CHAPTER 3

MATERIAL AND METHODS

This chapter discusses about the methodology opted to evaluate the effectiveness of the MPCM technique in improving the indoor thermal behavior of the building in tropical environment of India. It also presents the material required for the experimental work. Additionally, a detailed discussion on experimental setup and various parameters which are to be evaluated for understanding the effectiveness of the MPCM in improving indoor thermal profile of the building.

3.1 Methodology

The aim of this study is to evaluate the indoor thermal behavior of the building outfitted with macrocapsules of PCM in the tropical environment along with enhancement in thermal energy storage performance of the PCM. To accomplish the aforesaid aim of the study, three investigations was conducted i.e. one day/24 hours analysis (short duration study), Yearly analysis (long duration study), and thermal energy storage analysis of the PCM. Field tests were carried out to evaluate the indoor thermal performance of the cubicles for 24 hours analysis and yearly analysis. To evaluate thermal energy storage performance of the PCM thermo-physical characterization was conducted. Field tests were carried out by developing two cubicles of dimensions 1.12 m × 1.12 m × 1.12 m at the GLA University located in Mathura (India). The University is located in India and lying at geographical co-ordinates 27.6056° N and 77.5930° E. The experimental site experienced a tropical climate throughout the year. Tropical countries are known for their hot and humid climate along with intense sunlight. The Indian subcontinent also receives plentiful solar radiation throughout the year due to its favorable location in the solar belt (7,9). Nearly 58% of the Indian subcontinent receives the annual average global solar irradiance of 5 kWh/m^2/day (267). Intense solar radiation will increase the solar heat gain of the building envelope (268,269) causing thermal discomfort and consequently increases the energy required for space cooling (270). High solar radiation will increase the cooling requirement of as much as 25% for a building (271). Therefore, in these regions, the energy required for space cooling is much intense than space heating. Thus, the location of the experimental site is well suited to evaluate the effect on the indoor thermal profile of the building by increasing the latent heat storage capacity using MPCM. The high atmospheric temperature will make the PCM completely charged (melt) and consequently leads to maximum utilization of the latent heat of the PCM. Both the cubicles are provided with the windows and the doors for proper ventilation of the indoor air, especially during the discharging of the PCM. To establish a comparative analysis in indoor thermal behavior between the building envelope with PCM and the building envelope without PCM, one cubicle is embedded with macroencapsulated PCM while the other is without macroencapsulated PCM. During the data collection, the window and the door of both the cubicles remain open from 06:00 PM to 06:00 AM, to provide effective passive ventilation to the heat released from the PCM during the discharging. However, during the day the door and window of both the cubicles remain closed.

The indoor temperature of both the cubicles and the sol-air temperature were recorded using thermocouples. The heat flux sensor is used to record the heat-flux across all the orientations of the cubicles. Indoor temperature profile of both the cubicles is also evaluated using infrared thermography. Lastly, Pyranometer is used to measure the global solar radiant energy of the experimental site. For the short duration (one day) experiment following data of July 12, 2018 was observed and recorded: (a) Indoor surface temperature of east, west, north, south walls and roof of both the cubicles (b) Indoor ambient temperature of both the cubicles (c) Heat flux across all the walls and roofs of both the cubicles (c) Indoor surface thermography of east, west, north, south walls and roof of both the cubicles. For long duration (yearly) experiment, indoor ambient temperature and heat flux across all the walls and roofs of both the cubicles for 24 hours of each months of the year 2019 was observed and recorded. Based on above mentioned measurements following parameters for short duration experiment and long duration experiment of both the cubicles were analyzed:

1. Peak temperature reduction
2. Percentage Reduction in thermal amplitude
3. Time lag
4. Decrement factor
5. Peak heat flux reduction or cooling load reduction
6. Cost savings in cooling load
7. Infrared thermography analysis (only for one day study)

Based on the above parameters a detailed comparative study was conducted to evaluate the effect of incorporating macroencapsulated PCM in the building envelop in tropical climate.

Thermal energy storage in buildings using phase change material has attracted many researcher and scientist in recent decades to improve building energy efficiency (272,273). Embedding PCM in the building envelope/building material will enhances the latent heat storage capacity of the building which in turn improve the indoor thermal behavior of the buildings (274) because of following merits of PCM over other materials to be used for thermal energy storage (11) in buildings (a) Ability to change phase at almost constant temperature (b) High heat of fusion (c) High energy storage density (d) Large availability in different temperature ranges (275). However, it has been analyzed after conducting a detailed literature review that PCM suffers with two major drawbacks when used in buildings for enhancing thermal energy storage. The first drawback is lower thermal conductivity (276) and the second is leakage during phase transition (240). Low thermal conductivity of PCM reduces the heat transfer rate which adversely affects the charging and discharging of PCM. Consequently, it limits the complete utilization of latent heat storage capacity of the PCM. On the other hand, leakage during phase change process may leads to wastage/loss of PCM from thermal energy storage system. Therefore, to overcome these drawbacks, series of composite PCM was prepared and investigated for improved thermo-physical properties. The composite PCM was prepared by mixing PCM (OM37) with a material having high thermal conductivity along with a material having high porosity. Following characteristics of the prepared composite PCM was investigated:

1. Thermal transient response
2. Latent heat storage capacity
3. Thermal decomposition
4. Thermal reliability
5. Thermal conductivity
6. Leakage-proof performance

3.2 Materials for field testing

The phase change material used was commercial PCM called SavE® OM37 PCM (purchased from Pluss Advanced Technologies Pvt. Ltd.) and is shown in Figure 3.1. The

PCM is a chemical-based PCM and it stores the thermal energy in the form of latent heat in its crystalline form. On changing the phase this latent heat is released or absorbed, allowing the ambient temperature within the system to be maintained. The PCM is constituted of the right mix of various salts, additives, and nucleating agents, allowing equilibrium between solid and liquid phases to be attained at the melting point. The PCM is chemically and physically stable in both the solid and liquid phase and can be encapsulated in various forms.

Figure 3.1 Commercially available OM37 PCM

The phase change temperature range of the PCM is almost similar to the average ambient air temperature during the summer months (March – June) (277) in India. This gives an advantage to the PCM to get charged/discharged completely causing maximum utilization of its latent heat storage capacity. The thermo-physical properties of the PCM are shown in Table 3.1. The container used for macro-encapsulation of PCM can be of various shapes such as tubes, spherical ball, and panels which are then integrated into the building material. In this study tubular shape of containers made of aluminum 8011 alloy, as shown in Figure 3.2, are used for the encapsulation of the PCM. Aluminum was used as a shell material because of its high thermal conductivity and easy availability. The thermo-physical properties of aluminum tubes, as provided by the manufacturer, are shown in Table 3.2.

Table 3.1 Thermo-physical properties of the PCM (Provided by the manufacturer)

Property	Value
Melting Temperature range (°C)	35-40
Latent heat (J/kg)	218
Density at 40°C (kg/m^3)	860
Density at 30°C (kg/m^3)	960
Thermal conductivity at 40°C (W/mK)	0.13
Thermal conductivity at 30°C (W/mK)	0.16
Thermal stability (Cycles)	~3000

Table 3.2 Thermo-physical properties of aluminum 8011 alloy (Provided by the manufacturer)

Property	Value
Thermal conductivity (W/mK)	232
Specific heat (J/kgK)	900
Density (g/cm^3)	2.7
Thermal expansion (%)	23
Ultimate tensile strength (MPa)	100-180

Aluminum tubes of thickness 0.5 mm, diameter 16.7 mm, and length 900 mm were used to encapsulate the PCM. PCM was filled in aluminum tubes in molten form and both the ends of the tubes were sealed by plastic caps using Araldite to avoid the leakage of the PCM. A total of 17.5 kg of the PCM was filled inside 35 tubes (25 tubes of 900 mm and 10 tubes of 370 mm). The 370 mm length of tubes is placed adjacent to the window and the door.

Figure 3.2 Prepared macrocapsules

The indoor ambient temperature and indoor surface temperature of walls was measured by using K-type thermocouple. A 12 channel paperless data logger was used to record the variation in temperature. All the thermocouples were placed at a height of 0.55 m from the ground. Ten heat flux sensors, five for the reference cubicle and five for the experiment cubicle, were used to measure the rate of heat transfer across the east, west, north, south, and the roof. The sensor on the roof is placed at the equidistance from all the four walls. For the measurement of solar radiation intensity, a pyranometer from Hukseflux is used. The details of all the sensors used for the measurement are shown in Table 3.3.

Table 3.3 List of sensors used for the data collection

S.No.	Sensors	Quantity	Range	Accuracy
1	K-type thermocouple	12	0˚C to 100˚C	0.6˚C
2	Temperature data logger	1	-10˚C to 250˚C	±0.51%
3	Heat flux meter	10	$0\text{-}3.1 \times 10^5$ W/m^2	2%
4	Pyranometer	1	2.8 µm to 3µm	≤1.8%
5	Thermal imager	01	-20˚C to 280 °C	±0.2˚C

3.3 Experimental setup

The experimental setup has two building structures, namely reference cubicle and experimental cubicle, which resembles a small scale representation of living houses. The experimental setup is a miniature building structure having miniature door and window. It is a small scale setup of a real size building. Both the building structures was developed by using common building material and are having similar dimensions (1.12 m × 1.12 m × 1.12 m). Bureau of Indian Standard code (IS 383-2016) has been followed in using building material for constructing both the cubicles. Table 3.4 depicts the composition of the building materials used to develop the cubicles. The reinforced concrete mixture of M25 grade was followed to construct the walls and roofs of both the cubicles. The Portland Pozzolana cement of Ultratech cement make, Sand from the river bed, Granite coarse aggregate of more than 20 mm and fine aggregate of less than 9 mm are used for the preparation of the concrete mix. Figure 3.3 shows the pictures of the construction site during the development of the cubicles.

Table 3.4 Composition of building material used to develop cubicles

S.No.	Building Material	Quantity (kg)	Specific gravity	Type
1	Cement	285	3.15	Portland Pozzolana cement
2	River sand	285	2.65-2.70	Saturated surface dry sand
3	Coarse aggregate	350	2.84	Granite
4	Fine aggregate	220	2.64	Granite

Figure 3.3 Various pictures of the experimental site during the construction (a) Embedding MPCM on the roof (b) Embedding MPCM in the wall (c) Installations of thermocouple inside the cubicles (d) Photograph of the construction site

The thickness of the walls and the roof is 0.101 m. Both the cubicles have a window (0.15 meters × 0.15 meters) and a door (0.15 meters × 0.46 meters). Figure 3.4 and Figure 3.5 shows the schematic of the reference and experimental cubicle developed for the study. The east wall was provided with the door and the west wall has the window. The window and door are provided to eliminate the heat released during the discharging of the PCM in the night (278).

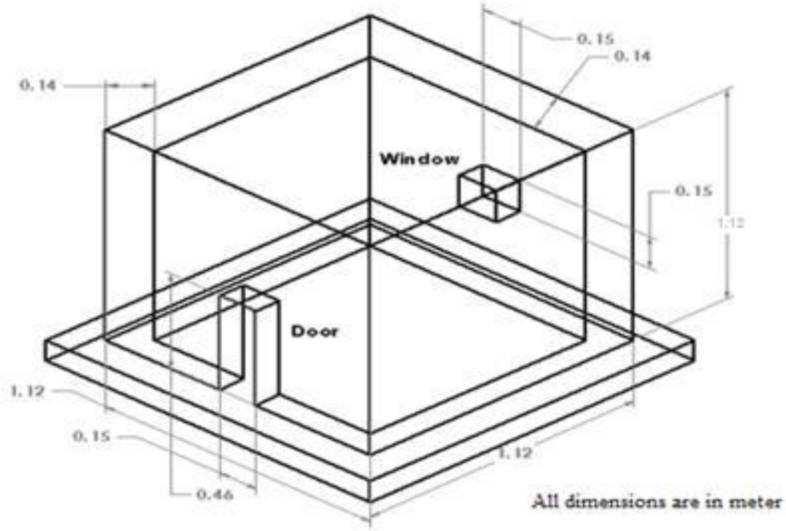

Figure 3.4 Schematic of the building structure

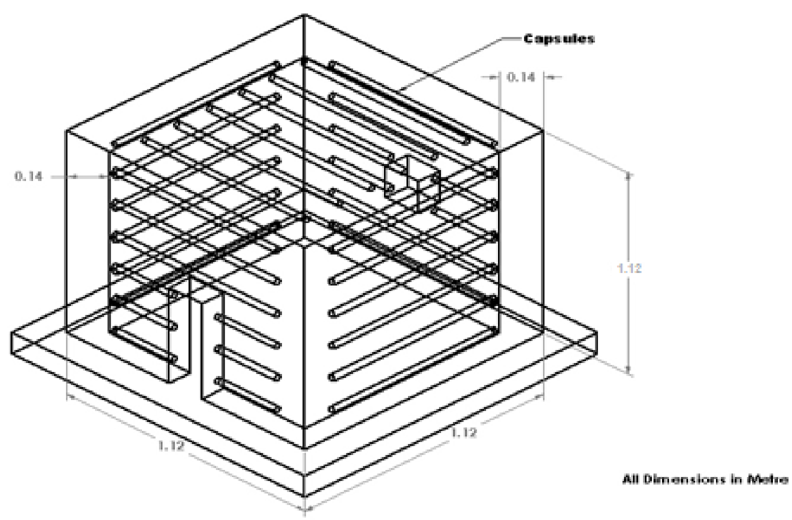

Figure 3.5 Schematic diagram of the experimental cubicle with macroencapsulated PCM

The MPCM was integrated in the experimental cubicle during the construction process, i.e when the concrete mixture is wet. Each wall and the roof of the experimental cubicle contain six macrocapsules of the PCM as shown in the Figure 3.6 (a). Figure 3.6 (b) shows the dimensions of the macrocapsule.

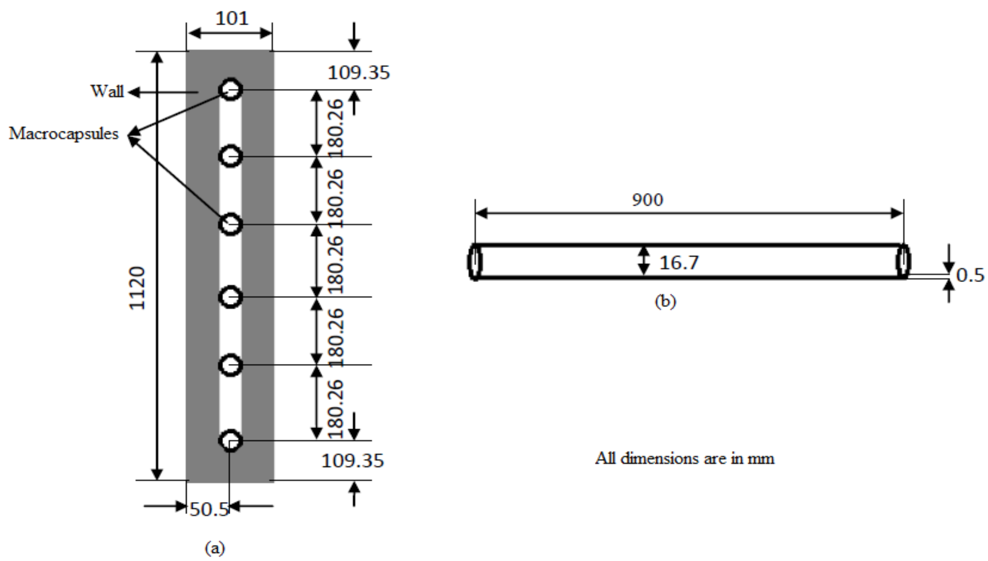

Figure 3.6 (a) Section view of the wall containing MPCM (b) Aluminum pipe used as Macrocapsule

Each macrocapsules of the PCM were placed horizontally at the center of the walls to optimize the charging and discharging rate of the PCM through heat gain and heat loss. If the PCM is placed near the outside surface of the wall, the charging time will reduce because of easy heat gain and if the PCM is placed near the inside surface of the wall, then charging time will reduce because of poor heat gain. Additionally, placing the macrocapsules near the inside surface of the wall will allow heat to penetrate inside the cubicle easily during discharging or during night. Before starting the actual experiment, 240 hours of continuous reading of indoor surface temperature, indoor ambient temperature, and heat flux across all the orientation was measured at various locations inside both the cubicles. Investigation shows a variation of under ±1.5 °C and ±1.8 W/m^2 for the indoor temperature and heat flux respectively. The developed experimental cubicle and reference cubicle are shown in the Figure 3.7.

Figure 3.7 Experimental setup photographs of developed experimental and reference cubicle

3.4 Material and equipment for the measurement of thermal energy storage performance

3.4.1 Materials

Inorganic PCM OM37, purchased from Pluss Advanced Technologies Pvt. Ltd., was used to prepare the shape stabilized composite PCM (ss-CPCM). It has thermal conductivity of 0.145 W/mk, density of 860 kg/m^3, and melting temperature range of 35-40°C. OM37 has high latent energy storage capacity and have low degree of super cooling. Expanded vermiculite (EV) was purchased from Saara Polycoats Pvt. Ltd. from Pune, India. EV has thermal conductivity in the range of 0.058-0.064 W/mK, density of 50-120 kg/m^3, and melting temperature of 1350°C. Various constituent of EV are SiO_2 (42.4%), Al_2O_3 (12.7%), Fe_2O_3 (10.6%), CaO (4.2%), MgO (21.2%), K_2O (2.8%), H_2O (6.1%). Graphite powder with an expandable rate of 220 ml/g – 300 ml/g was provided by Reinste Nano Ventures Pvt. Ltd. located at Noida, India. EG (Thermal conductivity: 50-100 W/mK, average particle size: ≤50 μm, density: 300 kg/m^3, surface area: 72 m^2/g) was prepared from graphite powder through chemical oxidation and then dried in a vacuum oven at 70°C for 20h, followed by heating at 900°C in a furnace for 90 sec which causes rapid expansion and exfoliation resulting in production of expanded graphite.

3.4.2 Preparation of PCM/EV/EG ss-CPCM

The PCM/EV/EG ss-CPCM was prepared in two steps. Initially, the PCM/EG composite mixture was prepared using ultrasonication method. The PCM was taken in a beaker in molten form and EG was added in it and subsequently ultrasonication was performed at 500 W with frequency of 20 kHz. This step was conducted at 90 °C for 20 min. Secondly, PCM/EG composite and EV was then undergoes vacuum impregnation to prepare the ss-CPCM. The EV at specified amount was taken in a flask which is attached with vacuum pump. Vacuum pump has evacuated all the air from the flask. The vacuum pump initiated the impregnation process and worked for 80 min and at 75 kPa. Then the PCM/EG composite was added drop-by-drop in the EV by using funnel. The prepared PCM/EV/EG ss-CPCM was then cooled at 10 °C. Four different samples, as shown in Table 3.5, of PCM/EV/EG ss-CPCM was prepared through the above mentioned procedure. The samples were named as ss-CPCM-1, ss-CPCM-3, ss-CPCM-5, and ss-CPCM-7.

Table 3.5 Weight and percentage amount of various components of ss-CPCM

Sample	PCM (g)	EV (g)	EG (g)	PCM (wt%)	EV (wt%)	EG (wt%)
ss-CPCM-1	7.50	7.50	0.0	50.0	50.0	0.0
ss-CPCM-3	7.27	7.27	0.45	48.5	48.5	3.0
ss-CPCM-5	7.12	7.12	0.75	47.5	47.5	5.0
ss-CPCM-7	6.97	6.97	1.05	46.5	46.5	7.0

The heat storage capacity of the ss-CPCM was analyzed using Differential Scanning Calorimeter (DSC, TAQ20). Melting and heating temperature along with corresponding latent heat storage capacity of the ss-CPCM samples are measured using DSC analysis. The experiment was carried out under the effect of nitrogen gas at a constant heating and cooling rate of 6 °C/min and 2 °C/min. The peak value of melting curve and freezing curve are recorded as melting and freezing point of ss-CPCM. Five repetitive measurements were recorded with mean deviation of ±0.11 °C for phase change temperature and ±1.15 J/g for latent heat storage capacity. The thermal stability of the ss-CPCM was measured using Perkin Elmer thermal gravimetric analyzer (TGA7). The TGA measurement was done in the temperature range 20 °C to 600 °C at a ramping rate of 10 °C/min. The thermal conductivity of ss-CPCM was tested using hot disk method through Transient Plane Source (TPS) technique. This method ensures accurate measurement under range of 0.005 to 500 W/mk over temperature ranges from 30 K to 1200 K. The TPS sensor (TPS 2500) is made of Nickel metal foil, cladded by kapton and was placed in between the specimen as shown in Figure 3.8. Compressive force is applied using weights and the data is recorded using thermal conductivity analyzer. This data is then analyzed in the computer. The sensor act as both heat source and a thermometer. The transient plane source method has precision better than 1% and accuracy better than 5%. Each sample was tested for three times and the average values of measurements were considered for final results. The thermal cycling test was performed under controlled laboratory environment using Thermal Air TA-500A model. The ss-CPCM undergoes 1500 heating-cooling cycles to evaluate the thermal reliability of the prepared composite. The experimental set up of thermal cycling consist copper plate for heating-cooling, which can be temperature controlled in a range of -90 °C to 250 °C. During the thermal cycling the temperature was maintained between 10 °C and 70 °C having a maximum temperature gradient of 10 °C/min. The cooling of the ss-CPCM was done using liquid nitrogen and heating was done electrically. The leakage test was performed on ss-CPCM to monitor leakage-proof performance. In this test the ss-CPCM samples was wrapped in a qualitative filter paper and placed inside the vacuum oven at 30°C for 10 minutes and at 40°C for 30 minutes. The whole process was monitored using digital camera.

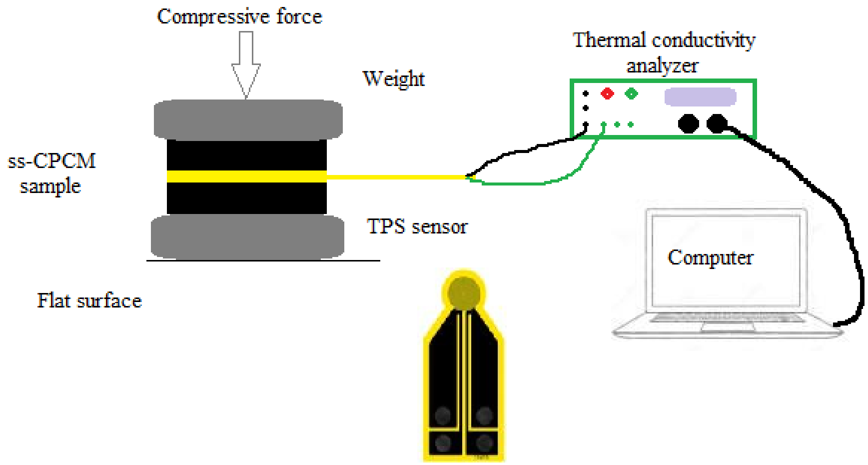

Figure 3.8 Schematic of thermal conductivity test using hot disk technique