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PCM Modified Gypsum Hempcrete with Increased Heat Capacity for Nearly Zero Energy Buildings

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***Abstract* – Low energy building materials based on natural and renewable resources have become popular among customers. The natural occurrence of the raw materials and the idea to move closer to nature with its natural products have brought high interest in hempcrete. Hempcrete is a kind of binder-aggregate material where besides mineral binder, hemp shive aggregate act as a filler. A good technical performance of such composites has been reported before, while the term an 'advanced material' for traditional hempcrete lacks some superior properties for civil engineers. This research offers advanced hempcrete-type material where gypsum binder and hemp shives are used as the main components. Additionally, phase change material (PCM) was incorporated into the mixture composition to increase their thermal mass. PCM gives additional thermal heat storage for buildings which makes the building envelope more homogenous regarding thermal stability under outer temperature fluctuations. This could give additional comfort during heating and cooling seasons. Up to 20 wt.% of microencapsulated PCM suspension had been added to the hempcrete mixture composition and heat capacity was calculated and validated with differential scanning calorimetry measurements. Physical and mechanical performance was also evaluated. Results indicate that in the temperature interval from 20 to 30 °C, the heat capacity of hempcrete can be increased to 1901 J/(gK) (by 70.4 %) and it correlates well with theoretical thermal mass calculation. This gives promising results for further development of the material and proves the feasibility of PCM integration in hempcrete.**

***Keywords* – Calorimetry; gypsum; hempcrete; hemp shives; PCM**

1. INTRODUCTION

Climate changes have been determining the way how the construction sector is evolving. Therefore, new approaches to sustainable construction material development could reduce carbon footprint and preserve natural resources is in scope. This comprises environmentally friendly thermal insulators, materials for reducing building cooling needs, materials with lower embodied energy, materials that can reuse a large amount of waste, and high-tech energy-efficient building materials [1]. The production of construction materials with a high waste content that may be reused is an important research field for waste disposal as a resource. The sustainable building envelope is especially essential because recent studies have found a strong link between the absence of green infrastructure in urban areas and an increase in inhabitant health problems [1]. The solution can be found in nature and also in the direction of locally available waste. Agricultural by-products can offer a source of natural aggregates and in a mixture with the binder, a bio-composite so-called 'hempcrete' can be obtained [2]. Hemp shives are the most commonly utilized because they are less susceptible to biological

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deterioration than other organic materials (e.g., straw) [3]. Traditional hempcrete binder is lime, while other binders such as gypsum, organic binders, and geopolymers could be used [4]. Hempcrete is commonly used as a filament in timber frame, slabs, wall sidings, bricks, or blocks in construction [3]. Hempcrete based on lime and hemp shives with a density from 220 to 375 kg/m^3 (λ = 0.08–0.11 W/(mK)) can be used in roofing, walls, and floors, while hemp shive plaster has higher binder content and its density can reach up to 950 kg/m^3 [5]. The problem with lime-based hempcrete is its slow drying time, which can take between 6 and 8 weeks depending on the weather content and air humidity. Insulation materials made of natural fibers typically have a low thermal mass (density around 20–100 kg/m^3, specific heat capacity 1000–1300 J/(kgK)), thus intensifying high and low-temperature fluctuation risks [6]. Hemp insulation absorbs nearly twice as much heat as mineral insulation, substantially enhancing the comfort conditions in the interior. This is associated with the degree of water content, which plays significant role in lightweight materials [5]. This is explained by the fact, that the specific heat capacity of water is higher than hemp shives or mineral binder, and so the moisture contributes significantly for partially or fully saturated lightweight building materials. The moisture buffer capacities for plasterboard made from gypsum and hemp fiber is higher compared to hemp-lime bio-composite which means that they can stabilize the humidity of a room by adsorbing when the relative humidity increases and desorbing as it decreases [7]. Adsorbed water in the material is a natural effect while it does not provide an effective and long-term solution for thermal mass increase. Phase change material (PCM) could be part of above-mentioned solutions. PCM are new type of materials which possess large specific latent heat capacity. PCM is typically utilized in a building envelope and it should melt and solidify between 18 °C and 30 °C to reach maximal efficiency. These materials can absorb and release up to 15 times more thermal energy than conventional (non-PCM) materials. Regardless of the origin of PCM, the heat of fusion is between 120 and 280 kJ/kg [7]. PCM should have good thermal conductivity to improve its charging and discharging properties (i), efficient in a small volume of space (ii), with high specific heat (iii) and inert to volumetric changed during phase shift to reduce possible contamination of bearing or lining material [11], [12]. PCM incorporated in building materials can absorb redundant heat, thus reducing the load of HVAC systems and save energy consumed. Previously it was reported that the thermal properties of concrete and plasters can be improved with incorporations of PCM [8]–[10].

The problem associated with the PCM application is its proper integration inbuilding environment [13]. One of the most popular approaches is related to PCM microencapsulation. In microencapsulation technology, the PCM is introduced in a polymeric film, which must remain thin, sealed and typically are with high molecular weight, which allows to maintain the shape (usually round microgranules) and therefore prevents the PCM from leakage during the phase change process [14]. This is one of the most common ways of introducing PCM in newly constructed building envelopes without deterioration of base material.

There were some efforts to incorporate PCM in bio-based building materials. Urea-formaldehyde resins with hemp shives and PCM showed specific heat capacity from 1444 to 2369 J/(gK) with a density from 284 to 302 kg/m^3 and thermal conductivity from 0.064 to 0.074 W/(mK). The specific heat capacity value of such hemp shive wallboard, with 5 wt.% encapsulated PCM was increased by 62 % [15]. Such an approach proved feasible and sustainable and further evaluation on mineral binder-based hemp shive bio-composite should be tested for broader application of such material.

Gypsum and hemp shive hempcrete are the objects of this research. This study aims to evaluate the improvement of thermal energy storage gypsum hempcrete with the integration of commercial microencapsulated PCM. In this stage, the bio-based insulation materials with increased thermal mass and low thermal conductivity were developed and tested.

2. MATERIALS AND METHODS

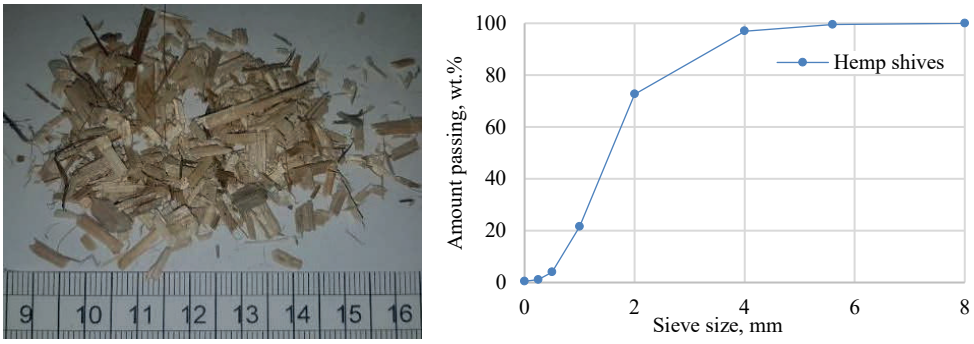
2.1. Raw materials

Commercial gypsum produced by Knauf Latvia Ltd. (BG) was used as binding material. BG particle size was characterized by d_{10} = 0.08 mm, d_{50} = 0.13 mm and d_{90} = 0.22 mm. The early age strength at 2 h was 8 MPa and the final strength at dry state was 16 MPa (according to LVS EN 13279-2). The set time t_{in} was 12:30 min and the final set time t_{fin} was 18:00 min. The set time of GB was taken into account during the production of hempcrete to avoid early hydration of hempcrete before casting was finished. The chemical composition of BG is given in Table 1. 88.97 % of the mixture is gypsum, also SiO_2, Al_2O_3, MgO, Fe_2O_3 are present. Loss on ignition for hydrated gypsum is 22.43 wt.%.

TABLE 1. CHEMICAL COMPOSITION OF COMMERCIAL GYPSUM, WEIGHT %

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	TiO_2	P_2O_5	LOI	Other	Total
Gypsum	3.73	1.68	0.46	35.64	3.92	30.9	0.31	0.05	–	22.43	0.58	100

Hemp shives (HS) were employed as a bio-based filler to create gypsum hempcrete. Fig. 1 illustrates the morphology of HS and its particle size distribution. Flat and elongated particles ranging in size from 2 mm to 40 mm make up hemp shives. Stevulova *et al.* studied the effect of HS mean particle length on hempcrete compressive strength and found that compressive strength increases as the mean particle length of HS decreases [16]. This was linked to a denser hempcrete structure with fewer pores, as well as higher heat conductivity. d_{10} of 0.7 mm, d_{50} of 1.6 mm, and d_{90} of 3.4 mm were the particle size distribution's characteristics. Depending on the chosen compaction method, large compaction intervals can be achieved. The bulk density of HS varies between 96 and 158 kg/m^3. HS had a maximum water absorption of 500 weight percent. Water absorption should be taken into account when combining HS with a binder, as extra water is required to compensate for HS's water absorption. The water solution with HS had a pH of 5.4–5.69.



a) b)

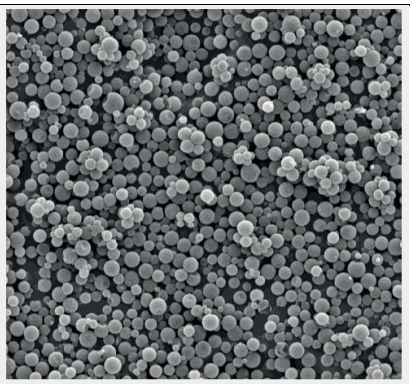
Fig. 1. Hemp shive macrostructure (a) and particle size distribution (b).

MikroCaps PCM25-S50 (Slovenia) were used as phase change material (PCM). Selected PCM is microencapsulated and is delivered in concentrated dispersion with an off-white slurry appearance. PCM is encapsulated in a formaldehyde-free polyurethane membrane and total PCM content in dispersion is between 41–45 wt.%. The characteristics of PCM are given in

Table 2. For selected microcapsules, characteristic values of latent heat of fusion were from 140 to 175 kJ/kg.

Differential scanning calorimeter (DSC) analysis of PCM was carried out for four samples. Samples were prepared from a dried PCM emulsion under room conditions in airtight aluminum crucibles. The tests were carried out in the heating-cooling-heating mode. The heat of fusion was determined from the cooling and reheating segments. At first, PCM stabilization at $T = -90$ °C was carried out, then heating up to $T = 60$ °C with a heating rate of 10 K/min was done, followed by cooling down to $T = -90$ °C with the rate of 5 K/min and then heating up to $T = 60$ °C with the rate of 10 K/min was repeated. Results are given in Fig. 2.

TABLE 2. CHARACTERISTIC PROPERTIES OF PCM

Property	**Value**	**Appearance of microencapsulated PCM**
Type of PCM	Paraffin wax	
Type of membrane:	PU	
PCM content in the dispersion, wt.%	41–45	
PCM content in dry capsule, wt.%	82–86	
Dry content in the dispersion, wt.%	49–52	
PCM melting area, °C	23–27	
Heat storage capacity, J/g	140–175	
pH	6.0–9.5	
Density, kg/m^3	880–950	
Viscosity at 25°C, cPs	500–1500	
Average particle size, µm	1–15	

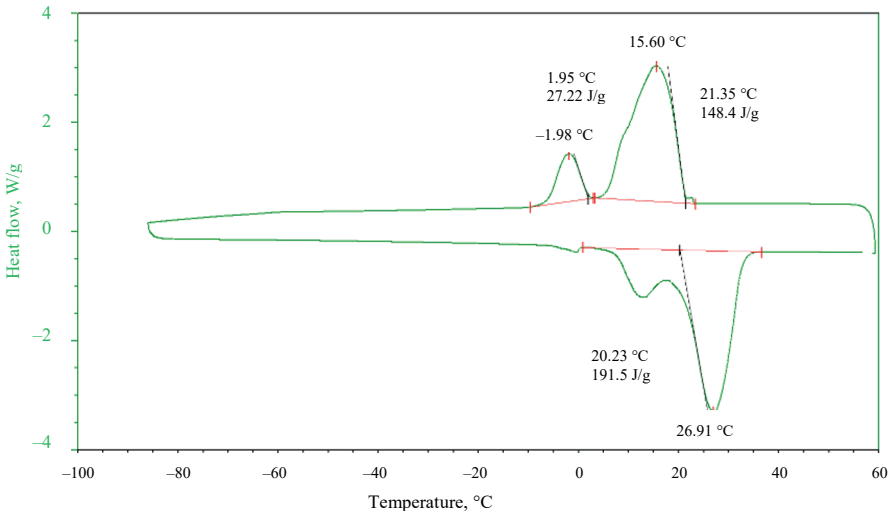


Fig. 2. Differential scanning calorimeter analysis of phase change material Microcaps PCM25-S50.

Two enthalpy ranges were observed. During the cooling process, the water freezing was observed at –1.98 °C, then phase change of PCM was observed at 15.6 °C. The phase change temperature (melting temperature: Tm and crystalline temperature: Tc) and latent heat (melting enthalpy: ΔHm and crystalline enthalpy: ΔHc) are two of the phase change properties that

define the application range and value of PCMs. Thermograms show two crystalline phases whose phase change takes place at different temperatures ($T \approx 21$ °C and $T \approx 27$ °C). The total phase change of two crystalline phases shows the heat of fusion ranging from 184.4 J/g to 193.5 J/g (for heating), similar to the datasheet provided by the producer. The heat of crystallization of the phase showed larger peak ranges from 144.8 J/g to 153.7 J/g.

2.2. Methods

The thermal conductivity of gypsum hempcrete was determined using a NETZSCH model HFM 446 Lambda heat flow meter system with NETZSCH-Proteus-HFM-Software for samples with the dimension of 20 cm × 20 cm × 4 cm. The system works according to ASTM C 518, ISO 8301, DIN EN 12667, DIN EN 13163, and JIS A 1412. In the literature, the measurement principle for thermal transmission characteristics is presented [15]. The dynamics heat flow meter method (DHFMA) was used for non-direct measurement and determination of the particular heat capacity Cp (J/(gK)) in addition to the determination of thermal conductivity [15], [17]. When utilizing PCMs with construction materials, their melting temperature must be consistent with the working temperature of the room, which is usually between 22 and 28 °C. In such a way, the heating was delivered in three ranges in the test procedure: between 10°C and 20°C, between 20 °C and 30 °C (this range should include phase change of PCM), and between 30 °C and 40 °C.

After plate sample testing, samples were cut to 4 cm × 4 cm × 4 cm cubes for the compressive strength test. A Zwick Z100 universal testing machine was used to conduct the tests. The pressure was applied at a rate of ten millimetres per minute. The compressive strength test was carried out until 10 % relative deformations occurred (according to the LVS EN 826). Moisture content was determined after demolding by the immediate weighting of plate samples and further drying of plates at 60 °C until a constant mass was achieved. Then after drying, samples were conditioned at room temperature at 22±2 °C at RH 60±5 % and before thermal conductivity tests, the samples were weighted to calculate adsorbed water. Hitachi TM3000 Tabletop Scanning Electron Microscope was used to analyze the microstructure of gypsum hempcrete.

2.3. Mixture composition and sample preparation

Four mixture compositions were prepared. Reference composition consisted of BG 300 kg/m^3, HS of 122 kg/m^3, and 300 kg/m^3 of mixing water for wetting and workability of gypsum hempcrete (Table 3). PCM suspension with a ratio of 5 %, 10 %, and 20 % of gypsum weight was incorporated in mixture composition (compositions PCM5, PCM10, and PCM20). The total amount of PCM suspension was 15, 30, and 60 kg/m^3 (PCM content was from 6.5 to 25.8 kg/m^3).

TABLE 3. MIXTURE COMPOSITION OF GYPSUM HEMPCRETE WITH PCM

Composition	HS, kg/m^3	BG, kg/m^3	Water, kg/m^3	PCM suspension kg/m^3			Total, kg/m^3
				Suspension	Water	PCM	
REF	122	300	300	0	0	0	722
PCM5	122	300	300	15	8.6	6.5	737
PCM10	122	300	300	30	17.1	12.9	752
PCM20	122	300	300	60	34.2	25.8	782

The sample preparation procedure is given in Fig. 3. First, all components were weighted and PCM suspension was mixed with water. Then the prepared solution was poured into the

hemp shives and mixed for one min to wet HS. Then gypsum powder was incorporated and the mixture was mixed throughout for two min. The homogenized mixture was cast in a 20 cm × 20 cm × 5 cm plywood formwork, and the mixture was pushed together with screws in such a way that 20 % deformations from initial case position occurred. The whole time for mixing and casting was from 7 to 8 minutes. After the gypsum set, the samples were released and removed from formwork and placed in a 60 °C drying room. Specimens of 20 cm × 20 cm × 4 cm dimensions were made. The samples were put through previously described tests after achieving a consistent mass.

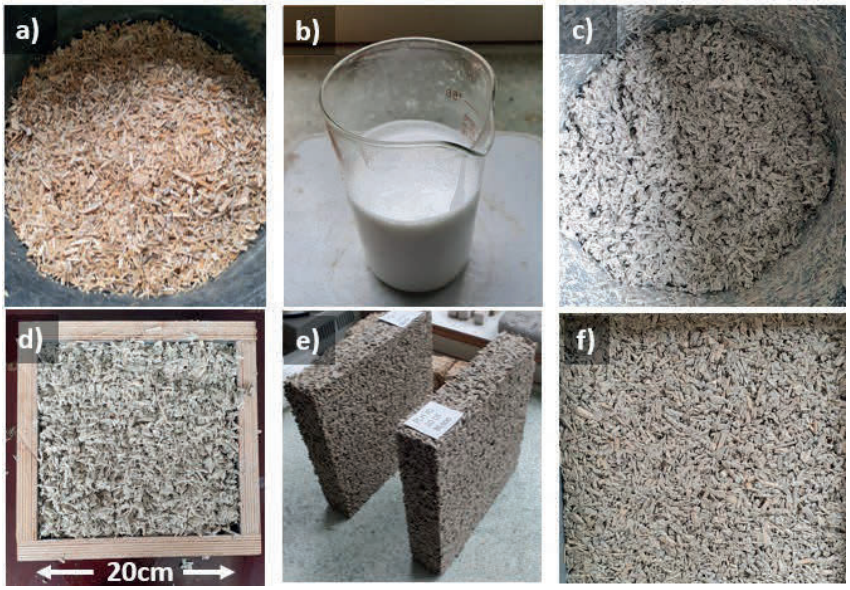
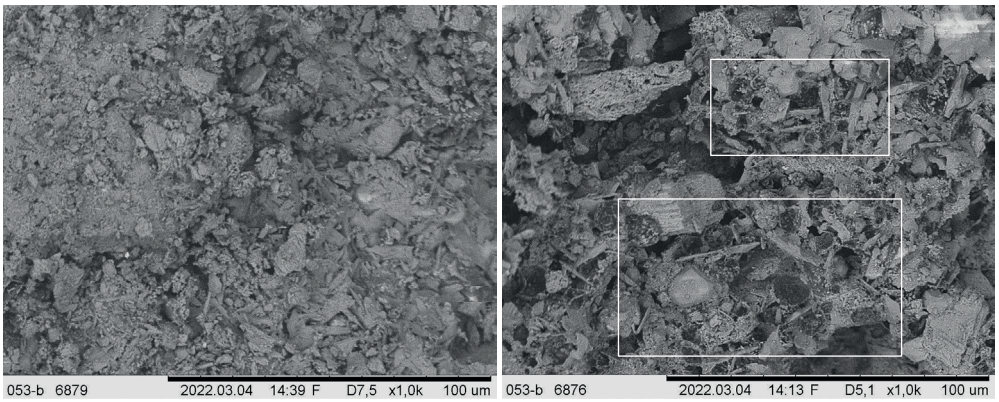


Fig. 3. The preparation procedure of gypsum hempcrete. a) hemp shives, b) PCM solution; c) fresh mixture of gypsum hempcrete, d) casted gypsum hempcrete samples; e) samples after demolding; f) macrostructure of gypsum hempcrete.

3. RESULTS AND DISCUSSION

3.1. Microstructure

The microstructure of REF with pure gypsum binder is visible in Fig. 4(a). Individual gypsum grains are visible with some voids and spaces between them. Gypsum mainly covers HS aggregate while gypsum hempcrete remains open macro-structure between grains. In Fig. 4(b), it is visible more porous gypsum paste with circular voids and spheres in its structure. PCM slurry, which had been mixed in composition, was mixed with gypsum binder, therefore, PCM microcapsules remained between gypsum paste and it is visible under magnification of ×1000. Clear identification of separate microencapsulated PCM was hardly detected while traces of PCM microcapsule voids were visible in structure. The appearance of the gypsum binder phase correlates well with results regarding physical and mechanical properties.



(a) (b)

Fig. 4. The microstructure of gypsum hempcrete: a) reference composition without PCM; b) PCM20 with 20wt.% of PCM.

3.2. Physical properties

The physical properties of gypsum hempcrete are given in Table 4. The moisture content of freshly produced and demolded gypsum hempcrete was from 61–65 wt.%. With the increase of PCM content, free moisture content increased in the structure of gypsum hempcrete as additional free water was added with the PCM solution. Dried samples had been conditioned in a room environment and the adsorbed water content of hempcrete was from 1.7 to 2.2 wt.%. Adsorbed water was slightly reduced by the addition of PCM. The bulk density of gypsum hempcrete was from 366–392 kg/m^3. Density was slightly reduced due to the PCM as polymer material content increased and the mineral phase decreased.

The average specific heat capacity (*C*p) of hempcrete was measured in three temperature intervals to evaluate the phase change effect of PCM. Results are given in table 4 and Fig. 5. It is visible that *C*p of gypsum hempcrete increased in the PCM working range between 20 and 30 °C. *C*p of gypsum hempcrete was from1.074 to 1.270 J/(gK) at 10–20 °C, and *C*p from 1.066 to 1.199 J/(gK) was at 30–40 °C. A small *C*p increase below 20 ℃ was associated with the early-phase change of PCM and this effect corresponds to DSC results and did not increase *C*p significantly. An increase of *C*p in the temperature range from 20–30 ℃ occurred due to the phase change of PCM. *C*p increased by 16.7 % for PCM5, 31.8 % for PCM10 and 70.4 % for PCM 20. A good correlation between incorporated PCM and *C*p increase was observed.

Thermal conductivity was not affected by PCM content in hempcrete. It was from 0.098 to 0.102 W/mK. Such results show improvement of hempcrete properties made with gypsum and PCM in its composition. In comparison to other reports on lime-based hempcrete, the density of hempcrete from 291 to 336 kg/m^3 resulted in thermal conductivity from 0.0868 to 0.1007 W/(mK) and specific heat capacity from 1.250 to 1.557 (J/gK) [18].

TABLE 4. PHYSICAL AND THERMAL PROPERTIES OF GYPSUM HEMPCRETE

	Moisture content, wt.%		**λ_d**	**Density**	***C*p, J/(gK)**		
	Produced	**Adsorbed**	**W/mK**	**kg/m^3**	**10–20 °C**	**20–30 °C**	**30–40 °C**
REF	61	2.2	0.102	378	1.074	1.115	1.161
PCM5	61	2.2	0.099	392	1.141	1.301	1.141
PCM10	63	1.7	0.101	378	1.091	1.374	1.066
PCM20	65	1.9	0.098	366	1.272	1.901	1.199

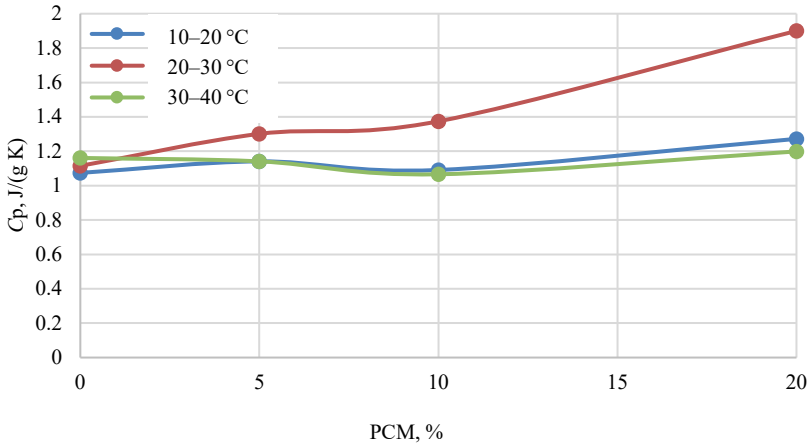


Fig. 5. Average specific heat capacity of gypsum hempcrete with PCM at different temperature ranges.

The efficiency of PCM in hempcrete was evaluated by calculating theoretical heat capacity according to mixture composition and weight of compounds in the sample and comparing it to experimentally obtained results. To assess the theoretical heat capacity of prepared samples, mixture composition was recalculated for every mixture depending on the dry weight of samples. In such a way, certain weight of compounds was known for further calculation.

For theoretical calculations, *C*p of individual compounds was taken from the literature. The specific heat capacity of hemp shives typically is between 1.476 and 1.675 J/(gK) [19], [20] and in this scenario, it was assumed to be 1.500 J/(gK). Gypsum used in the production of gypsum board has *C*p between 0.852 and 1.034 J/(gK) [21]. Here it was assumed to be 1.000 J/(gK). The heat capacity of PCM was taken from DSC experimental results and was assumed to be 191 J/g during phase shift interval. The *C*p for adsorbed water value of 4.100 J/(gK) was used [22].

Results given in table 5 were calculated to the interval of 10 °C so that the phase change of PCM in the gypsum hempcrete is completed. Then the heat capacity was calculated as an average of 1 °C in the selected interval, similar to results provided by DSC. It is visible from the results that the total energy needed to heat each gypsum hempcrete composition increases as PCM content is increased.

TABLE 5. THEORETICAL SPECIFIC HEAT OF GYPSUM HEMPCRETE ACCORDING TO MATERIAL CONTENT AND COMPOSITION IN GYPSUM HEMPCRETE SAMPLES

Component mass in sample, g				
	REF	PCM5	PCM10	PCM20
HS	184	189	191	195
BG	453	464	470	479
Water	14	14	15	15
PCM	0	10	20	41
Cumulative specific heat calculation in gypsum hempcrete, J				
	REF	PCM5	PCM10	PCM20
HS	276	283	287	292
BG	521	533	541	550
Water	58	59	60	61
PCM	0	1905	3862	7861
Total heat at dT = 10 °C	8122	10127	12102	16051
Total heat at dT = 1 °C	812	1013	1210	1605

Specific heat capacity calculations and experimental results are given in Table 6. It was calculated that the specific heat of reference gypsum hempcrete without PCM was 1.15 J/(gK) while the measured value was 1.12 J/(gK) and the difference was 3.3 %. A good correlation was obtained also for mixtures with PCM. The difference between measured values was from 7.8 to 13.4 %. A closer correlation to experimental values can be obtained, if PCM producers provided specific heat capacity would be taken (150 J/g), then the correlation would fit between 95 and 98 %. This proves that phase change shift of integrated PCM occurs in the structure of gypsum hempcrete and such an approach of PCM microcapsule integration efficienty increases amount of energy storage in a certain temperature fluctuation interval. Depending on PCM amount in composition, PCM in gypsum hempcrete contributes from 15.4 to 42.7 % of the total specific heat value in the interval from 20 to 30 °C.

TABLE 6. COMPARISON OF CALCULATED AND MEASURED PCM EFFICIENCY IN GYPSUM HEMPCRETE

	Specific heat capacity, calculated, J/(gK)	**Specific heat capacity, Measured, J/(gK)**	**Difference, %**
REF	1.15	1.12	3.3
PCM5	1.40	1.30	7.8
PCM10	1.65	1.47	12.5
PCM20	2.15	1.90	13.4

3.3. Compressive strength of gypsum hempcrete

The compressive strength of gypsum hempcrete with different amounts of PCM in the composition is given in Fig. 6. Compressive strength was determined in casting direction and across casting direction until 10 % of deformation occurred from the initial sample height. In casting direction, strength for REF mixture composition was 552 kPa. The incorporation of small PCM amount (PCM5) did not take any effect on strength of bio-composite (544 kPa), while with the higher rate reduced strength to 411 kPa (PCM10) and 387 kPa (PCM20).

Strength reduction was from 25 to 30 % which is associated with higher water content in mixture composition, which comes from PCM slurry and the PCM capsules themselves can weaken the bonding of gypsum binder with hemp shive aggregates. Strength across casting direction was 17–41 % lower than strength obtained in casting direction. This is attributed to the hemp shive orientation during casting and applied pressure. The longitudinal orientation, which matches the load application direction gave lower strength results. On the other hand, PCM did not make a significant influence on the strength results across casting direction – it was 281–327 kPa. Previous reports on hempcrete based on hydrated lime, metakaolin, and hydraulic lime achieved strength 110–470 kPa with density 290–338 kg/m^3 which gives a similar strength-density ratio with gypsum binder used in this research [18].

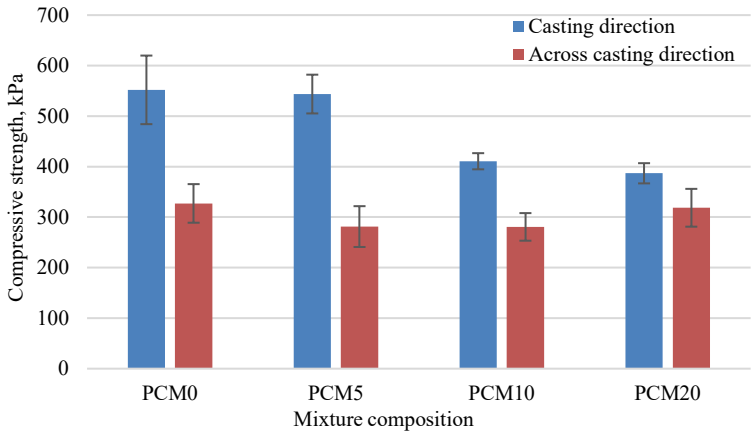


Fig. 6. Compressive strength of gypsum hempcrete with PCM at 10% deformation and depending on casting direction.

4. Conclusion

Gypsum-hemp shive bio-composite proved to be suitable and effective in combination with integrated micro-encapsulated PCM with its content up to 20 wt.% of gypsum binder. PCM slurry did not have a significant effect on the density of the bio-composite (366–392 kg/m^3) and thermal conductivity (0.098–0.102 W/(mK)). Compressive strength was 387–552 kPa in casting direction and 281–327 kPa across casting direction (17–41 % difference). The strength of bio-composite was reduced by 25–30 % when PCM content was between 10–20 wt.% which could indicate weakened bonding of hemp shives with gypsum binder. Heat capacity in the PCM working range was increased up to 70.4 % and it increased 1.115–1.901 J/(gK) which gives promising results for improved thermal capacity and efficiency results of such bio-composites in building envelope. The experimental results of heat capacity correlate well with the theoretical calculation. This proves the phase change of PCM in the selected interval and is a significant step in reaching a nearly zero-emission building envelope. Increased heat capacity of bio-based gypsum-hemp shive composite could reduce the energy consumption of heating and improve thermal comfort in the building, which will be further investigated in a climate chamber under a controlled environment.

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