

Article

Thermal Performance Measurement Procedure and Its Accuracy for Shape-Stabilized Phase-Change Material and Microcapsule Phase-Change Material Combined with Building Materials

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Abstract: The accuracy of differential scanning calorimetry (DSC) used in the dynamic method, which is the method most widely used to measure the thermal performance of existing phase-change materials (PCMs), is limited when measuring the phase-change range and peak temperature of PCMs combined with building materials. Therefore, we measured the thermal performance in a thermochamber; the samples were a sheet of shape-stabilized phase-change material (SSPCM) and a microencapsulated PCM-impregnated gypsum board fabricated by combining PCM building materials with paraffin. Then, we investigated ways to improve the measurement accuracy. We confirmed the setting time of the thermochamber temperature change based on the internal temperature of the PCM and the effect of the PCM capacity on its thermal performance using the dynamic method. The temperature was increased or decreased in uniform steps at regular time intervals. The error of the heat absorption and release was less than 2% when a stabilization time of at least 4 h elapsed before the start of the heating or cooling process. Overall trends in the specific heat and enthalpy, such as the phase-change section and peak temperature of the PCM, were similar regardless of the setting time. Thus, it was confirmed that the latent heat performance did not increase proportionally with the increase in the PCM capacity. The proposed approach can be used to measure the specific heat and enthalpy of various types of PCMs and building materials.

Keywords: phase-change material; shape-stabilized PCM; microencapsulated PCM; thermal performance; specific heat measurement



Citation: Kim, H.B.; Mae, M.; Choi, Y. Thermal Performance Measurement Procedure and Its Accuracy for Shape-Stabilized Phase-Change Material and Microcapsule Phase-Change Material Combined with Building Materials. *Sustainability* **2021**, *13*, 6671. <https://doi.org/10.3390/su13126671>

Academic Editor: Antonio Caggiano

Received: 19 May 2021

Accepted: 10 June 2021

Published: 11 June 2021

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Highlights

- Paraffin-based PCM combined with building materials were measured for thermal performance in a thermochamber.
- The samples were a sheet of shape-stabilized phase-change material (SSPCM) and a microencapsulated PCM-impregnated gypsum board.
- The effect of thermochamber temperature change and PCM capacity on thermal performance was confirmed using a dynamic method.
- The proposed approach can be used to measure the specific heat and enthalpy of various types of PCMs and building materials.

1. Introduction

The use of thermal energy storage (TES) systems in the building and industrial sectors has great potential for energy conservation [1]. TES systems can reduce the imbalance between energy demand and supply and reduce costs associated with energy loss [2,3]. Latent heat storage using a phase-change material (PCM) in a TES system can reduce the volume and weight since the unit volume has more heat than sensible heat storage [4,5].

PCMs are advantageous for heat utilization because they absorb or release heat at a constant temperature [6]. PCMs are also suitable for indoor use as they can easily be applied to floors, walls, and ceilings as building materials [7]. When a PCM is applied indoors, the internal temperature of the building changes as the PCM undergoes phase changes between solid and liquid [8]. In the application of a PCM to a building, accurate measurement of its performance serves as the cornerstone of the overall system. Thus, it is critical to understand the thermal properties of PCMs [9]. The phase-change range and latent heat are among the most important parameters of PCMs as they affect the performance of PCM applications and systems integrated with PCMs [10]. For example, when a PCM is integrated into a wall, fluctuations in the room temperature and thermal comfort depend on the phase-change range [11]. This has a significant effect on heating and cooling loads, demonstrating the importance of measuring the thermal performance of PCMs.

Differential scanning calorimetry (DSC) and differential thermal analysis (DTA) are standard tools for measuring the thermal performance of PCMs [12]. In DSC, a reference material and a sample are heated and cooled at a constant rate, and their thermal properties, such as heat storage, specific heat, and thermal conductivity, are measured [13]. In DTA, the temperature difference between a reference and a sample is measured during heating [14]. As PCMs can contain various additives, Yinping et al. [15] published a T-history method for measuring their thermal performance based on the heat transfer relationship between a contrast agent and the fixed ambient temperature. However, the specific heat or enthalpy of the PCM is generally measured by DSC using the dynamic method [10,16]. The measurement accuracy depends on the heating and cooling rates and the mass of the sample [17,18]. Since DSC is most suitable for small samples (5–10 mg) [19], it is often not suitable for the measurement of PCM building materials bonded to a gypsum board or flooring. Therefore, to measure the thermal performance of PCM building materials, methods have been proposed using a thermochamber or thermobath, which can regulate the temperature. Tiago Silva et al. [20] prepared a sample by placing macrocapsules containing a PCM inside a clay brick; then, they compared the changes in the surface temperature and internal temperature of a normal brick with those of the brick containing the PCM in a thermochamber. Lai et al. [21] measured the specific heat of gypsum boards incorporating a microencapsulated PCM with a melting point of 28 °C in a thermobath at different temperatures and investigated its heat transfer and heat storage behavior. These studies are considered to be insufficient for demonstrating the variation in the experimental results with changes in the temperature of the thermochamber or thermobath, changes in the stabilization setting time, and the method of using the internal temperature of the PCM sample to measure specific heat.

In this study, we investigated the use of a thermal chamber without DSC equipment to improve the measurement accuracy of the thermal performance of PCM building materials. For this purpose, the specific heat and enthalpy of an SSPCM sheet and microencapsulated PCM-impregnated gypsum board fabricated by combining PCM building materials with paraffin were measured under various conditions using the dynamic method. We confirmed the setting time of the thermochamber temperature change based on the internal temperature of the PCM and the effect of the PCM capacity on its thermal performance using the dynamic method, for which the temperature was increased or decreased uniformly at regular time intervals. In addition, we compared the efficiencies of the PCM building materials by examining the relationship between the PCM capacity and its latent heat performance. In this manner, we could improve the accuracy of the thermal performance measurement of PCM building materials and identify an effective measurement method.

2. Overview of the Experiment

2.1. PCM Sample

An SSPCM sheet (hereinafter SSPCM) and a microencapsulated PCM-impregnated gypsum board (hereinafter MPCM), as depicted in Figure 1, were selected as samples to measure the specific heat of the paraffinic PCM using a thermochamber. The samples

were 300.0 mm wide and 300.0 mm long. The SSPCM sample was prepared by stacking four sheets, each with a thickness of 2.5 mm, to match the thickness of the MPCM sample (10.0 mm). The SSPCM exhibits a phase-change temperature of 19–25 °C, whereas the MPCM has a phase-change temperature of 25 °C, as presented in Table 1. Multiple MPCM samples were used with PCM capacities of latent heat as 0 (a gypsum board without PCM), 11.5, 14.7, and 20.1 KJ/kg; they were compared in terms of their capacities and phase-change temperatures.

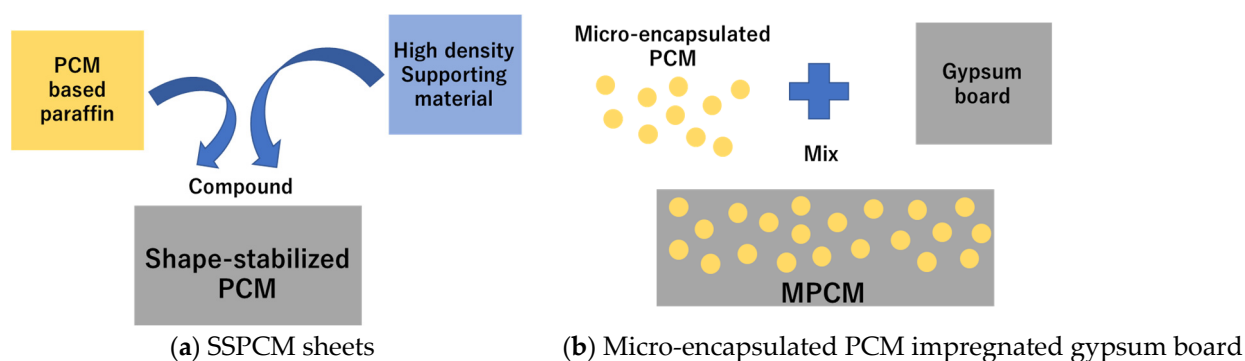


Figure 1. Concept of PCM samples.

Table 1. PCM samples used in the experiment.

PCM Type	Number	PCM Sample
SSPCM sheet	1	Phase change temp. 19–25 °C, latent heat (62.8 KJ/kg)
Microencapsulated PCM	2	Phase change temp. 25 °C, latent heat (0 KJ/kg)
impregnated gypsum board	3	Phase change temp. 25 °C, latent heat (11.5 KJ/kg)
(MPCM)	4	Phase change temp. 25 °C, latent heat (14.7 KJ/kg)
	5	Phase change temp. 25 °C, latent heat (20.1 KJ/kg)

2.2. Measurement Method

The specific heat measurement experiment was conducted using a PCM sample in a thermo-chamber (Figure 2a), where the temperature could be controlled over time. To calculate its specific heat, a thermocouple and a heat flow meter were installed above and below the PCM sample to measure the surface temperature and heat transfer of its upper and lower surfaces, as illustrated in Figure 2b. Aluminum plates were installed above and below the PCM to prevent heat transfer between the inside of the thermo-chamber and the sample. In addition, to prevent heat leakage from the sample, the sides were insulated.

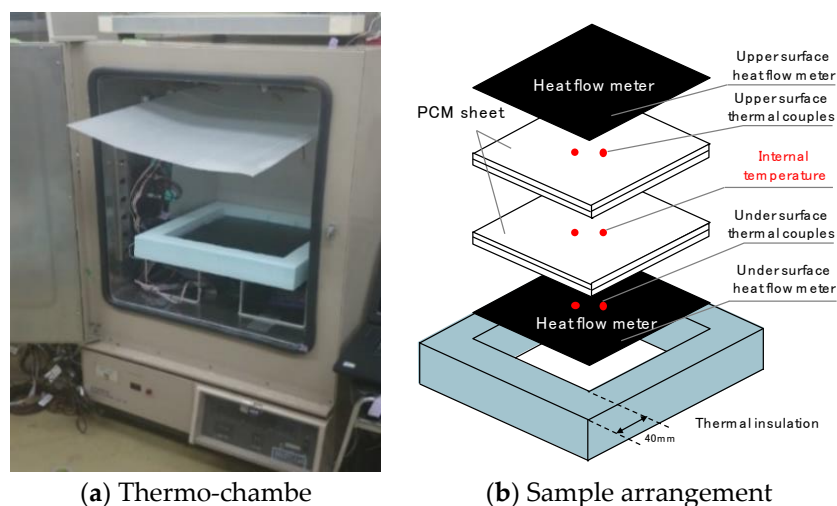


Figure 2. Method of measurement.

Figure 3 depicts the internal temperature settings of the thermochamber for measuring the specific heat of the PCMs. The heating and cooling temperatures of the inside of the thermochamber ranged from 10 °C to 35 °C, that is, with a margin of approximately 10 °C from the phase-change temperature of the PCM samples (SSPCM: 19–25 °C, MPCM: 25 °C) to measure the latent heat in the phase-change range and the sensible heat outside the phase-change range. The time interval during which the internal temperature of the thermochamber was changed varied. A stabilization time of 4 h before and after heating and cooling from 10 °C to 35 °C (or 35 °C to 10 °C) was used to stabilize the internal temperature of the thermochamber and the temperature of the PCM sample. Using the thermocouple installed on the PCM sample and the measured values of the surface temperature and heat transfer from the heat flow meter, the specific heat of the PCM was calculated according to the heating and cooling times, as shown in Equation (1):

$$c = \frac{Q \cdot \Delta t}{M \cdot \Delta \theta} \quad (1)$$

where c is the specific heat capacity (J/g·K), Q is the heat flow on the surface of the PCM (W), M is the weight of the PCM (g), $\Delta \theta$ is the temperature change (K), and Δt is the time taken for $\Delta \theta$ to occur (s).

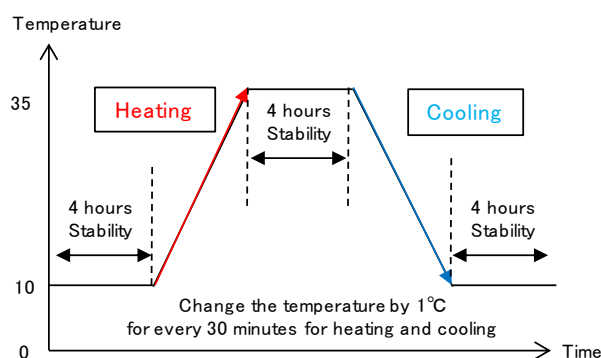


Figure 3. Temperature control of the thermochamber.

3. Specific Heat Measurement of PCM

3.1. Adjustment of Measured Value of Thermochamber

An acrylic plate with a constant specific heat was used to measure the accuracies of the thermochamber and sensors used in the PCM specific heat measurement experiment. The acrylic plate was created by stacking two sheets. Figure 4 depicts the basic measurement settings, such as the internal temperature of the thermochamber, and the top and bottom surface temperatures, heat flow, and internal temperature of the sample. The test was conducted by heating the chamber in increments of 1 °C at intervals of 20 min from 10 °C to 35 °C, followed by a stabilization time of 4 h at 35 °C. The chamber was then cooled in decrements of 1 °C at intervals of 20 min from 35 °C to 10 °C, followed by a 4-h stabilization time at 10 °C, concluding the measurement. To improve the accuracy of the experimental results, a specific heat between 15 °C and 25 °C was used as the measurement result, and the temperatures at the beginning and end of the heating and cooling processes were excluded. According to the measurement results, the specific heat of the acrylic plate was 1.43 J/g·K when heated and 1.44 J/g·K when cooled. Therefore, the accuracy was approximately 97% since the theoretical value is 1.47 J/g·K, which is a basic property of the material. Thus, we confirmed the reliability of the experimental devices, such as the thermochamber, and the experimental settings for the specific heat measurement.

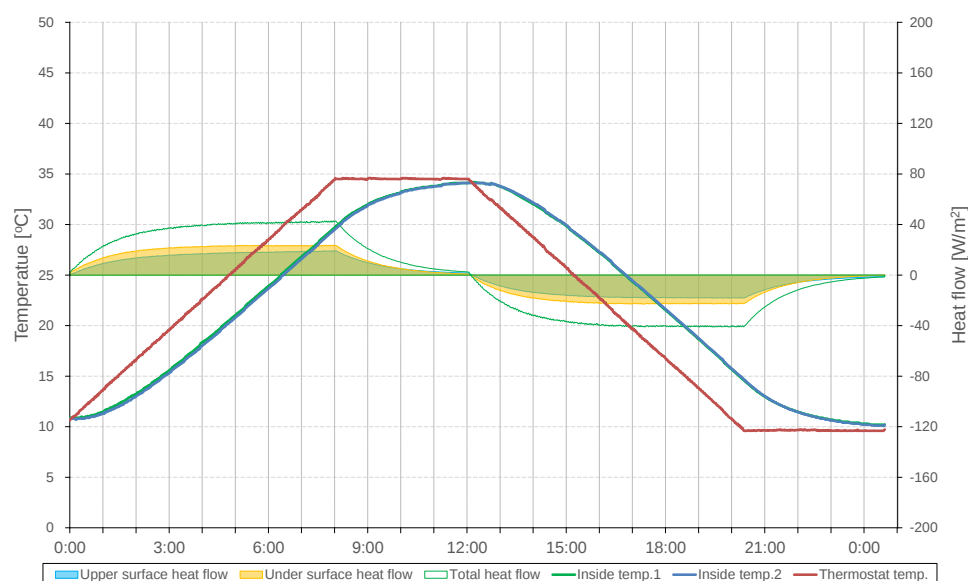


Figure 4. Temperature and heat flow data for the acrylic panel.

3.2. Specific Heat Measurement of PCM Using Dynamic Method

The specific heat measurement of a PCM depends on the method of controlling the temperature in the thermochamber and the method of using the internal temperature of the sample. We reviewed the temperature control times of the thermochamber suitable for measuring the specific heat of the PCM and methods of using the internal temperature of the PCM sample. In addition, for each experimental method, a means to improve the accuracy of the thermal performance measurement of the PCM was identified.

3.2.1. Temperature Change Setting Time of Thermochamber

Examples of specific heat measurement methods by controlling the temperature of the thermochamber are the dynamic method and the step method [22]. In the dynamic method, the specific heat is measured while heating or cooling the interior of the thermochamber by a fixed temperature increment at regular time intervals, as illustrated in Figure 4. In the step method, the temperature of the thermochamber is changed as much as the set temperature; there is a stabilization period after each temperature change, and the heating or cooling is conducted in a stepwise manner. Although the accuracy of the dynamic method is slightly inferior to that of the step method, it has the advantage of significantly shortening the experiment time. Therefore, in this study, the measurements were conducted using the dynamic method. The heat absorbed during heating (melting process) and the amount of heat released during cooling (solidifying process) were measured using an SSPCM sample at 19–25 °C (Sample 1 in Table 1). During both heating and cooling in steps of 1 °C in the thermochamber, setting times of 12 s, 30 s, 1 min, 3 min, 5 min, 7 min, 10 min, 20 min, and 30 min were tested. When comparing the heat absorption and release according to the setting time, the error between the maximum and minimum values was found to be within 2%, as presented in Table 2. This is because regardless of the heating or cooling time, a stabilization time of 4 h elapsed after the temperature change was complete, allowing the internal temperature of the PCM to increase or decrease and reach the set temperature. Therefore, it was found that if a sufficient stabilization time elapsed before and after heating and cooling of the thermochamber, as in this experiment, similar amounts of heat absorption and heat release were observed regardless of the temperature change time of the dynamic method.

3.2.2. Methods of Measuring Internal Temperature of PCM

The internal temperature of a PCM sample is required to determine the specific heat and thermal performance of the PCM material. When measuring the specific heat of a

PCM, there are two methods of measuring the internal temperature of the sample: directly measuring the internal temperature of the sample and averaging the upper and lower surface temperatures. In this subsection, we compare the results for these two methods.

3.2.2.1. Use of Internal Temperature of PCM

When measuring the specific heat of a PCM, more accurate measurements can be achieved by installing a temperature sensor inside the sample. However, because it is difficult to install a sensor inside the PCM, unless planned in the manufacturing stage, for this study, we produced and measured several PCMs as a single sample. We prepared four sheets of SSPCM and measured the internal temperature by installing thermocouples in the center with two sheets on either side. Figure 5 depicts the specific heat measured based on the internal temperature of the SSPCM for various setting times. The setting times were 12 s, 30 s, 1 min, 3 min, 5 min, 7 min, 10 min, 20 min, and 30 min for a temperature change of 1 °C in the thermochamber, as described in Section 3.2.1. The specific heats measured at each temperature during heating and cooling for the various setting times were generally similar. However, differences in the peak temperature and specific heat were observed: a difference of approximately 2 °C in the peak temperature of 23.5–24.5 °C was observed depending on the setting time. The maximum value of the specific heat was 22.2 J/g·K at a peak temperature of 24.0 °C when the setting time was 10 min, whereas the minimum value was 17.1 J/g·K for a setting time of 3 min and a peak temperature of 24.5 °C. The difference between the two values was approximately 5.1 J/g·K (approximately 23%). During the cooling process, the peak temperature appeared to be constant at 24.0 °C regardless of the setting time. The maximum value of the specific heat was 26.3 J/g·K for a setting time of 3 min, and the minimum value was 18.5 J/g·K for a setting time of 1 min; both peaked at 24.0 °C. This indicates a difference of approximately 7.8 J/g·K (approximately 30%). Figure 6 depicts the enthalpy based on the internal temperature of the SSPCM for various setting times. As the enthalpy is based on the specific heat measurement result, the basic features and results are the same as those in Figure 5. The enthalpy was similar to the specific heat in the gradient and trend of the overall graph for the various setting times; the phase change occurred most actively between 24 and 26 °C, and the peak temperature was observed. Based on the overall results for the specific heat and enthalpy, we found that the temperature at the start and end of the phase-change section and the specific heat in the sensible heat section were significantly different when the setting time was less than 5 min. For stable specific heat measurement by the dynamic method using the PCM internal temperature, a setting time of at least 5 min is required when the internal temperature of the thermochamber varies in increments/decrements of 1 °C.

Table 2. Heat absorption and release according to set times of the dynamic method (SSPCM 19–25 °C).

Dynamic Method: Temp. Change per 1 °C	Heat Absorption [KJ]	Heat Release [KJ]
	19–25 °C SSPCM Sheet	
30 min	1126.2	−1090.2
20 min	1120.9	−1094.6
10 min	1115.2	−1095.1
7 min	1083.4	−1086.6
5 min	1102.2	−1086.9
3 min	1108.0	−1086.7
1 min	1097.2	−1092.7
30 s	1109.8	−1076.9
12 s	1086.5	−1086.0

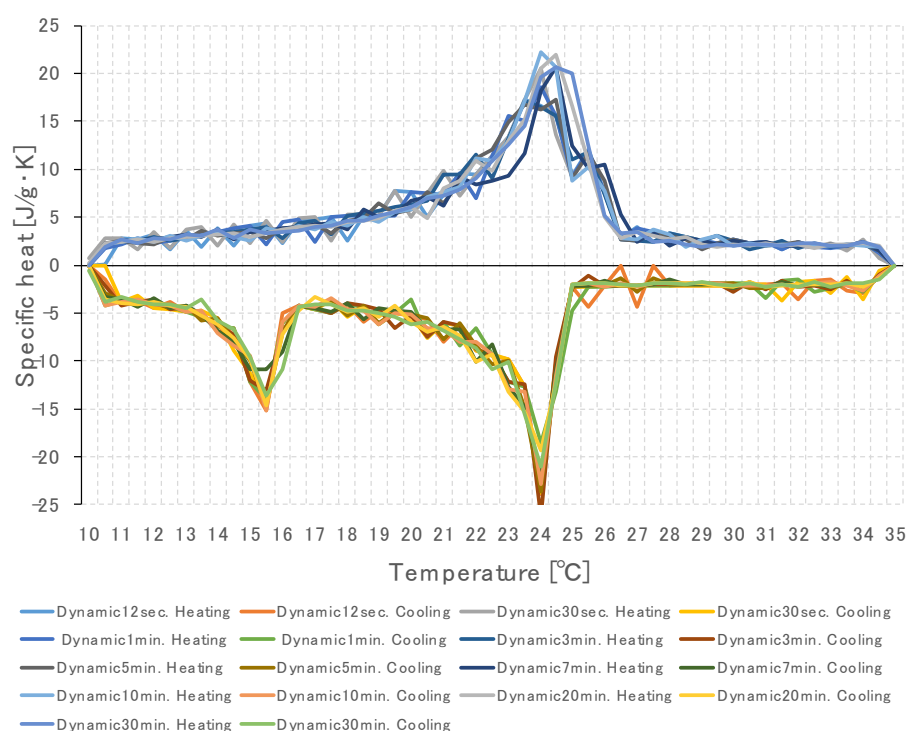


Figure 5. Specific heat determined using the internal temperature of the SSPCM (19–25 °C) for various setting times in the dynamic method.

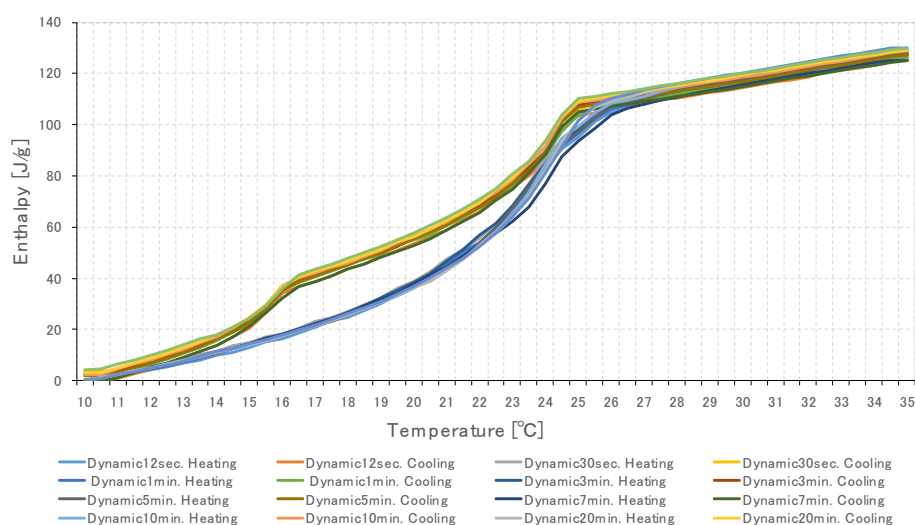


Figure 6. Enthalpy determined using the internal temperature of the SSPCM (19–25 °C) for various setting times in the dynamic method.

3.2.2.2. Use of Average Temperature of Upper and Lower Surfaces of PCM

The specific heat can be measured by using the average of the upper and lower surface temperatures of the PCM when there is only one sample or when the internal temperature cannot be measured. As for the experiment based on the internal temperature of the sample, four SSPCMs were stacked to create one sample, and thermocouples were installed on the upper and lower surfaces for measurement. Figure 7 depicts the specific heat determined based on the average of the upper and lower surface temperatures of the SSPCM for various setting times. The results indicated that the trends of the specific heat, start and end temperatures of the phase change, and peak temperature were significantly different for the different setting times of the dynamic method. In addition, in comparison with the results

obtained using the internal temperature of the sample in Section 3.2.2.1, the variation in the results due to the change in the setting time was more pronounced for this method. This implies that when the thermochamber is being heated, the surface temperature of the PCM first increases, and then the internal temperature increases. The same delay occurs during the cooling process. Therefore, the use of the upper and lower surface temperatures results in a delay in the phase-change section and the peak temperature of the PCM. This delay increased as the setting time was shortened from 30 min to 12 s.

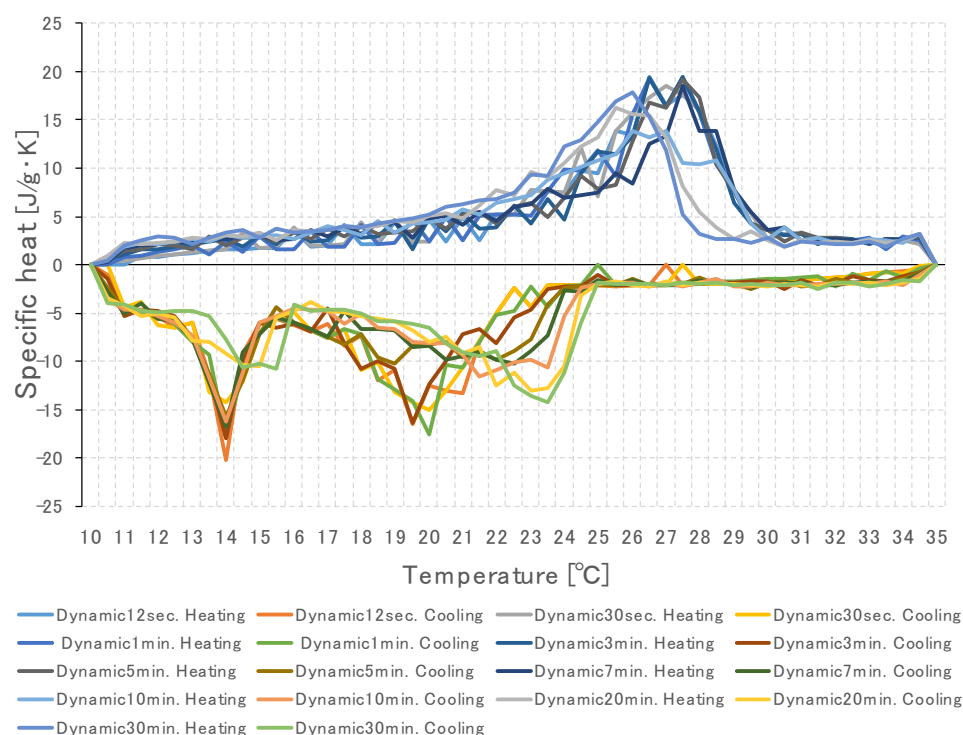


Figure 7. Specific heat determined using the average of the upper and lower surface temperatures of the SSPCM (19–25 °C) for various setting times in the dynamic method.

Figure 8 depicts the enthalpy determined using the upper and lower surface temperatures of the SSPCM for various setting times. As for the specific heat, the enthalpy results varied significantly depending on the setting time for both the heating and cooling processes. Specifically, as the temperature change setting time was reduced, the delays in the phase-change section and in the peak temperature increased. When measuring the specific heat by the dynamic method using the average of the upper and lower surface temperatures of the PCM, the accuracy decreased significantly as the setting time decreased. Therefore, accurate and stable measurements can only be achieved when the temperature change setting time is 30 min or more for both the heating and cooling processes.

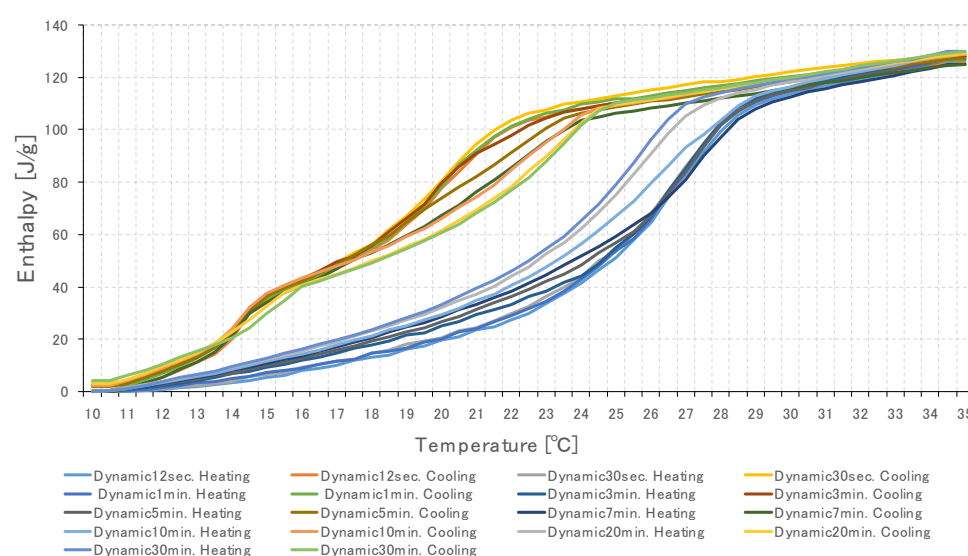


Figure 8. Enthalpy determined using the average of the upper and lower surface temperatures of the SSPCM (19–25 °C) for various setting times in the dynamic method.

3.3. Specific Heat Measurement According to PCM Capacity

The specific heat of the microencapsulated PCM-impregnated gypsum board was measured using setting times of 10, 20, and 30 min, which allowed for stable measurement via the dynamic method (Samples 2, 3, 4, and 5 in Table 1). The specific heats of the gypsum board without PCM and of the MPCM with three capacities of latent heat (11.5, 14.7, and 20.1 kJ/kg), all with phase changes at 25 °C, were measured. The MPCM used in this study was developed with different finishes on its two sides for indoor installation. Therefore, the average temperature of the upper and lower surfaces was considered as the internal temperature because the more accurate method of measuring the internal temperature by stacking PCM layers could not be used. Figure 9 depicts the specific heat obtained based on the MPCM capacity and the setting time used in the dynamic method. For each setting time, there were slight errors in the results; however, a similar overall trend could be observed. Moreover, the phase-change section and peak temperature appeared clearly when the MPCM capacity exceeded 14.7 kJ/kg.

Table 3 presents the heat absorption and release according to the setting time of the dynamic method and the MPCM capacity. The errors between the maximum and minimum values for the different setting times were within 2% in all samples. This is because a stabilization time of 4 h elapsed after the temperature change was complete, regardless of the heating or cooling time, as in the previous experiment. Hence, the accuracy of measuring the amount of heat absorbed and released according to the stabilization time was confirmed for both the SSPCM and MPCM. The heat absorption and heat dissipation according to the MPCM capacity was compared with that of the gypsum board without PCM. The heat absorbed during the heating process and the heat released during the cooling process increased by approximately 37% and 36% for 11.5 kJ/kg, 44% and 43% for 14.7 kJ/kg, and 56% and 57% for 20.1 kJ/kg. These results indicate that the PCM capacity does not increase in proportion to its thermal storage performance. Therefore, it is necessary to review the appropriate installation capacity for each PCM when using it as an indoor building material.

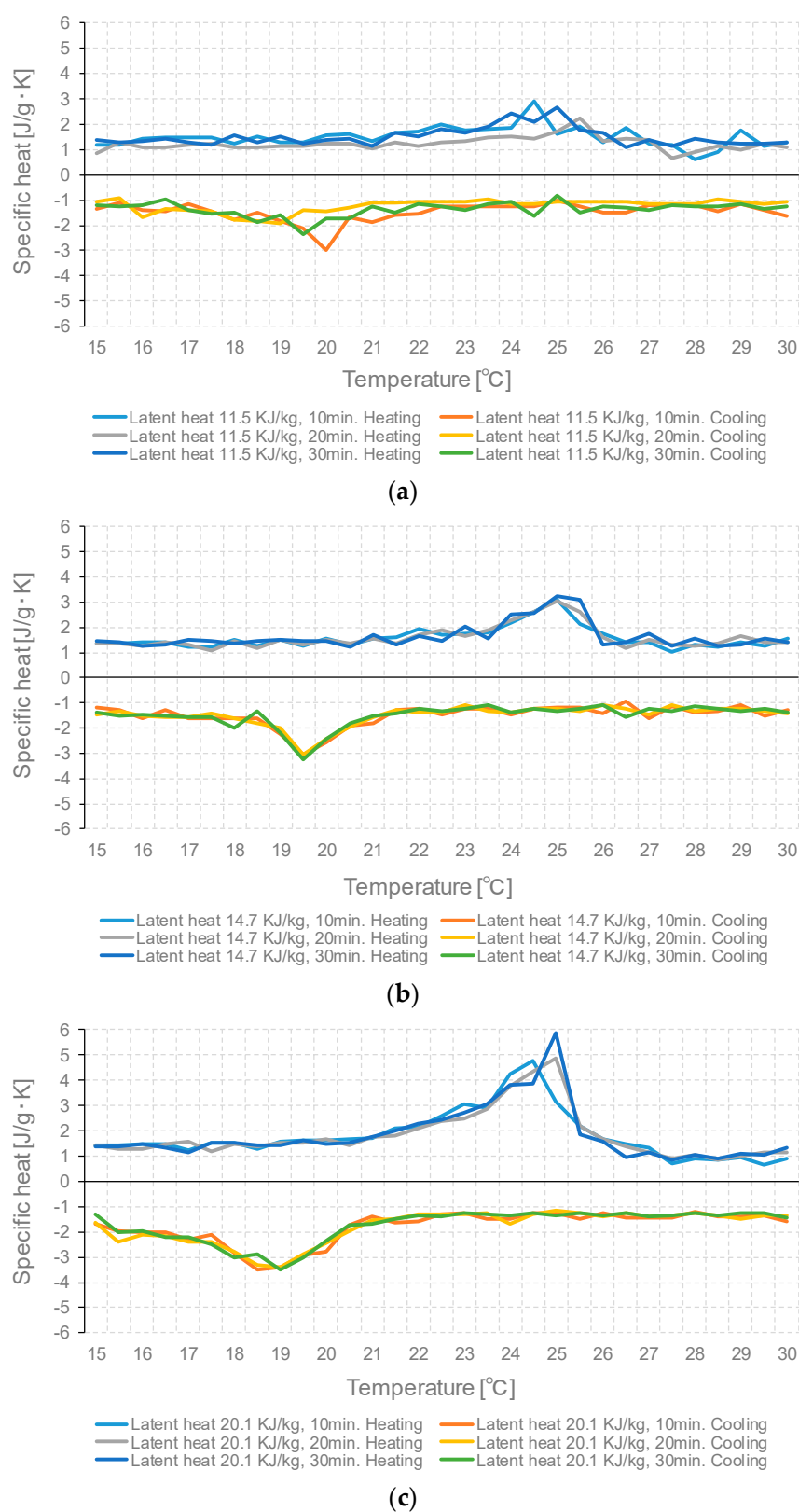


Figure 9. Specific heat according to the capacity of MPCM (25 °C) and setting times of the dynamic method. (a) Microencapsulated PCM impregnated gypsum board, latent heat 11.5 KJ/kg (10 min, 20 min, 30 min); (b) Microencapsulated PCM impregnated gypsum board, latent heat 14.7 KJ/kg (10 min, 20 min, 30 min); (c) Microencapsulated PCM impregnated gypsum board, latent heat 20.1 KJ/kg (10 min, 20 min, 30 min).

Table 3. Heat absorption and release according to the set times of the dynamic method and the capacity of PCM (MPCM 25 °C).

Dynamic Method: Temp. Change per 1 °C	Heat Absorption [KJ]	Heat Release [KJ]
Sample type	25 °C, 0 KJ/kg	
30 min	566.9	−528.8
20 min	560.6	−533.2
10 min	556.2	−538.2
Sample type	25 °C, 11.5 KJ/kg	
30 min	777.7	−716.5
20 min	764.4	−725.4
10 min	763.3	−735.5
Sample type	25 °C, 14.7 KJ/kg	
30 min	815.1	−751.9
20 min	802.7	−761.4
10 min	799.7	−774.5
Sample type	25 °C, 20.1 KJ/kg	
30 min	884.3	−833.1
20 min	869.7	−838.9
10 min	868.4	−847.0

4. Conclusions

The specific heat and enthalpy of an SSPCM and a microencapsulated PCM-impregnated gypsum board fabricated by combining a PCM building material with paraffin were measured under various conditions using the dynamic method. The main findings of this study are as follows.

The setting time of the dynamic method did not significantly affect the measurement of the latent heat of the PCM samples when a sufficient stabilization time elapsed before the heating and cooling processes in the thermochamber. The overall trends in the phase-change section and the peak temperature of the PCM were similar regardless of the setting time when the internal temperature of the PCM samples was used to determine the specific heat and enthalpy at each temperature. However, when the average of the upper and lower surface temperatures of the PCM sample was considered, large errors were observed owing to the delay in the phase change, which could be attributed to the difference between the surface and internal temperatures of the PCM. This delay increased as the setting time decreased from 30 min to 12 s. The capacity of the PCM did not increase in proportion to the thermal storage performance according to the measurements of the latent heat performance according to the capacity of the PCM. Therefore, it is necessary to review the appropriate installation capacity of a PCM to improve its efficiency when combined with building materials and applied indoors.

This study examined methods for improving the accuracy of specific heat and enthalpy measurements of PCM building materials when using a thermochamber without DSC equipment. It is expected that the measurement time and accuracy of the measured thermal performance of newly produced samples can be improved by testing a greater variety of samples in subsequent research and by reviewing the appropriate heating, cooling, and stabilization time settings.

Author Contributions: Conceptualization, H.B.K.; methodology, H.B.K.; validation, H.B.K. and Y.C.; formal analysis, H.B.K. and Y.C.; investigation, H.B.K.; data curation, H.B.K.; writing original draft preparation, H.B.K.; visualization, H.B.K.; supervision, M.M.; project administration, M.M.; funding acquisition, Y.C. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIT) (No. 2020-0298).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: No data is available for this work.

Conflicts of Interest: The authors declare no conflict of interest.

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