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Thermal Performance Evaluation of a PCM-Integrated Gypsum Plaster Board †

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Abstract: In order to design and optimize building materials containing phase changing material (PCM), it is important to accurately characterize the thermal properties of these composites: the enthalpy curve and its hysteresis. This paper presents the initial tests of these properties for a composite panel: gypsum plasterboard with an evenly distributed layer of PCM. Performance testing of the selected material was carried out by means of the dynamic method. This method (dynamic heat flow meter apparatus (DHFMA)) involves the measurement of non-stationary heat flow to determine the stored energy (enthalpy change) as a function of temperature using a stationary heat flow measurement apparatus (HFMA). This method allows for the measurement of the sensible and latent heat capacity of the products containing phase change material. In addition to presenting the obtained results, the study will discuss the practical aspects of this test method, recently introduced in the standard ASTM C 1784-20. The preliminary experiments described in the article were aimed at learning a new research technique, determining the required conditions for conducting research and the capabilities of the possessed apparatus in this regard.

Keywords: phase change material; PCM; dynamic thermal properties; differential scanning calorimetry; DSC method; DHFMA method

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1. Introduction

Modern buildings are characterized by significantly improved thermal insulation and the other solutions aimed at minimizing energy demand. A frequently used techno- logy for erecting buildings is a technology based on durable structural elements forming the skeleton filled with thermal insulation. Heat transfer through such partitions is minimized, but the disadvantage of this technology is the low thermal mass. In addition, oversized glazing often causes a disproportion between the solar gains and the limited thermal capacity of the building. The inability to counteract significant temperature fluctuations in the rooms has a very negative effect on the microclimate and the feelings of people staying in them, as well as on the efficiency of solar energy use.

The ability to store excess heat energy and release it when the room cools down results in the better use of solar radiation in the winter and reduces the risk of space overheating in the winter and summer. The occurrence of a too-high temperature is usually connected with the use of expensive and energy-intensive air-conditioning equipment. In low energy buildings, the ability to store energy becomes particularly important.

The lack of a time coincidence of the demand and possible supply of energy gains contribute to the development of research on heat and cold storage. One of the effective ways of passive accumulation of thermal energy in buildings is the use of materials that change the state of aggregation, usually marked with the abbreviation PCM (phase changing material).

During the change of aggregation phase, a large amount of heat is accumulated or released. Particularly in buildings erected using light construction technologies, the application of additional energy storage in the form of PCM proves to be very effective [1]. PCM can contribute to reducing the peak demand on cooling and allow for more even operation of the air conditioning system, also reducing the need for heating at night. Thanks to the use of PCM, it is possible to avoid or at least reduce the currently frequent effects of the overheating of buildings with excessive glazing [1,2].

Phase change materials offered for building use can take the form of:

- Microcapsules added to plasters and mortars, concrete screeds or plasterboards;
- Small containers in the form of flexible mats with cavities filled with Bio-PCM or aluminum sheet plates filled with phase change material (Dupont);
- Fibrous insulation with the addition of PCM in the form of capsules [3–5].

This article presents the results of preliminary tests of the thermal capacity of a plasterboard integrated with PCM. The purpose of these studies was to test a new research technique, the necessary conditions for conducting research and a completely different than designed application of the FOX 314 apparatus for measuring the thermal conductivity of building materials. The apparatus and its original software were designed to conduct tests in stationary conditions, while thermal capacity tests require dynamic conditions.

2. Measurement Methods of PCM Thermal Characteristics

The proper selection of the PCM type and its quantity requires the precise experimental determination of the thermal characteristics of heterogeneous products.

Differential scanning calorimetry (DSC) is one of the most widely used PCM measurement methods because of the ease with which different thermodynamic data can be obtained. DSC measures the amount of heat needed to raise the temperature of the test sample in a given temperature range [4]. On this basis, the phase change temperature, enthalpy, heat capacity and specific heat can be determined. However, the DSC method is applicable to millimeter-scale samples with weight in the order of a few milligrams. The DSC method also requires relatively homogeneous samples. Due to the large heterogeneity of composites on a small-scale sample, determining the average PCM content in a mass-produced product requires many tests, which is troublesome and expensive, and may be subject to a large error of sample randomness [3]. In the case of composite materials (PCM—enhanced building products), with unevenly distributed PCM in the structure, testing according to the DSC method does not make sense.

In search of a possibly fast and at the same time precise method that can be used in determining the thermodynamic parameters of large samples of PCM-reinforced building components, a dynamic method using a plate apparatus was developed.

In the Oak Ridge National Laboratory, a dynamic method using a plate apparatus was developed to measure the content of phase-change material in composite samples [6,7]. The plate apparatus is basically used to measure the thermal conductivity of materials in a steady state of heat conduction. However, it can also be used to measure the dynamic thermal properties of samples of the tested materials, in which the heat flux stabilization is relatively slow due to the ongoing phase change. The developed method is referred to as dynamic HFMA or DHFMA. Since the beginning of its development, the DHFMA method has been subject to constant modifications. The theory of the method and test procedure has been described by the creators of the method [8].

In an isothermal process such as a phase transition, the enthalpy change (ΔH) is equal to the amount of heat absorbed (heat input) or released (heat output) during the process (ΔQ). The heat capacity (c_p) is the temperature derivative of the enthalpy (H), i.e., $c_p = dH/dT$.

Assuming there is a linear relationship between enthalpy and temperature for small temperature increments (ΔT):

$$c_p = dH/dT \approx \Delta H/\Delta T = \Delta Q/\Delta T, \quad (1)$$

where:

ΔH—enthalpy change [J/m^2];

ΔQ—energy amount absorbed or released during a given process [J/m^2];

ΔT—temperature increase [K].

In the DHFMA method, the temperature of the sample is changed in small steps and the resulting heat flow into or out of the sample is measured during this process. The heat capacity at an average temperature is then determined using Equation (1). The DHFMA method uses a conventional HFMA instrument containing at least one heat flow sensor on each of the isothermal plates. The sample is placed parallel to the plates while the sides are thermally insulated to achieve near-adiabatic conditions. Both isothermal plates are kept at the same temperature. A temperature step is then applied to both plates and the heat flows through the plates are measured until a thermal equilibrium is reached where the heat flow values become negligible. In the case of a step change in temperature, the change in enthalpy per unit area of the sample is calculated by integrating the heat flow rates over time (2):

$$\Delta H = \Delta Q = \sum[\{(qU_i - qU_{final}) + (qL_i - qL_{final})\}\tau] \quad (2)$$

where:

qU_i—heat flux flowing through the upper plate, recorded at time intervals τ [W/m^2];

qL_i—heat flux flowing through the bottom plate, recorded at time intervals τ [W/m^2];

qU_{final}—the value of the remaining heat flux of the upper plate, caused by side heat losses in the equilibrium state [W/m^2];

qL_{final}—the value of the remaining heat flux of the lower plate, caused by side heat losses in the equilibrium state [W/m^2];

τ—time step between readings [s].

The volumetric heat capacity of the PCM-enhanced component is determined as follows (3):

$$c_V = (1/l)\cdot(\Delta Q/\Delta T), \quad (3)$$

where l is the thickness of the sample. It should be noted that the DHFMA method can be used to measure the heat capacity of any solid or liquid material. Recently, ASTM introduced the test standard C1784 for measuring the thermal storage properties of phase change materials and products, based on the DHFMA method [9].

3. Thermal Capacity Testing of Micronal® PCM SmartBoard™ 23

The composite material Micronal® PCM SmartBoard™ 23, in the form of a plasterboard with the addition of a phase change material (approximately 3 kg of dry Micronal per 1 m^2 of the board), was tested. According to the authors of [10], this microencapsulated paraffin avoids leakage during the liquid phase and makes heat transfer easier by increasing the contact area. The authors of [10] analyzed gypsum boards with a mass PCM content up to 45% [11].

3.1. DSC Test

Before starting the dynamic DHFMA testing of the building material, thanks to the kindness of Netzsch, it was possible to perform a calorimetric study of the Micronal itself. It is an organic material which, according to the manufacturer's data, has a melting point of 23 °C. PCM makes up about 30% of the weight of the SmartBoard panel. The phase change material added to the plasterboard matrix is in the form of microcapsules (with a diameter of 2–20 μm) filled with paraffin.

The thermal characteristics of the PCM materials used, obtained as a result of DSC tests, are shown in Figure 1. The tests performed by Netzsch were carried out in the DSC 214 Polyma calorimeter with an aluminum melting pot, for the range from −50 °C to +50 °C. Calorimetric studies allowed the value of the heat of the phase change to be determined: PCM 23: 127.7 J/g. This value is similar to the properties of PCM described in article [9], where Micronal with a heat of fusion of 110 J/g was used and was significantly lower than the paraffin presented in [11] of 151 J/g. The phase transition range is a wide

temperature range from 17.8 °C to 31.5 °C, practically identical in the case of heating and cooling of the material. However, the peak heat flux was achieved at 25.1 °C, not 23 °C. During the DSC test, there was no sub-cooling effect, but there was a noticeable difference between the heat capacity in the entire heating and solidification process.

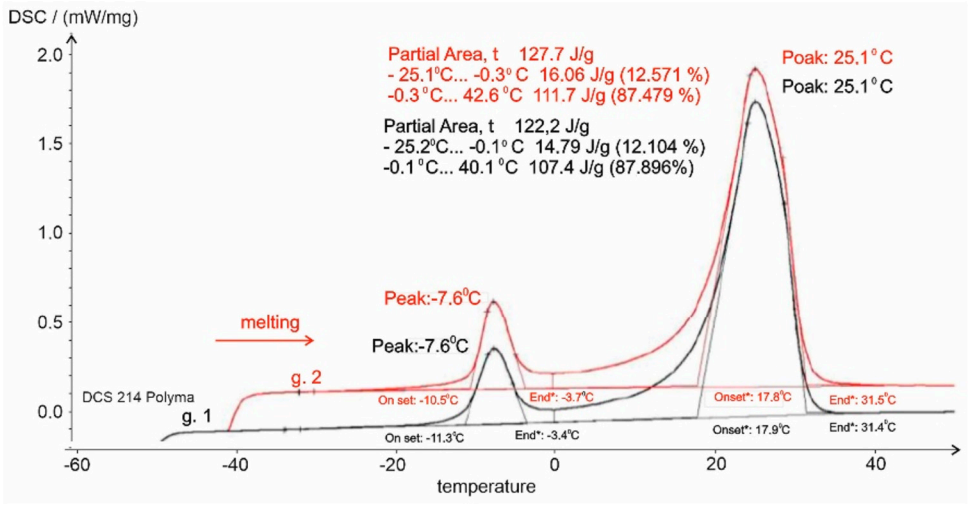

Figure 1. Results of DSC test.

3.2. FOX 314 Tests

The equipment used for dynamic tests of thermal conductivity and heat capacity is a device called FOX 314, manufactured by LaserComp USA, serial number: 1043. The design of the FOX 314 device is based on the method of measuring thermal resistance using heat flux sensors in stationary conditions and is compatible with the following standards:

- EN ISO 8301:1998: Thermal insulation, determination of steady-state thermal resistance and related properties and heat flow meter apparatus;
- EN 12667:2002: Thermal performance of building materials and products, determination of thermal resistance by means of guarded hot plate and heat flow meter methods and products of high and medium thermal resistance;
- ASTM C518-91: Standard test method for steady-state heat flux, and measurements and thermal transmission properties by means of the heat flow meter apparatus.

The characteristics of the sample tested in the FOX 314 apparatus are:

geometrical dimensions: 300 × 301 × 14.8 mm; volume: 0.001336 m^3; weight: 1089.36 g; density: 815.12 kg/m^3.

3.2.1. Thermal Conductivity Tests

The values of the thermal conductivity coefficient shown in Table 1 were tested for two sets of temperature differences:

- 0 °C to 20 °C, the average temperature of the sample during this test equal to 10 °C, corresponds to the condition of full PCM solidification;
- In the range of 10 °C to 30 °C, partial liquefaction of the PCM in the cross-section of the plate should be expected. Such average conditions occur in reality during the summer period in Polish climatic conditions.

Table 1. Thermal conductivity coefficients of Micronal® PCM SmartBoard™ 23 plasterboard in two temperature ranges: from 0 to 20 °C and from 10 to 30 °C.

T_1/T_2, [°C]	λ, [W/mK]
0/20	0.1658
10/30	0.1651

3.2.2. Thermal Capacity Testing

Due to the results of the Micronal tests obtained by the DSC method, a very wide range of temperatures in the DHFMA tests of the plate containing this material was adopted. Since this type of research is extremely time-consuming, a temperature change step of 2K was assumed for the preliminary testing.

The heating stage started at 10 °C and finished at 32 °C. The cooling of the material was carried out immediately after the heating stage, so the first measurement value obtained after reversing the process was the temperature of 30 °C, and the last one was 8 °C. Table 2 shows the temperature values that were actually obtained in the apparatus during the measurements and mass heat capacity. Temperature values given in Table 2 differ insignificantly from the measurement conditions set in the FOX 314 apparatus. Figure 2 and Table 2 summarize the results of the measurement obtained during the heating stage, while Figure 3 and Table 3 summarize the results during the cooling stage.

Table 2. Mass thermal capacity of Micronal® PCM SmartBoard™ 23, melting stage: 10–32 °C.

Melting	
Temperature [°C]	**c_p [J/kgK]**
10.01	1518.801
12.01	1544.036
14.01	1553.112
16.03	1581.835
18.01	1707.86
20.02	1970.278
22.02	3789.865
24.02	8795.741
26.01	1380.011
28.01	1384.403
30.02	1396.335
32.02	1401.394

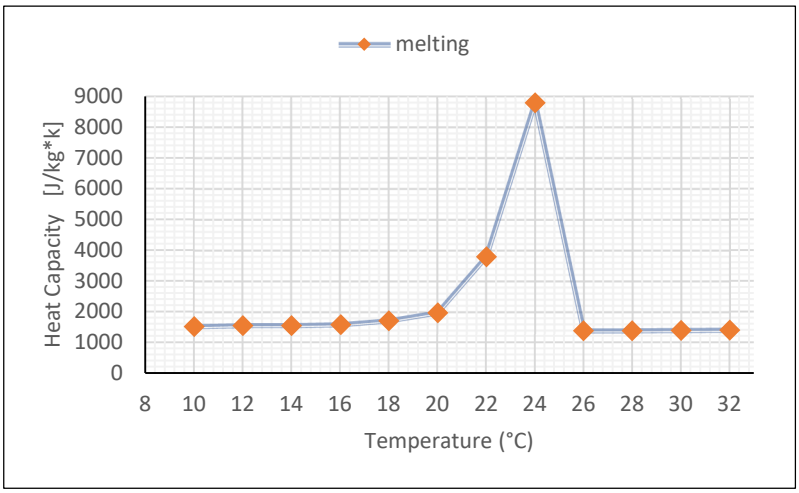

Figure 2. Mass thermal capacity of Micronal® PCM SmartBoard™ 23, melting stage: 10–32 °C.

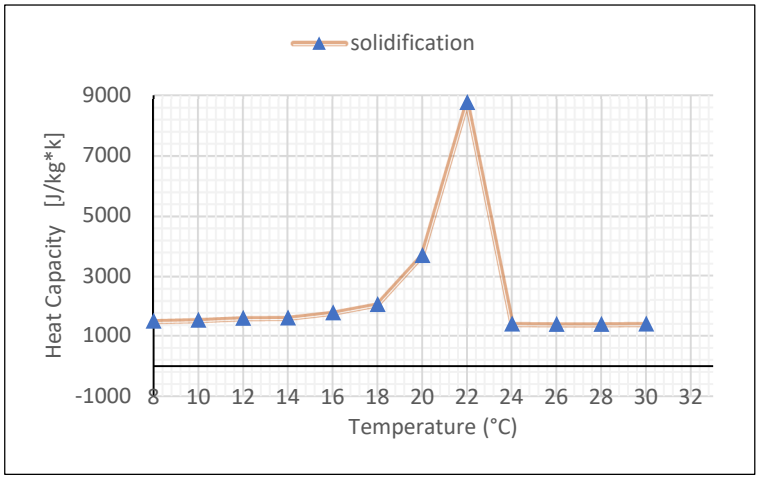

Figure 3. Mass thermal capacity of Micronal® PCM SmartBoard™ 23, cooling stage: 30–8 °C.

Table 3. Mass thermal capacity of Micronal® PCM SmartBoard™ 23, cooling stage: 30–8 °C.

Solidification	
Temperature [°C]	**c_p [J/kgK]**
8	1500.192
10.01	1537.904
12.01	1596.831
14.01	1607.684
16.03	1778.953
18.01	2060.286
20.02	3689.62
22.02	8771.898
24.02	1412.638
26.01	1396.558
28.01	1399.615
30.02	1405.134

Based on the measurement in the FOX apparatus, the volumetric heat capacity [J/m^3·K] was obtained. On this basis, after conversion, the value of the mass heat capacity [J/kg·K] was calculated

The apparatus used for the tests described here does not have specialized or very expensive software for dynamic tests. Therefore, obtaining the results was an extremely time- and labor-intensive process. It was necessary to manually control the course of the research and observe the process of stabilization after changing the boundary conditions. The measurement step of the apparatus is approximately 0.7 s. The tested sample is a relatively thin gypsum board, for which the average stabilization time was close to 4 h. For this period, almost 21,000 measurement records were obtained, which had to be further copied and processed. With 12 measurement steps used in the research, the calculation sheet counted over 220,000 records for only one series of tests.

Figure 4 shows the combined results obtained during the heating and cooling of the Micronal® PCM SmartBoard™ 23 in one graph.

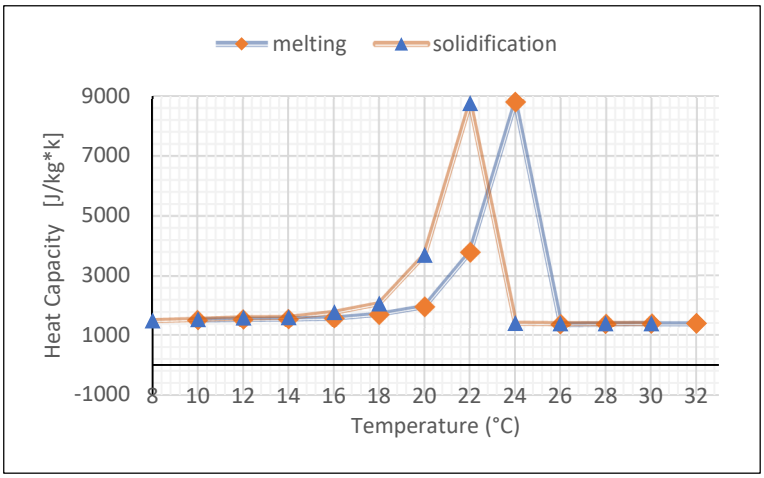

Figure 4. Thermal capacity of Micronal® PCM SmartBoard™ 23 for melting and cooling stages.

The temperature range in which the phase activity of the material can be observed starts at 16 °C and extends up to 26 °C. Due to the long duration and preliminary nature of the tests, a large (2K) step of temperature changes over time was adopted, and unfortunately, the stages of heating and cooling were carried out for the same temperature values, without any shift. Thus, the results and graphs obtained in this way are approximates only; they do not allow a reproduction of the real shape of the enthalpy curve or to clearly specify the phase transition temperature. Even with a 2K step, offsetting the heating and cooling cycles by 1K would yield additional information. According to the ASTM guidelines [9], the step of temperature changes can be as small as 0.5K. However, in order to maintain the accuracy of the measurement, several separate measurement series should be carried out for this purpose with a larger step, but mutually shifted by 1K or 0.5K.

The preliminary results of the PCM tests based on a new procedure and the non-standard use of the FOX 314 apparatus allowed estimations that the phase change in the tested material takes place close to the temperature declared by the manufacturer. The maximum heat capacity of a gypsum board with PCM was 8772 J/kgK, and in terms of volumetric capacity, it was 7.15 MJ/m^{3}K. The authors of the article [8] obtained, in the case of a gypsum board containing 25% paraffin-based PCM, ca. 6 MJ/m^{3}K. The heat capacity values of the tested material during melting and solidification were not identical, showing the hysteresis effect. Solidification took place at a lower temperature than melting, while the maximum heat capacity values obtained were almost identical. Both observations differ from the characteristics of Micronal itself, obtained from the DSC study. The material containing PCM already in the liquid phase has a lower heat capacity than in the solid phase. A similar result was obtained by the authors of [8].

4. Conclusions

The conducted pilot tests of the thermal characteristics of the phase change material allow the following conclusions:

- Using the ordinary FOX 314 apparatus, designed to measure thermal conductivity in stationary conditions, it is possible to conduct dynamic enthalpy studies of PCM;
- Research conducted for a wide range of temperatures is very time-consuming, and without a special software, extending the apparatus application, which is also very laborious, requires the processing of hundreds of thousands of measurement data;
- The measured maximum heat capacity of the plate during the phase transformation, equal to 8772 J/kgK, is close to the results reported in the literature. However, the adopted step change of the test temperature every 2K was too large to faithfully reproduce the thermal properties of the sample and enable detailed comparisons;

- The described dynamic method gives the opportunity to test large samples of composite building materials with non-homogeneous PCM distribution. Such data cannot be obtained by means of DSC method.

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References

1. Berardi, U.; Manca, M. The energy saving and indoor comfort improvements with latent thermal energy storage in building retrofits in Canada, 8th Int. Conference on Sustainability in Energy and Buildings, Turin. *Energy Procedia* **2017**, *111*, 462–471. [CrossRef]
2. Soudian, S.; Berardi, U. Assessing the effect of night ventilation on PCM performance in high-rise residential buildings. *J. Build. Phys.* **2019**, *43*, 229–249. [CrossRef]
3. Kossecka, E.; Kośny, J. Dynamiczna metoda pomiaru zawartości materiału fazowo-zmiennego w izolacji włóknistej. *Fiz. Budowli Teor. Prakt.* **2009**, *IV*, 109–112.
4. Kośny, J. *PCM- Enhanced Building Components: An application of Phase Change Materials in Building Envelopes and Internal Structures*; Springer International Publishing: Cham, Switzerland, 2015. [CrossRef]
5. Zastawna-Rumin, A. The Analysis of the Appliaction Efficiency of Phase Change Materials in Partitions in Polish Low-Energy Buildings. Doctoral Thesis, Cracow University of Technology, Cracow, Poland, 2018.
6. Kośny, J.; Yarbrough, D.W.; Miller, W.A.; Wilkes, K.E.; Lee, E.S. Analysis of the dynamic thermal performance of fibrous insulations containing phase change materials. In Proceedings of the 11th International Conference on Thermal Energy Storage, Effstock 2009—Thermal Energy Storage for Energy Efficiency and Sustainability, Stockholm, Sweden, 14–17 June 2009.
7. Kośny, J.; Yarbrough, D.W.; Kossecka, E. Use of a Heat-Flow Meter to Determine Active PCM Content in an Insulation. In Proceedings of the 30th International Thermal Conductivity Conference, Pittsburgh, PA, USA, 29 August–2 September 2009.
8. Shukla, N.; Kosny, J. DHFMA Method for Dynamic Thermal Property Measurement of PCM-integrated Building Materials. *Curr Sustain. Renew. Energy Rep.* **2015**, *2*, 41–46. [CrossRef]
9. *ASTM C174-20*; Standard Test Method for Using a Heat Flow Meter Apparatus for Measuring Thermal Storage Properties of Phase Change Materials and Products. ASTM: West Conshohocken, PA, USA, 2014.
10. Oliver, A. Thermal characterization of gypsum boards with PCM included: Thermal energy storage in buildings through latent heat. *Energy Build.* **2012**, *48*, 1–7. [CrossRef]
11. Sharifi, N.P.; Shaikh, A.A.N.; Sakulich, A.R. Application of phase change materials in gypsum boards to meet building energy conservation goals. *Energy Build.* **2017**, *138*, 455–467. [CrossRef]