RIGA TECHNICAL UNIVERSITY

Inga Zotova

LIGNO-CELLULOSIC INSULATION BOARDS OF IMPROVED PERFORMANCE AND THE TECHNOLOGY OF THEIR MANUFACTURE

Summary of the Doctoral Thesis

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RIGA TECHNICAL UNIVERSITY
Faculty of Materials Science and Applied Chemistry
Institute of Design Technologies

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DOCTORAL THESIS PROPOSED TO RIGA TECHNICAL UNIVERSITY FOR THE PROMOTION TO THE SCIENTIFIC DEGREE OF DOCTOR OF SCIENCE

To be granted the scientific degree of Doctor of Science (Ph. D.), the present Doctoral Thesis has been submitted for defence at the open meeting of RTU Promotion Council on 28 November 2023 at 15.00 at the Faculty of Materials Science and Applied Chemistry of Riga Technical University, 6 Kipsala Street, Room 206.

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DECLARATION OF ACADEMIC INTEGRITY

I hereby declare that the Doctoral Thesis submitted for review to Riga Technical University for promotion to the scientific degree of Doctor of Science (Ph. D.) is my own. I confirm that this Doctoral Thesis has not been submitted to any other university for promotion to a scientific degree.

Inga Zotova …….
Date: ……

The Doctoral Thesis has been written in Latvian. It consists of an Introduction, 3 chapters, Conclusions, 60 figures, 25 tables; the total number of pages is 101. The Bibliography contains 177 titles.

CONTENTS

INTRODUCTION

The European Union and the whole world are currently facing climate change caused by greenhouse gas emissions [1]. According to the standing scenario, global energy demand in buildings will at least double by 2050 compared to the present level and the largest expenditure will fall on heating and cooling [2]. The Intergovernmental Panel on Climate Change (IPCC) predicts that by 2050 the average global temperature will rise by 6 °C. In light of this, it is necessary to find more efficient ways to reduce energy consumption in the building sector and, at the same time reduce the amount of greenhouse gas emissions [3]. The conditions can be met by employing advanced materials. Natural resources are not inexhaustible, so new combinations of natural materials must be found, combining primary and reusable resources and ensuring their capacity and aesthetics. With the growing global consensus that the planet may be affected by irreversible climate change, ecological and sustainability requirements have become dominant in new product development. 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. If the product is to correspond to the requirements of sustainability criteria, it must be developed with mutual synergies between the product's characteristics and its impact on the economy, society and environment [4].

According to the action plan “Agenda 21” developed by the United Nations Organization regarding sustainable development, environmentally friendly technologies are considered to be those that protect and reduce adverse effects on the environment throughout the life cycle of material, starting from the extraction of raw materials and ending with the further use or recycling of the product/material [5–6]. Analysis of the use of various natural resources in board materials application has been carried out in a series of research [7–9]. One of the very important aims is to achieve good specific heat capacity characteristics by using innovative technologies for high efficiency, for example, by adding microencapsulated phase change materials (PCM) in the composition [10–13]. By including hemp shives and wood longitudinal milling chips in thermal insulation materials with PCM, the thermal inertia of the building can be increased without practically enlarging the mass of the building. Based on the great potential of using latent heat, it can offer a possible new technology development solution for autoregulation of indoor microclimate. Phase change materials have proved effective in enlarging building thermal mass and improving energy management, thereby increasing energy efficiency [14–18]. PCM can be incorporated into lightweight [19] and construction materials [20–26].

Microencapsulated phase change materials (PCM) are now widely recognised as potential additions to building insulation materials that provide a thermal mass effect to help save energy and maintain comfortable indoor temperatures. At the same time, PCM does not replace traditional insulation materials. So, the possibility of combining PCM with traditional insulation materials is a significant direction of research that has not yet received enough attention.

The present Doctoral Thesis aims to concentrate on experimental research for the creation of a prototype and technology of a smart thermal insulation material. Based on the acquired know-how, the aim of the work was to create a temperature-stabilizing insulation material from

industrial hemp shives and wood longitudinal milling chips, integrating into it a dispersion containing microencapsulated phase change material (temperature regulation range 23–28 ºC) to increase the specific heat capacity, thermal inertia and thermal conductivity. During the Thesis research, searches were carried out for combinations of material structures to ensure a set of functional peculiarities (constructive stability, insulation properties), as well as tests of appropriate binders' amount and physical and mechanical peculiarities of their connections; parameters for production process were optimised, the peculiarities of the obtained prototypes were tested, areas were specified for possible application and limitations, experimental technologies were developed and their parameters optimised. The experimental insulation materials were made from hemp shives and wood longitudinal milling chips by blending directly into the mass 5 %, 10 %, and 15 % of encapsulated PCM dispersion. Cold pressing and urea-formaldehyde resin glue (UF) as a binder were used to reduce CO_2 emissions in the production of boards. The experimental samples were made as single-layer 25 mm thick board materials with a density of 290 ± 20 kg/m^{3,} which qualifies as low-density boards.

Actuality of the theme

Despite the existing research on the use of phase change materials (PCM), 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; this prototype can form a solution for the development of new technologies related to indoor microclimate regulation, without increasing the mass of the building.

Research objective

To conduct complex research to improve the capacity of hemp by-product-based indoor finishing insulation boards by integrating into their composition microencapsulated biological-based phase change materials to intensify their functional peculiarities.

Research tasks

- Analysis of published and unpublished materials on the use of renewable resources in insulation boards combined using microencapsulated phase change materials.
- Identify and analyse the methods and technologies of production of existing board materials.
- Elaborate the technology for incorporating microencapsulated phase change materials (PCM) into the board structure.
- Develop solutions for experimental board prototype structures with and without PCM from hemp shives and wood longitude milling chips.
- Perform sample testing, analysis and interpretation of obtained results.
- Carry out a comparative analysis of material peculiarities and formulate recommendations.

Scientific novelty of the research

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 board.

Practical significance of the research

Boards from agricultural and wood industry by-products with microcapsules of phase change materials integrated into their composition for latent storage of thermal energy provide an opportunity to improve the microclimate in indoor spaces (air temperature), to increase the energy efficiency of the building's thermal energy in general, making a positive impact on moving towards passive house and the implementation of the principles of circular bioeconomy. The use of agricultural and wood industry by-products for the development of materials with high added value will be an important contribution to the development of the relevant industries and the development of the national economy of Latvia.

Thesis to be defended

The created board prototypes and the appropriate technology for their production allow to obtain sustainable insulation materials with 2.53 times higher performance for ensuring indoor temperature regulation of buildings from agricultural and wood processing by-products.

Approbation of the research

Publications:

- Kirilovs, E., **Zotova, I**., Kukle, S., Pugovics, K. "Low density hemp shive particleboards for latent thermal energy storage performance." **In**: Journal of Energy Systems, 2021, 5(1), pp. 1–9. (SCOPUS) DOI: doi.org/10.30521/jes.805791
- Kirilovs, E., **Zotova, I**., Gendelis, S., Kukle, S., Stramkale, V. "Experimental study of using micro-encapsulated phase-change material integrated into hemp shive wallboard." **In**: MDPI Journal Buildings, 2020, 10(12), pp. 1–14. (SCOPUS) DOI: doi.org/10.3390/buildings10120228
- Kirilovs, E., Kukle, S., Gusovius, H.-J., **Zotova, I**., Stramkale, V. "Development of wet-preserved hemp fibreboard with thermal and sound insulation properties." **In**: International Multidisciplinary Scientific GeoConference Surveying Geology and Mining Ecology Management, SGEM, 2019, pp. 74–79. (SCOPUS) DOI: 10.5593/sgem2019/6.2/S26.010
- Kirilovs, E., Kukle, S., **Zotova, I**., Nagle, A. "Structures of sound absorbing and thermal conductivity composite from raw renewable materials." **In**: International Multidisciplinary Scientific GeoConference Surveying Geology and Mining Ecology Management, SGEM, 2018, pp. 333–340. (SCOPUS) DOI: 10.5593/sgem2018/6.3/S26.044

Conferences:

- **Zotova, I**., Kukle, S., Kirilovs, E., Gutmane, I. "Analysis of one-layer Hemp Shive and Wood Chips Insulation Wallboards." Advanced Materials and Technologies 2020, Lithuania, Palanga, 24–28 August 2020.
- Kirilovs, E., Kukle, S., Gusovius, H-J., **Zotova, I**. "Phase change material integrated in to indoor two-layer shive hemp wallboard." Advanced Materials and Technologies 2020, Lithuania, Palanga, 24–28 August 2020.
- Kirilovs, E., Gusovius, H-J., **Zotova, I**., Kukle, S. "Development of hemp shive wallboard with integrated phase change material" 8. European conference on renewable energy systems 2020, Turkey, Istanbul, 24–25 August 2020.
- Kirilovs, E., Kukle, S., Gusovius, H-J., **Zotova, I**. "Development of smart insulation materials with PCMs for indoor microclimate regulation" International Conference on Materials Engineering and Nanotechnology, ICMEN 2019, Kuala Lumpur, Malaysia, 2–5 December 2019.
- Kirilovs, E., Kukle, S., Gusovius, H-J., **Zotova, I**. "Development of Innovative Low-Density Thermal Insulation from Wet-Preserved Hemp." Advanced Materials and Technologies 2019. Lithuania, Palanga, 19–23 August 2019.
- Kirilovs, E., Kukle, S., Gusovius, H-J., **Zotova, I**., Stramkale, V. "Innovative board material development of raw material with different wet-preservation time." 21st International Conference Materials, Methods & Technologies 2019, Burgas, Bulgaria, 1–5 July 2019.

1. REVIEW OF SOURCES

The review and analysis of published and unpublished materials proves that the main topics are:

- usage of renewable resources and production by-products;
- phase change materials – reviews of historical development, principles of their operation, classification, types of installation, and fields of application;
- insulation materials – both existing and experimentally newly created by scientists;
- board materials – their production methods, technologies and properties, performance improvement and use of renewable components.

1.1. Increasing heat capacity with phase change material

Phase change materials are ideal products for thermal management solutions. This is because they store and release heat energy during the process of melting and solidification (changing phase from one to the other). During a phase change, the molecules rearrange themselves, causing a chaotic change in the entropy of the material system. In thermodynamics, a material must absorb or release thermal energy or heat due to this change in entropy, and this heat associated with a unit mass of the material is defined as the latent heat of the material. When such a material solidifies, it releases a large amount of energy in the form of latent heat of fusion or energy of crystallisation. And vice versa, when the material is melted, the same amount of energy is absorbed from the direct environment, changing from a solid to a liquid consistency (see Fig. 1.1). This property of PCM can be used in several ways, such as thermal energy storage, building energy efficiency, food refrigeration, spacecraft thermal systems, solar power plants, and thermal protection of microelectronics.

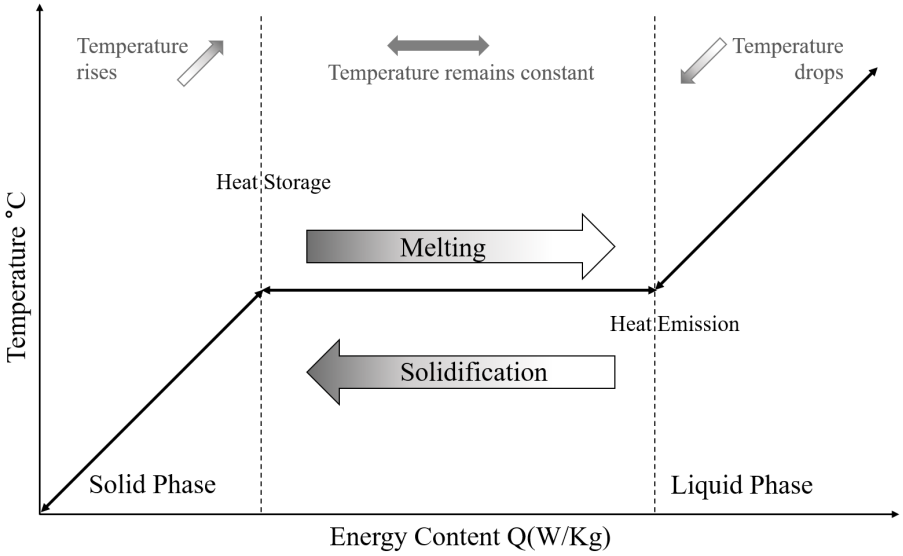

Fig. 1.1. PCM operating principle [27–31].

2. MATERIALS AND METHODS OF THE EXPERIMENTS

The chapter gathers information on the selection of materials for making samples, production technology, methods used in testing, and preparation of samples according to standards. Insulation materials have important properties such as thermal conductivity, heat capacity, sound absorption, fire resistance, environment and people safety, sustainability and durability. In order to ensure the abovementioned properties, appropriate tests were performed on the developed board material.

Components are selected taking into consideration the following criteria: local availability, functional compliance, and impact on the environment (acquisition, production, use).

2.1. Raw materials and substances for sample production

- Hemp shives – a by-product of the hemp fibre variety "Bialobrzeskie" grown in Latvia.
- Pine longitudinal milling chips – a by-product of furniture production and carpentry.
- Urea-formaldehyde resin adhesive (UF) with hardener for cold pressing.
- Produced encapsulated phase change material of biological origin that does not contain formaldehyde.

2.2. Production of board samples

- 47 groups of samples were produced and analysed, which had varied raw material structures and layered boards, the amount of raw materials, the amount of binder, the sizes of fractions, the formation of surface reliefs, veneering and various PCM materials embedded. In order to clarify the properties of the boards, 9 sample groups were chosen for further development and testing according to the methods specified in the standard. When improving the selected board types, a constant amount of raw material and binder was defined for all of them in order to evaluate the overall properties of the board and how the properties are affected by the addition of microencapsulated PCM into the board composition.
- 9 groups of samples were prepared for testing varying the types of raw materials and the percentage of microencapsulated PCM from 5 to 15 % with a step of 5 %, maintaining the specified limits of the target density. Three groups of boards were produced without PCM to have inputs H, W, and HW (a 50 : 50 mix of hemp straw and wood planning chips materials), but three H groups H and three HW groups were produced varying the amount of PCM to allow the comparison of data.
- A sample production plan was elaborated in which the amount of components was calculated and determined. In the production of boards, the amount of binder used was 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, to 231.5 g of raw materials were added 23.15 g of UF powder binder and 18.94 g of water, which form 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 the boards to which the microencapsulated PCM dispersion is added with the volume of dry capsules ranging from 49 % to 53 %, 52 % was assumed in the calculations making the ratio 52 : 48 (capsules: dispersant). Based on the research in literature it was chosen to add three % volume capsules – 5 %, 10 %, and 15 %.

The percentage of microencapsulated PCM is also calculated based on the absolute dry mass of the raw material, where to the 5 % board would be added 22.26 g of dispersion, 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 with a composition of 23.15 g of capsule and 21.37 g of dispersant were added. But to the 15 % board microencapsulated PCM 66.77 g, which consist of 34.72 g of capsules and 32.05 grams of dispersant were added.

Table 2.1

Plan of Making Samples

Sample 200 mm × 200 mm × 25 mm	**Raw material**	**Binder % from mass of dry raw material**	**% of PCM material from mass of dry raw material**	
Proportions of materials	250 g, of which dry mass 231.48 g, 8 % humidity of raw material 18.52 g	Proportion – binder : water 55 : 45 UF powder 23.15 g; Water 23.15 g	Ratio of PCM capsules to dispersion 52 : 48	
H	Hemp shives 100 %	UF 10 %	–	
W	Pine chips 100 %	UF 10 %	–	
HW	Hemp shives 50 % and pine chips 50 %	UF 10 %	–	
H_5%PCM	Hemp shives 100 %	UF 10 %	**5 %** 22.26 g dispersion	Capsules 11.57 g Dispersant 10.68 g
H_10%PCM	Hemp shives 100 %	UF 10 %	**10 %** 44.52 g dispersion	Capsules 23.15 g Dispersant 21,37 g
H_15%PCM	Hemp shives 100 %	UF 10 %	**15 %** 66.77 g dispersion	Capsules 34.72 g Dispersant 32.05 g
HW_5%PCM	Hemp shives 50 % and pine chips 50 %	UF 10 %	**5 %** 22.26 g dispersion	Capsules 11.57 g Dispersant 10.68 g
HW_10%PCM	Hemp shives 50 % and pine chips 50 %	UF 10 %	**10 %** 44.52 g dispersion	Capsules 23.15 g Dispersant 21.37 g

Sample 200 mm × 200 mm × 25 mm	Raw material	Binder % from mass of dry raw material	% of PCM material from mass of dry raw material	
HW_15%PCM	Hemp shives 50 % and pine chips 50 %	UF 10 %	**15 %** 66.77 g dispersion	Capsules 34.72 g Dispersant 32.05 g

- In the production of boards, 10 % binder was used and the amount was chosen according to the data used in production and by evaluating the organoleptic criteria of the boards with the amount of binder from 4 % to 14 %, with a step of 2 % (in the tests, the amount of binder for the specific raw material was checked, so that it corresponds to the data of industrially developed materials, and at what percentage of the binder samples have the best processing possibilities of mechanical properties and the smallest deformation).

2.3. Technological offer for the production of board samples

Cold pressing is used for the production of board samples (see Fig. 2.1)

1. Weighing of lignocellulose raw material. If several layers are envisaged for production, each layer of raw material is weighed in a separate container.

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 °C ± 1 °C, is weighed separately. The binder components are mixed using an electric hand 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 lignocellulose raw material.
- **With PCM.** The initial process of binder preparation is the same as for the preparation without PCM. After mixing for 120 s until a homogeneous mass of binder is obtained, PCM is added in the amount of 5 %, 10 %, or 15 % of the mass of the raw material. An additional 120 s of mixing is performed to mix the components to obtain a homogeneous consistency. The total mixing time is 240 s.

3. The binder is mixed with the raw material in a mixing tank. The raw material is poured and stirred with an electric 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 levelled, the mixed mass is formed into a matrix. If there are several layers, it must be ensured that each layer is levelled before laying the next layer.

5. The board is pressed into the matrix to the specified thickness according to the plan of the experiment.

6. According to the data sheet of the binder manufacturer, it is accepted that 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.

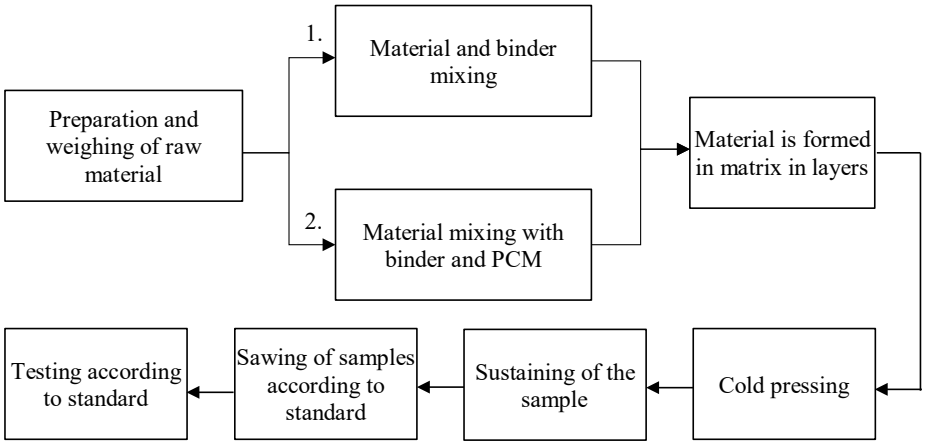

Fig. 2.1. Scheme of the production of board materials.

2.4. Requirements for raw materials

- The moisture content is determined according to the LVS EN 322 standard, using a digital scale *"Kern" EMB 600-2* (to the nearest of 0.01 g) and a drying cabinet *BinderKBF 115.*
- Granulometric analysis is performed in accordance with LVS EN 933-1 and LVS EN 933-2 standards, using a digitally controlled electromagnetic sieve shaker *"Matest A059-01 KIT"* and digital balance *"Kern" EMB 600-2* (to the nearest of 0.01 g).

2.5. Tests conducted to check exploitation properties of insulation boards

- Moisture was determined according to the LVS EN 322 standard, using a digital scale *"Kern" EMB 600-2* (to the nearest 0.01 g) and a drying cabinet *Binder KBF 115.*
- Density was determined according to the LVS EN 323 standard, using digital balance *"Kern" EMB 600-2*, digital calliper *KS Tools 300.0532* to the nearest 0.01 mm.
- Water absorption and swelling were evaluated by combining LVS EN 317 and LVS EN ISO 29767 standards, using digital balance *"Kern" EMB 600-2*, digital calliper *KS Tools 300.0532* to the nearest of 0.01 mm.
- Scanning electron microscopy was performed using a *Tescan Vega* SEM device, and *LEICA EM ACE 200* was used for coating.
- Thermal properties were determined using methods according to standards LVS ISO 8302 and LVS ISO 8301, determining heat capacity, thermal conductivity, and thermal resistance, using *HFM 446 Lambda Series – NETZSCH Analyzing & Testing* equipment, digital balance *"Kern" EMB 600-2* (to the nearest of 0.01 g).

- LVS ISO 5660-1 standard was used to determine the physicochemical properties, determining fire reaction properties using a conical calorimeter *GD-ISO5660*, digital balance *"Kern" EMB 600-2* (to the closest of 0.01 g), a digital calliper *KS Tools 300.0532* to the closest of 0.01 mm.
- Acoustic properties were determined according to the LVS EN ISO 10534 standard, assessing the sound absorption coefficient, NRC coefficient, weighted sound coefficient and sound absorption class, using the acoustic impedance and transmission loss measurement set – *Brüel & Kjær Type 4206*, digital calliper *KS Tools 300.0532* to the nearest 0.01 mm.

2.6. Determination of mechanical properties

- Ultimate bending strength was determined according to LVS EN 310 and LVS EN 12089 standards using the universal testing device FORMTEST UBP 86/200.
- Resistance determination test for axial withdrawal of screws was performed according to the LVS EN 320 standard, using the universal testing device FORMTEST UBP 86/200.

3. RESULTS AND ANALYSIS OF EXPERIMENTS

According to the summarised experimental methods, experiments have been carried out with board samples. Their results lead to the data characterising the material, summarising, interpretation, and comparative analysis with similar material data from industry and published literature.

3.1. Characteristics of raw materials

In the production of samples, the most important features characterising the raw material are the following: the moisture content of the raw material so that it is appropriate for the selected binder, and the distribution of fractions in the total mass of the raw material so that it is possible to envisage how much dust and small fractions there are in the total mass, which reduces the mechanical stability of the board.

3.1.1. Moisture content of raw material components

The average moisture content of hemp shives is 7.9 %, and the average moisture content of pine chips is 8.8 %. The moisture content of pine chips is 11% higher than that of hemp shives. It should be kept in mind that the average moisture content of wood chips is estimated with an absolute error of 0.92 compared to 0.21 for hemp shives. The measurement of the material moisture content was carried out in order to precisely determine whether it is permissible to use the selected UF binder for the raw materials, and it was concluded that their average moisture content falls within the 8±2 % moisture interval of the adhesive material specified by the manufacturer of the binder.

3.1.2. Granulometric analysis of raw materials

In Fig. 3.4, the average results of sieving of raw material particle samples are summarised. For hemp shives (H), a quarter (25 %) of the fraction is in the size range of 150–2000 μm; on the other hand, for pine chips (W) material, these fractions are 39 % less. Less than 20 % of the H material has fractions with a size greater than 5600 μm, while W has 38 % more fractions of this size. The amount of W fractions in the range of 500–1000 μm is 17.7 %, which is 17 % less than in the W material. The amount of fractions in the range from 2000 to 5600 μm in raw material for W material is between 12 and 14 %. In the size fraction below 500 μm, the fraction of fine and dust in the H raw material is about 3.5 %, which is 45 % less than in the W material. Fractions of this size affect the mechanical properties of the board because when mixing the material with a binder, most of the binder is attracted and a smaller amount of it falls on the larger fractions, so it was concluded that fractions of this size should be separated from the total mass before mixing with the binder by additional sieving.

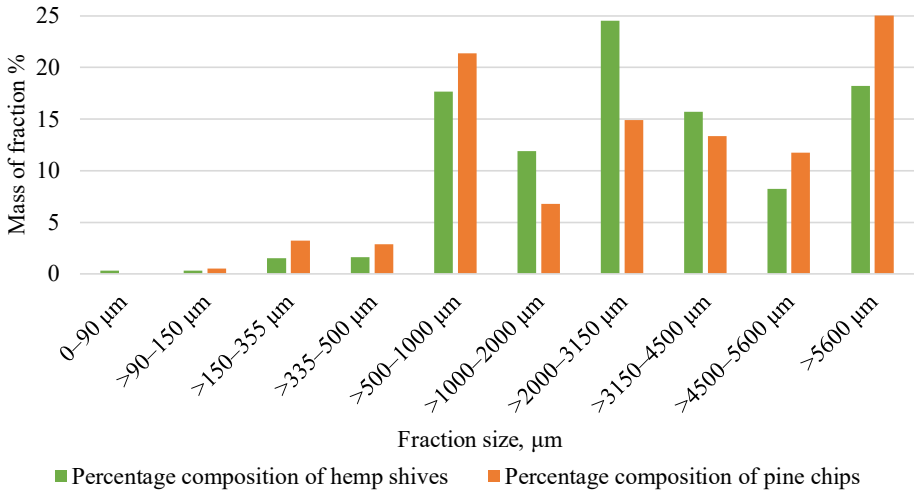

Fig. 3.1. Results of granulometric analysis of hemp shives and wood chips.

3.2. Properties of board materials

When developing boards, it is important to determine their basic properties in order to interpret the mechanical and physical properties based on these results.

3.2.1. Determination and comparison of board material moisture

The moisture content of the sample is determined using the mass method. The mass of the board after pressing is 286 g after 24 h, and when removed from the drying cabinet, the mass is 212 g, losing 26 % of the initial mass or 35 % of moisture (the water used for the binder and the moisture of the raw material of hemp shives 8 %). During the continuation of the experiment, the sample was weighed every 60 min for the first 5 h, and the mass of the sample increased by 2 g every 1 h on average. The mass of the sample reached 267 g after 72 h, correspondingly, the mass increased by 25 % from the moment of removal. After fixing this result, the sample is continuously weighed for 14 days with minimal changes ranging from 267 g ± 1 g. From the obtained results, it was concluded that the sample should be kept in the laboratory room conditions for three days after drying in order to obtain equilibrium moisture; 7 days is recommended.

On average, the mass change of the board sample is within 6.5 %, while the moisture content of the sample is 6.9 %. The obtained result corresponds to the required moisture of the board for its use indoors, and the obtained board moisture is equivalent to the moisture of the raw material components – 8 ± 2 %. Figure 3.2 shows the change in the mass of the board after the drying cabinet.

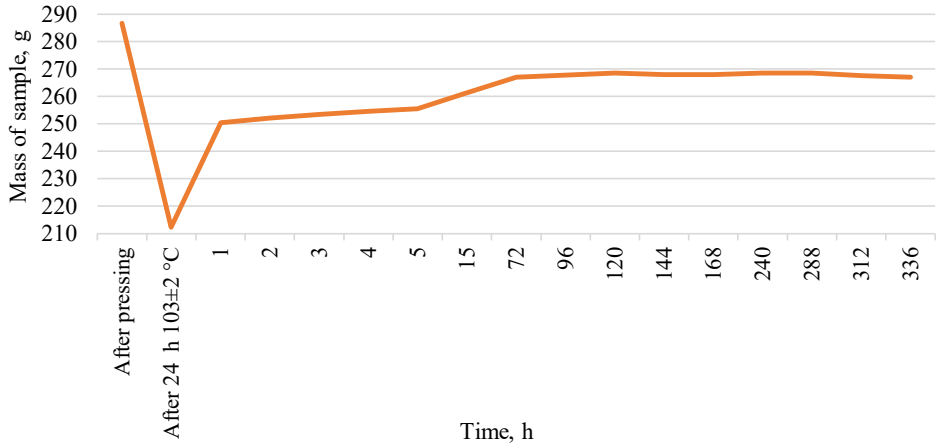

Fig. 3.2. Mass of the sample immediately after pressing for 24 h in a drying cabinet and during 14 days under laboratory conditions.

For comparison, the moisture determination of the second type of sample was conducted after removing the sample from the press and keeping 14 days in laboratory conditions. The initial mass of the sample is 286 g, but after 72 h, the mass of the sample has decreased by 3 %, reaching 277 g.

When fixing the mass for 14 days, variations of the mass are observed within 1.5 %, which is permissible for a material that is made from natural raw materials and "breathes" – absorbs and releases moisture (see Fig. 3.3).

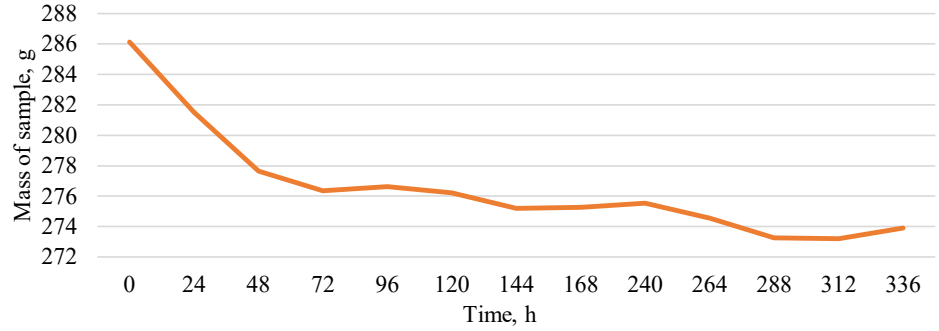

Fig. 3.3. Change in sample mass after pressing for 336 h under laboratory conditions.

To fix the moisture content of the samples using both moisture determination methods, after 14 days, both samples are placed in a drying cabinet for 24 h at 105 ± 2 °C. The initial mass of the first sample is 286 g, and of the second sample 273 g. As a result of drying, the samples lost proportionally 8 % and 10 % of the mass, and the moisture content of the samples was 9 % and 11 %, respectively. Control measurements were made for the samples in the laboratory, and it was determined that after 336 h, the initial mass for each board was obtained.

Comparing the two methods of determining the moisture content of boards, it was observed that for slabs after cold pressing, it is recommended that the drying process proceeds slowly

and evenly; otherwise, as the slab dries quickly, it starts to crack and deform. When implementing such a process of drying the board, it can be observed that after 336 h, small fluctuations in moisture content and mass occur in the board, which on implementing the first method can be expressed within 0.5 %, but on implementing the second method can be expressed within 1.5 %.

3.2.2. Density uniformity in boards

To characterise the internal unevenness of the samples, the density uniformity was determined to find out the distribution of the density dispersion of the boards. The average density of 200 mm × 200 mm boards of group H is 285 kg/m^3. Densities range from 269 kg/m^3 to 296 kg/m^3. The absolute error of the manufactured boards is 24.94 kg/m^3, and the relative error is 8.8 %. These differences occur due to various technological development processes, for example, imprecise incorporation of raw materials or directly during manufacturing or board drying when the thickness changes. The average density of the prepared 50 mm × 50 mm samples is 290 kg/m^3; the density varies from 272 kg/m^3 to 307 kg/m^3 with an absolute error of 9.98 kg/m^3 and a relative error of 3.4 %. The largest variation of the 50 mm × 50 mm samples is for the H_55 group, where the relative error is, on average, three times larger than for the other H group boards. When comparing the density of all H boards with the average density of 50 mm × 50 mm size samples it was concluded that the variation of the density of the board sample between the small samples and the average density of the boards is within the normal limits of 2 % amplitude.

The density of the HW group 200 mm × 200 mm size board samples varies from 276 to 292 kg/m^3; the average density of the boards is 285 kg/m^3 with an absolute error of 14.74 kg/m^3 and a relative error of 5.2 % between the boards individually. The densities of the prepared 50 mm × 50 mm samples range from 259 to 294 kg/m^3, which average is 276 kg/m^3, absolute error of 15.21 kg/m^3. This difference constitutes a relative error of 5.5 %, respectively 3.6 % for the HW_45 group, 7.2 % for the HW_46 group and 11.5 % for HW_47 group. Errors occur because of both the technological production and the fact that the boards contain two types of raw material, the hemp shives are denser and have a larger fraction size, while the wood chips are compressed more unevenly. For HW boards of the 50 mm ×50 mm samples, the average result, as compared to the average result of the boards, shows an unevenness of 3 % in volume, but this result does not show the overall internal unevenness of the board.

The density of the W group 200 mm × 200 mm boards is between 286.83 kg/m^3 and 308.5 kg/m^3. The average density of the board is 300 kg/m^3, with an absolute error of 20.90 kg/m^3. A relative error of 7 % is from these boards. The average density of the prepared 50 mm × 50 mm samples is 302 kg/m^3 ± 24.74 kg/m^3, where the sample densities vary between 283 and 326 kg/m^3; their relative error is 8.2 %. The largest relative error of 12.9 % is in the W_53 group, which is almost three times larger than in the W_52 group, which is 4.1 %. Analysing the average results of the samples with the board densities average result, the difference is 1 %, but it is observed that the boards have a more uneven density closer to the edges, and the unevenness is more expressed for the W group boards.

3.2.3. Evaluation of material water absorption and swelling

Evaluation of water absorption and swelling of materials was conducted for three groups of samples: H, W, and HW. The H group samples demonstrated the fastest change in mass in the first 15 min reaching 205 %, or a three times increase from the initial mass, see Fig. 3.4. On continuing the experiment after the first 15 min, during the next 5 h the mass change is within 18 %. From 120 min to 1440 min, the mass increases by another 4 g, which is 20 % of the initial mass. The total change of mass was 343 % within 24 h.

For HW samples, the mass change after the first 15 min increases by 239 %, or 3.4 times of the initial mass. In the following 120 min, the increase in mass is minimal, up to 3 %. The change of the sample mass in 24 h increased 3.7 times, on average by 268 %. The sample absorbs water in large quantities, but it does not fractionate and holds together well.

The increase of W samples is the largest compared with the other groups of samples, reaching 272 % of their initial mass, or within 24 h, the change in mass increases by 3.8 times. The main increase in mass occurs in the first 15 min, and while continuing the test, the mass change difference after 24 h is small because the samples have absorbed all the moisture initially.

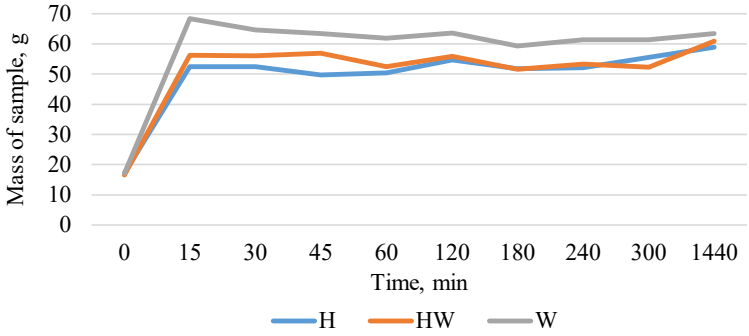

Fig. 3.4. Mass changes of H, HW, and W samples after 24 h in water.

The changes in the thickness of the H samples have been most dramatic compared to all sample groups, see Fig. 3.5. In the first 15 min, the thickness of the sample increased by 71 % of the initial thickness; continuing to measure the thickness changes, the average thickness changed by 4–12 % or 1–3 mm in the first 300 min, the same changes occurred from 120–1440 min. During this experiment, the sample thickness increased by an average of 192 %, or almost twice the initial thickness. On visual observation of the samples, they had absorbed moisture very quickly in the first 15 min, the samples changed their geometry rapidly and unevenly in different directions, fractions separated from most of the samples, and some of the pieces being large and a couple of samples even split in half when unsuccessfully removed from the vessel measurements during the experiment.

For HW samples, the thickness changes compared to the mass changes are not so drastic; on the whole, the thickness has changed by less than 10 mm, which is 40 % of the initial thickness. The sharpest changes in the thickness of the samples occurred in the first 15 min, where the thickness of the samples changed by an average of 31 %. Visually, these samples

have changed in thickness but hold together quite well, although considering the amount of moisture absorption, they are not intended for rooms with direct contact with water or high humidity.

Changes in the W samples are observed on the average increase of 7.5 mm, which is less than 34 %. For this group of samples, the increase in thickness per h is less than 1 mm, which indicates that these samples are most resistant to swelling. Visually, these samples, although they take the largest mass according to the calculations, change their geometric position the least, and from their visual appearance, it can be concluded that they have slightly changed their thickness.

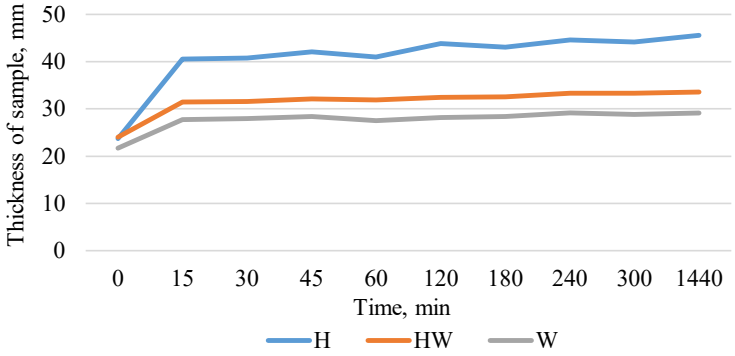

Fig. 3.5. Thickness changes of H, HW, and W samples after 24 h in water.

When assessing the absorption capacity and swelling for 24 h (see Fig. 3.6), it was concluded that the largest mass increase is for W samples reaching 46 g and the smallest for H samples reaching 41 g. All samples are made of fractions in the range of 500–5600 µm, porous and with a water-soluble binder, which are theoretically not intended for long-term exposure to water. But it is worth knowing to foresee what might happen, for example, to the wall panels if the room floods or is in constant contact with a high moisture content.

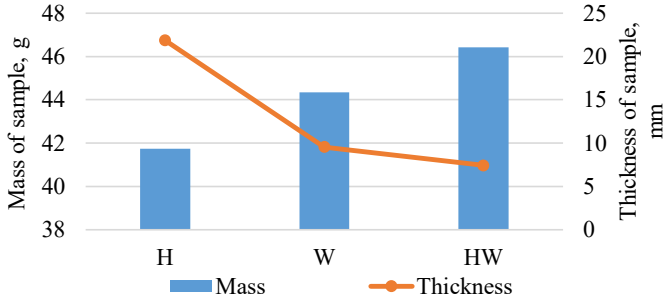

Fig. 3.6. Changes in thickness and mass of H, HW and W samples in water for 24 h.

3.2.4. Scanning electron microscopy

The prepared samples underwent scanning electron microscopy. Figure 3.7 shows a bundle of hemp fibres and a binder, but in the marked places a) a small-sized microcapsule on the surface of the hemp shive and b) a ~15 µm size microcapsule.

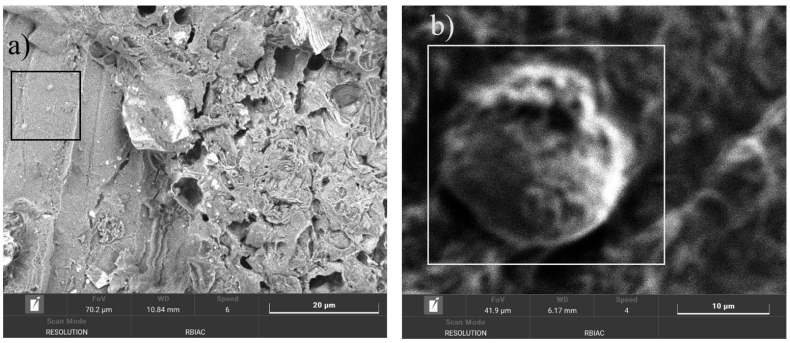

Fig 3.7. SEM image of the PCM material location in the samples.

3.3. Exploitational characteristics of insulation board

As part of the work, the thermal, physicochemical, acoustic and mechanical properties of board samples were determined and analysed.

3.3.1. Thermal properties

Determination of thermal properties is an essential stage of board evaluation. One of the most accurate and widely used measuring methods is the use of a heat flow meter. Thermal conductivity, heat capacity and thermal resistance values were calculated for the board samples, determining the performance characteristics of the boards and checking the effect of PCM on the performance results.

3.3.1.1. Thermal conductivity

The coefficient of thermal conductivity of the samples is determined at a temperature ranging from 10 ºC to 30 ºC with an interval of 5 ºC. Figure 3.8. shows that from 10 ºC to 20 ºC, lambda λ increases linearly proportionally for samples with 10 % and 15 % PCM, but for the sample with 5 %, lambda λ increases linearly proportionally up to 25 ºC. The samples with 10 % and 15 % PCM show sharper fluctuations in the results after 20 ºC, whereas with 5 % PCM, the fluctuations start only at 25 ºC. 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 more significant amount of water-based dispersion in the sample. Up to 20 ºC, the moisture of the sample is released, which affects the lambda λ coefficient; after 20 ºC, the moisture of the material has stabilised, and the lambda coefficient improves by an average of 1.41 %.

On assessing the samples without PCM admixture at 20 ºC, 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 result decreases by 4.69 %, reaching 0.067 W/m·K.

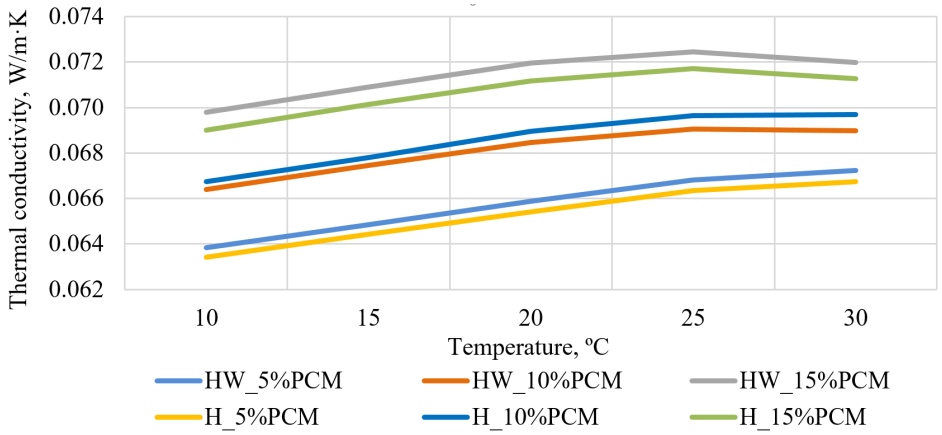

Fig. 3.8. Average results of thermal conductivity at 10 ºC for groups of samples with PCM.

Determining the thermal conductivity coefficient at 20 ºC of the samples with added PCM, the best result (lowest lambda 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 (26 mm) and the lowest density (273 kg/m^3), which provides material with the property that it is more porous and conducts heat better. 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/m3.

On analysing the results, a tendency of coincidence can be observed that a lower coefficient of thermal conductivity or better thermal insulation is in the 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 is 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 also because hemp shives create a more porous material structure in the sample than chips.

3.3.1.2. 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 3.9 shows that at 15 °C and 35 °C the PCM activity is not observed, and changes in the results in both groups of materials (HW and H) in the temperature range 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.

To give an indicative understanding of the heat capacity value of samples with PCM at a temperature of 25 °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 analysing 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 between 3–4 %, and between 25 °C and 35 °C also 3–4 %, which is a relatively uniform heat capacity of the material with the increase in temperature.

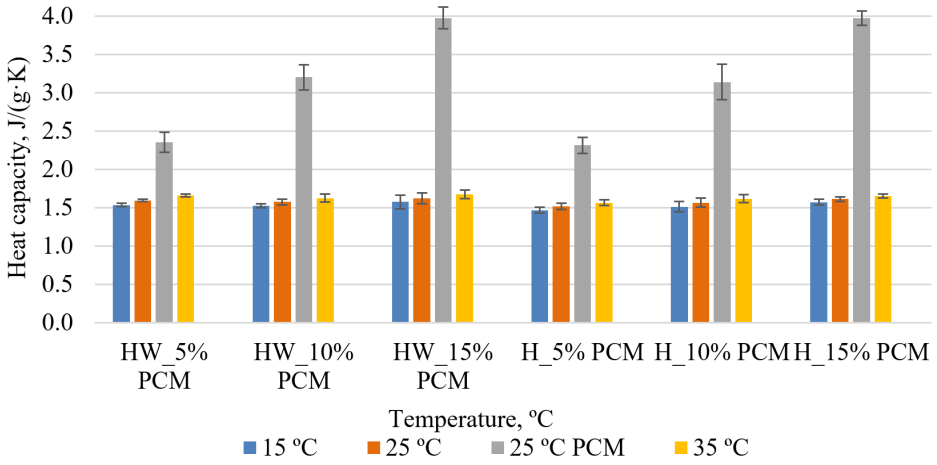

Fig. 3.9. Changes in the heat capacity of the samples with different amounts of PCM at 25 °C and the average values and standard deviation of the calculated heat capacity of these samples.

Figure 3.9 illustrates that an apparent increase in heat capacity is observed at a temperature of 25 °C, which coincides with the melting temperature of PCM capsules both in material groups and within one group; with the increase of PCM amount, the amount of heat capacity increases. In material groups HW and H, when the amount of PCM increases by 5 %, the heat capacity index rises on average by 1.3 times, and at 15 % of the amount, it reaches 3.97 J/(g·K).

On comparison of 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 by 2.1 times, and at 15 % PCM, capacity increases from 1.57 J/(g·K) to 3.97 J/(g·K), reaching a 2.53 times increase. The H group samples have identical changes, only at 5 % PCM, the volume capacity increases by 57 % from 1.47 to 2.31 J/(g·K), while for HW group samples, the heat capacity is 1.53 J/(g·K), which is 7 % less.

Comparison of data from 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 47 % for the H group; at 10 % PCM, the heat capacity for the HW group increases by 2.03 times and for the H group by 2.01 times, and at 15 % PCM, the heat capacity of the W group increases by 2.45 times and in the H group by 2.47 times.

3.3.1.3. Thermal resistance

Figure 3.10 shows that the thermal resistance of H samples, when the PCM content varies from 5 % to 15 %, exceeds the average values of HW samples corresponding to the entire range by 3.9 to 4.1 percentage points. As the temperature increases, the average thermal resistance of all tested variants decreases, HW_5%PCM (relative error >9 %) and HW_10%PCM, as well as H_15%PCM (relative error ~7 %) variants have a large measurement dispersion, possibly related to uneven dispersion of particles of material components in the cross-section of the samples, as well as explained by a small number of samples in each group, causing an absolute error of 0.02–0.04. The average thermal resistance of H samples decreases by 4.9 % in the considered temperature range, while in the HW samples by 5.1 %, and these differences are not considered significant.

Overall, the highest results are for the H_5%PCM group, R is 0.39–0.41 ($m^2 \cdot K$)/W, with the lowest density of all sample groups providing the best insulation properties in the room. The next material with the highest result is HW_5%PCM, which is also explained by the relationship between density and thermal conductivity properties, which are the best for both sample groups with 5 % PCM admixture.

With 10 % PCM admixture, the material properties of both groups are practically the same, reaching 0.37 ($m^2 \cdot K$)/W. At 15 %, the PCM admixture group achieves a result of 0.36 ($m^2 \cdot K$)/W and a slightly worse result of 0.35 ($m^2 \cdot K$)/W for the HW_15%PCM group.

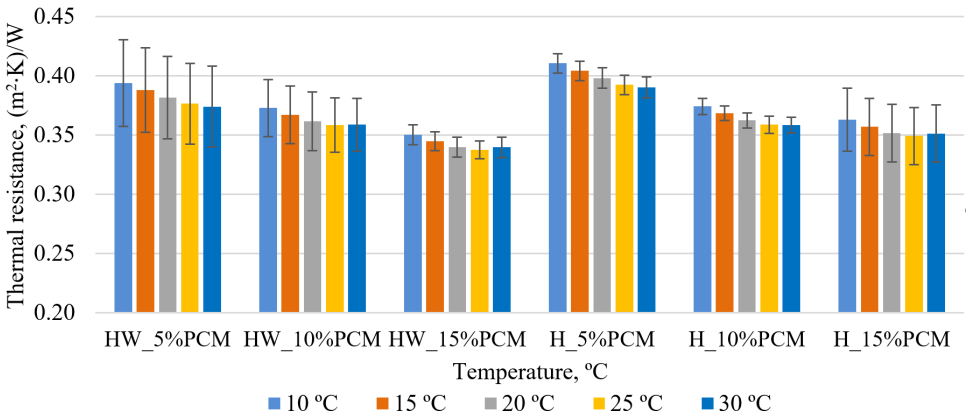

Fig. 3.10. Thermal resistance results at different temperatures.

Overall, the resistance result is in the range of 0.34–0.41 ($m^2 \cdot K$)/W, of which the hemp sample group showed slightly higher results at 0.35–0.41 ($m^2 \cdot K$)/W.

Figure 3.11 shows the thermal resistance results for W, HW and H groups without PCM admixture. The results in groups H and W are proportional, but the results of the W group are higher than those of the H group. Combining these two materials reduces their thermal resistance property by 4 %. The result of HW_5%PCM is practically the same as that of the W samples. And HW has a 3 % difference from the H_15%PCM result.

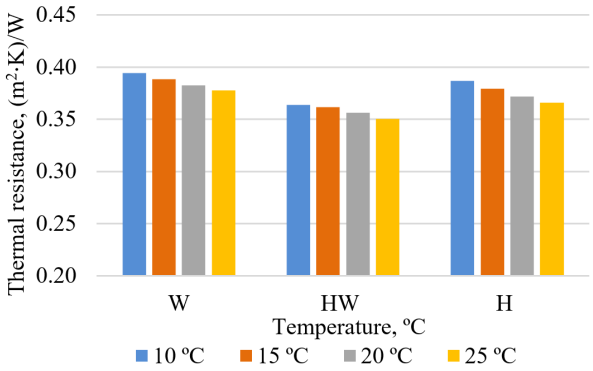

Fig. 3.11. Heat resistance results at different temperatures.

3.3.2. Physicochemical properties

An important aspect is the determination of the flammability properties of the boards and the effect of PCM on the overall results. As mentioned above, there is a risk of flammability for phase change materials, and their fire reaction has not been evaluated when incorporating PCM into interior wall finishing boards. The results obtained can be used by designers to balance the potential energy savings of using PCM with a complete understanding and predictability of the associated fire risk when using the proposed boards. It also allows for appropriate risk mitigation strategies.

3.3.2.1. Fire reaction tests on conical calorimeter

The fire reaction characteristics of the materials were determined for the H and H_10%PCM sample groups.

The heat release of both sample groups is similar, but the H_10%PCM group has 6 MJ/m² or 15 % higher heat output than the H group. For both sample groups, the total amount of heat released is in the range of 45–51 MJ/m², but the consumed oxygen is in the range of 28– 32 g, where the H_10%PCM group has consumed 14 % or 4.5 g more oxygen in comparison with the H group samples. This could be explained by the addition of raw material, which also requires more oxygen for combustion. The difference in mass loss for both groups of samples is within 1 %. The specific loss factor differs by 0.3 % between the sample groups, where the H group has a loss of 6.89 g/(cm^2) and the H_10%PCM group has a loss of 6.97 g/(cm^2). The samples with PCM in the composition ignited 1.49 times faster than those without PCM, which means that the capsules contribute to the combustion of the samples, and additional protection against ignition should be considered. The H group samples ignited in 9.67 s, and the H_10%PCM group in 6.5 s.

As a result of the smoke release, the total amount of smoke in the period from 0–605 s is 33.70 m²/m² for the H group and 43.95 m²/m² for the H_10%PCM group. As a result, H_10%PCM emitted 30 % more than the H group during the entire test smoke. The H sample group released 3.6 times more smoke than the H_10%PCM sample group in the first 10 s, which is the non-flaming phase. But in the period from 10 s to 605 s, in the flaming phase, 1.4 times

more smoke was released by the samples with PCM composition, respectively, the H sample group released 31.67 m²/m², and H_10%PCM released 43.4 m²/m². The results lead to the conclusion that the amount of smoke is affected by the PCM composition in the samples (see Fig. 3.12).

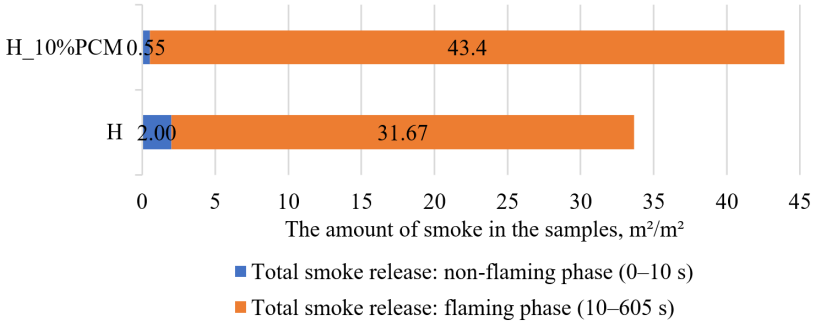

Fig. 3.12. The amount of smoke in the samples.

On analysing the heat release data, the conclusion can be made that the sample group with PCM at 60 s has a higher result of 136.92 kW/m², and for the H group, 120.7 kW/m², which also decreased with a similar difference in results, obtaining that at 605 s it still has a higher heat release speed 87.11 against 75.82 for the H group (see Fig. 3.13).

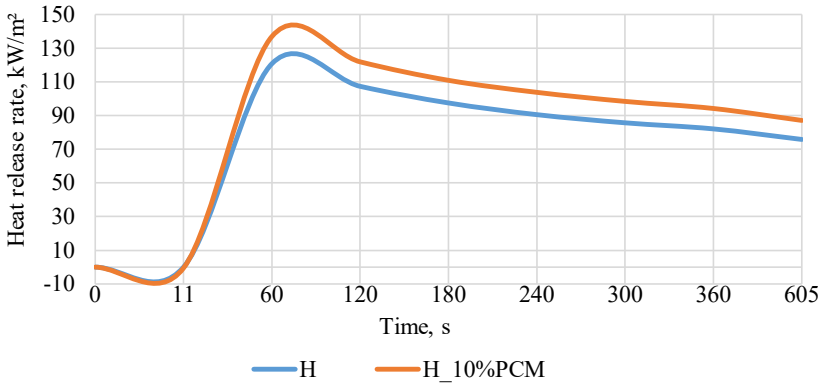

Fig. 3.13. Heat release rate.

Figure 3.14. shows the results of the effective heat of combustion, where in the first 60 s the heat of combustion is released rapidly; after 10 s, when the flaming phase begins, the heat of combustion increases rapidly up to 60 s. In the period from 60 s to 605 s, changes occur, stabilising the result, reaching 12 MJ/kg for the H_10%PCM group and 10.41 MJ/kg for the H group.

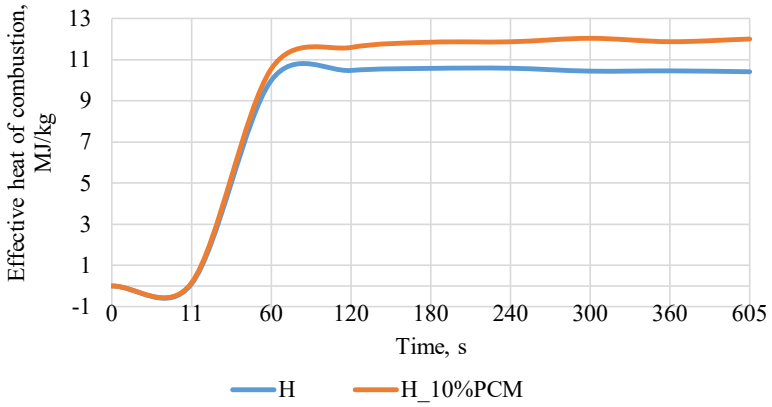

Fig. 3.14. Effective heat of combustion.

The coefficient of specific extinction is drastically different for both groups; as can be seen for the group with PCM, it happens more slowly and evenly in the first 60 s, reaching a maximum value of 41.78 m²/kg. Then this value decreases steadily, while for the samples of H group, the maximum value is already reached in the first 11 s, reaching 65.3 m²/kg, and by 60 s it is already rapidly falling and then steadily decreased. The value of group H goes four times higher than the specific extinction coefficient in the first 11 s (see Fig. 3.15).

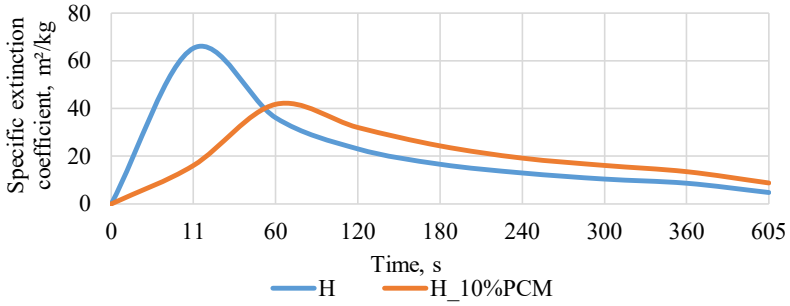

Fig. 3.15. Specific extinction coefficient.

Mass loss measurements are used to analyse the development of smouldering in hemp shives insulation board. The mass loss rate rapidly peaks at 12.1 g/(s·m²) for the H group and 12.64 g/(s·m²) for the H_10%PCM group. The mass loss for both groups of samples starts to decrease from 60 s, and continues to decline equally for the rest of the test time. Despite the low release temperatures recorded, most of the total mass loss is during the downward smouldering regime. The reason is that the initial combustion involves the pyrolysis process, during which most of the mass is lost; the further process involves charring oxidation reactions, and thus the mass loss is less.

The first 60 s is the fastest burning process during which carbon dioxide (CO_2) is released. It can be seen that the amount of CO_2 emitted during the subsequent combustion process

decreases. The results of the H_10%PCM group are slightly higher, which could be influenced by the added microencapsulated PCM dispersion (see Fig. 3.16).

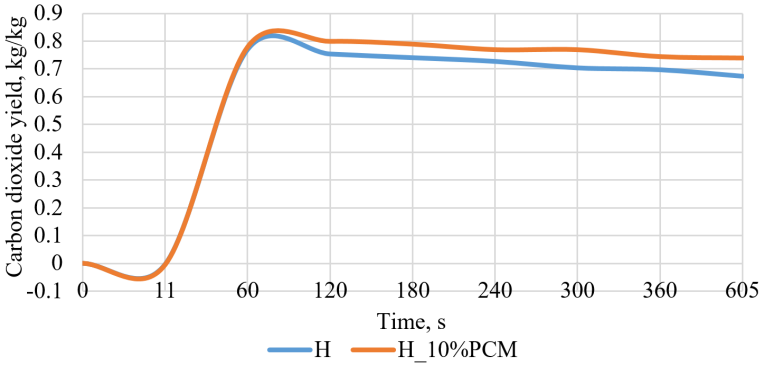

Fig. 3.16. Carbon dioxide yield.

3.3.3. Acoustic properties

Noise pollution is considered a stress factor that can adversely affect human health for a long time. Porous absorbing materials are used for passive control of various sounds and noises. Knowing the high porosity of technical hemp shives, it is essential to determine the acoustic properties of the manufactured board. The acoustic properties of the samples were described by determining whether the material is sound absorbent, the sound absorption and NRC coefficients were calculated, and the absorption class was determined.

3.3.3.1. Sound absorption coefficient, sound absorption classes, NRC coefficient

The material is heterogeneous and relatively porous; the samples differ from each other in the internal structure of the material and the orientation of the fractions to the sound wave. The set of these factors impacts the absorption of the material, which affects the measurement results.

There is an unevenness in the results of the sound absorption tests. It can be explained both by the differences in the material structures and the preparation of the material samples and their placement in the impedance tube. The deviation in the results is because, for a given apparatus, two sizes of samples must be produced in order to obtain a wide frequency range. The obtained results must be combined in a single graph in a computer program. The merging of the data into one graph can be seen at 1600 Hz, marked with a dashed line in Fig. 3.17. Analysing the data leads to the conclusion that the density affects the degree of absorption, that the group of samples by diameter has a uniformity of different results, but general trends of the properties remain at the specific frequencies.

For the group of HW samples, the material can be considered absorbing, where the absorption coefficient is above 0.5 at frequencies starting from ~650 Hz. The developed material is basically reflective up to 400 Hz. The absorption curve increases up to 1600 Hz, and it reaches the highest value of 0.93, where further the curve develops a decrease in absorption

up to 3150 Hz, obtaining a value of 0.5, where again, the absorption increases in the frequency range 5000–6300 Hz, reaching an absorption coefficient in the range of 0.78 to 0.83. There is slight variation between samples in the HW group, with the largest differences between samples around 4000 Hz, where the absolute unevenness reaches 0.17.

The W group samples have very heterogeneous results because this group of materials was the most difficult to process and obtain small diameter samples, where large absolute error deviations in the frequency range of 0.12–0.36 – 1000–6300 Hz are also observed. The W samples work as an absorber in the frequency band from 550–6300 Hz with a coefficient of 0.65–0.9.

Samples G, H, I and 7, 8, and 9 of the H sample group demonstrate similar results between themselves, not exceeding an absolute error of 0.07 up to 1500 Hz, but at frequencies between 2000–6300 Hz, the error is 0.11–0.19. The material can be considered absorbing from 700 Hz onwards. The sample with the highest density shows a slightly lower degree of absorption. The best results and absorption coefficient are in the range of 0.75–0.95 at the frequency ranges 1000–2000 Hz and 4000–6300 Hz. After combining the samples in the 1600–3000 Hz range, the average absorption coefficient is 0.55–0.66.

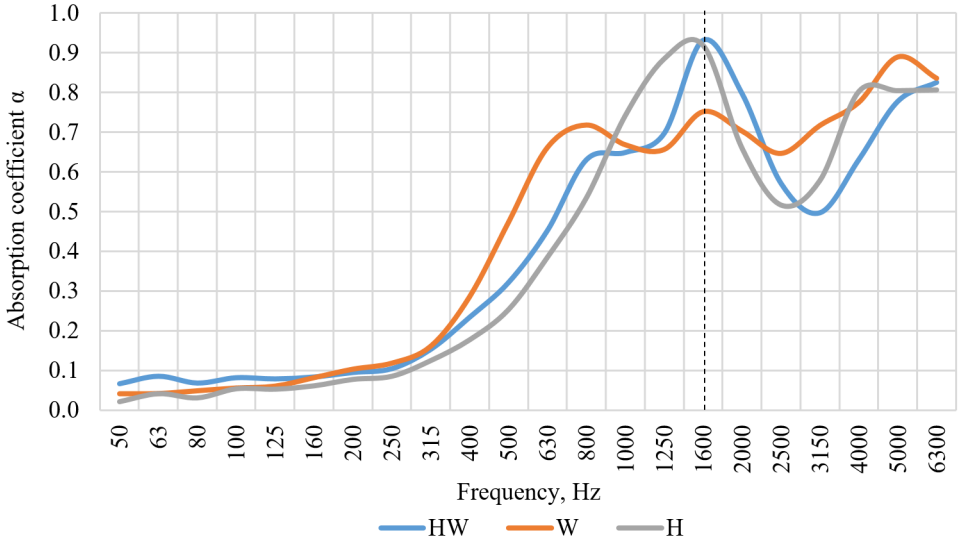

Fig. 3.17. Average results of absorption coefficient α 1/3 octave frequency bands of sample groups HW, W and H.

The materials of all three groups work similarly as an absorber; the samples differ insignificantly with a coefficient value above 0.5 in the frequency band from 700 to 6300 Hz. The most effective absorption is at frequencies 1000–2000 Hz and 4000–6300 Hz. After 500 Hz, the W samples absorb the most evenly, and the results of H and HW samples are very similar. Materials of all three groups can be called sound absorbers.

The average sound absorption coefficient, by coincidence, for the H group at 50–6300 Hz, it is 0.48 and for the HW group, it is 0.47; the results are very similar, considering the similar

structure and sample density. The result of the W group is higher in all frequency bands, taking into account that for materials of such a structure, the result is viewed at the highest frequency, but for samples H, HW and W at 500–6300 Hz, the absorption coefficient is in the range of 0.65–0.69.

The NRC coefficient is determined in the frequency range 250–2000 Hz. For the HW material group, the coefficient is 0.47, for the H group, 0.44, and for the W group, 0.49. The results are similar to the absorption coefficient at 50–6300 Hz, but considering that the low and high frequencies are not taken into account when calculating the NRC, the results are not the same.

Table 3.1

Sound Absorption Class at Different Frequencies

	250 Hz		**500 Hz**		**1000 Hz**		**2000 Hz**		**4000 Hz**	
HW group	0.10	–	0.32	**D**	0.65	**C**	0.80	**B**	0.63	**C**
H group	0.09	–	0.25	**E**	0.74	**C**	0.66	**C**	0.80	**B**
W group	0.12	–	0.47	**D**	0.67	**C**	0.70	**C**	0.78	**C**

The sound absorption class is determined in each group of samples, separately assessing at which frequencies the material absorbs best and to clarify whether this developed material also works as an absorption material, see Table 3.1.

3.3.4. Mechanical properties

Indicative measurements of the mechanical properties of the prepared board samples were performed to determine the board's stability in bending and the resistance to axial screw withdrawal. These two tests describe the use of the board material as a free-standing element and the possibility of fixing it to the surface with appropriate fasteners.

3.3.4.1. Bending strength

The ultimate strength in bending test was performed for H and HW samples and for groups with added 10 % PCM (see Fig. 3.18) with a thickness of 25 mm and a density between 278 kg/m^3 and 298 kg/m^3. Comparing the H and HW results, the ultimate strength differs by 0.04 MPa, showing the best result for the HW samples at 1.39 MPa. For sample groups H and H_10%PCM, the results show a difference of 0.04 MPa, showing a worse result with PCM in the composition. On the other hand, comparing samples HW and HW_10%PCM, a decrease of 13 % in the result is observed in samples with PCM. Although the density of the PCM material increases by 7 % when adding PCM, the difference in the result can be explained by changes in the composition and the amount of water in the binder because the composition of the PCM dispersion also contains water, which generally affects the strength of samples with longitudinal milling chips. The samples have a relatively low average bending strength of 1.38 MPa, which means they are best used in the construction of frame fillings, or to form a non-load-bearing structure, for example, by screwing to a stable surface. The specific material cannot be used as a free-standing horizontal element.

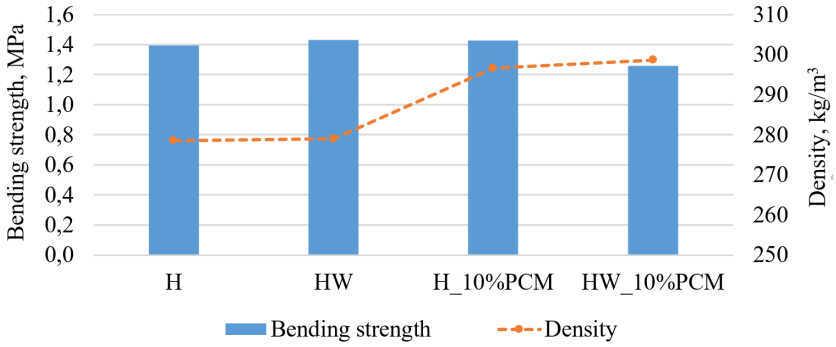

Fig. 3.18. Bending strength.

3.3.4.2. Determination of resistance to axial screws withdrawal

During the test, screws were initially screwed into the samples without pre-drilling because the material is of low density. After adding 10 % PCM to the samples, the axial strength decreased by 1.6 times. The H group samples are 1.3 times stronger than HW samples, with the H samples reaching 23 MPa and the HW samples reaching 18 MPa (see Fig. 3.19).

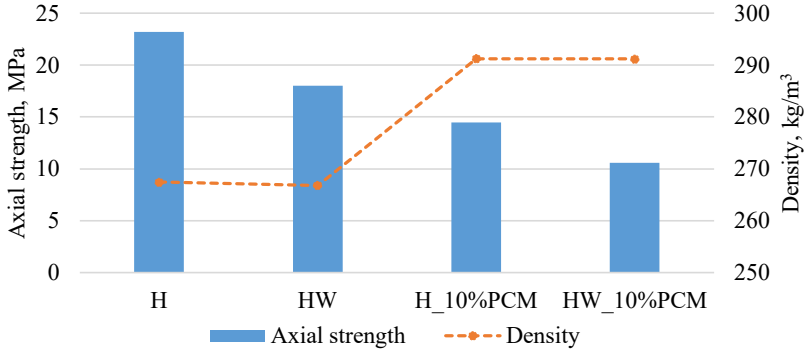

Fig. 3.19. Determination of resistance to axial withdrawal of screws.

3.4. Comparison of properties with other insulation materials

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 weaknesses 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, it is 185–416 kg/m^3, and for the author's boards, it is 296–316 kg/m^3.

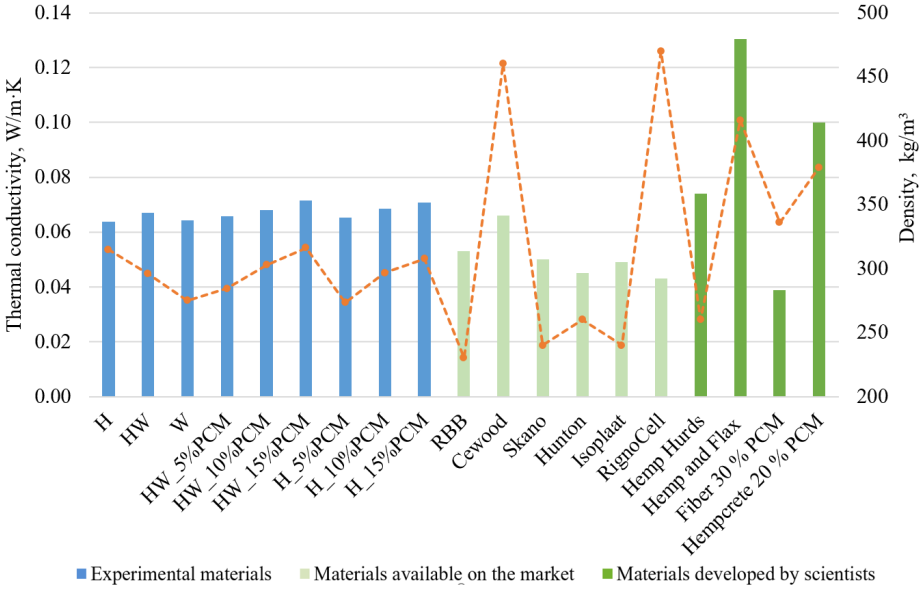

Fig. 3.20. Comparative data of thermal conductivity.

The author compared the thermal conductivity of six in

sulation materials (see Fig. 3.20) that are available on the market and four materials developed by scientists, two of which were with incorporated PCM.

The best thermal conductivity results are for thermal insulation materials in the range of 0.043–0.053 W/(m·K) available on the market. But with the available *Cewood* acoustic boards and the hemp shives boards created by Lithuanian scientists with a starch binder, the result is the most similar to the prototypes developed by the author – 0.066–0.074 W/(m·K).

Average thermal conductivity results on the market are 0.051 W/(m·K), for materials developed by scientists 0.086 W/(m·K), and for author's materials 0.067 W/(m·K). The thermal conductivity of the materials developed by the author is by 32 % inferior to the materials on the market, where most materials have a lower density, but by 28 % superior to the majority of materials developed by scientists, where one of the components is agricultural residues. For one of the research materials, where 20 % PCM was added to gypsum hemp concrete, a negative effect of PCM on the thermal conductivity result was observed.

Figure 3.21 illustrates that the developed materials have 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 reached 1.97 J/(g·K), and 1.55 J/(g·K) of the materials developed by scientists.

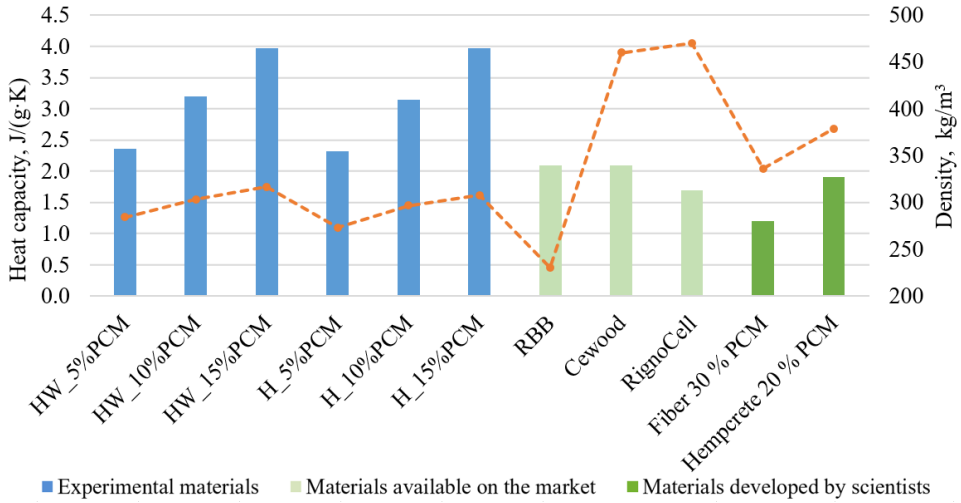

Fig. 3.21. Comparative data of heat capacity.

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 the range of 1.7–2.1 J/(g·K), but comparing the average results of the H and HW samples with the materials available on the market, the obtained result is 1.6 times better. When comparing the author's best result with materials developed by scientists with added PCM from 20 % to 30 %, where the obtained result is in the range of 1.2–1.9 J/(g·K), the author's boards demonstrate a 2.6 times better result.

Table 3.2

Comparative Data of Fire Reaction Class

Material	**H**	**Cewood**	**HW_10%**	**RBB**	**ISOPLAAT**	**SKANO**	**HUNTON**
Fire reaction class	B*	B	C*	E	E	E	F

On comparing the developed material with insulation materials (see Table 3.2), which can be purchased in construction stores, the fire reaction class, which is theoretical at the moment and obtained after some tests, is even relatively high. It is more difficult to compete with materials developed from concrete or lime. Here is an opportunity to look for solutions in the future with additional fillers to reduce the ignition response and speed.

The *Cewood* company writes that sound absorption can reach 0.9, but it is different, as in the author's research, the material reacts differently at different frequencies, reaching an average coefficient of 0.6–0.7. It is important that materials with insulating properties additionally ensure the acoustic comfort of the room, reduce sound vibrations and improve the insulation of the room. On investigation of various acoustic board properties, it was concluded that the most important thing is that the material should be absorbent and the coefficient should be above 0.5.

In comparison, the ultimate bending strength of the experimental boards with industrially produced materials and materials developed by scientists, it was observed that the developed materials are at the same level as the RBB fibre board, reaching 1.3 MPa, but 1.8 times higher than the *Cewood* and *Isoplaat* materials (see Fig. 3.22). The highest results for the developed materials range from 1.26 MPa to 1.43 MPa. The ultimate bending strength of materials developed by scientists is between 0.0068 MPa and 0.24 MPa.

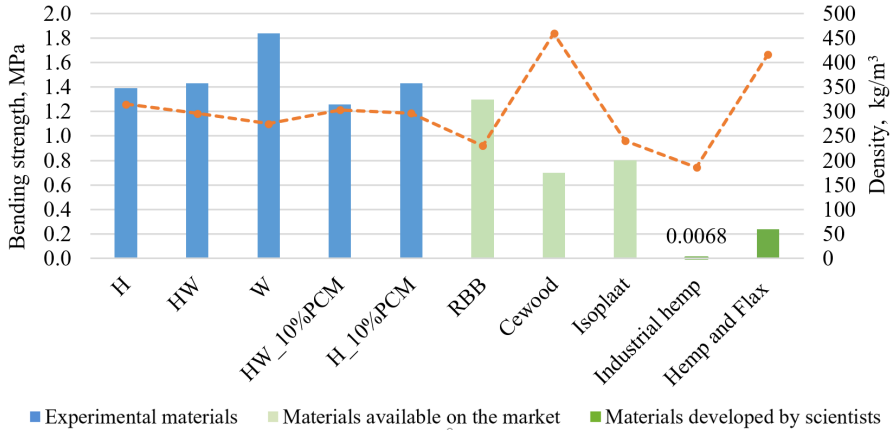

Fig. 3.22. Ultimate bending strength – comparative data.

3.5. Brief description of the material and intended use

Lignocellulose insulation board for providing indoor microclimate

Sustainability and efficient use of local production surpluses or renewable natural resources is an essential motive in the production of board insulation materials. During the research, low-density insulation boards for internal walls were developed from agricultural and wood processing by-products, varying the amount of structure and microencapsulated phase change material in the board up to 15 %.

Innovation

The innovation of the new product is based on the high potential of utilising latent heat by integrating phase change material into production by-products-based insulation material.

Raw materials and production

Hemp shives, pine longitudinal milling chips, UF binder, microencapsulated phase change material dispersion *MikroCaps* PCM-S50 (Slovenia).

PCM is used as a latent thermal energy storage for indoor microclimate self-regulation depending on the ambient temperature. The board samples were produced using cold pressing technology, which ensures reduced CO_2 emissions in the production process. Changing the phase of the PCM at the selected interval is an important step towards achieving a near-zero emission of a building's separating construction. Phase change materials are recognised as an effective way to improve the energy management of a building; due to the phase transition, such materials can store a large amount of energy. By combining heat insulation materials of

hemp shives and wood longitudinal milling chips with PCM material, it is possible to increase the heat capacity of the building up to two and a half times, ensuring a uniform climate in the room, practically without increasing the mass of the building, and the porous structure of the board can reduce the noise level.

Visual and processing capabilities of the material

Material can be used as a functional – for latent heat accumulation and room noise reduction, improving acoustics, as well as a design element – in the pressing process, creating a relief surface in decorative wall and ceiling panels. Panels fixed in the frame structure can also serve as room separating elements.

It is possible to perform mechanical processing – sawing, grinding, milling, and drilling.

Finishing solutions – it is possible to paint both in mass and only on the outer surface (the best option is with a paint gun), and pasting with various decorative materials.

Environment for application

The material is intended for indoor use where the relative humidity does not exceed 60 ± 5 % as a functional decorative panel (for improving climate and acoustics) or insulation board to improve energy efficiency by interior walls and ceiling decoration.

Fastening by screwing, glueing, and placing in a frame structure.

Technical specification

- Thermal conductivity: 0.064–0.072 W/m·K
- Heat capacity: 0.352–0.394 J/(g·K)
- Thermal resistance: 2.35–3.97 (m^2·K)/W
- Sound absorption: 500–6300 Hz α = 0.65–0.69
- NRC coefficient: 250–2000 Hz α = 0.44–0.49
- Sound absorption classes: 1000–4000 Hz B and C
- Density: 290 ± 20 kg/m^3
- Thickness: 25 mm
- Moisture content in the material: 8 ± 2 %
- Fire reaction class: B–C
- Ultimate bending strength: 1.26–1.43 MPa
- Axial resistance: 10.6–23.2 MPa

Summary of Chapter 3

Characteristics of raw materials.

- The moisture of the hemp straw (H) fractions is 7.9 %, while the average moisture of the pine chips (W) fractions is 8.8 %; it is permissible to use the selected UF binder as raw material as their average moisture content falls within the 8 ± 2 % moisture range of the adhesive material specified by the binder manufacturer.
- The granulometric analysis revealed that the fractions which are less than 500 μm in hemp shives contain 3.7 % and 6.6 % in wood longitudinal milling chips, and they are separated before the production of board as in the production of boards fractions larger than 500 μm are used.

Production of boards.

- During the research, we worked on the experimental boards by varying the board variables, such as thickness, proportions of raw materials, the sizes of the fractions of raw materials, creating different types of layered boards, surface structures, as well as glueing surfaces. It was chosen to make boards from one material or by mixing raw materials 50 : 50.

Determination of binder volume and analysis of related properties

- In determining the amount of binder and analysing the related properties, the conclusion was made that it is necessary to produce boards with at least 10 % binder, and they must be kept under the press for at least 10 h in order to reduce geometric deformation. With fast drying and short time under load, the geometric shape of the board changes.
- The mass of the board sample changes up to 4 % when moisture leaves from the sample; accordingly, the higher the percentage of binder is chosen, the greater the mass change will be. Materials must be kept in the laboratory conditions for at least 5 days, but preferably for 7–10 days, when the mass does not change more than 1.5 % per day, before performing other tests.

Properties of board materials:

- Further processing of the boards can be performed 10–14 days after pressing when the equilibrium moisture of the material after pressing is obtained.
- The uniformity of the density of 200 mm × 200 mm boards compared to 50 mm × 50 mm samples is even within 1 %. It was observed that the boards have a more uneven density closer to the edges, and the unevenness is more pronounced for boards of the W group.
- The water absorption and swelling evaluation test of the material determined that all three groups of samples absorbed water sharply; the samples of group H changed their thickness 1.9 times from the original thickness, degraded, crumbled and dissolved in water. The largest mass change was for the the W group, 3.8 times the average mass of the original sample group, but this did not affect its properties and did not fractionate in water. HW samples are a good compromise between the two raw materials and prevent the hemp boards from swelling and breaking into fractions due to the added longitudinal milling chips.
- In SEM images microcapsules of different sizes were observed on the hemp shives surfaces.

Thermal properties

- Thermal conductivity capacity decreases with the addition of PCM; the higher the percentage, the larger the decrease. The best results are for the sample groups without PCM. At the temperature of 10 ºC – H 0.062 W/m·K; HW 0.066 W/m·K; W 0.062 W/m·K; for the H group samples with 5–15 % of PCM it is 0.063–0.069 W/m·K; for the HW group samples with 5%–15% of PCM it is 0.064–0.070 W/m·K. At the temperature of 20 ºC – H 0.064 W/m·K; HW 0.067 W/m·K; W 0.064 W/m·K; for the H group samples with 5 %–15 % of PCM it is 0.065–0.071 W/m·K; for the HW group samples with 5 %–15 % of PCM is 0.066–0.072 W/m·K.
- Heat capacity increases in the samples at the PCM melting temperature of 23–28 °C. By determining the increase in the capacity at 25 °C for samples with 5–15% percentage of PCM, it is proved that the capacity can be increased up to 2.5 times. For the HW sample group with 5 % of PCM, it increased 1.48 times, which is 2.35 J/(g·K); with 10 % of PMC – 2.03 times, which is 3.20 J/(g·K), and with 15 % of PCM – 2.45 times, achieving the capacity of 3.97 J/(g·K). For the H group, heat capacity increased 1.53 times with 5 % of PCM –, which is 2.31 J/(g·K); with 10% of PCM – 2.01 times, which is 3.14 J/(g·K) and with 15 % of PCM – 2.47 times, which is 3.97 J/(g·K).
- The heat resistance result by sample groups ranges from 0.34 to 0.41 $(m^2 \cdot K)/W$, of which the highest result was achieved by the hemp straw (H) sample group – 0.41 $(m^2 \cdot K)/W$.

Fire reaction test with a conical calorimeter

- Both groups of samples showed the total amount of heat released in the range of 45–51 MJ/m^2, and the difference in mass loss is within 1 %. The rate of mass loss speed reaches a maximum of 12.1 $g/(s \cdot m^2)$ for the H group and 12.64 $g/(s \cdot m^2)$ for the H_PCM group. The specific loss factor differs by 0.3 %.
- Oxygen consumption ranges from 28 g to 32 g, where the sample group H_10%PCM shows oxygen consumption of 14 % or 4.5 g more.
- The H group samples ignited in 9.67 s and the H_PCM group samples in 6.5 s, which is 1.49 times faster.
- The amount of smoke released from 0–605 s was 33.70 m^2/m^2 for the H group and 43.95 m^2/m^2 for the H_10%PCM group, so the H_10%PCM group compared to the H group during the entire test emitted 30 % more smoke.
- The heat release results for the samples with PCM at 60 s are higher – 136.92 kW/m^2, and for group H, it is 120.7 kW/m^2.

Acoustic properties

- All sample groups are sound absorbers, absorbing best/most effectively in the frequency band from 1000–2000 Hz and 4000–6300 Hz.
- At frequencies 500–6300 Hz, the absorption coefficient for the H group is 0.65, for the HW group – 0.65, and for the W group – 0.69.
- The NRC coefficient is determined at frequencies 250–2000 Hz. For the HW material group, the coefficient is 0.47; for the H group, it is 0.44; and for the W group, it is 0.49.
- Sound absorption classes from 1000–4000 Hz are B and C.

Mechanical properties

- The ultimate bending strength of the developed board prototype reaches1.26–1.43 MPa.
- The resistance to axial withdrawal of screws for the H group samples is 23.2 MPa, but for the HW group samples – 18 MPa; for the H_10% samples – 14.5 MPa, and for the HW_10%PCM samples – 10.6 MPa.

Comparison of properties with other insulation materials

- The thermal conductivity of the developed materials is 28 % superior than those developed by most scientists but 32 % inferior than in industrially produced materials. On analysing the results, a negative trend was observed – the thermal conductivity deteriorated for the materials with added PCM.
- The heat capacity of the insulation materials made with 15 % PCM admixture is the highest, reaching 3.97 J/(g·K), which is 2 times higher than the result of the materials available on the market, which are in the range of 1.7–2.1 J/(g·K). Compared to the materials developed by scientists with added PCM from 20 % to 30 %, where the obtained result is in the range of 1.2–1.9 J/(g·K), the newly produced boards achieve a 2.6 times better result.
- The fire reaction class is competitive and higher than that of insulation materials on the market.

Intended use, environment and processing:

- The material is intended for indoor use where the relative moisture does not exceed 60 ± 5 %.
- It can be used as a functional (for latent heat accumulation and room noise reduction, improving acoustics) and aesthetic (as a design element) insulation board for interior walls and ceiling decoration, or by fixing panels in the frame structure, it can also serve as a room separating element.
- Finishing solutions – it is possible to paint both in mass and only on the outer surface (the best option is with a paint gun), as well as pasting with various decorative materials.
- It is possible to perform mechanical processing – sawing, grinding, milling, drilling.
- Fastening by screwing, glueing, placing in a frame structure.

CONCLUSIONS

- The review and analysis of published and unpublished materials shows that the main topics are: 1) the use of renewable resources and production by-products; 2) phase change materials, principles of their operation, reviews of historical development, classification, application, fields of application; 3) insulation materials, both existing and experimentally created by scientists; 4) manufacturing methods, technologies and properties of board materials, performance improvement and use of renewable components.
- In the Doctoral Thesis, based on the research review and practical analysis, for the first time in laboratory conditions, using the cold pressing method, industrial hemp straw boards with microencapsulated PCM dispersion mixed into the mass up to 15 % in volume were made, and the physical and mechanical properties of the plate variants were determined.
- The aim of the Doctoral Thesis was achieved as a result of complex research by integrating hemp by-products into the composition of insulation boards for indoor decoration, improving their performance by incorporating microencapsulated phase change materials of biological origin into the board structure for the intensification of functional properties.
- The developed cold pressing board manufacturing technology for embedding microencapsulated PCM dispersion in the board manufacturing process and the materials used ensure temperature regulation in indoor spaces, ensuring an even climate in the room, practically without increasing the mass of the building.
- The developed prototypes of boards from hemp straws and pine longitudinal milling chips in a ratio of 50 : 50 with 10 % urea-formaldehyde resin glue and a percentage of microencapsulated PCM dispersion from 5 % to 15 % are more resistant to water absorption.
- Certain thermal, physical, acoustic and mechanical properties show that the boards are a good thermal and acoustic insulation material.
- Thermal conductivity decreases as the percentage of PCM increases; the best thermal conductivity results achieved without PCM for the H and HW sample groups are 0.062–0.063 W/m·K, and the best thermal resistance results are H 0.41 (m^2·K)/W.
- The incorporated phase change microcapsules are able to increase the heat capacity of the board, reaching the best result of 3.97 J/(g·K) at 15 % PCM, increasing the indicator by 147 % compared to boards without PCM.
- Good sound absorption properties at frequencies 500–6300 Hz, the best absorption coefficient W 0.69, and the porous structure of the board can improve the acoustic properties. Boards most effectively absorb sound in the frequency bands of 1000–2000 Hz and 4000–6300 Hz; that corresponds to sound absorption classes B and C (1000–4000 Hz).
- Fire reaction tests with a conical calorimeter prove that the boards with microencapsulated PCM emit 30 % more smoke (43.95 m^2/m^2), consume 14 % more oxygen, ignite 1.49 times faster; the total amount of heat released is between 45 and 51 MJ/m^2, and the difference in mass loss is within 1 %, the specific loss coefficient is different by 0.3 %; heat release for the samples with PCM in 60 s ranges from 120.7 to 136.92 kW/m^2.
- As a result of the analysis of the mechanical properties, it was concluded that the ultimate bending strength of the insulation board prototypes is relatively low at 1.26–1.43 MPa,

lower than for the samples with PCM in the composition; it is not recommended to use them as free-standing, but to use them in frame constructions as fillings or to strengthen the load-bearing ones in constructions, for example, by fixing with screws to the surface. The highest achieved axial withdrawal resistance of the screws for the H group samples is 23.2 MPa.

- The developed technology allows to tint the boards, add other fillers, as well as create a surface with relief.
- The developed board prototypes can be used indoors in a dry environment; thanks to their porosity they can absorb noise; the built-in PCM creates a uniform climate in the room.

Proposals, future vision – topics for further studies

- Increasing the material's properties against burning by adding microcapsules with flame retardant filling.
- The possibilities of utilisation by grinding or burning, the impact of the material on the environment.
- To develop a technology for the production of multi-layer boards by varying the thickness of the layers and the optimal position of the PCM layer in the room.

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