Thermal Performance of Lignocellulose's By-Product Wallboards with Bio-Based Microencapsulated Phase Change Materials

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Abstract: The growing availability and decreasing cost of microencapsulated phase change materials (PCMs) present an opportunity to develop innovative insulation materials for latent heat energy storage. By integrating PCMs with traditional insulation materials, it is possible to enhance the thermal capacity of a building by up to 2.5-times, virtually without increasing the building's mass. To improve buildings' indoor structural performance, as well as improving their energy performance, microencapsulated PCMs are integrated into wallboards. The integration of microencapsulated PCMs into the wallboard solves the PCM leakage problem and assures a good bond with the building materials to achieve better structural performance. The novelty of this research is the application of encapsulated phase change material dispersion and technology for its incorporation into the structure of hemp shives and longitudinally milled wood chip-based insulation boards, using cold pressing technology to reduce the energy consumption of board production. As a result, low-density insulation boards for indoor application were produced by varying their structure and the amount of phase change materials in the range of 5% to 15% by board mass. The obtained board prototypes can be used as microclimate and thermoregulation elements of interiors, as well as functional aesthetic elements of interior design.

Keywords: encapsuled phase change material; renewable resources; thermal conductivity; heat capacity; phase transition heat; latent heat; hemp shives; longitudinally milled wood chips

1. Introduction

The Intergovernmental Panel on Climate Change predicts a 6 °C rise in the average global temperature by 2050 [1]. In light of this, it is necessary to find more efficient ways to reduce energy consumption in the building sector. The rapid rise in global energy consumption has raised concerns because of the increased dependency on natural resources and the negative environmental impacts. The building sector is the largest energy consumption sector, accounting for over one-third of the total energy consumption, and is an important source of carbon dioxide emissions [2,3]. In addition to population growth, improved building services, indoor comfort levels and the increased time spent inside buildings have dramatically increased energy consumption in the building sector [4]. More and more often, in their research, scientists investigate the connections of different, non-traditional materials when making insulation board materials, in order to find new solutions. This research implements the principles of sustainability, creating increasingly excellent solutions without harming the environment [5,6]. The vital topic is the production of board insulation materials, with an emphasis on the efficient use of local production by-products and rapidly renewable natural resources [7]. Lignocellulosic materials are made from material particles/fractions of different sizes and orientations, varying different glues, glue volumes and pressing technologies, obtaining boards of different thicknesses and

densities [8,9]. One of the very important aims is to achieve good specific heat capacity characteristics using innovative technologies for high efficiency, for example, by adding microencapsulated PCM into the composition [10–13].

Research on phase change materials (PCMs) has gained attention since the 1940s, especially during the energy crisis in the late 1970s and early 1980s [14–16]. Phase change materials can be classified into two groups that are mainly used in buildings: solid–liquid and solid–solid states [17]. Only solid–liquid phase PCMs are used in materials, especially for integration into wall panels, and are available on the market with a wide range of phase change temperatures [18]. With PCM, it is possible to innovate traditional insulation materials to store and return latent heat energy, using a phase change material supplemented with microcapsules and incorporating it into the insulation materials of various structures.

By including hemp shives and longitudinally milled wood chips in thermal insulation materials with PCM, the thermal inertia of the building can be increased without enlarging the mass of the building [19]. To provide the same heat capacity as a 10 mm thick PCM layer, a 110 mm thick brick wall is required [20–22]. Based on the great potential of using latent heat, it can offer a possible new technological development solution for the autoregulation of indoor microclimates [23–29]. PCM can be incorporated into both lightweight [30–33] and construction materials [34–37].

When such a material solidifies, it releases a large amount of energy, in the form of latent heat, from the energy of crystallization; vice versa, when the material is melted, the same amount of energy is absorbed from the surrounding environment when changing from a solid to a liquid consistency (Figure 1) [38–42]. To be protected from the environment, PCMs need to be encapsulated. This also prevents any leakage when the PCM is in the liquid state. Microencapsulation has become a common method to protect PCMs and, thus, to enable their incorporation in various components [43].
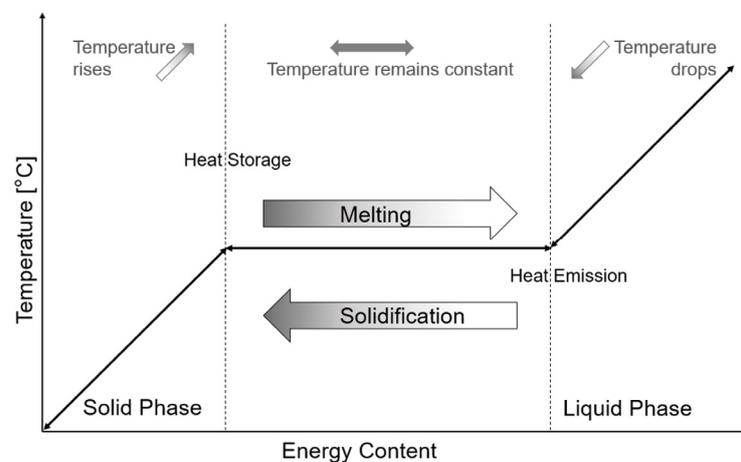

Figure 1. PCM operating principle.

Impregnation [44,45] and immersion [46] are the simplest and most cost-effective methods, requiring very few additional processes. When blending liquid or powdered PCM with construction materials, challenges like leakage and material incompatibility may arise. Researchers address this by integrating PCM into microstructural support materials, creating a shape-stable microencapsulated composite. This approach enables precise control over the amount of conventional PCM produced [47].

Plant-based organic PCMs are used in this study as a renewable and environmentally friendly alternative with better fire resistance to paraffin PCM, which offers advantages such as non-toxicity and recyclability for thousands of cycles without material degradation. These hydrogenated hydrocarbons possess a saturated electronic configuration, ensuring chemical stability, non-corrosiveness and potential for long-term use. Nonetheless, bio-based PCM may present some limitations, including a restricted temperature range, lim-

itations in energy storage due to low thermal conductivity, limited commercial availability and lower thermal conductivity [48–52].

Inspired by existing research on the use of PCMs, the relevance of new prototypes is based on the great potential of using latent heat by integrating microencapsulated phase change dispersion into insulating hemp by-product boards. The objective of this work is to carry out complex research to improve the capacity of hemp by-product-based interior insulation boards by integrating microencapsulated biological-based phase change materials into their composition for the intensification of functional peculiarities.

2. Materials and Methods

2.1. Materials

The following materials were used:

Hemp shives—a by-product of the hemp fiber variety Białobrzeskie grown in Latvia. Pine longitudinal milling chips—a by-product of furniture production and carpentry. Urea-formaldehyde resin adhesive (UF) in powder form with hardener for cold pressing.

The phase change material microcapsule (PCM) MikroCaps Ltd. (Ljubljana, Slovenia) products were selected and incorporated directly into the insulation wallboard material prototypes. According to the datasheet of MikroCaps Ltd. (Ljubljana, Slovenia), PCM-S50 has a melting temperature of 23–28 °C, an average microcapsule particle size of 1–15 μm and a bio-based type of PCM.

The experimental insulation materials were made from hemp shives and wood longitudinal milling chips blending directly into the mass 5%, 10% and 15% of encapsulated PCM dispersion. The aim of this work was to create a temperature-stabilizing insulation material by integrating into it a dispersion containing microencapsulated phase change material to increase the specific heat capacity, thermal inertia and thermal conductivity. The experimental samples were made as single-layer 25 mm thick boards with a density of 290 ± 20 kg/m^3, which qualifies as low-density boards.

Material manufacturing composition: The amount of binder added was the same in all samples at 10% calculated on an absolute dry basis (from H or HW).

The sample variable included only the phase change material (PCM). The phase change material was calculated in the same way, on an absolute dry basis (from H or HW), giving 5%, 10% and 15% added PCM.

The average density results in the group of experimental plates are as follows: HW_5%PCM 284 kg/m^3; HW_10%PCM 303 kg/m^3; HW_15%PCM 316 kg/m^3; H_5%PCM 273 kg/m^3, H_10%PCM 296 kg/m^3; H_15%PCM 307 kg/m^3.

2.2. Technological Offer to Produce Board Samples

Cold pressing is used to produce board samples in the following way:

1. Weighing of lignocellulosic raw material with electronic balance Kern EMB 600-2 (to the nearest of 0.01 g).
2. Preparation of binder.

Without PCM: The mass method with a 55:45 ratio of glue powder and water is used for the preparation of the binder. The UF glue powder with the hardener is weighed according to the manufacturer's technical data sheet and the mass of water, which is at room temperature (21 ± 1 °C) and is weighed separately. The binder components are mixed using a hand electric mixer, gradually mixing the powdered binder into the water. Mixing is continued for 120 s until a homogeneous binder mass is obtained. The maximum open time of the binder is 20 min, which is necessary to incorporate the binder into the lignocellulosic raw material.

With PCM: The initial process of binder preparation is the same as for preparation without PCM. After mixing for 120 s, PCM is added in amounts of 5%, 10% or 15% of the mass of the raw material. An additional 120 s of mixing was performed to mix the components to obtain a homogeneous consistency. Total mixing time is 240 s.

3. The binder is mixed with the raw material in a mixing tank, pouring the raw material and stirring with electric hand mixer, and the binder mixture is slowly and evenly added. The mixing process is carried out for another 120 s after adding all the binder.
4. Being evenly leveled, the mixed mass is formed into a matrix.
5. The board is pressed into the matrix to 25 mm thickness.
6. According to the data sheet of the binder manufacturer, the samples must be kept under pressure in the matrix for a minimum of 12 h.
7. The obtained board is kept in laboratory conditions for 10–14 days, then removed from the matrix and sawed according to the standards of the test to be performed.

2.3. Components of the Raw Material of the Boards

A sample production plan was developed, in which the amount of components was calculated and determined. In the production of boards, the amount of binder used is 10% of the absolutely dry mass of raw materials, based on the proportion of binder used in industry for the production of similar boards. Accordingly, for 231.5 g of raw materials, 23.15 g of UF powder binder and 18.94 g of water were added, which formed a ratio of 55:45. When determining the mass of the board after pressing for 336 h, it was concluded that all the water added to the binder had completely evaporated from the board.

For boards to which the microencapsulated PCM dispersion is added with the ratio 52:48 (capsules: dispersant), the percentage of microencapsulated PCM is also calculated based on the absolute dry mass of the raw material, where to the 5% board, 22.26 g of dispersion would be added, consisting of 11.57 g of microcapsules and 10.68 g of dispersant, which would evaporate. To the 10% board, 44.52 g of dispersion was added, with a composition of 23.15 g of capsule and 21.37 g of dispersant. But to the 15% microencapsulated PCM board, 66.77 g was added, consisting of 34.72 g of capsules and 32.05 g of dispersant.

Explanation of samples and percentage of raw material: H—hemp shives 100%; W—pine chips 100%; HW—hemp shives 50% and pine chips 50%; H_5%PCM—hemp shives 100%, 5%PCM; H_10%PCM—hemp shives 100%, 10%PCM; H_15%PCM—hemp shives 100%, 15%PCM; HW_5%PCM—hemp shives 50% and pine chips 50%, 5%PCM; HW_10%PCM—hemp shives 50% and pine chips 50%, 10%PCM; HW_15%PCM—hemp shives 50% and pine chips 50%, 15%PCM.

2.4. Thermal Conductivity

Thermal conductivity λ (W/(m·K)) is the property that characterizes the efficiency of insulation materials, and it is experimentally measured according to the ISO 8301 standard using heat flow meter apparatus (HFMA) *Netzsch HFM 446 Lambda Small*. According to the standard and equipment requirements, 200×200 mm size samples are prepared for testing and placed in the equipment between two horizontal plates, which are two calibrated heat flow sensors on both sides of the sample. The mean temperature T_{mean} (K) of the sample is ensured by maintaining the temperature difference ΔT (typically 10 K) between both plates. By measuring the heat flow Q (W) flowing during thermal equilibrium conditions through the sample with the thickness L (m) and surface area A (m^2), the thermal conductivity is calculated using an equation:

$$\lambda = \frac{Q}{A} \cdot \frac{L}{\Delta T} \tag{1}$$

2.5. Heat Capacity

A parameter that characterizes the thermal inertia of materials is the specific heat capacity c_p (J/(kg·K)), which describes the ability to absorb and release the heat energy with temperature change. Heat capacity measurements were performed in the same *Netzsch HFM 446 Lambda Small* apparatus based on the dynamic heat flow measurement method (DHFMA). The principle used in such measurements is a rapid temperature increase, with the dynamic analysis of thermal information generated by the heat flux instrument [53–55]. In a phase transition process, the enthalpy change ΔH is equal to the heat absorbed ΔQ.

Assuming a linear relationship between enthalpy and temperature, the heat capacity can be calculated as:

$$c_p = \frac{dH}{dT} \approx \frac{\Delta H}{\Delta T} = \frac{\Delta Q}{\Delta T} \tag{2}$$

Initially, the top and bottom heating/cooling HFMA plates are kept at the same initial temperatures. With the following temperature increase, the heat fluxes through the plates are measured until equilibrium is reached at the chosen temperature. The change in enthalpy (or absorbed heat ΔQ) is calculated by integrating the heat flow measurements over time. More detailed information about this indirect c_p measurement can be found in publications [56,57].

2.6. Latent Heat

The most important thermophysical parameter describing the effectiveness of phase change material is phase transitions or latent heat, which give a quantitative characteristic of the ability to store or release amounts of energy during the phase change process [58–60]. Supplying an amount of heat to the sample containing PCM in the solid state changes it to a liquid, and vice versa. During a phase change, energy is added or subtracted from the system at constant temperature. The temperature will change only when the phase change is fully completed. The temperature at which the phase transitions occur is at a melting or freezing point. The energy amount Q (W) required to change the phase of the sample mass m (kg) is given by the equation:

$$Q = mL \tag{3}$$

where L is latent heat (J/kg) of heat of fusion (energy needed for the melting process).

For the PCM, differential scanning calorimetry (DSC) is the most precise method for a comprehensive analysis of the melting process [61], determining the exact temperature at which the PCM starts melting and at which the melting process ends, thereby obtaining the latent heat as a function of temperature. But, for an approximate and fast estimation, the simplified approach described in [56] can be used. It is based on the difference in heat capacity values measured at temperatures below and above the melting point (without phase change), as well as around it. An estimation of the latent heat is the increase in heat flux ΔQ over the temperature range that includes the phase change compared to a linear interpolation (Figure 2).
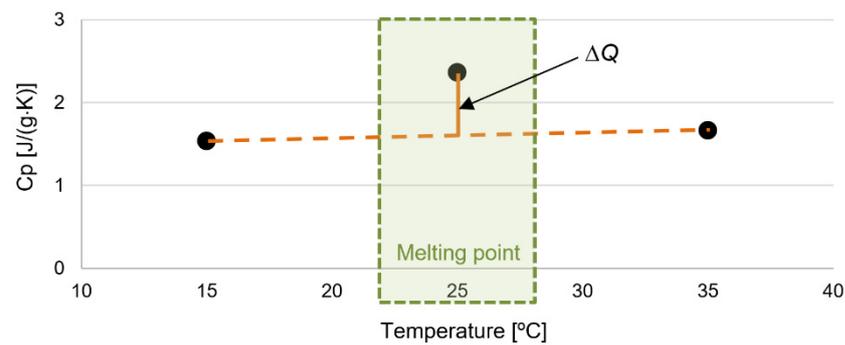

Figure 2. Increase in the measured heat capacity between 20 and 30 °C during a measurement of the specific heat capacity around the melting point.

When using PCM incapsulated into building materials, it is important that the melting temperature is within the range of indoor temperatures, typically 22–27 °C. In the experiments performed, cp measurements are made in three ranges: 10 to 20 °C, 20 to 30 °C (including phase change process) and 30 to 40 °C. Figure 2 shows all three points (15, 20 and 25 °C are the mean temperatures), with increased values between 20 and 30 °C, meaning that it also includes the phase transition heat. If the mass of added PCM is known, the latent heat can be calculated using Equation (3).

3. Results

3.1. Thermal Conductivity

The coefficient of thermal conductivity λ of the samples is determined at a temperature ranging from 10 °C to 30 °C, with an interval of 5 °C. Figure 3 shows that from 10 to 20 °C, λ increases linearly proportionally for samples with 10% and 15% PCM, but for the sample with 5%, λ increases linearly proportionally up to 25 °C. Samples with 10% and 15% PCM show sharper fluctuations in the results after 20 °C, whereas with 5% PCM, the fluctuations start only at 25 °C. It can be concluded that as the sample has a larger volume with PCM, the fluctuations are larger, which is explained by the fact that with a larger volume of PCM, there is a larger amount of water-based dispersion in the sample. Up to 20 °C, the moisture of the sample is released, which affects the thermal conductivity; after 20 °C, the moisture of the material stabilizes, and the thermal conductivity improves by an average of 1.4%.
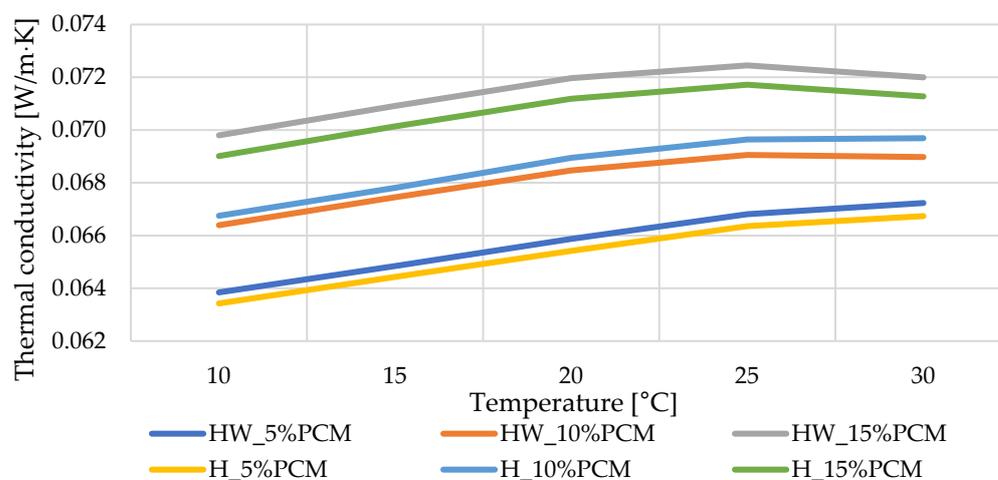

Figure 3. Measured thermal conductivity for groups of samples with PCM.

On assessing the samples without PCM admixture at 20 °C, the best result is shown by the sample with hemp shives and longitudinal milling chips, reaching 0.064 W/(m·K). When creating the structure of the sample using a mixture of 50% hemp shives and 50% wood longitudinal milling chips, the thermal conductivity increases by 4.7%, reaching 0.067 W/(m·K).

Determining the thermal conductivity at 20 °C for samples with added PCM, the best result (lowest λ) is the hemp shives sample with 5% PCM, which has a thermal conductivity λ of 0.065 W/(m·K), but this sample also has the largest thickness of 26 mm and the lowest density 273 kg/m^3, which make the material more porous and have better heat conductivity. A very close result is HW_5%PCM, which has a thermal conductivity coefficient λ = 0.066 W/(m·K), with a similar density to H_5%PCM. But, among all samples, the highest λ = 0.07 W/(m·K) is for sample groups H_15%PCM and HW_15%PCM, with the highest density of 308–316 kg/m^3.

On analyzing the results, a tendency of coincidence can be observed, in that a lower coefficient of thermal conductivity or better thermal insulation is seen in samples without PCM. When applying the capsules, λ increases, which is clearly observed for the group of HW samples. With the increase in temperature, λ increases in all variants. Better thermal conductivity at a similar density can be observed for the H group samples in comparison with the HW group samples, which could be connected to both the fact that hemp shives have a better thermal conductivity than wood and because hemp shives create a more porous material structure in the sample than chips.

3.2. Heat Capacity

The analysis of the heat capacity results is based on the information about the melting temperature range of PCM, which is 23–28 °C. Figure 4 shows that at 15 °C and 35 °C,

PCM activity is not observed and changes in the results in both groups of materials in the temperature range HW and Hm which are 7%, but within one material group, the differences between the amount of PCM in the samples are 2% in volume. The heat capacity of the HW material group is 4% higher than that of the H group samples.
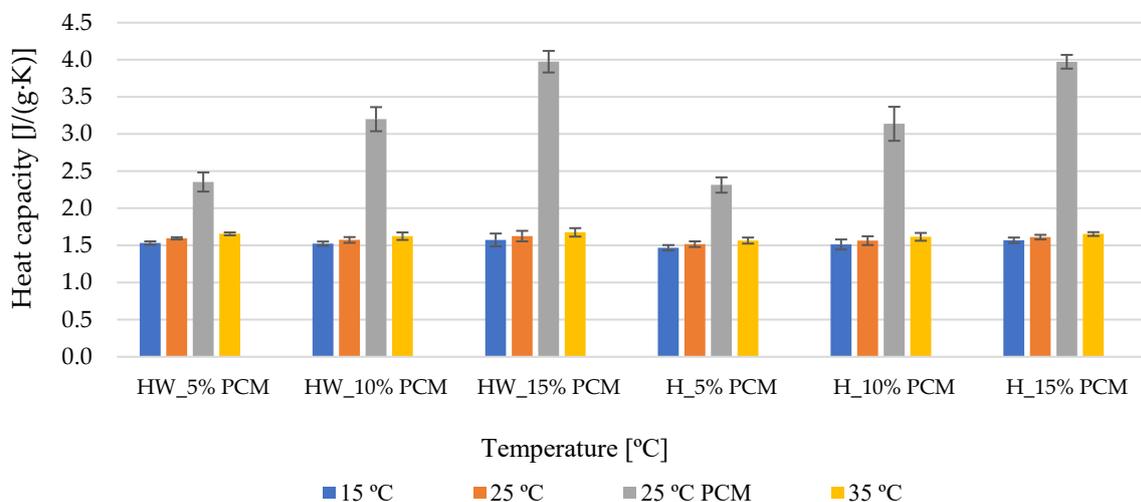

Figure 4. Measured heat capacity for group of samples with different amount of PCM.

In order to give an indicative understanding of the heat capacity value of samples with PCM at a temperature of 25 °C (heating process from 10 to 30 °C), it is calculated as a weighted average value between 15 °C and 35 °C. The resulting linear relationship shows that the heat capacity within the entire sample increases proportionally to the temperature.

On analyzing the results without considering the addition of PCM, the heat capacity of the material would increase by 3–4% on average for every 10 °C. Between 15 °C and 25 °C, the heat capacity of all six sample groups increased by between 3 and 4% and between 25 and 35 °C, as well as by 3 and 4%, which is a relatively uniform heat capacity of the material with the increase in temperature.

Figure 4 illustrates that a clear increase in heat capacity is observed between 20 and 30 °C (the mean value of 25 °C), which coincides with the melting temperature of PCM capsules, both in material groups and within one group, with the increase in the PCM amount the same amount that heat capacity increases. In material groups HW and H, when the amount of PCM increases by 5%, the heat capacity index increases on average 1.3-times, and at 15% reaches 3.97 J/(g·K).

In comparison with the results in the HW group between 15 °C, when the PCM does not work, and 25 °C, when the PCM works, the following changes are observed: at 5% PCM, the capacity of the material increases by 54%, at 10% the capacity increases 2.1-times, and at 15%, PCM capacity increases from 1.57 (g·K) to 3.97 (g·K), reaching a 2.53-times increase. H group samples have identical changes; only at 5% PCM, the volume capacity increases by 57% from 1.47 to 2.31 (g·K), while for HW group samples, the heat capacity is 1.53 J/(g·K), which is 7% less.

When comparing data, the samples at a temperature of 25 °C, with the data when the PCM operation is activated and the calculated heat capacity of the materials, an increase in the heat capacity of the PCM from 1.48- to 2.47-times is observed. At 5% PCM, the capacity increases by 48% for the HW group and by 47% for H; at 10% PCM, the heat capacity for HW and H groups increases twice; and at 15% PCM, the heat capacity of the W and H groups increases by 2.5-times.

By conducting research between insulation materials available on the market and materials developed by scientists, both with and without incorporated PCM, a comparative analysis was carried out to determine the advantages and disadvantages of the developed material. Materials with similar densities were selected for comparison—the density of

materials available on the market is within 230–460 kg/m^3; for materials developed by scientists [61–63], the value was 185–416 kg/m^3, and for author's boards, 296–316 kg/m^3.

Figure 5 illustrates that the developed materials achieved a higher heat capacity, with an average result of 3.16 J/(g·K), whereas the average heat capacity of the materials available on the market (RBB wood fibreboard, Cewood wood wool panel, RignoCell steam-exploded hemp shives board) reached on average 1.97 J/(g·K), and 1.55 J/(g·K) was reached on average in the materials developed by scientists.
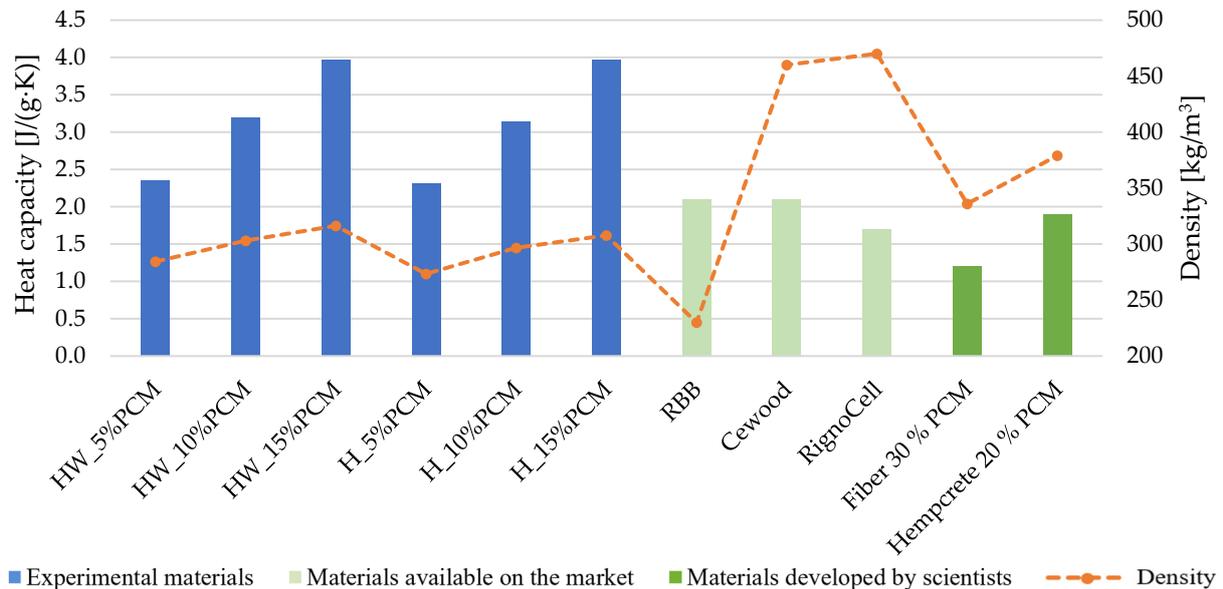

Figure 5. Comparative data of heat capacity.

The heat capacity of the manufactured insulation materials with the same thickness and composition of 15% PCM is the highest, reaching 3.97 J/(g·K), which is twice the result of the materials available on the market, which are in a range of 1.7–2.1 J/(g·K), but comparing the average results of H and HW with the materials available on the market, the obtained result is 1.6-times better. When comparing the authors' best result with materials developed by scientists with added PCM from 20 to 30%, where the obtained result is in the range of 1.2 (30%)–1.9 (20%) J/(g·K), the authors' boards achieved a 2.6-times better result.

3.3. Latent Heat

The latent heat of fusion (or enthalpy) is calculated from the heat capacity measurements using the method described in Section 2.6—as a difference in measured heat capacity between 20 and 30 °C for samples with and without the PCM admixture. Heat is applied to 10 °C and appropriate mass of PCM using (3). With an incremental increase in PCM content in the samples every 5%, the resulting latent heat for the whole sample shows a proportional rise (orange bars on Figure 6), ranging from 14 J/g (for 5% PCM) to 46 J/g (for 15% PCM).

The additionally calculated enthalpy values for pure PCM, which are normalized to the weight of PCM content in each sample (blue bars in Figure 6), have a very small dispersion, from 152 to 163 J/g, showing good reproducibility of the results and compliance with literature data for pure PCM—140–180 J/g [12,55]. It indicates the reliability of the results and the applicability of the indirect method used for such calculations.

A possible reason for a potential shift in absolute latent heat values is the very precise weighting of phase change material in a sample, e.g., change in PCM mass of 1%, resulting in the change of calculated latent heat value by up to 20%.
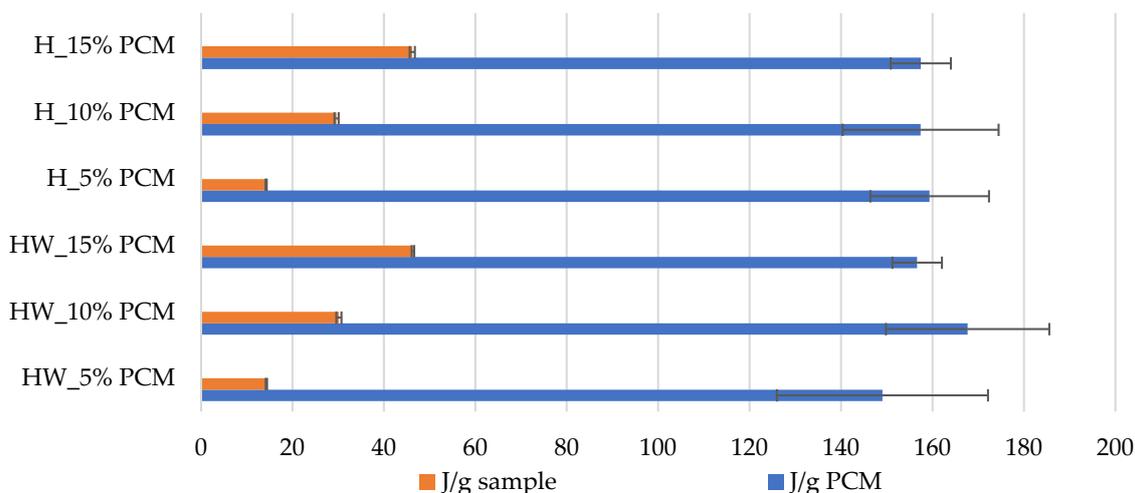
Figure 6. Calculated latent heat for group of samples with different amount of PCM.

4. Discussion

- A microencapsulated bio-based phase change material was chosen and employed to enable precise quantification of the incorporated phase change amount, eliminating the risk of loss when the room temperature rises between 23 °C and 28 °C. Increasing the quantity of PCM in a sample leads to an increase in the materials' thermal conductivity, changing the function of the material.

- However, since this material is intended not only to exhibit high thermal conductivity but also to possess a significant heat capacity, it was crucial to achieve a balance. The goal was to ensure that the material does not absorb excessive heat, allowing it to correlate with room temperature. It should also function as a thermal capacity material when the room temperature rises between 23 °C and 28 °C. If the thermal conductivity coefficient is much lower, it is likely that this material, with included PCM, would not effectively function as a heat storage material, impeding the transfer of heat to the capsules.

- The incorporation of phase change microcapsules proved to be a transformative addition, resulting in a substantial increase in the heat capacity of the board, reaching an impressive enhancement of up to 147%. This boost in heat storage capacity holds promising implications for the overall energy performance of the material, offering potential benefits for various applications that require efficient heat management.

- As the content of phase change material (PCM) increases, a noticeable reduction in thermal conductivity is observed. Remarkably, samples lacking PCM exhibited the most favorable thermal conductivity, approximately 0.062 W/(m·K). This finding is pivotal to understanding the influence of PCM inclusion on the materials' heat transfer properties, providing valuable insight for material selection and design in specific thermal applications.

- Furthermore, the specific heat capacity of samples incorporating 15% encapsulated phase change material displayed a remarkable increase, surging by 2.5-times to reach 3.97 J/(g·K). This substantial improvement in specific heat capacity bestows upon the material the capability to significantly mitigate indoor temperature fluctuations. This attribute is particularly valuable in scenarios involving direct solar radiation, where the material can effectively absorb and release heat, contributing to the creation of more stable and comfortable indoor environments.

- At the moment, it is difficult to find lignocellulosic-based thermal insulation materials with PCM on the market. However, for comparison purposes, RBB wood fiber with a density of 230 kg/m^3 and a heat capacity of 2.1 J/(g·K) is chosen, along with a comparable cedar planer cement board with a density of 450 kg/m^3. Additionally, RignoCell steam-exploded hemp fiberboard, having a similar density but a heat

capacity percentile of 1.70 J/(g·K), is also selected. Compared to materials developed by scientists with 20 to 30% added PCM (1.2–1.9 J/(g·K)), our material achieves a 2.6-times better result.

- Adding 5% PCM to HW and H samples yields an average result that is 19% higher than the results of commercially available materials. Furthermore, the experimental materials produced a result that is 51% higher than that achieved by material results developed by other researchers. This difference is likely attributed to the lower PCM content, greater material thickness and higher density of the dispersion used in the studies of those researchers.
- In addition to the findings, our rigorous calculations of latent heat of fusion yielded results with minimal dispersion, signifying the reliability and robustness of our experimental approach. The consistency in our results establishes confidence in the characteristic values of latent heat, which consistently hover around 160 J/g. This congruence with existing literature data for the specific PCM type employed in our study validates the accuracy and relevance of our research outcomes.
- The material was tested in fire tests, revealing its combustibility. However, in comparison to similar materials, it achieved Class B–D ratings (with A being the highest). This indicates that additional fire protection measures should be considered in the future.

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