



Article

# The Morphological and Thermal Characteristics of Hollow-Glass-Microsphere-Coated Phase Change Material–Cow Pie Embedded Recycled Plastic Tiles for Cool Roofs

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**Abstract:** This study addresses the global plastic waste crisis and the urban heat island effect by developing an innovative solution: recycled plastic roof tiles embedded with phase change material (PCM) and coated with hollow-glass-microsphere-based white paint. The samples were fabricated with cow pie fibers, OM37 and OM42 PCM materials with different *wt./vol.* values, i.e., 15/50, 20/50, 25/50, 30/50 ratios. The fabricated tiles were coated with hollow glass microspheres to provide a reflective layer. The tiles' effectiveness was evaluated through morphological examination and thermal analysis. The SEM analysis revealed an excellent bonding ability for the PCM blend, i.e., OM37 and OM42 at a 20/50 ratio (*wt./vol.*) with cow pie fibers. Adding cow pie fibers to the PCM shifted the melting points of OM37 and OM42, indicating an increased heat storage capacity in both blends. The thermal conductivity results revealed decreased thermal conductivity with an increased cow pie fiber percentage. The recycled plastic roof tile of the PCM composite at a 20/50 (*wt./vol.*) ratio showed good thermal properties. Upon testing in real-time conditions in a physical setup, the roof tiles showed a temperature reduction of 8 °C from outdoors to indoors during the peak of summer. In winter, cozy temperatures were maintained indoors due to the heat regulation from the roof.



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## 1. Introduction

Plastics are a group of synthetic materials made from polymers, which are long chains of molecules [1,2]. They have become integral to our modern society due to their low density, high chemical stability, and hydrophobicity [3,4]. Plastics also have excellent longevity and strength-to-weight ratios [5,6]. Plastics are popularly used in various applications, including packaging, construction, transportation, electronics, textiles, and healthcare [7,8]. Plastic consumption has risen four times, and its production has doubled. As for the exciting statistics, 40% of plastic waste is generated from packaging, 12% from consumer goods, and 11% from textiles, of which only 9% is recycled. Nearly 22% of collected plastic waste is burned in open sites or landfills [9,10].

Reduce, reuse, recycle, and recover (4R) are waste management techniques that focus on sustainable alternatives and aim for a Circular Economy. Mechanical recycling is the most popular solution for plastic recycling due to its low cost and ease of maintenance. Although recycling results in property degradation due to polymer chain scission, there are effective techniques for enhancing these properties. This degradation may be due to the presence of contaminants and other chemical residues while recycling [11]. The recycling industry has implemented new approaches to improving the desired properties through blending with additives, chain extenders, compatibilizers, etc. [12]. Nanoparticles are also innovatively blended with recycled plastics, and there is an improvement in the thermal and



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mechanical properties of these blends, extending the application of recycled plastics more efficiently in construction and roofing [13]. In their research, Ganesan et al. [14] proposed that maximizing 100% recycled pellets can extend the recycling cycle to approximately 15–20 years while significantly reducing carbon emissions. However, the rise in spatial temperatures, attributed to the urban heat island effect, presents a contrasting challenge. Urbanization, population growth, and the widespread adoption of cooling systems have contributed to a temperature increase of 3 to 4 degrees Celsius over the past decade, accompanied by an escalating Human Discomfort Index [15].

Using phase change materials (PCMs) emerges as a viable solution to address these rising temperatures. PCMs possess notable latent heat during their solid–liquid phase transition, making them valuable for thermal storage applications and as cost-effective alternatives. Implementing PCMs for thermal storage makes it possible to buffer transient heat loads, meet the growing demand for renewable energy sources, facilitate waste heat recovery, and contribute to environmental sustainability efforts, ultimately working toward achieving carbon neutrality [16]. PCMs are used in roofing as they can regulate the heat from indoors to outdoors. Recently, more focus has been placed in this area, as this significantly reduces the thermal loads on the grid, greenhouse gases, costs, etc. Roofs are generally subjected to various thermal changes, like convection, conduction, and radiation. Various passive techniques, like roof shading, roof coating, etc., have been implemented to improve the thermal performance of roofs, but they have been ineffective due to their high retro-fitting costs and yearly maintenance. Still, PCMs are improving in enhancing thermal comfort [17]. Implementing PCMs for roofing/walls in households can mitigate overheating indoors during power outages. However, their effectiveness depends on the climatic conditions and the melting temperatures of the PCMs [18]. Phase change materials can be blended with conductive nanomaterials [19] to improve their cooling and stability characteristics. There is an enhancement of latent heat and heat storage efficiency with the addition of 0.3% nanoparticles [20].

Nowadays, more focus is placed on sustainable energy sources. Cow pie fibers are one of those special materials obtained from the undigested matter of cattle. Since ancient times, there has been the tradition of using cow pie cakes/cow pie paste on buildings or roofs in some parts of the world, especially in India. This acts as a natural insulator, protecting spaces from heat loss/gains [21]. Studies have shown that cow pie fibers have potential applications in the industrial sector [22]. When cow pie fibers are used as the reinforcement in cementitious composites, it has been observed that their shrinkage is reduced, and pretreatment of the cow pie fibers furthermore enhances these properties. They are a potential material for sustainable buildings [23]. Moreover, it is noteworthy that specific individuals employ cool white paint on their rooftops during summer. Nevertheless, it is vital to recognize that the painting process and subsequent maintenance are arduous undertakings, further compounded by the inherent unpredictability of the paint's longevity in the presence of rapidly changing weather patterns. Hollow glass microspheres (HGMs) are low-density materials that can be used as fillers in waterproof coatings and pigments. Since they reflect solar radiation and reduce temperatures, they are highly used today. It is found that the smaller the size of these microspheres, the higher the reflective capacity the material possesses, and they can achieve innovative ambient radiative cooling when used as a coating on buildings [24].

This experimental study attempts to fabricate novel recycled plastic roof tiles embedded with PCMs mixed with cow pie fibers and coated with hollow glass microsphere white paint, which is conceptualized, designed, and developed to improve indoor thermal comfort. This work innovatively covers the major issues today: the heavy load on power grids due to the uncontrolled usage of air-cooling systems, plastic waste, and greenhouse gases. With plastics recycled in the form of tiles, there is sustainable utilization of recycled materials. The reflective capacity of the tiles has been addressed with the top coating. Analyzing the thermal characteristics of recycled plastics is a topic that many researchers have studied; however, our analysis is focused on PCM's unique and unexplored morphological

and thermal characteristics when mixed with cow pie fibers. The roof tile is examined in a physical setup constructed under actual conditions. The real-time study was conducted using PCM along with cow pie fiber mix compositions. The best blend was determined based on its thermal performance, and the chosen blend was then used to create the roof tiles. The proposed roof tiles provide thermal comfort and are affordable and ecologically beneficial.

## 2. Materials and Methods

### 2.1. Materials

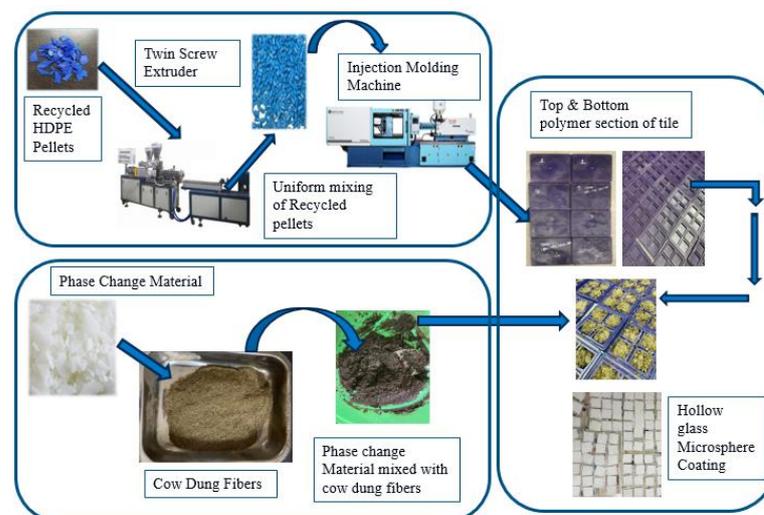
Recycled high-density polyethylene (rHDPE) chips (size: 3–4 mm) were used as the major raw material for the preparation of the roof tiles [4,13]. Plastic waste collected from certain localities was segregated, cleaned, shredded into small chips, and pelletized through a twin-screw extruder. Phase change material mixed with cow pie fibers was used as the secondary material to enhance the insulating properties of the tiles. The phase change materials (OM37 and OM42) were sourced from Pluss Advanced Technologies, Gurugram. The properties of OM37 and OM42 are given in Table 1 [25].

**Table 1.** Properties of OM37 and OM42 PCM [25].

Properties	Units	OM37	OM42
Specific heat (Cp)	(kJ/kg K)	2.63	2.78
Density ( $\rho$ )	(kg/m <sup>3</sup> )	860	863
Thermal conductivity (k)	(W/mK)	0.13	0.10
Latent heat (L)	(kJ/kg)	186	199
Melting temp. (t)	(°C)	38	44

### 2.2. Processing and Preparation of the Roof Tile Specimens

Recycled HDPE pellets obtained from a recycler were processed using a twin-screw extruder (Make: M/s. Specific Engineering Model: ZV20, Facility: CIPET, CSTS—Vijayawada, Surampalli, India) for uniform mixing of the materials, and then they were pelletized to achieve a uniform shape. In the next stage, the pellets were processed through an injection molding machine (Make: Toshiba Machine Pvt. Ltd., Model: STS 80-430, Facility: CIPET, CSTS Vijayawada, Surampalli, India). The die design was conceptualized, modeled, and manufactured at CIPET, CSTS Vijayawada. Figure 1 shows the life cycle of the roof tile from the raw material stage to the completed product.



**Figure 1.** Schematic flowchart of the preparation of novel recycled plastic roof tiles.

After processing, the recycled HDPE material and the phase change materials (OM37 and OM42, State: Solid) were heated in a hot air oven above their melting temperatures. Then, the liquid was mixed with the cow pie fibers and made into a slurry. Four samples with different *wt./vol.* PCM–cow pie compositions [viz., 15/50, 20/50, 25/50, 30/50] were prepared and drawn to compare the thermal behavior of different compositions. A prototype of the roof tile ( $15 \times 15 \text{ cm}^2$ ) was prepared from the composition that exhibited the optimum thermal properties. The PCM and cow pie fiber slurry were arranged in the bottom portion of the tile compartments as per the tile design. Then, the top portion was sealed with the bottom portion using industrial adhesive (3M™ High Performance Industrial Plastic Adhesive). Hollow glass microspheres were mixed with the standard white paint and coated onto the top portion of the tile. This coating helped reduce the heat transfer between the top and bottom portions, improving indoor comfort.

### 2.3. Scanning Electron Microscopy (SEM)

A scanning electron microscope (JSM-IT500 InTouchScope™/JEOL, Tokyo, Japan) was employed to perform microstructural analysis of the PCM–cow pie composites.

Voltage ranges from 10 kV to 15 kV were used to study the samples at 100  $\mu\text{m}$ , 200  $\mu\text{m}$ , 500  $\mu\text{m}$  magnification levels on a 15 mm desk. Five images for each blend were examined for validation of the analysis.

### 2.4. Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) is a thermoanalytic technique that measures heat flow at the molecular level. A differential scanning calorimeter (Make: TA Instruments DSC-250, New Castle, DE, USA) was used under a nitrogen environment (60 mL/min) with a heating rate of 10  $^{\circ}\text{C}/\text{min}$  over a range of 500  $^{\circ}\text{C}$  for the study. Five samples were tested for each condition to confirm the repeatability of the results.

### 2.5. Coefficient of Thermal Expansion (CTE), Volumetric Heat Capacity, and Thermal Conductivity

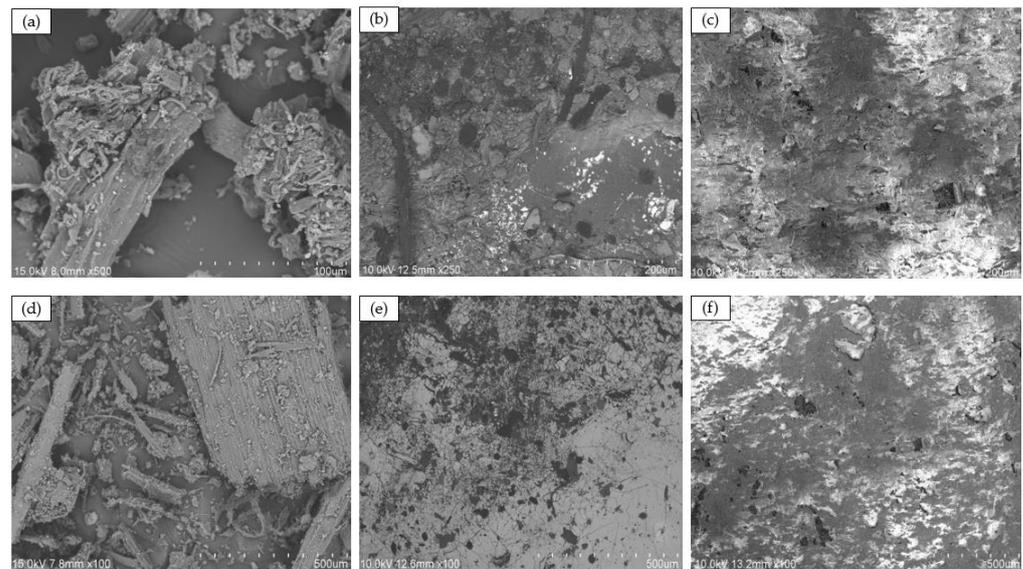
In this study, TA Instruments' analyzer TMA 450, DE, USA, was used to estimate the samples' thermal expansion coefficient. The testing was performed in a nitrogen environment at a heating rate of 10  $^{\circ}\text{C}/\text{min}$  up to 500  $^{\circ}\text{C}$ . Volumetric heat capacity and thermal conductivity tests were carried out using (Hot disk instruments (TPS 2200), Elk Grove, CA, USA) and using Kapton insulated sensors in the operating range of 25  $^{\circ}\text{C}$  to 120  $^{\circ}\text{C}$ . Five samples were tested.

## 3. Results and Discussion

### 3.1. Morphological Investigations

The morphology of the samples was analyzed under SEM at different magnifications; the respective images are shown in Figure 2a–f. Figure 2a,d represent the SEM images of the cow pie samples (not subjected to any chemical process). In these images, irregular surfaces, node structures, and rugged patterns were observed, along with skeleton-like patterns due to the cellulose content. At the same time, the presence of pie fibers and wheat straws generally enhances the adhesive bonding (internal) for different fiber interfaces [26].

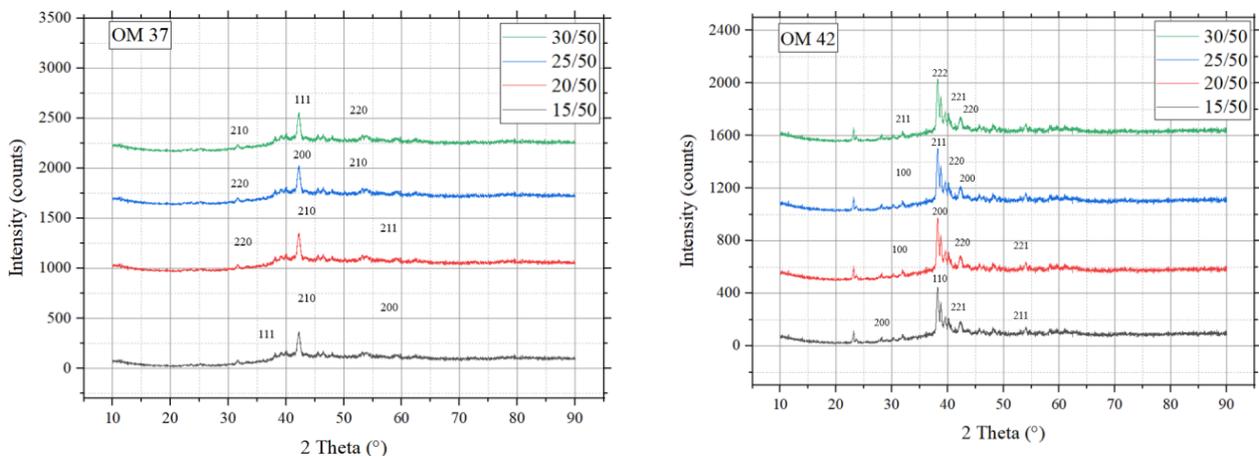
When the cow pie powder was mixed with the OM37 and OM42 phase change materials, the surface was found to have micro-pores and furrow-like structures, majorly noticeable for the OM37 and OM42 samples. Figure 2 depicts that all the inner layers have interconnected thin-walled tubules [27] and black spots due to waxy residues, lignin, and pectin [28]. A good compact surface and a glossy appearance (due to its solidification) were observed when the PCM was blended with the cow pie powder at different ratios. As shown in Figure 2b,c,e,f, the optimal blend is obtained at a *w/v* ratio of 20/50 for both the OM37 and OM42 PCMs. Good adhesion is exhibited by the PCM and fibers. Nevertheless, holes/black spots are visible at some locations due to improper blending, impurities, and sediments, such as undigested grains, soil, etc. (unavoidable).



**Figure 2.** SEM images of (a) pure cow pie powder at 100 μm magnification; (b) OM37 blend PCM at a 20/50 ratio at 200 μm magnification; (c) OM42 blend at a 20/50 ratio at 200 μm magnification; (d) pure cow pie powder at 500 μm magnification; (e) OM37 blend at 20/50 ratio at 500 μm magnification; (f) OM42 blend at 20/50 ratio at 500 μm magnification.

### 3.2. XRD Analysis

The crystallization of the PCM blends (at different weight-to-volume ratios) was examined using XRD. Figure 3 shows the XRD patterns of the PCM + cow pie powder samples. Table 2 clearly shows the diffraction peaks at different levels, which are majorly due to the diffraction of the crystal planes of the phase change materials [29] and also due to the presence of amorphous and hemi cellulose, along with rumen microorganisms, which can be found in cow pie powder [30]. From the XRD traces, it is noticeable that the two observable crystalline structures are face-centered cubic (FCC) and body-centered cubic (BCC), and this suggests both the blends have almost the same crystallization. However, the OM37 blend with a 25/50 wt./vol. ratio and the OM42 blends with 25/50 (wt./vol.) and 20/50 (wt./vol.) ratios showed a slight advantage due to the presence of FCC and BCC structures, indicating good mechanical and thermal properties. The remaining miscellaneous peaks are due to the impurities in the cow pie powder.



**Figure 3.** XRD graphs of OM37 and OM42 blends.

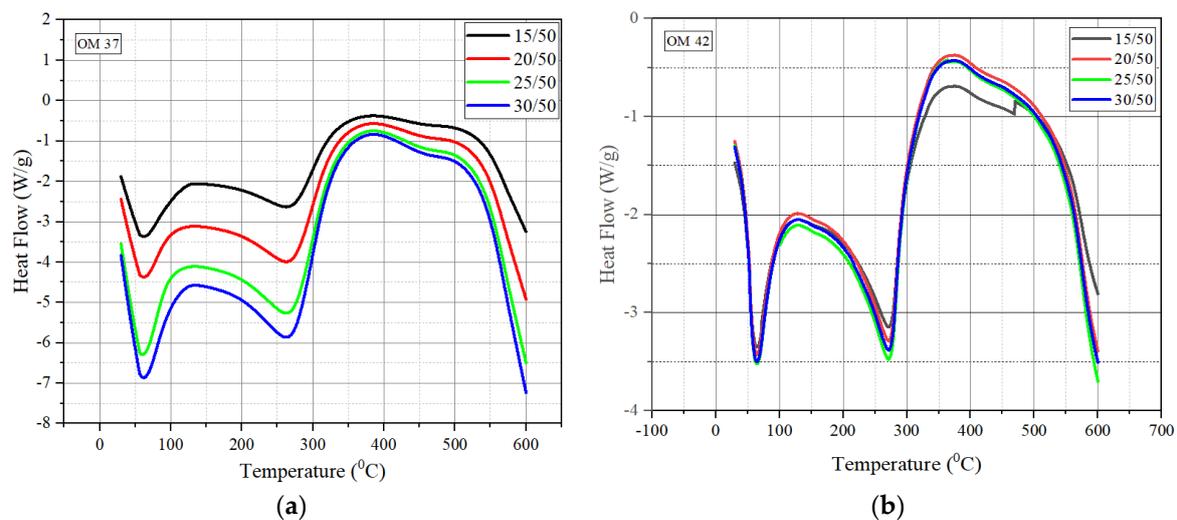
**Table 2.** Crystallinity of PCM blends.

Composite	wt./vol.	2 $\Theta$ —Peak 1	2 $\Theta$ —Peak 2	2 $\Theta$ —Peak 3	2 $\Theta$ —Peak 4
OM37 + Cow Pie Powder	30/50	320	430	540	
	25/50	32.50	42.50	540	
	20/50	32.50	42.50	540	
	15/50	330	42.50	540	
OM42 + Cow Pie Powder	30/50	320	380	400	430
	25/50	32.50	390	400	430
	20/50	32.50	390	430	540
	15/50	330	38.50	430	540

### 3.3. Thermal Analysis

#### 3.3.1. Differential Scanning Calorimetry (DSC)

Figure 4 represents the thermograms of all the samples. To avoid the disturbances that are developed due to the thermal resistance of the PCM, the process was started at a minimum temperature and then gradually increased to higher temperatures (at 0.5 K/min). The traces of the OM37 and OM42 samples which were blended with cow pie powder (with different weight-to-volume ratios) exhibited strong endothermic peaks between 55 and 60 °C, and the thermal events after 250 °C were due to the degradation of the phase change material or due to impurities.

**Figure 4.** DSC curves of (a) OM37 blend and (b) OM42 blend.

The first and second peaks observed were due to the melting of the PCM and cow pie powder. The reason for the good endothermic peaks is the strong attractive interaction between the PCM and the inner surfaces of the pores (cow pie powder). Generally, these interactions play a crucial role in the shift direction of the PCM temperature [31]. In Figure 4, compared to the 15/50 (wt./vol.) sample, the remaining samples show a negligible melting peak shift at lower temperatures. This behavior is probably due to the plasticization effect, and similarly, slight changes in the first peak shift shows a decline in the phase transition temperatures [32]. Nonetheless, adding cow pie powder to the PCM in all the samples showed a similar trend and only slight changes to the characteristics of the PCM blends.

### 3.3.2. Coefficient of Thermal Expansion (CTE), Thermal Conductivity (TC), and Volumetric Heat Capacity (VHC)

From Figure 5a, it is evident that the coefficient of thermal expansion of the OM42 PCM blends is higher than that of the OM37 blends, indicating that the OM42 blends have a larger expansion rate than the OM37 blends. The difference in these values probably could be due to their intermolecular structure. For the OM37 blends, the sample with a 30/50 (wt./vol.) ratio showed the highest expansion, followed by that with a 20/50 (wt./vol.) ratio, and the same pattern was observed for the OM42 blends. The results have proven that adding cow pie fibers to the respective PCMs significantly improved the thermal expansion rate as the mass of the component increased and evinced that it can be used in thermal management applications (when ambient temperatures are confined to 35 to 45 °C).

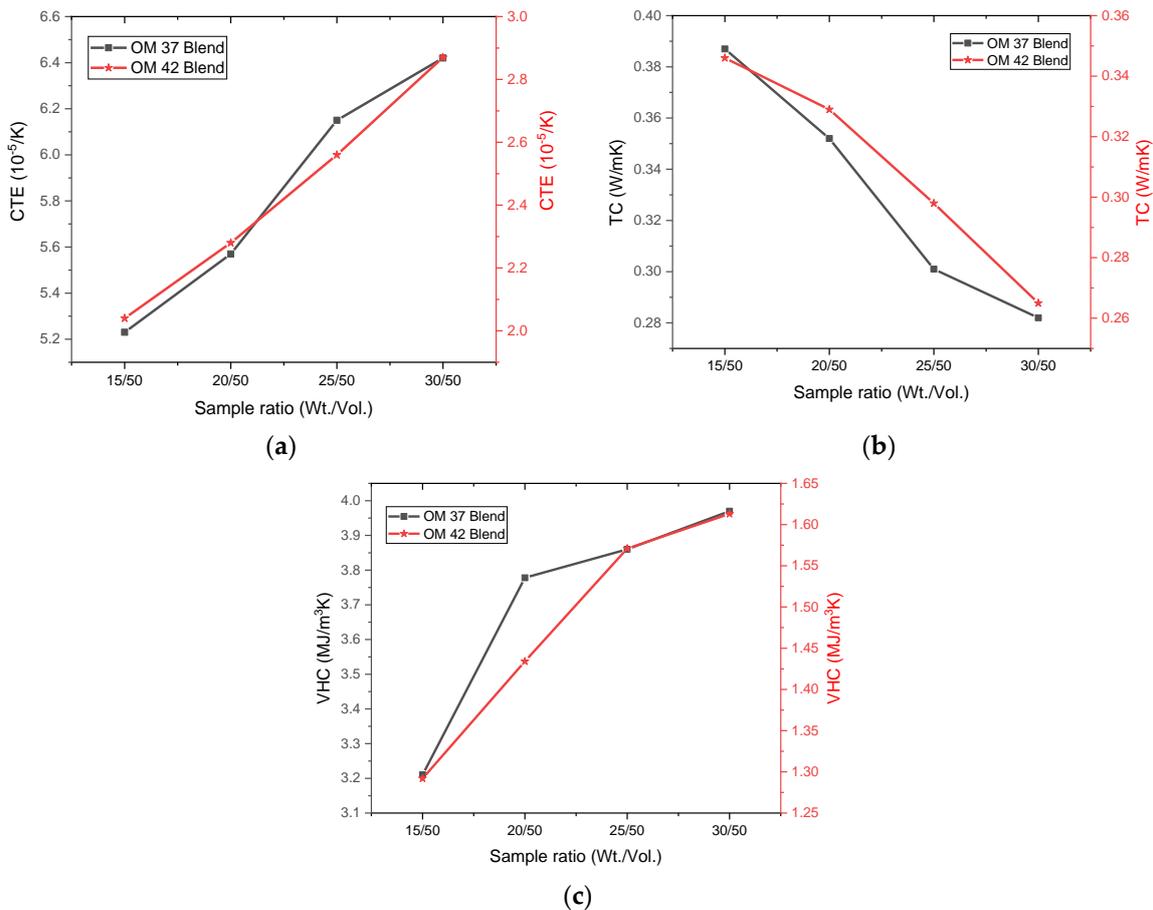


Figure 5. (a) Coefficient of thermal expansion, (b) thermal conductivity, (c) volumetric heat capacity.

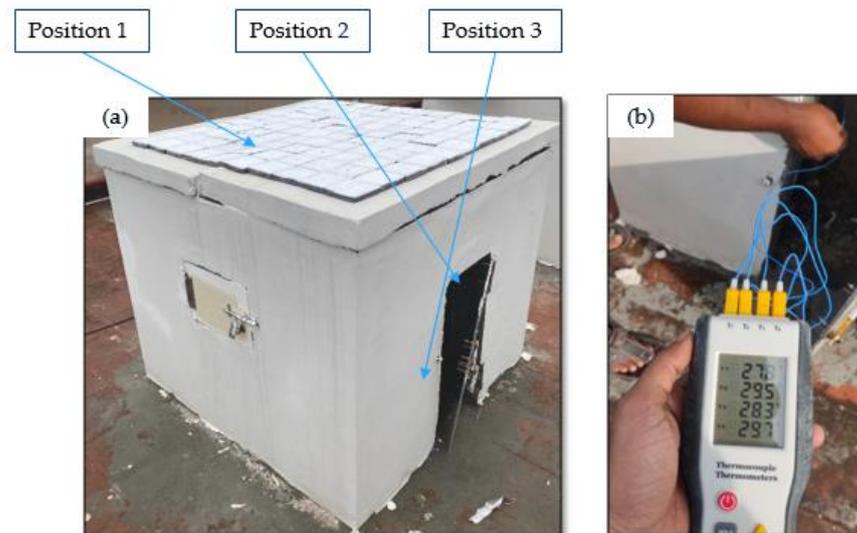
Similarly, Figure 5b shows that the thermal conductivity is reduced by adding cow pie fibers. Pure OM37 has a thermal conductivity of 0.13 to 0.16 W/mK; after adding the fibers, its thermal conductivity increased to 0.387, and eventually, with the addition of the cow pie fibers, the thermal conductivity was reduced, which is due to the porous structure engendered and the multiplication of the porosity of the mixture. Generally, pores reduce thermal conductivity [33]. Like that of OM37 blends, a similar pattern/structure was observed in the OM42 blend. The results confirmed that both the blends exhibited the same (almost) amount of thermal conductivity in all the samples.

Figure 5c shows the volumetric heat capacity testing; it was noticed that the volumetric heat capacity of the blends (OM37 and OM42) increased with the addition of the cow pie fibers to the pure PCM, which was mainly due to an increase in the density of the blends. This resulted in an increase in their thermal storage capacities (within the specific volumes) [34]. Among the OM37 and OM42 blends, OM37 has shown a higher

thermal storage capacity since OM37 has a higher density value ( $980 \text{ kg/m}^3$ ) than OM42 ( $900 \text{ kg/m}^3$ ). This indicates that the OM37 blends could be used in thermal management due to their higher thermal storage capacities.

### 3.4. Real-Time Temperature Distribution on the Roof Tiles

In this study, to test the real-time performance of the recycled HDPE roof tiles, a prototype house was built on the terrace of a university building, as shown in Figure 6a. The construction was built with cement and Plaster of Paris (dimensions:  $150 \text{ cm} \times 150 \text{ cm} \times 150 \text{ cm}$ ), replicating a regular single room. Proper ventilation was set up by providing two windows and one door.



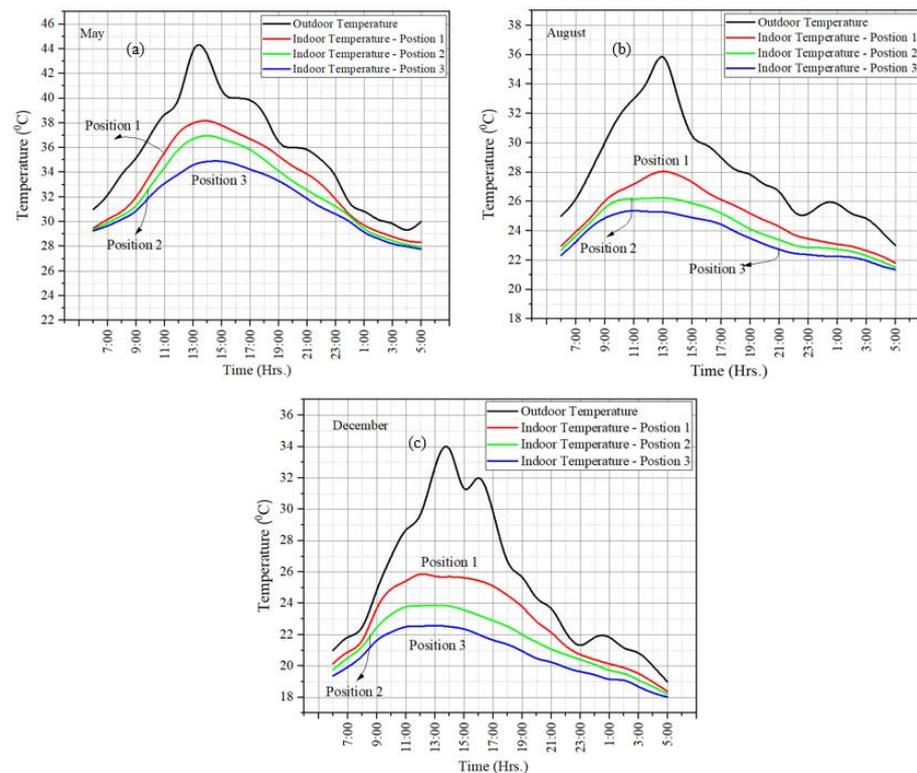
**Figure 6.** (a) Physical setup built for testing the tiles; (b) temperature measurement using a thermocouple.

The roof tiles were arranged as part of the physical setup, and the study was conducted using the below test conditions as tabulated (Table 3). The temperature readings were noted using a 4-channel K-type digital thermocouple (Make: Leaton Thermocouple) by placing the probes in three different locations, viz. The probes were placed on the outside roof and on the inside wall and on the floor, as shown in Figure 6a. This setup was monitored bi-hourly for 10 successive days each season for a 24 h time frame.

**Table 3.** Test conditions for the recycled plastic roof tiles.

SNo.	Season	Test Condition
1	Summer (May)	Doors and windows were kept closed in the morning and open at night
2	Rainy (August)	Doors and windows were kept open during morning and night
3	Winter (December)	Doors and windows were kept open during the morning and closed during night-time

Figure 7a–c show the real-time temperature distribution of the roof tiles. The outdoor temperatures were collected from meteorological department data. The temperature values are bi-hourly from the different positions of the thermocouple probe. It can be observed that during summer, there is a temperature reduction of  $6\text{--}8 \text{ }^\circ\text{C}$  indoors in the afternoon and a reduction of  $2\text{--}3 \text{ }^\circ\text{C}$  at night under the test conditions.



**Figure 7.** Real-time temperature distribution of recycled plastic roof tiles in (a) summer (May), (b) rainy (August), and (c) winter (December) seasons.

During the daytime, sunlight hits the roof tiles, and part of it is reflected due to the presence of the hollow glass microsphere coating on the tiles' surfaces. The top portion of the tiles conducts the remaining heat to the middle section, where the PCM composite takes the heat and stores it, which acts as thermal storage for the latent heat, emitting only part of the heat indoors through the roof. At night, the stored heat is dissipated to the surroundings, regulating the heat from indoors to outdoors. In the rainy season, there is a 7–9 °C reduction in temperature during the afternoon and a 2 °C reduction in temperature at night. In the winter, there is a significant effect on the temperature distributions, especially during nights. Since the outdoor temperatures reduce drastically, the stored heat from the tiles will radiate out to the surroundings and partially provide some heat to the indoors, thus creating a cozy temperature inside.

The choice of phase change material (PCM) significantly influences the attainment of the targeted outcomes. The OM37 blend emerges as an optimal selection for residential settings, ensuring favorable indoor thermal comfort. Conversely, in regions characterized by tropical climates with elevated temperatures, the OM42 blend stands as the preferred option. This specific blend exhibits higher melting points, enhancing its capacity to store a more significant amount of heat, making it well suited to cooling purposes.

#### 4. Conclusions

In this study, morphological and thermal analysis was carried out on recycled Plastic roof tiles embedded with phase change materials and cow pie fibers and coated with hollow glass microsphere blended paint. The following observations were made from the experimental work:

1. Good bonding was observed during the SEM analysis. PCM, along with cow pie fibers, at a 20/50 (*wt./vol.*) ratio blend showed the best adhesive bonding for OM37 and OM42.

2. The mixing of cow pie fibers into the PCM increased the heat storage capacity of the blends due to a shift in the melting point for the OM37 and OM42 composites.
3. The thermal conductivity was reduced with an increase in the cow pie fiber percentage due to the material's increased porosity. In contrast, the volumetric heat capacity increased with an increase in the cow pie fibers. This further enhanced the thermal storage capacity of the material.
4. In the summer season, it was observed that there is a temperature reduction of 6–8 °C indoors in the afternoon, reduced to a 2–3 °C reduction at night, under the test conditions.
5. In the rainy season, there is a 7–9 °C reduction during afternoons and a 2 °C reduction at night. During winter, there is no significant change in the temperatures during the day, but the indoor temperatures are cozy at night.
6. The selection of the PCM will be crucial to energy-saving applications and is based on geographical location.

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**Data Availability Statement:** All the data generated or analyzed during this study are included in this published article.

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