM, thereby reducing room temperature fluctuations. Singh and Bhat [28] compared temperature regulation performance of conventional gypsum board and a dual PCM-impregnated gypsum board. Poly-bags were used to fill the PCMs. According to the authors, noteworthy discount in indoor temperature swings was detected. Imani and Madhkhan [6] used PEG600 as PCM at 10 and 15 wt% to produce the gypsum sandwich-panels in various thicknesses of 7 and 10 cm. They tested the thermal performance of panels in different cities. They observed 5.7 °C reduction of the maximum daily temperature.

On the other hand, a deeply investigations on analyzing of influence of microencapsulated PCM (mPCM) in gypsum elements are needed because mPCMs is directly used in construction materials such as gypsum plaster or concrete without any leakage. However, there is still a clear gap in the developing and characterization of new type TES gypsum plasterboards containing mPCM gypsum composites have mechanical strength for service life of construction aims not to have problems. The PCMs no matter their form such as shape stabilized or encapsulated have a negative effect on mechanical strength of the composites. It is well known that fibers can be a good choice in point of mechanical performance increment. As reported by Medina and Barbero-Barrera [29], several types of fibers have been used for it in composites. There is highly limited information about the combined effect of mPCM and glass fiber on characteristics of gypsum composites. With this sense, the presented work was intended to produce a novel gypsum plasterboard which contained glass fibers mPCM having improved thermal and physico-mechanical properties for building TES practices. Before incorporation of mPCM, the structural durability of the gypsum was increased by adding glass fiber. The produced new type of plasterboard was subjected to the detailed chemical and morphological characterizations besides determining compressive strengths, flexural strengths, unit weight, porosity, heat storage characteristics, thermal



Fig. 1. Production of gypsum/mPCM composite samples.

conductivity and thermal stability properties. TES performance of the developed mPCM-gypsum plasterboard was also examined under the actual climatic conditions. As per authors' knowledge, such type of intensive work about development, characterization and estimation of TES performance of the mPCM-gypsum plasterboard under real weather conditions has been carried out for the first time.

2. Materials and methods

2.1. Gypsum

An α-gypsum, provided commercially by Ata Alçı company, was used in the production of composites. The characteristics of the used gypsum are presented in Table 1.

2.2. Polymer additive

Polymer additive was provided by Forton company. It modifies physical and mechanical characteristics of gypsum products. This polymer additive is especially produced for the application of alpha gypsum. Polymer additive consists of three additive components. Component one is the water based polymer emulsion (50% solids) which significantly improves physical characteristics of α-gypsum elements. Component 2 enhances the physical properties of the overall mix. Component 1, which is 50% solids polymer, crosslinks with component 2. Component 3 is ammonium chloride hardener used as pH modifier for obtaining good crosslinking of component 1 and component 2.

2.3. Glass fiber

The glass fiber (17 μm filament diameter and 12 mm length) having high alkali resistance was developed by Owens Corning Composite Materials, LLC. Its specific gravity, softening pint, modulus of elasticity and tensile strength are 2.68 g/cm^3, 860 °C, 72 GPa, and 1000–1700 MPa, respectively. AR-Glass Fiber has been preferred for its resistance in case of impurities that may be present in gypsum and composite components or exposure to alkalis such as Na, K. If damage occurs as a result of possible alkali exposure, significant damage is happened in the composite. Since the produced composites are plate elements, flexural strength expresses the critical strength. The critical strength of the produced composite provides the glass fiber used in the composite.

2.4. mPCM

The mPCM available in dry powder form (≥97% solids) was purchased from Microtek Laboratories Inc as a commercial product (Nextek

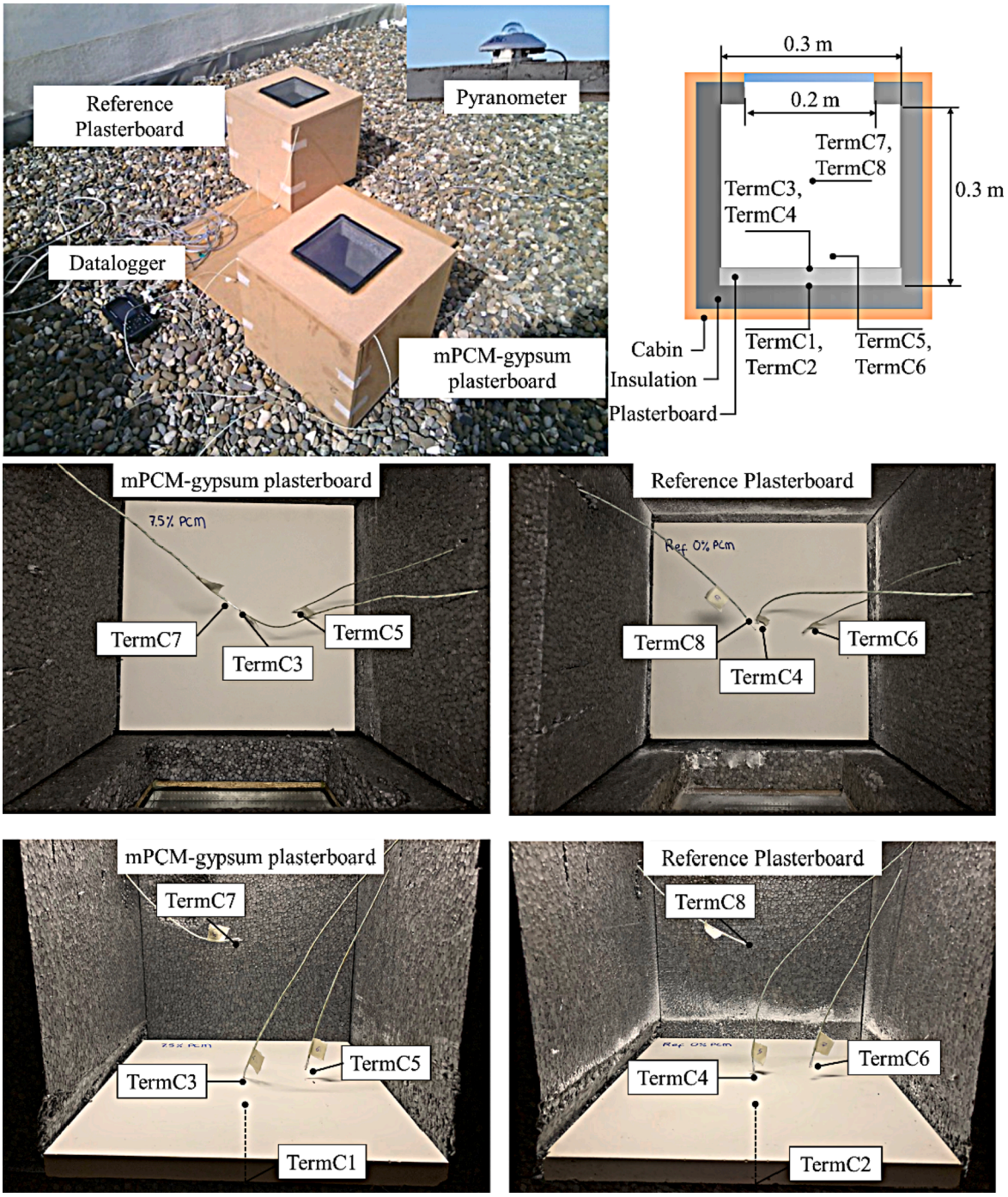


Fig. 2. Thermoregulation performance test setup image and thermocouples positions.

18D). It is consisted of melamine–formaldehyde as shell material and the branched-chain hydrocarbon mixtures as PCM core material.

2.5. Mixture proportions and production of mPCM/Gypsum plasterboards

Mixture quantities of gypsum composites are shown in Table 2. mPCM was used at 1.5%, 3% and 4.5% by weight of the total mixture. 1.5%, 3% and 4.5 ratios were preferred because they are the ratios that are thought to contribute to the energy storage performance and will not cause much loss in strength and workability parameters. Polymer additive was added at 3% of the total mixture weight of gypsum composite. Glass fiber was used as 3.8% of the total mixture weight of gypsum composite. The mixtures were prepared using a spiral blade mixer with a speed of 500 rpm. For mixing, firstly, polymer additive was mixed with water. Then α-gypsum was introduced into mix and mixed during 2 min. Thereafter, mPCMs were added and mixed for one minute. Finally, glass fibers were introduced into the mix and mixed for 2 min. Flow ability values of the produced paste were measured by using slump apparatus. After measuring the flow ability of the composites, it was placed in different moulds having sizes of 50 × 50 × 50 mm, 40 × 40 × 160 mm and 300 × 300 × 20 mm for tests shown in Table 3. Loading rates of 0.5 MPa/s and 0.05 MPa/s were applied in Compressive and Flexural Strength, respectively. After 24 h, gypsum composites were removed from the moulds and left in the laboratory for 28 days. The details of the production of gypsum composites are shown in Fig. 1. It is clearly shown

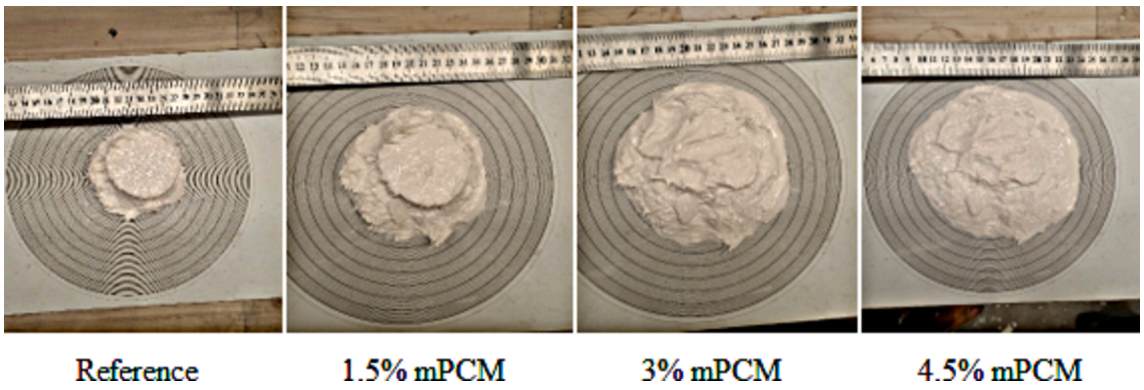


Fig. 3. Slump flow of the produced composite.

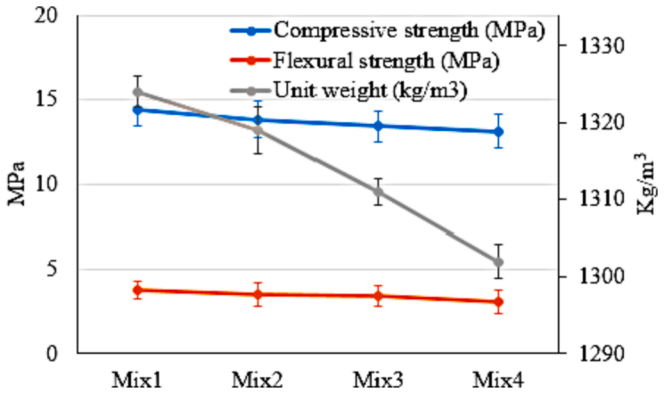


Fig. 4. Unit weight and strengths of mPCM/Gypsum composites.

that workability of composite increased by increment in percentage of mPCM. This may be attributed to the addition of capsule of mPCM which does not soak water. The wall shell of mPCM is generally is made up of polymer-based such as the polymethyl methacrylate. It is well-known that this kind of polymers does not absorb the water. So, it can affect the viscosity of the composites with mPCM that needs further studies. During the mixing process, it was observed that PCM leaked from broken mPCM which may play a plasticizer role and hence increasing the workability of the composites. Al-Absi et al. [30] reported that increment in mPCM results in a higher viscosity of mixture.

2.6. Characterization

The phase transition temperatures and the enthalpy values of mPCM and mPCM/Gypsum samples were measured using a Hitachi7020 model DSC. All samples were tested between 0 and 35 °C having the heating and cooling rates of 3 °C/min in nitrogen gas flow media of 30 ml/min. From a DSC thermogram, three distinctive temperatures regarding phase transition of solid–liquid and liquid–solid are identified; these are extrapolated onset-end temperatures and peak temperatures. The extrapolated onset temperature is usually taken into account as the phase transition temperature. However, in the present study, all characteristic temperatures were determined to analyze phase change behavior of the samples. The Archimedes method was used to determine apparent porosity according to ASTM C20 [36]. Thermal conductivity measurements of the gypsum composites were done according to ASTM D7984 [37].

Thermal stabilities of mPCM, Mix1 and Mix4 were evaluated using TGA (Perkin Elmer) technique at rising ratio of 20 °C/min in nitrogen gas meadia. FTIR spectra of mPCM, Mix1 and Mix4 were determined between 4000 and 500 cm^{-1} by a Shimadzu IRSpirit model Fourier transform infrared spectrometer.

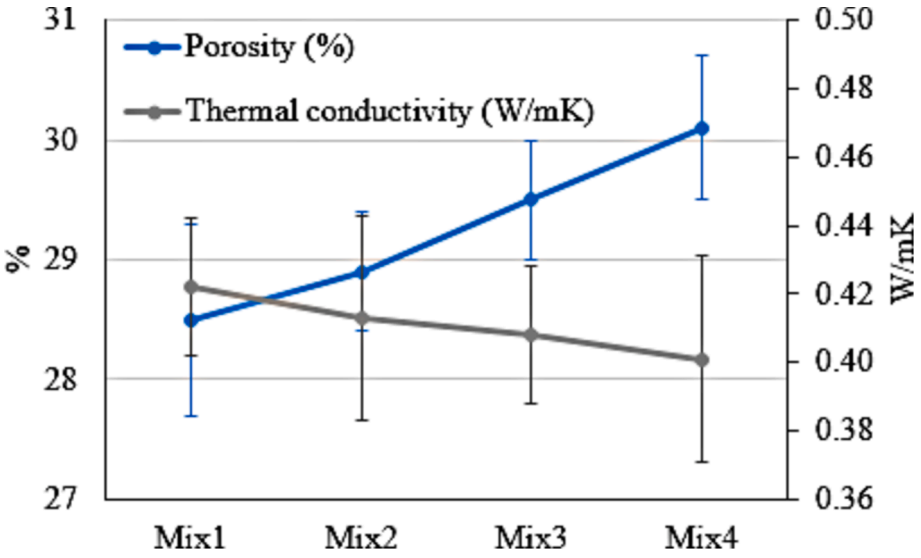


Fig. 5. Porosity and thermal conductivity of mPCM/Gypsum composites.

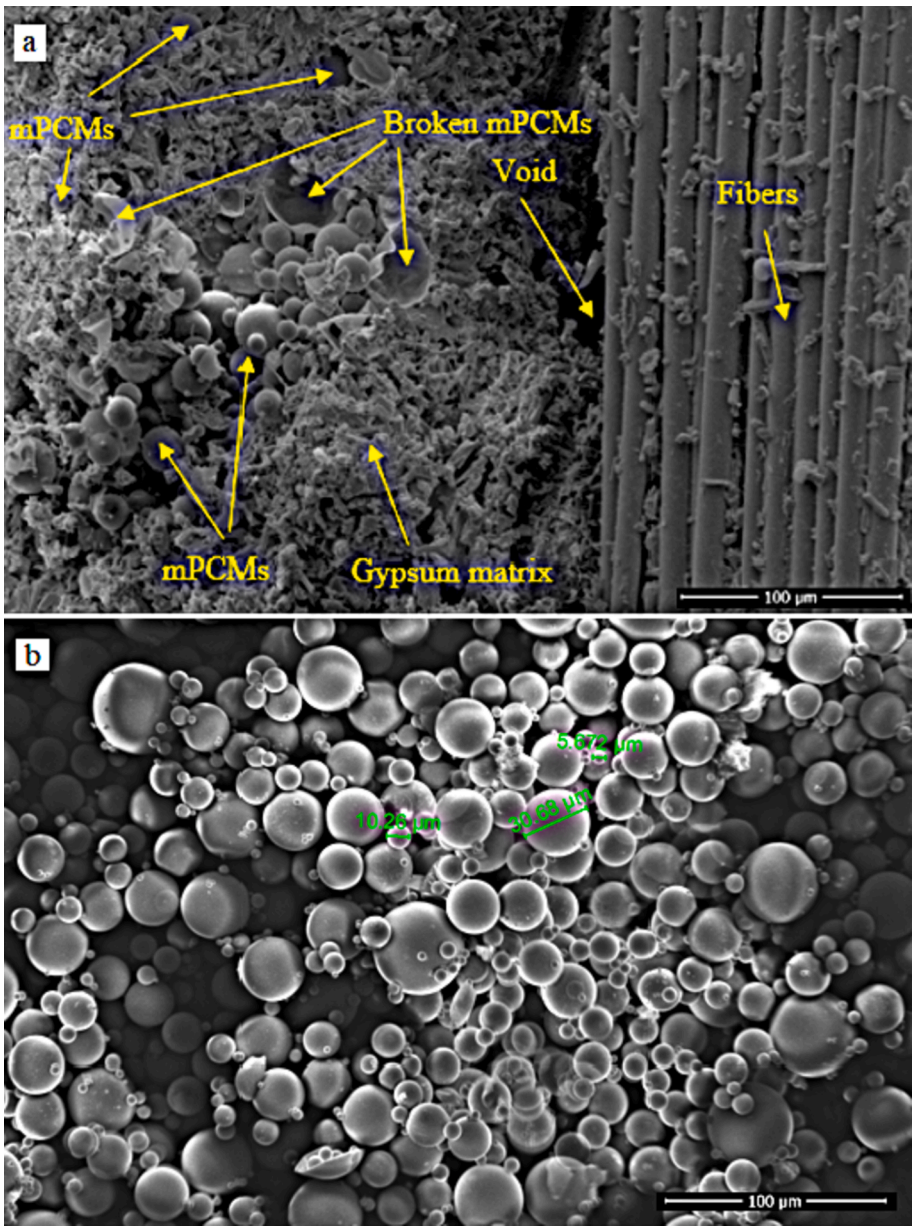


Fig. 6. (a) Microstructure of composite, (b) Microencapsulated PCM.

2.7. Thermoregulation performance measurements of mPCM/Gypsum plasterboard

Fig. 2 shows the experimental setup and the test rooms including mPCM-gypsum plasterboard and reference board. The experiment was carried out in (Bartın, Turkey) on a sunny-cloudy day on 02.10.2021. To determine the incident global solar radiation (H), which is totality of direct (H_b) and diffuse (H_d) solar radiation on the horizontal surface, a pyranometer was used. The direct and diffuse solar radiation were obtained using the clearness index of the weather ($K_T = H/H_0$). It can be described as global solar radiation rate to extraterrestrial radiation [38]. When K_T is between 0.3 and 0.85, the direct solar radiation (I_b) becomes equal to $-520 + 1800K_T$, otherwise, it is taken as zero [39]. The zero value indicates cloudy weather without direct solar radiation from the sun. The direct solar radiation on the horizontal surface was the cosine of I_b. Two identical cabins were used for the experiment. One was used as a reference cabin including the reference plasterboard without PCM while the other one test cabin including mPCM-gypsum plasterboard.

The plasterboards were produced with dimensions of 0.3 × 0.3 × 0.02 m. Double glazed windows having sizes of 0.2 × 0.2 m were placed on the cabins. The inner surfaces of the cabins were coated by the expanded polystyrene foams (EPS) with 0.05 m thickness. The cabins were made of medium-density fiberboard (MDF) with 0.02 m. Window placed surfaces were directed to southward. A thermocouple (TermC9) was utilized for recording ambient temperature. TermC1 and TermC2 were placed between the plasterboards and EPS while TermC3 and TermC4 were positioned on the center-upper surface of plasterboards in test room with PCM impregnated plasterboard and reference room, respectively. TermC5 and TermC6 were placed about 0.01 m far from surface of the plasterboards while TermC7 and TermC8 were placed on the room centers.

3. Results and discussion

3.1. Physico-mechanical properties and microstructure investigation

The schematic of flow ability of different composites is shown in Fig. 3. Slump flow values were 8.5 cm for Mix1, 11.5 cm for Mix2, 13.5

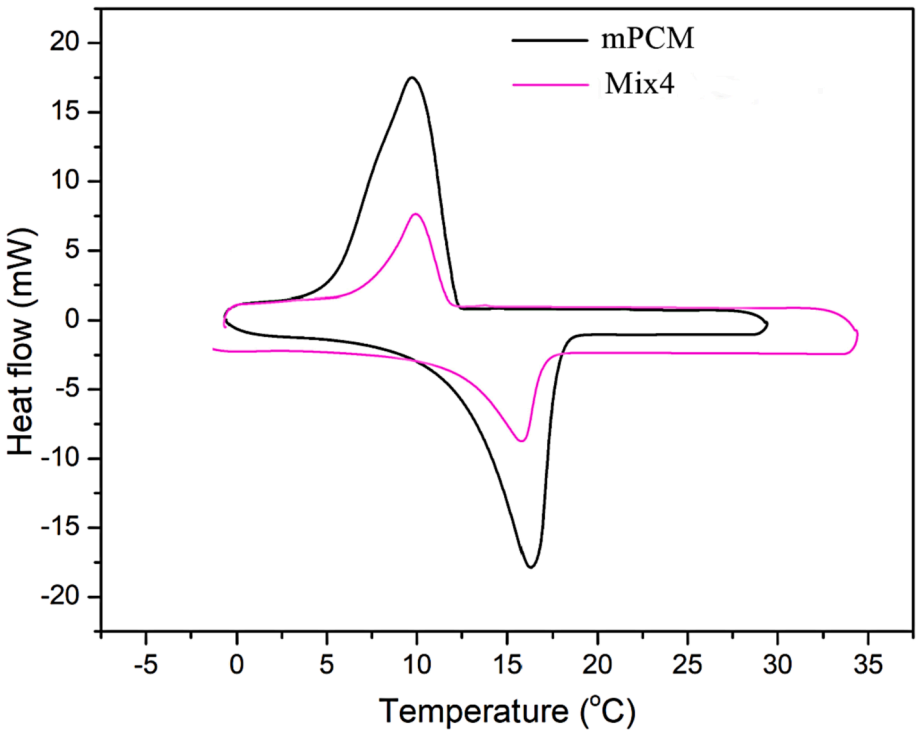


Fig. 7. The DSC thermograms of mPCM and mPCM/Gypsum mixture called as Mix4.

Table 4
Characteristic temperatures and latent heats for mPCM and Mix4.

Material	Heating mode				Cooling mode			
	T_{onset} (°C)	T_{peak} (°C)	T_{end} (°C)	Latent heat (J/g)	T_{onset} (°C)	T_{peak} (°C)	T_{end} (°C)	Latent heat (J/g)
mPCM	11.90	16.33	18.71	202	12.09	9.79	5.23	203
Mix4	12.32	15.86	17.31	16.7	11.69	9.97	6.73	16.6

cm for Mix3 and 15.5 cm for Mix4. It is clearly shown that workability of composite rised by increment in percentage of mPCM. Thus, the structure of mPCM has a significant role in improving the workability of the composites. The capsule is polymer-based and it does not have capability to absorb water. Hence, the viscosity decreases and plasticity increases.

The results of dry unit weight, flexural strength and compressive strength of gypsum composites were exhibited in Fig. 4. For all mixes, the unit weight values ranged from 1302 to 1324 kg/m^3. This shows that the usage of mPCM slightly decreased the bulk density. Srinivasaraonaik et al. [40] observed a drastic 30% reduction in density when 5% mPCM was used. Bake et al. [24] observed reduction in bulk density of PCM-gypsum plasterboard with the increment in mPCM quantity because of lower density of mPCM. Bake et al. [24] obtained 1549 kg/m^3 bulk density with 15% mPCM. The reason of the reduction of density is correlated with the porosity. Borreguero et al. [25] obtained a variation of bulk density of gypsum composites containing mPCM between 1104 and 1285 kg/m^3. In their findings, 15% mPCM usage resulted in 14.1% reduction of bulk density. Toppi et al. [41] obtained a variation of bulk density in variety of 708–1107 kg/m^3. They observed that increment of mPCM content in gypsum composite significantly decreased the bulk density. The values of bulk density obtained in this research are considered as lightweight since they are below 1800 kg/m^3. Besides, the obtained values are above 600 kg/m^3 according to the requirement of EN 13279-2 [42].

Compressive strength of mixes was in the range of 13.1–14.4 MPa. It is clear that the incorporation of mPCM into the gypsum resulted in a slight reduction in the compressive strength of composite. Reason of reduction in strength is related to the increase in the porosity of gypsum composite mixes as would be presented in the next paragraph. The reason of reduction is related with the increment of porosity. The decrease in strength is also related to the breaking of mPCM during compressive strength test. Also, existence of fibers can be another reason because the fibers can cause both reduction and increment in compressive strength by depending on factor such as the type of fiber, percentage, orientation, distribution, mixture design etc. The results obtained in this research regarding the decrease in compressive strength with increment in mPCM quantity addition into gypsum board are in line with available literature [24,25,40]. For example, Bake et al. [24] obtained 5.36 MPa compressive strength at 5% mPCM and 4.34 MPa compressive strength at 15% mPCM usage in their gypsum composites. Borreguero et al. [25] obtained ~9 MPa compressive strength at 7.5% mPCM and ~11 MPa compressive strength at 15% mPCM usage. The lowest value of compressive strength in this study is 13.1 MPa and is above the values of compressive strength for gypsum boards reported in literature as well as 2 MPa, which is the minimum value of compressive strength required by EN 13279-2 [42]. The values of flexural strength varied from 3.1 MPa to 3.8 MPa. This shows that the effect of mPCM on flexural strength is not as strong as on compressive strength because of bridging influence of glass fibers.

Results of porosity and thermal conductivity were given in Fig. 5. The value of porosity ranged from 28.5% to 30.1%. This shows that the porosity raised with increment in mPCM quantity addition into gypsum composite. In assessment of control sample, increment in porosity was

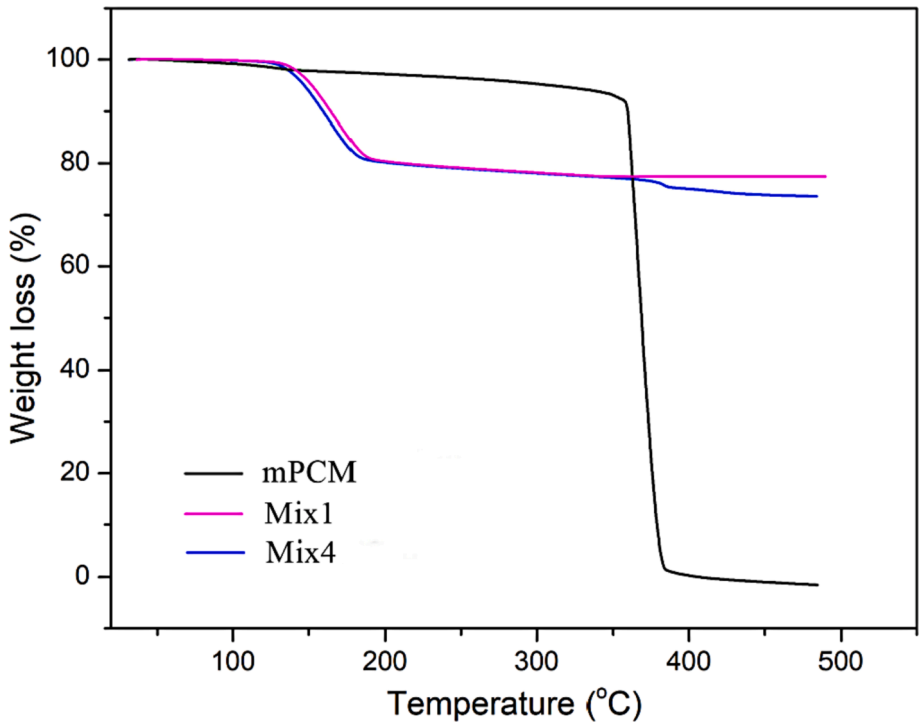


Fig. 8. TGA curve of micro-PCM, Mix1 and Mix4.

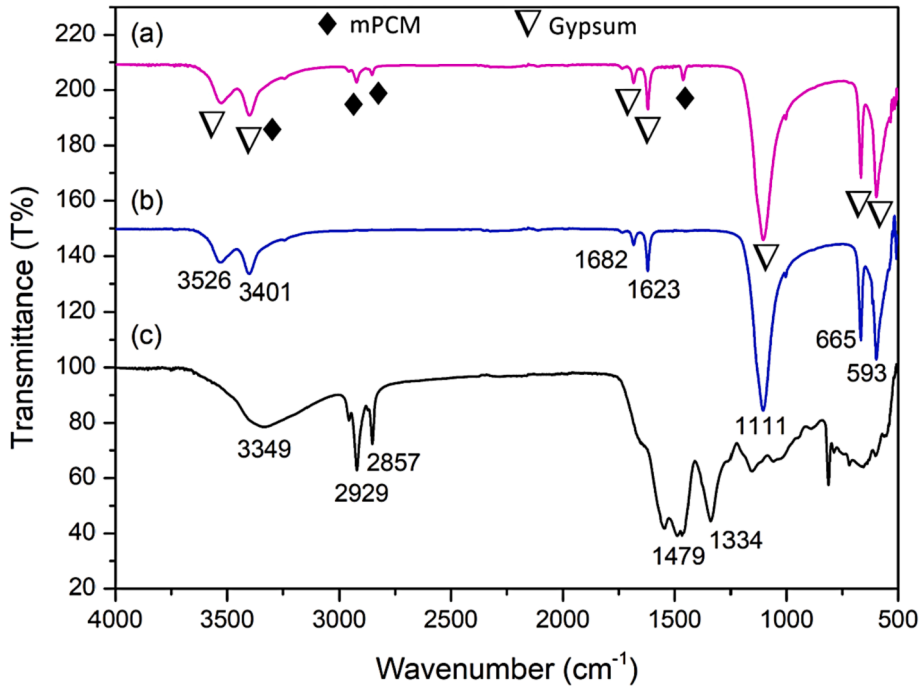


Fig. 9. FTIR spectra of mPCM (a) Mix1 (b) and Mix4 (c).

found to be 1.4%, 3.5% and 5.6% with the addition of 1.5%, 3% and 4.5% mPCM in gypsum composite. This behavior is usually observed for both cement based and gypsum based composites containing mPCM. Toppi et al. [41] reported that mPCM usage increased gypsum porosity due to the reason that its structure is different from gypsum matrix. The increase in porosity is desirable in point of reducing the coefficient of thermal conductivity which is required for thermal isolation.

The thermal conductivity values obtained in this research varied from 0.401 W/mK to 0.422 W/mK. In comparison to control specimen (without mPCM), the coefficient of thermal conductivity decreased by 2.1%, 3.3% and 5% with the addition of 1.5%, 3% and 4.5% mPCM respectively. Bake et al. [24] observed up to 13% decrease in the rate of thermal conductivity via mPCM incorporation. Lower thermal conductivity value of mPCM donates to decline of thermal conductivity, too. Borreguero et al. [25] obtained value of 0.482 W/mK at 7.5% mPCM and 0.484 W/mK at 15 mPCM usage in gypsum composites. Toppi et al. [41] reported a variation of thermal conductivity in variety of 0.193–0.396 W/mK. They detected that thermal conductivity of gypsum composites

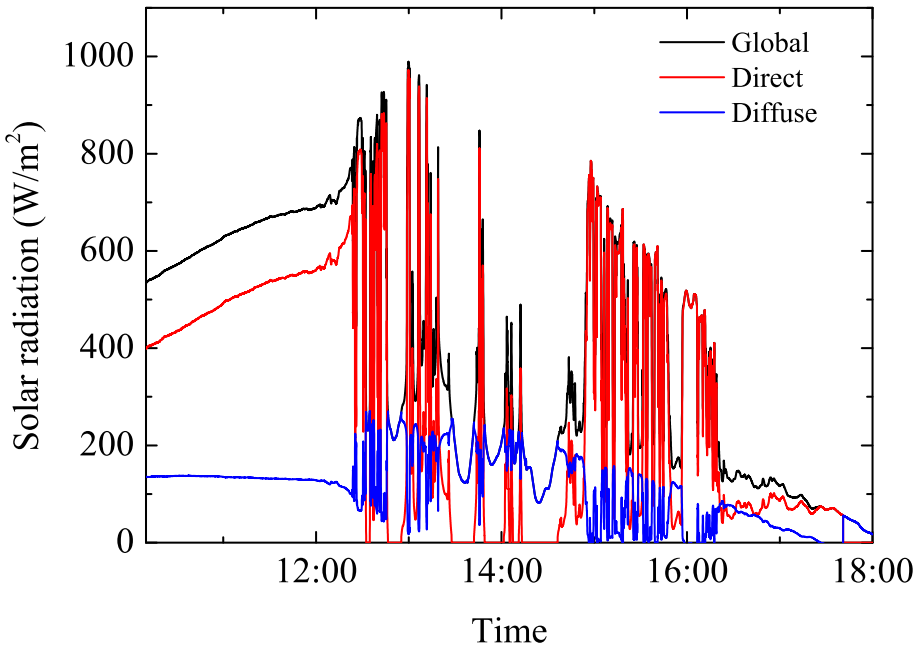


Fig. 10. Radiation information for 02.10.2021 sunny-cloudy sky.

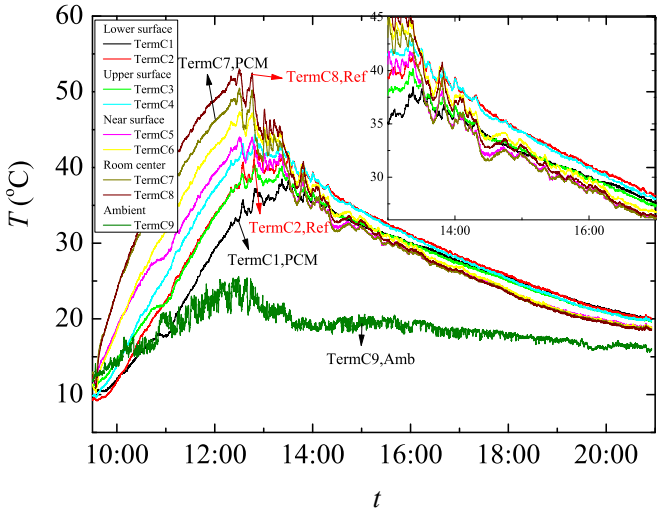


Fig. 11. Temperature changes for 02.10.2021, sunny-cloudy sky.

drops via increment in mPCM quantity in gypsum composite. Borreguero et al. [24] reported in their study that the thermal conductivity of gypsum composites can vary from 0.19 to 0.76 W/mK. It is highly dependent on the factors such as the used materials, mixture proportions etc.

Microstructure of glass fiber reinforced mPCM/gypsum composite and microencapsulated PCM is presented in Fig. 6. The mPCM particles are round in shape and their mean particle size range from 5 to 30 μm. Some voids in addition to voids due to the broken mPCMs are detected in matrix of gypsum composite which in turn is responsible for increasing the porosity of the system. Weak bond in some parts between glass fibers and the matrix can be observed which might have negatively influenced the mechanical properties of gypsum composite.

The leakage of PCM from broken mPCM might have contributed to a weaker bond between the components of the composite. The agglomeration of mPCMs may also be one of the reasons to influence the mechanical properties of gypsum composite. The observations obtained in this investigation are in line with those stated in literature [25,24,43].

3.2. Phase change behaviors of mPCM and mPCM/Gypsum composites

The melting/solidification DSC thermograms of pure mPCM and Mix4 as the representative of other mixtures are shown in Fig. 7 while the detailed results of characteristic temperatures and latent heats were presented in Table 4. The onset temperatures indicate the beginning of the phase change (melting or solidification) as the peak temperatures indicate the completion of the relevant phase change. In DSC thermograms, the negative heat flow characterizes heating stage while positive heat flow characterizes cooling stage. As can be seen from the DSC thermogram of the mPCM, there is one temperature peak each during heating and cooling cycles representing the solid–liquid and liquid–solid phase change. On the other side, Mix4 showed similar temperature peaks as pure mPCM, but showed lower latent heat values. The onset melting and solidification temperature of mPCM was found to be 11.90 and 12.09 °C, whereas they were measured as 12.32 and 11.69 °C for the Mix4. Considering the phase change temperatures of the prepared Mix4, it can be evaluated as a thermo-regulator material for buildings, especially for the air conditioning of building envelopes, depending on climatic conditions. The latent heat of mPCM for the period of heating and cooling mode were 202 and 203 J/g, whereas latent heat of Mix4 was calculated as 16.7 J/g and 16.6 J/g during heating and cooling mode. Due to the relatively high latent heat values obtained for Mix4 during heating and cooling mode, Mix4 has a high potential to be used directly in buildings as a gypsum-based thermo-regulator construction material.

3.3. TGA results of mPCM and mPCM/Gypsum composites

Thermal stability is an essential consideration to determine appropriateness of PCM or PCM-included material for building application. Hence, the PCM or PCM-included material should have highly resistive against thermal decomposition within its servicing temperature. In this regard, TGA test for pure mPCM, Mix1 and Mix4 were conducted, and their TGA curves are presented in Fig. 8. The mPCM thermally decomposed in only one step in the temperature range of 343–385 °C. For Mix1, a one-step mass loss of 20.21% can be noticed between 136 °C and 190 °C. Moreover, TGA curve of Mix4 is quite similar to that of Mix1, except for the portion above 370 °C. This is because the mPCM within the Mix4 starts to thermally degrade after 370 °C and hence mass loss occurs.

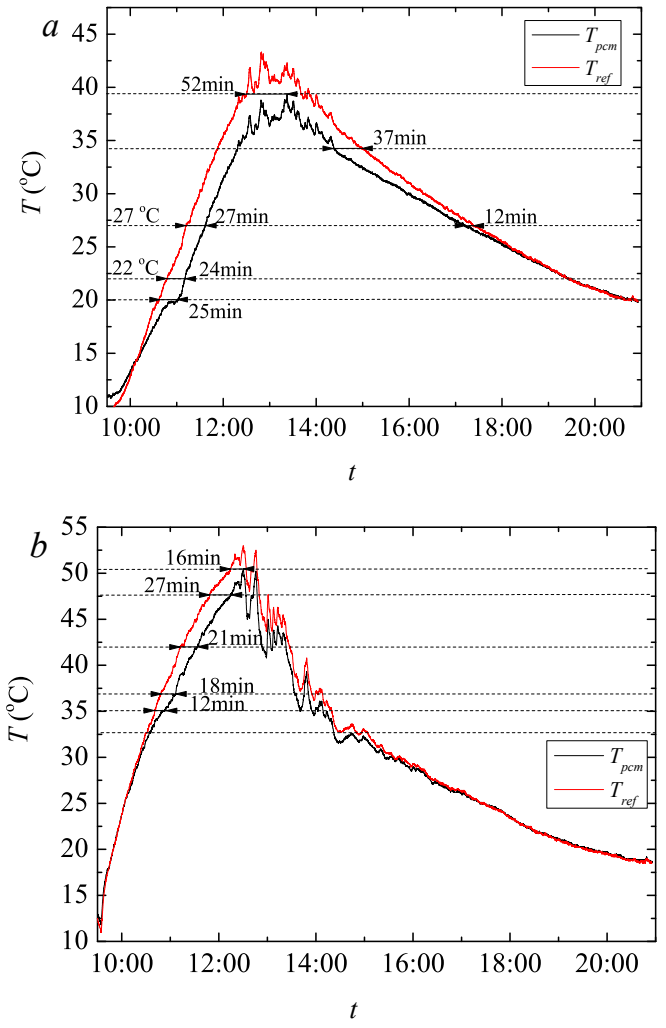


Fig. 12. Time lags between the mPCM-gypsum and reference cases for the plasterboards mean temperatures (*a*) and room center temperatures (*b*) for 02.10.2021, sunny-cloudy sky.

3.4. FTIR results of mPCM and micro-PCM/Gypsum composites

The chemical structures of pure mPCM, Mix1 and Mix4 were described by FTIR. Spectral outcomes were given in Fig. 9. In the FTIR spectra of pure mPCM, the peaks observed at 2929 and 2857 cm^{-1} represent the stretching vibrations band of C–H groups while large peak at 3349 cm^{-1} is typical stretching vibrations of OH groups. Peaks at 1334 and 1479 cm^{-1} are associated with C–H bending vibration bands. In FTIR spectrum of Mix1, as expected, typical peaks of gypsum are seen. A pair of peaks observed at 3526 and 3401 cm^{-1} is affected via asymmetric stretching of water remaining in the gypsum while the peak at 1111 cm^{-1} is because of symmetric stretching vibrations of O–H group in the water. The peaks observed at 1682 and 1625 cm^{-1} denote bending vibrations of S-O group in $CaSO_4$ while the peaks at 665 and 593 cm^{-1} are connected for asymmetric bending vibrations of SO_4. In the FTIR spectrum of Mix4, main characteristic peaks related mPCM and gypsum can be observed. No new peak was observed in Mix 4 depicting that mPCM within the gypsum mix maintain its chemical structure. In other words, the FTIR spectrum of Mix4 showed similar peaks observed in pure mPCM and Mix1, and the related peaks of these components are marked with symbols in Fig. 9.

3.5. Thermoregulation performance of the mPCM-gypsum plasterboard

The thermal performance outdoor testing was done on 02.10.2021. For this purpose, the rooms made with mPCM-gypsum plasterboard and reference board were placed at rooftop of building (41.6°N, 32.4°E). Fig. 10 illustrates the global, direct, and diffuse solar radiation on a sunny-cloudy day. The day was sunny until 12:20 h. At this time, the direct and diffuse solar radiation values were around 650 W/m^2 and 120 W/m^2, respectively. Then, clouds appeared, and the solar radiation values fluctuated between 12:23–16:20. Then, clouds started to appear the values of solar radiation started to fluctuate. The direct solar radiation almost totally disappeared due to the appearance of heavy clouds between 12:46–12:58, 13:20–13:44, 13:55–14:55 h. The solar radiation reached the peak value around 13:00 h and then gradually decreased until 16:20 hrs. After that, the shadow appeared on the test rooms. According to the ambient temperature profile presented in Fig. 11 (TermC9), it increased and reached the peak value of 25.4 °C at 12:45 h. Then, the ambient temperature started to decrease in accordance with the incident solar radiation profile until 13:40 h and became 19.2 °C. After that, the ambient temperature decreased only by 3 °C until 21:00 hrs.

Temperature profiles in rooms made with mPCM-gypsum plasterboard and reference board are presented in Fig. 11. TermC1 and TermC2 indicate the lower surface temperature while TermC3 and TermC4 indicate the upper surface temperatures of mPCM-gypsum plasterboard and reference board, respectively. TermC5 and TermC6 represent the near-surface temperatures while TermC7 and TermC8 show the room temperatures at center of room made having mPCM-gypsum plasterboard and reference plasterboard, respectively. The temperature difference between the PCM impregnated plaster board and reference plaster board gradually increased until 12:30 h and then the temperature gap decreased. After 18:00, temperatures of the mPCM-gypsum plasterboard and reference board were almost equal in the upper and lower surfaces. The upper and lower surface temperatures were 39.6 °C and 37.7 °C at 13:21, respectively. These became 19.7–19.9 °C at 20:59. It is quite above the melting temperature of about 12 °C and it continued for quite a long time. The duration above the melting temperature and the point the lower and upper surfaces reached are sufficient for a melting fraction of 100% for a quite low amount of mPCM ratio of 4.5%.

The surface temperature of the proposed mPCM-gypsum plasterboard reached above 27 °C after 11:45 while that of the reference wallboard reached this temperature at 11:22 (Fig. 12a). It means that the mPCM wallboard can keep within the comfort temperature about 27 min more. It took about 52 min more to reach the maximum surface temperature of 39 °C for the case of mPCM-gypsum plasterboard. It took around 12–27 min more to keep room center cooler (Fig. 12b). To clearly demonstrate the temperature difference, the inner (lower) surface, the outer (upper) surface, near-surface, and the temperature recorded at center of reference room without mPCM-gypsum plasterboard was extracted from the those of test room with mPCM-gypsum plasterboard.

Fig. 13 displays the temperature difference between the test and reference rooms. A negative value indicates that temperature values in the test room having mPCM-gypsum plasterboard are less than those of the reference room, while positive values indicate warmer temperatures in the test room in proportion to the reference room. Temperatures in the test room with PCM were warmer than those in the reference room without PCM until 10:10, while the ambient temperature was under 16.5 °C. The lower surface temperatures were around 12.5 °C for both cases at 10:10. Due to the heat transfer and storage processes of mPCM-gypsum plasterboard at the melting temperature, the temperature differences started to increase. The temperatures of the test room with mPCM-gypsum plasterboard increased more slowly until 11:05. The temperature difference variation was not that significant between 11:05–13:00. During peak room temperature hours, the mPCM-gypsum plasterboard achieved 4.6 °C, 3.7 °C, 3.56 °C and 3 °C lower

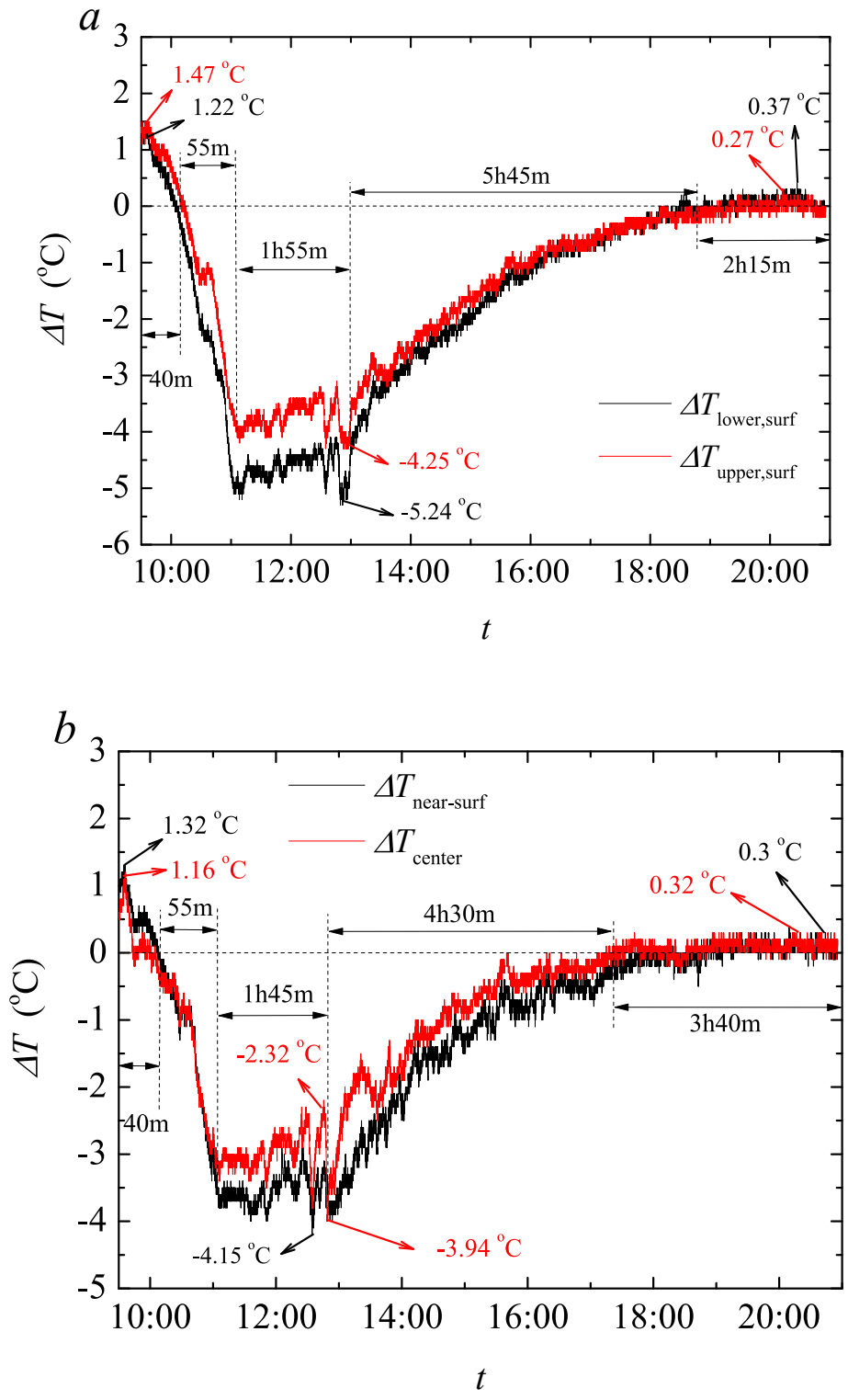


Fig. 13. Temperature differences between mPCM-gypsum plasterboard and reference plasterboard for undersurface and upper surfaces (*a*) and temperature differences between near surfaces and room centers of the test room with mPCM-gypsum plasterboard and reference room (*b*) on 02.10.2021 sunny-cloudy sky.

temperatures than the reference room for lower, upper, near surfaces and room centers, respectively. After 13:00, the temperature difference gradually diminished. The mPCM-gypsum plasterboard provided a cooler surface temperature for about 8 h 35 min and a cooler room temperature for 7 h 10 m. The temperatures of the room centers were similar at 17:20. After 17:20, the temperature of the room with mPCM wallboard decreased below 25 °C and become around 18 °C at 21:00. Then, the test room with PCM becomes about 0.3 °C warmer than the reference room for 3 h 40 min. The most significant effect of the discharge process of the mPCM-gypsum was observed before 10:10 due to cold weather conditions. Before 10:10, it stayed about 1.16 °C warmer for 40 min when the room temperature decreased below 22 °C. Although the phase change temperature was found to be around 12.32 and 11.69 °C, mPCM-gypsum plasterboard provided a significant advantage even at the higher temperatures than melting temperature of PCM. Room center temperature was about 3.9 °C cooler than that of the

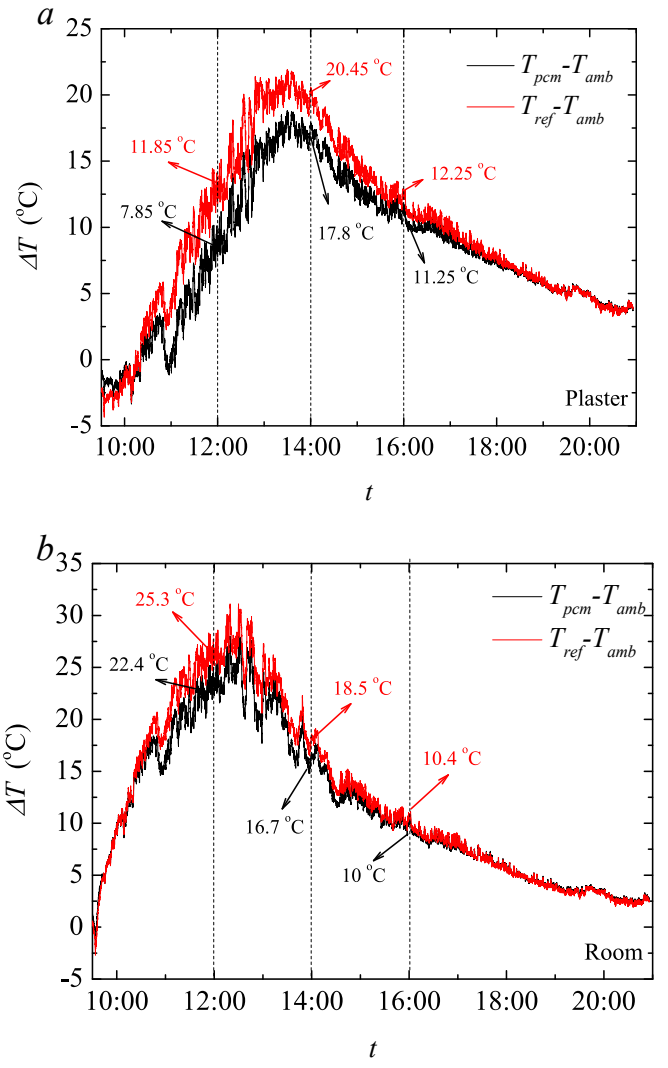


Fig. 14. Temperature differences between plasterboards and ambient (*a*) and between room center and ambient on 02.10.2021 sunny-cloudy sky.

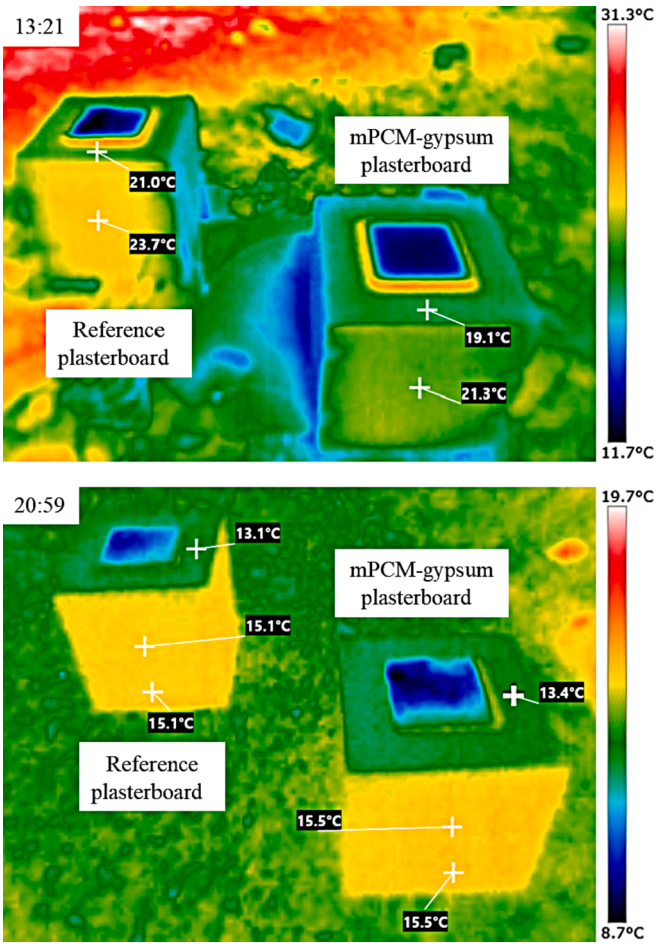


Fig. 15. Thermal camera image of the test room mPCM-gypsum plasterboard and reference room.

reference room at the maximum case. It can significantly decrease the cooling load of the building. Consequently, the proposed system can significantly reduce the cooling load while it provided a warmer temperature in cold weather. Fig. 14 illustrates the temperature differences between plasterboard and ambient and between room center and ambient. The plasterboard temperature indicates the average value of the upper and lower surface temperature of the plasterboard. The difference becomes more apparent in the case of plasterboard. The peak differences between the plasterboard and ambient were around 13:30. It was around 12:20 for the case of room temperatures. It has arisen from the late warming of plasters. The differences gradually decreased while the solar flux and ambient temperatures decreased. The advantage of mPCM-gypsum plasterboard during the day and cold weather was also demonstrated by using the thermal image technique. The results of the thermal image taken at 13:21 and 20:50 h for the reference room and the room made with mPCM-gypsum plasterboard are presented in Fig. 15. Although there is an insulation layer covering the inner surface of the rooms, the room with mPCM-gypsum plasterboard showed a cooler outer surface temperature at the peak solar flux hours while a warmer temperature at 20:50 h when the weather was cold.

4. Conclusion

In the work, usage of mPCM in glass fiber reinforced gypsum composites was investigated in the context of manufacturing innovative building material for thermal energy storage purposes. The key findings are as follows:

- Slump flow measurement indicated that increasing mPCM increases from 8.5 to 15.5 cm. It means about 82.4% increment.
- Unit weight exhibits 1.7% reduction when 4.5% mPCM usage.
- The usage of mPCM increase porosity of gypsum composites up to 30.1%.
- The breakage of mPCMs was observed in the mixture that may increase the porosity.
- Compressive strength was observed as 13.1–14.4 MPa. When the mPCM was used at 4.5%, compressive strength decreased 9%. The same behaviour was observed for flexural strength of gypsum composites.
- The mPCM existence weakens the bond between the gypsum matrix and the glass fiber. That negatively affects the bridging behavior of fibers.
- Thermal conductivity value of gypsum composites was observed as 0.401–0.422 W/mK. Thermal conductivity value decreased 5% when 4.5% mPCM was used. The lowest thermal conductivity value was 0.401 W/mK.
- The mPCM presented admirable latent heat values of 202 and 203 J/g at an onset melting and solidification temperature of 11.90 and 12.09 °C, respectively.
- The latent heat capacities of gypsum composites containing mPCM were as high as 16.7 and 16.6 J/g during heating and cooling mode.

- FTIR analysis exhibited confirming of the chemical structural stability of mPCM after combining with the gypsum composite and the physical compatibility of mPCM.
- The mPCM-gypsum plasterboard provided a cooler room temperature for about 7 h during the daytime between 10:10–17:20.
- The temperature measured at center of test room having mPCM-gypsum plasterboard was about 3.9 °C.
- Outcomes showed that the proposed mPCM-gypsum plasterboard can substantially reduce the cooling load of a building during the daytime and offer a drop in total heat demand of a residential during cold weather conditions. A significant advantage can be attained in stabilizing the room temperature along with energy and carbon emission savings.
- As a future plan, it is recommended that the viscosity and pilot-scale thermoregulation performance of the glass fiber reinforced gypsum/mPCM composites should be studied.

CRediT authorship contribution statement

Osman Gencel: Conceptualization, Supervision, Writing – original draft, Writing – review & editing. **Gökhan Hekimoglu:** Investigation, Methodology, Writing – original draft. **Ahmet Sarı:** Supervision, Writing – original draft, Writing – review & editing. **Abid Ustaoglu:** Data curation, Validation, Writing – original draft, Writing – review & editing. **Serkan Subasi:** Data curation, Writing – original draft, Writing – review & editing. **Muhammed Marasli:** Formal analysis, Writing – review & editing. **Ertugrul Erdogmus:** Investigation. **Shazim Ali Memon:** Writing – original draft, Writing – review & editing.

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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