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Research of the physical properties of bio-based building materials with phase change material

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Abstract

This article presents the results of experimental measurements of the physical properties of new environmentally friendly bio-based composite building materials containing hemp shives bonded with a magnesium binder. Some of the tested materials contained an admixture of phase change material (PCM) of variable proportions in the binder to increase the heat capacity of building elements (walls), which can positively affect room temperature regulation. Densities and porosities are key parameters describing building materials, directly affecting mechanical, acoustic, and, most importantly, hygro-thermal properties, including thermal conductivity, water vapor permeability, water absorptivity, and sorption curves. The experiment was carried out for ten different samples of bio-based building composites, differing in the bulk density obtained during the manufacturing process and in the PCM proportion. As part of the experiment, true density tests were conducted on a helium pycnometer. Then, the geometric densities of the tested materials (which may differ from the bulk density obtained during production) were measured using the Archimedes method, making it possible to obtain the total, closed, and open porosity values. Tests were also carried out for selected traditional building materials, such as red brick and autoclaved aerated concrete, to compare the results obtained.

Keywords: Bio-based building materials; Bio-based composites; Magnesium binder; Hemp shives; Phase change material (PCM); Helium pycnometer; Porosity measurement

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1. Introduction

The growing interest in sustainability across all industries is prompting, among others, a search for innovative building materials in which environmentally friendly plant wastes can partially replace standard building materials. A major advantage of such solutions is concrete utilization reduction in the production of building materials, which contributes to decreasing carbon dioxide emissions into the atmosphere while ensuring the use of plant waste such as hemp shives [1,2]. Moreover, bio-based building composites are easier to recycle and often have an additional advantage as they have better thermal conductivity than traditional building materials, making buildings more energy efficient.

Nomenclature

m_{dry} – mass of a dry sample, kg
m_{sat} – mass of a saturated sample containing isopropyl alcohol, kg
$m_{sat,air}$ – mass of a saturated sample containing isopropyl alcohol, measured in isopropyl air, kg
$m_{sat,iso}$ – mass of a saturated sample containing isopropyl alcohol, measured in isopropyl alcohol, kg
V_g – geometric volume, m^3
V_p – open pore volume, m^3
V_t – total volume, m^3

Greek symbols

ϵ_c – closed porosity
ϵ_o – open porosity
ϵ_t – total porosity
ρ_g – geometric density, kg/m^3
ρ_l – liquid density, kg/m^3
ρ_t – true density, kg/m^3

Subscripts and Superscripts

Air – measurement performed in air
Iso – measurement performed in isopropyl alcohol

Abbreviations and Acronyms

PCM – phase change material

Moreover, this solution can still be improved by adding phase change materials (PCMs) to the binder, e.g., in the form of microcapsules, which are designed to increase the thermal capacity of building elements (e.g., walls) through phase change transformation [3–5]. In this way, energy losses to the environment can be reduced during cold weather, while heat gains can be decreased during hot periods [5], and, as a result, energy consumption needed for space heating and cooling can be lowered [6]. Hemp-based building materials have very good thermal insulation properties (i.e., their thermal conductivity is typically in the range of 0.08–0.2 W/(m K) [7]), good acoustic performance, and very good moisture accumulation and buffering behavior. However, traditional hemp concrete based on a lime binder is very fragile and has very weak mechanical properties, i.e. its compressive strength is typically below 0.5–0.6 MPa [7]. It cannot be used in places directly exposed to water, i.e. the hemp concrete wall must be protected from direct rainfall with plaster and by protruding roof eaves and from groundwater by horizontal waterproofing and a raised foundation. However, hemp concrete is characterized by low microbiological corrosion because hemp shives consist of only the woody inner part of the hemp stalk and contain no nutrient elements. It is mainly used as a wall filling material, for thermal insulation, and to regulate the indoor microclimate, but it cannot be considered a load-bearing material. Therefore, many modifications of this material were proposed in the literature [8] in order to improve its mechanical performance and increase its application range. One of the possible ways is to use alternative binders that have better mechanical properties than those based on lime ones [9]. For example, the use of magnesium binders, as in the case of the material analyzed in this paper, can increase the strength up to 3.8 MPa [9].

The prospects for using PCMs in the building sector, including application in bio-based composite building materials, are widely reported in the literature. These materials are studied on both the numerical simulation and experimental levels. For example, Sawadogo et al. [10] conducted experiments in which different properties of hemp concrete with PCM (i.e. capric acid), such as melting and solidification temperatures and enthalpies, thermal conductivity, and general thermal performance, were investigated. A similar experiment was conducted by Meng et al. [11] where, by using simulations and an experiment, they compared the parameters inside an ordinary room and a room additionally insulated with a PCM layer and presented possible efficiency gains with the use of this material.

Hemp concrete as a bio-based composite material does not have clearly and easily defined thermal and moisture parameters [12]. It is influenced by many factors, from the type of binder and its admixtures [8,13], the amount of binder used, the degree of grinding of the plants [7,14], and the harvesting period of the hemp seed [15]. The plurality of solutions available, whether in terms of available binders, PCMs, or the form of PCM added to the building material itself, makes it necessary to pay great attention to the development of available experimental databases that could be used for mathematical modeling of hygro-thermal performances [16,17] or comparison of bio-based building materials.

Key physical parameters describing the structure of building materials include density and porosity. Their values directly affect mechanical [18], acoustic [19], and hygro-thermal properties [20,21], including thermal conductivity [22,23], water vapor permeability [8], and moisture sorption [24,25]. It is supposed that some of these properties depend more specifically on the pore size or the type of pores in the structure of the materials [26,27], hence it is important not only to know the total porosity but also the open and closed porosity or the pore size distribution in the structure. Glé et al. [24] point out that open and closed porosity values have an especially important role in determining the acoustic properties of materials. Wei et al. [28] conclude that the lower open porosity results in lower water absorption and weaker evaporation performance.

Methodology for measuring the porosity of building materials containing bio-additives, like hemp shives, is a matter of concern due to the occurrence of pores in biocomposite materials ranging in size from a few nanometers to even millimeters [29]. Brewer et al. [29] used helium pycnometry combined with powder micro-granular pycnometry to calculate the densities and porosities of biochar. Jiang et al. [30] identify radiation-based (e.g. microscopy) and porosimetry-based (such as water immersion porosimetry) methods as the main experimental techniques while suggesting the use of several of them to be able to achieve a comprehensive research result. Glé et al. [24] extensively describe aspects of density and porosity measurements of hemp shives themselves and suggest pycnometry (powder, helium, air, nitrogen, or mercury-based) as the optimal method for measuring the properties of ground biocomposites. They also suggested

X-ray computed tomography as one option, but due to its time-consuming nature, it should be rather used for local structure studies. Delanoy et al. [31] studied the porosities of two different hemp concretes (with a lime-based binder and a natural cement) using an air porosimeter. Wei et al. [28] studied the properties of bio-based composite building materials created from corn residue and magnesium cement using the Archimedes method and acetone-based pycnometry. Ferroukhi et al. [32] used pycnometry to find densities and porosities of hemp concrete based on a lime binder containing PCM. However, experimental data on the physical properties of bio-based building composites studied in this paper, i.e. hemp concrete created with magnesium binder, is difficult to find in the literature, and additionally, such results for materials containing PCM are also notably lacking.

This article presents the results of an experimental study in which the values of geometric and true density, as well as total, open, and closed porosity, were measured for 10 samples of bio-based building composites made of hemp shives and magnesium binder. Three of them contained microencapsulated PCM in different proportions, seven were bio-based composite samples with a different bulk density, and additionally, two conventional building materials were studied for comparison purposes. A helium pycnometer was used to determine total porosity and true density, while the Archimedes method based on measurements in isopropyl alcohol was used to determine open porosity.

2. Description of the method and materials investigated

2.1. Investigated materials

Bio-based composite materials with different bulk densities obtained during the manufacturing of the biocomposite samples by the research team from the Riga Technical University were tested [9]. All samples were made by mixing hemp shives, magnesium binder (MgO), magnesium chloride ($MgCl_2$) water solution, and water needed for the initial preparation of materials. To 3 samples, microencapsulated PCM was added in the form of concentrated water dispersion. Different mass shares of constituents were used to obtain tested composites, as presented in Table 1.

In the applied hemp shives (Natūralus Pluoštas, Lithuania), almost 74% of all plant pieces were characterized by lengths ranging from 1 to 30 mm, with the longest pieces reaching up to 40 mm. The base bulk density of the hemp components was in the range of 80–110 kg/m^3, while compacted bulk density was 115 kg/m^3. Magnesia named RKMH-F (produced by RHI AG, Austria) and calcined at the temperature of 750°C was used to prepare the composite. Its composition consists of 74% MgO with an admixture of CaO, SiO_2, Fe_2O_3, and Al_2O_3 in a range of 1–4%. Magnesium chloride (Chemische Fabrik Kalk, Germany) in the form of flakes was applied and, before adding to the mixture, was initially diluted in water in a 1:1 by-mass proportion. Additionally, some water was used to initially moisturize dry hemp particles, which strongly absorb water [33].

PCM-containing samples were produced by adding concentrated water dispersion of microcapsules MikroCapsPCM25-S50 (MikroCaps d.o.o., Slovenia) with the paraffin wax enclosed in a polyurethane shell. The average size of the microcapsules varied from 1 to 15 μm, and the PCM content inside the dry capsule was 82–86%, while in the dispersion, it was 41-45% wt. Paraffin used was characterized by latent heat in the range of 140–175 kJ/kg and melting/solidification temperature of 23–27°C.

The PCM-free materials were designated D200, D300, D400, D500 and D600 (as listed in Table 1) referring to their approximate bulk densities. The densities of these samples varied due to the use of variable amounts of MgO and $MgCl_2$ solution relative to the other components (see Table 1). Also, the sample Ind400 was analyzed, which was specified for its different industrial methods of manufacture. Samples containing PCMs were named PCM0, PCM5, PCM10 and PCM20. Moreover, autoclaved aerated concrete and red brick, available in the Polish market, were used as comparative conventional building materials.

The compositions of all tested bio-based composite samples were calculated with reference to the mass proportion of hemp shives (see Table 1). The numbers in the names of samples with PCM, i.e. 0, 5, 10 and 20, refer to the mass ratio of the PCM-water dispersion to the mass of MgO used for composite preparation. In fact, sample PCM0 does not contain PCM, but is the

Table 1. Ingredients contents for preparing bio-based composite samples referring to 1 unit of hemp shives (mass proportions).

Sample name	Hemp shives	MgO	$MgCl_2$ solution 1:1 with water	Water to initially moisten the hemp	PCM water dispersion (41-45% wt. of PCM)	PCM mass fraction (%)
D200	1.00	0.50	0.32	1.30	-	-
D300	1.00	1.00	0.63	0.70	-	-
D400	1.00	1.93	1.22	0.50	-	-
D500	1.00	2.44	1.53	0.50	-	-
D600	1.00	3.00	1.89	0.50	-	-
Ind400	1.00	1.85	1.17	1.00	-	-
PCM0	1.00	2.00	1.26	1.00	0.00	0.0
PCM5	1.00	2.00	1.26	1.00	0.10	1.2
PCM10	1.00	2.00	1.26	1.00	0.20	2.3
PCM20	1.00	2.00	1.26	1.00	0.40	4.5

reference sample for other materials in this batch, the production of which required a different composition of ingredients than the samples named "D", as shown in Table 1. Sample PCM0 has a composition most similar to D400, but during production, a bit more binder and twice as much water to pre-wet the hemp shives were used. Taking into account the mass shares of the individual components, the mass shares of PCM in the samples were 0.0, 1.2, 2.3 and 4.5% for samples PCM0, PCM5, PCM10 and PCM20, respectively.

2.2. Total porosity investigation using a helium pycnometer

Four small fragments were extracted from different parts of a larger block of tested materials to measure the true density and total porosity of developed bio-based building composites, from which 13 finely ground samples were prepared for measuring on a helium pycnometer. The materials were ground to fine dust using a laboratory grinder, with liquid nitrogen applied to increase the brittleness of the bio-based fibers and facilitate the grinding of these samples. All samples were then dried in a laboratory drier at 60°C for at least 5 days. Weight measurements showed that this period was sufficient to consider the ground samples dried. After drying, density measurements were started using a helium pycnometer AccuPyc 1345.

The ground material (as shown in Fig. 1) was poured into a cleaned, dry-air rinsed and weighed 3.5 cm^3 steel vessel and then weighed. A laboratory scale with an accuracy of 0.001 g was used to weigh the samples. To increase the accuracy of the measurement, an effort was made to place as much powdered material as possible in the vessel, additionally kneading it (the device required a certain minimum mass of material to be placed inside the steel vessel). The sample was then placed inside the device and sealed tightly. Figure 1 also shows a filter, which is essential for conducting experiments on bulk substances. The measurement began with 10 cycles of purging the prepared sample with helium to remove air and any residual moisture and ensure homogeneous conditions inside, followed by 30 measurement cycles. Each cycle consisted of filling the vessel with a certain volume of helium until an overpressure of about 1.35 bar at 25°C was reached. When pressure stability was reached, an automatic valve to a vessel of known volume was opened, and the system was again waiting for equilibrium to be reached. Knowing the thermodynamic parameters, the device calculated the volume of the material, and using the known mass of the sample, its true density (ρ_t) could be determined. Based on the information about the true density and geometric density, the total porosity of the test samples was determined.

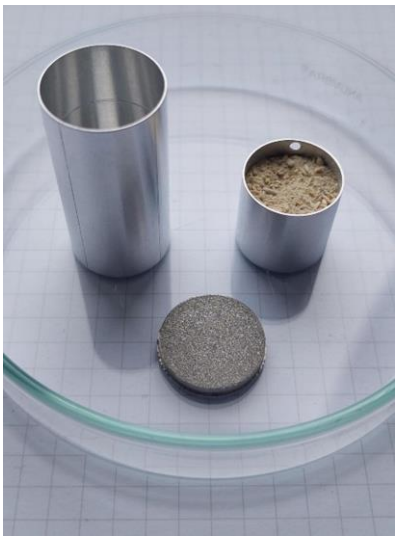

Fig. 1. Exemplary sample prepared for testing in helium pycnometer.

2.3. Open porosity investigation

The bio-based composite samples in the shape of cubes with sides of about 50 mm (some of which are shown in Fig. 2), 3 of each type that came from the same production batch as the samples investigated on the pycnometer, were tested. For conventional materials, 4 cuboidal samples were prepared from autoclaved aerated concrete and brick with volumes similar to the bio-based composite samples (i.e. in the range of 105–180 cm^3). Samples were cut out of the large blocks using an angle grinder, and then they were smoothed and aligned with sandpaper to obtain parallel walls. After the samples were prepared, they were placed in a laboratory drier and dried at about 60°C to remove moisture from them. Over the following days, the weights of the samples were recorded until they reached a constant mass of m_{dry} in three consecutive measurements. Then, the dimensions of the samples were measured to the nearest 0.01 mm, three times in each of the three dimensions, to be able to estimate their geometric volumes (V_g) and then calculate their geometric densities (ρ_g).

After drying, the samples were weighed, and then saturated with isopropyl alcohol. To do this, they were placed inside a sealed vessel filled with isopropyl alcohol to the point that the samples were completely submerged, and then a vacuum pump was used to create a vacuum (at –0.85 bar gauge pressure) to remove air from the pores. The samples were in the vessel until it was determined that no more air bubbles were coming out. Higher-density materials required several hours to remove air, while porous materials required at least 2–3 hours. After saturation, the samples were removed from the vessel and immediately placed in a dish with isopropyl alcohol located on a scale, where their apparent masses ($m_{sat,iso}$) were measured (the operation is shown in Fig. 3). During the measurements, care was taken that the sample placed in the glass beaker did not come into contact with the walls and the wires that held the dish. Measuring the mass in isopropyl alcohol took from 2 to as long as 5 hours. Samples with higher density needed much more time to stabilize. After the tests, the density of isopropyl alcohol (ρ_l) was determined using Archimedes' law and a standard of known volume. This one may have varied depending on the room conditions or as a result of the dissolution of substances contained in the bio-based building composite samples.

Fig. 2. Archimedes test samples (from the left: D300, D500, PCM20, red brick, and autoclaved aerated concrete).

After measuring the apparent mass, the samples were removed from the vessel, slightly wiped from the outside to remove the lingering liquid layer from the surface, and then weighed to obtain the mass of the sample saturated with isopropyl alcohol ($m_{sat,air}$). The open porosity was calculated based on mass measurements, and with known total porosity, the closed porosity value can be obtained. This was done using the following steps.

Geometric density is defined as the ratio of a dry sample's mass (m_{dry}) to its geometric volume (V_g):

$$\rho_g = \frac{m_{dry}}{V_g}. \tag{1}$$

The volume of open pores is calculated from the following equation:

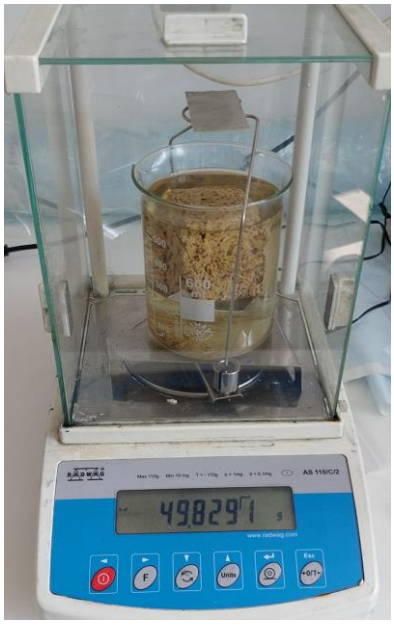

Fig. 3. A sample during measurement of apparent mass in isopropanol.

$$V_p = \frac{m_{sat,air} - m_{dry}}{\rho_l}, \tag{2}$$

where: ρ_l is isopropyl alcohol density, and $m_{sat,air}$ is the mass of the sample saturated with isopropyl alcohol measured in air.

The total volume, which is the volume of the solid, including the pores inside its structure, is defined as the difference in the saturated weight of the sample measured in air ($m_{sat,air}$) and in isopropyl alcohol ($m_{sat,iso}$):

$$V_t = \frac{m_{sat,air} - m_{sat,iso}}{\rho_l}. \tag{3}$$

Open porosity is defined as the ratio of open pore volume (V_p) to total volume (V_t):

$$\epsilon_o = \frac{V_p}{V_t}, \tag{4}$$

while total porosity is determined from the formula:

$$\epsilon_t = \left(1 - \frac{\rho_g}{\rho_t}\right) \cdot 100\%, \tag{5}$$

where ρ_t is a true density, which is the density of a solid phase.

Closed porosity is calculated as the difference between total porosity and open porosity:

$$\epsilon_c = \epsilon_t - \epsilon_o. \tag{6}$$

3. Results

From the results shown in Fig. 4 it can be deduced that the geometric density results of samples that do not contain PCM are very close to the assumed bulk density values included in the sample names. The largest difference occurs for D200 samples containing the lowest amount of binder. The largest standard deviation values were also achieved for these samples. This may be due to the brittleness of the samples since the small amount of binder used in their production made them extremely fragile, and they may have lost some material. As a result, their mass might be smaller and their geometric dimensions irregular, resulting in difficulties in measuring accurately. It is worth noting

that the geometric density value of Ind400 samples is slightly higher than that of D400. For the comparative materials, it can be seen that red brick is much denser, which means it is also more massive, while autoclaved aerated concrete places its density between D500 and D600 samples.

Samples containing PCM achieve similar geometric densities for each composite type (see Fig. 5). As the content of the PCM increases, no clear trend in the density variation is noticed. Moreover, the standard deviations for the samples with PCM are low, and all results obtained were similar for every sample type. Most interestingly, even though the basic PCM0 samples were manufactured with ingredients fractions very similar to the samples labeled D400, it can be seen from a comparison of Figs. 4 and 5 that the values of their geometric densities are much closer to those of D500. This is due to the higher amount of water used during the preparation of the PCM0 mixture than for D400, which resulted in a more liquid mixture consistency and greater susceptibility to compaction.

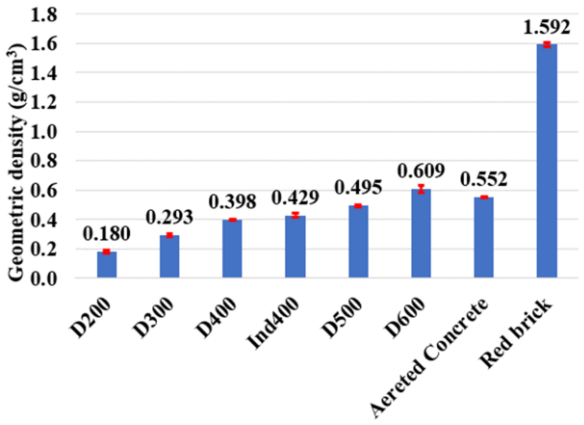

Fig. 4. Mean geometric density of the tested samples without PCM (minimal and maximal expanded uncertainty: 0.004 and 0.044 g/cm^3, respectively).

The results in Fig. 6 show the relationship between true density and bulk density (bio-based composite samples are ordered according to the rise in geometric density). An increase in true density with an increase in bulk density can be observed due to a higher amount of binder in denser samples and, thus, a lower proportion of hemp shives – the trend is clear. Moreover, the values of the true density of pure hemp shives and magnesium binder are also presented for comparison. Since magnesium binder is denser than plant fibers, samples with higher bulk density have more of it. Samples labeled D200 and D300 differ significantly in their true density from the observed trend for others, and a significant spread of results for D200 should be pointed out. These results show that achieving an even distribution of the binder in the low-density samples in the manufacturing process is difficult, and samples could be highly macroscopically non-homogenous. In contrast to the results of the geometric density test, the differences between D400, D500, D600 and Ind400 samples are relatively small. In this case, the density of the D400 samples is higher than the value for Ind400. However, it is worth noting that autoclaved aerated concrete has a higher

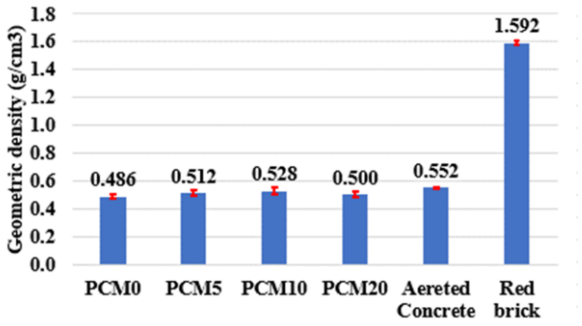

Fig. 5. Mean geometric density of the tested samples containing PCM (minimal and maximal expanded uncertainty: 0.004 and 0.055 g/cm^3, respectively).

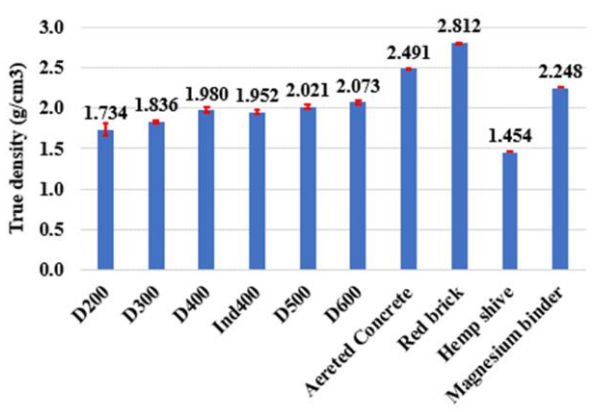

Fig. 6. Mean true density of the tested samples without PCM (minimal and maximal expanded uncertainty: 0.019 and 0.0149 g/cm^3, respectively).

true density than all bio-based composite samples, unlike its geometric density.

Samples containing PCM have almost the same true densities. However, comparing Figs. 6 and 7, it can be seen that they have values slightly smaller than those of the D400 and Ind400 samples and much lower than those of D500.

True density values (sometimes called solid density) for hemp concrete made of lime-metakaolin binder found in the literature range from 1.655 to 2.155 g/cm^3 [34]. These values should be considered only as a certain point of reference due to the different compositions of the materials tested and the use of different binders. Nevertheless, they allow for the conclusion that the results obtained in this work are realistic. Interesting results of density studies were presented by Ferroukhi et al. [32], who show that as the proportion of PCM in the bio-based composite structure increased (i.e., PCM amount was 0, 5, 10, and 15% of the mass of lime binder), the values of true density and geometric density significantly decreased. For samples investigated in this work, Figs. 5 and 7 show only small changes in density values with a change in the PCM proportion. Figure 5 shows an increase in density with an increase in PCM for samples PCM0, PCM5 and PCM10, but sample PCM 20 disrupts this trend, while in Fig. 7, true density decreases with the rise in the PCM amount, similarly as in [32]. Slightly different trends

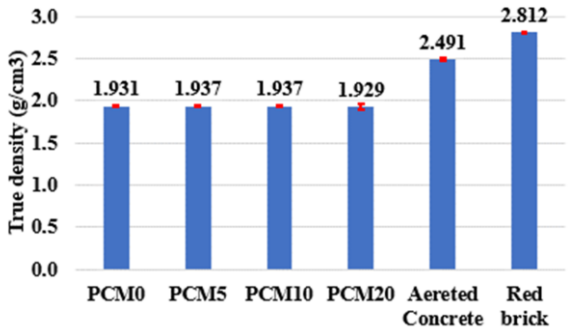

Fig. 7. Mean true density of the tested samples containing PCM (minimal and maximal expanded uncertainty: 0.019 and 0.057 g/cm^3, respectively).

in [32] can be due to much higher shares of PCM in the tested composites than in this work, i.e. 3.3, 6.6 and 9.2% wt. vs. 1.2, 2.3 and 4.5%, respectively, that can better reflect general composites behaviour.

The total porosity values of the bio-based composites shown in Fig. 8 vary from nearly 90% for the D200 samples to 70% for the D600 samples. These differences are naturally due to the varying mass content of hemp shives, which are much more porous than the magnesium binder, i.e., the lower the sample density, the higher the mass share of hemp shives, and the higher the porosity. Moreover, less binder resulted in more macropores in the composite. The porosity value of autoclaved aerated concrete does not differ from biocomposites and is very close to Ind400, while red brick is much less porous through its very compact structure.

The results in Fig. 9 show that the total porosity of the samples with PCM is similar to each other and close to that of the D500 samples but slightly lower than that of autoclaved aerated concrete. No correlation between the PCM content in the composite and its porosity value was observed.

The open porosity results in Fig. 10 show that this porosity decreases with the bulk density of the samples. Again, the values for D200 and D300 are significantly different (i.e., higher) than for the other composites. A similar level of open porosity as for

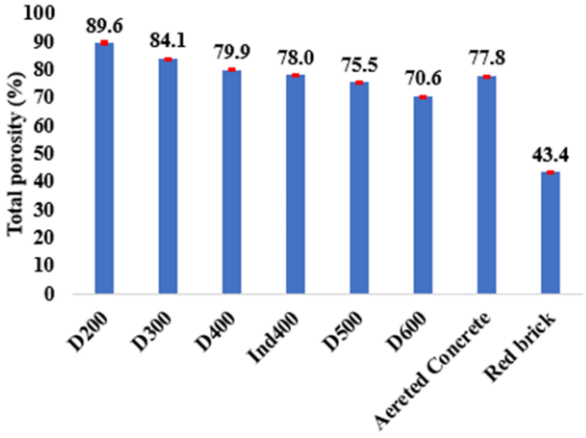

Fig. 8. Mean total porosity of the tested samples without PCM (minimal and maximal expanded uncertainty: 0.61 and 3.04 pp, respectively).

D200 and D300 achieves the autoclaved aerated concrete. Moreover, the Ind400 open porosity value clearly exceeds that of D400. Among all the samples tested, the values of open porosity significantly exceed those of closed porosity, which is largely related to the structure of the hemp shives that are highly porous with closed pores.

The lowest open and closed porosity has red brick. Its closed porosity value is only 3.2%, while the open one is 40.2%. The autoclaved aerated concrete has a slightly higher open porosity of 3.6%. This shows well the structure of autoclaved aerated concrete, which is blown during production, and many small cracks are formed in it. The closed porosity values of bio-based composites range from 10.3% to 16.5% and seem chaotic – no relationship between bulk density and closed porosity is visible.

For PCM samples, the open porosity fluctuates around 63%, which is comparable to D400 samples and much less than autoclaved aerated concrete (see Fig. 11). This time, however, an interesting relationship was obtained for the closed porosity results – as the proportion of PCM increases, the value of closed porosity decreases from 11.5% for PCM0 to 10.0% for PCM20.

Literature values for the total porosity of lime-metakaolin-based hemp concrete reach values in the range of 72–79% [34].

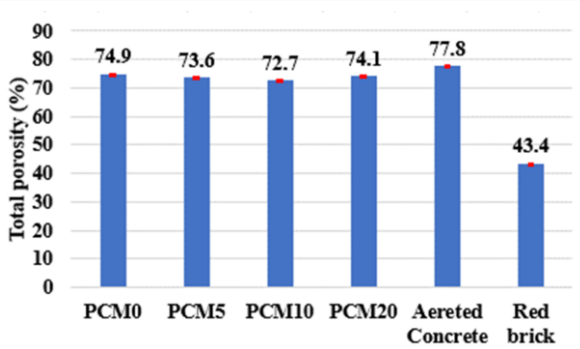

Fig. 9. Mean total porosity of the tested samples containing PCM (minimal and maximal expanded uncertainty: 0.61 and 3.51 pp, respectively).

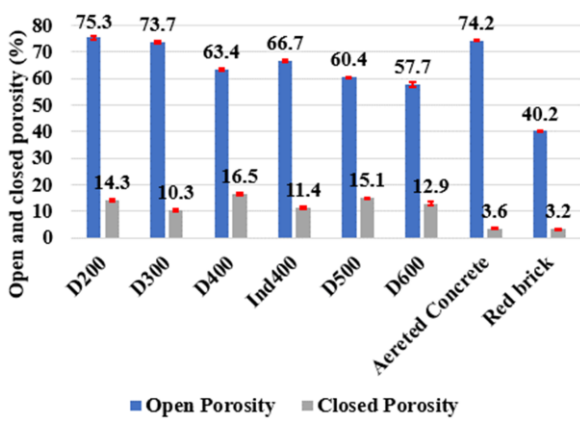

Fig. 10. Mean closed and open porosity of the tested samples without PCM (minimal and maximal expanded uncertainty: 0.74 and 1.74 pp for open porosity, and 1.54 and 4.09 pp for closed porosity, respectively).

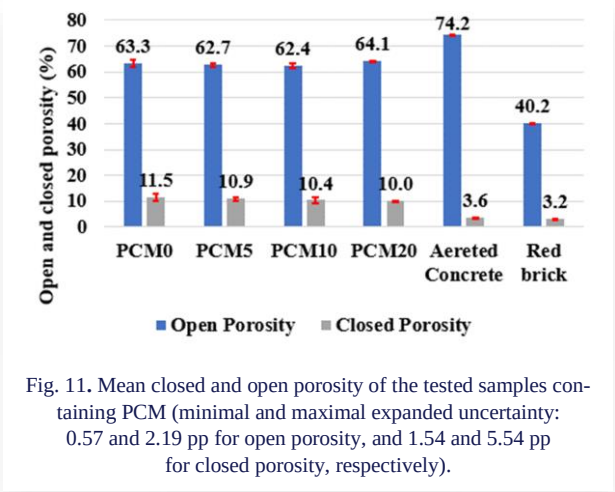

Fig. 11. Mean closed and open porosity of the tested samples containing PCM (minimal and maximal expanded uncertainty: 0.57 and 2.19 pp for open porosity, and 1.54 and 5.54 pp for closed porosity, respectively).

In this study, total porosities of 89.6% and 84.3% were achieved for specimens D200 and D300, respectively, with a much lower density than those tested in the literature, while other composites' results are in line with the data presented in [34]. An interesting aspect is the studies of open porosity presented in the literature, where results ranged from 50% for the vacuum saturation method [35] to 76% for the air porosimetry [36]. The porosity results achieved by Ferroukhi et al. [32] for PCM materials were in the range from 74.9% for a reference sample to 73.1% for a sample named HC15PCM. The values shown in Fig. 9 are very close to these, although one should consider the different compositions of the biocomposite samples and different constituents used in [31] and this study.

All combined standard uncertainties presented in Figs. 4–11 take into account both measurement uncertainties due to inaccuracies of the instruments (mainly related to weight measurements on balances, but also geometrical measurements, volume with a pycnometer or isopropyl alcohol density) and standard deviations of the results obtained from multiple samples. In the case of the uncertainty calculation for total porosity, where each of the 13 samples had a different uncertainty in volume measurement, the measurement with the largest relative uncertainty was selected, and this error was assumed for the whole material. Finally, an expanded uncertainty was calculated by applying a coverage factor $k = 2$, for which the confidence coefficient is better than 95%.

4. Conclusions

In the conducted experiments, the values of the basic parameters of bio-based composite building materials, such as densities (i.e. true and geometric) and porosities (total, open, and closed), were measured. The results obtained can find application as input data in numerical models simulating the hygro-thermal behavior of wall elements or help in assessing the utility of considered building materials. The basic properties of the biocomposites were also compared with autoclaved aerated concrete, some of whose properties were quite similar to bio-based materials, and with the much denser and less porous red brick.

The applied measurement techniques achieved results that can be considered realistic when compared to similar experiments described in the literature. For densities, total porosity, and open porosity measurements, results with satisfactory measurement uncertainties were obtained, while it should be highlighted that a significant part of these uncertainties may be due to the heterogeneous structure of the biocomposite building materials. However, it should be emphasized that the calculated values of the closed porosity of the tested materials, due to the large relative uncertainties (i.e. reaching even close to 100%), can only be considered indicative. To achieve more reliable closed porosity measurement results, a different measurement method should be developed, which is dependent on a lower number of intermediate measurements.

Most of the parameters studied show a clear relationship between the bulk density of the samples (which is controlled by the amount of binder added to their production) and the individual parameters. As the bulk density decreases, the true and geometric densities decrease also while the porosity increases. The situation is completely different in the case of samples containing PCM – a correlation between the mass share of microcapsules with PCM and the studied parameter was found only for the closed porosity. This may indicate a lack of regularity during manufacturing or a small effect of the microencapsulated PCM on the studied parameters. In terms of properties, the samples with PCM reach values between those obtained for D400 and D500.

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References

[1] Jami, T., Karade, S.R., & Singh, L.P. (2019). A review of the properties of hemp concrete for green building applications. *Journal of Cleaner Production*, 239, 117852. doi: 10.1016/J.JCLEPRO.2019.117852

[2] Ahmed, A.T.M.F., Islam, M.Z., Mahmud, M.S., Sarker, M.E., & Islam, M.R. (2022). Hemp as a potential raw material toward a sustainable world: A review. *Heliyon*, 8(1), e08753. doi: 10.1016/J.HELIYON.2022.E08753

[3] Wu, D., Rahim, M., El Ganaoui, M., Bennacer, R., Djedjig, R., & Liu, B. (2022). Dynamic hygrothermal behavior and energy performance analysis of a novel multilayer building envelope based on PCM and hemp concrete. *Construction and Building Materials*, 341, 127739. doi: 10.1016/J.CONBUILDMAT.2022.127739

[4] Rathore, P.K.S., & Shukla, S.K. (2021). Enhanced thermophysical properties of organic PCM through shape stabilization for thermal energy storage in buildings: A state of the art review. *Energy and Buildings*, 236, 110799. doi: 10.1016/J.ENBUILD.2021.110799

[5] Lamrani, B., Johannes, K., & Kuznik, F. (2021). Phase change materials integrated into building walls: An updated review. *Renewable and Sustainable Energy Reviews*, 140, 110751. doi: 10.1016/J.RSER.2021.110751

[6] Bennai, F., Ferroukhi, M.Y., Benmahiddine, F., Belarbi, R., & Nouviaire, A. (2022). Assessment of hygrothermal performance of hemp concrete compared to conventional building materials at overall building scale. *Construction and Building Materials*, 316, 126007. doi: 10.1016/J.CONBUILDMAT.2021.126007

[7] Brzyski, P., Gładecki, M., Rumińska, M., Pietrak, K., Kubiś, M., & Łapka, P. (2020). Influence of Hemp Shives Size on Hygro-Thermal and Mechanical Properties of a Hemp-Lime Composite. *Materials*, 13(23), 5383. doi: 10.3390/MA13235383

[8] Łapka, P., Brzyski, P., Pietrak, K., Cieślikiewicz, Ł., & Suchorab, Z. (2023). Hygro-thermal characterization of the hemp concrete modified with the gum Arabic admixture. *Construction and Building Materials*, 368, 130392. doi: 10.1016/J.CONBUILDMAT.2023.130392

[9] Zorica, J., Sinka, M., Sahmenko, G., Vitola, L., Korjakins, A., & Bajare, D. (2022). Hemp Biocomposite Boards Using Improved Magnesium Oxychloride Cement. *Energies*, 15(19), 7320. doi: 10.3390/EN15197320/S1

[10] Sawadogo, M., Benmahiddine, F., Hamami, A.E.A., Belarbi, R., Godin, A., & Duquesne, M. (2022). Investigation of a novel bio-based phase change material hemp concrete for passive energy storage in buildings. *Applied Thermal Engineering*, 212, 118620. doi: 10.1016/J.APPLTHERMALENG.2022.118620

[11] Meng, E., Yu, H., & Zhou, B. (2017). Study of the thermal behavior of the composite phase change material (PCM) room in summer and winter. *Applied Thermal Engineering*, 126, 212–225. doi: 10.1016/J.APPLTHERMALENG.2017.07.110

[12] Ghosn, S., Cherkawi, N., & Hamad, B. (2020). Studies on Hemp and Recycled Aggregate Concrete. *International Journal of Concrete Structures and Materials*, 14(1), 1–17. doi: 10.1186/S40069-020-00429-6

[13] Brzyski, P. (2021). The Influence of Gum Arabic Admixture on the Mechanical Properties of Lime-Metakaolin Paste Used as Binder in Hemp Concrete. *Materials*, 14(22), 6775. doi: 10.3390/MA14226775

[14] Williams, J., Lawrence, M., & Walker, P. (2018). The influence of constituents on the properties of the bio-aggregate composite hemp-lime. *Construction and Building Materials*, 159, 9–17. doi: 10.1016/J.CONBUILDMAT.2017.10.109

[15] Li, Y., Pickering, K.L., & Farrell, R. L. (2009). Analysis of green hemp fibre reinforced composites using bag retting and white rot fungal treatments. *Industrial Crops and Products*, 29(2–3), 420–426. doi: 10.1016/J.INDCROP.2008.08.005

[16] Dietrich, F., Łapka, P., Cieślikiewicz, Ł., Furmański, P., Sinka, M., Vitola, L., & Bajare, D. (2021). Micro-scale modeling-based approach for calculation of thermal conductivity of bio-based building composite. *AIP Conference Proceedings*, 2429, 020023. doi: 10.1063/5.0071466

[17] Łapka, P., Dietrich, F., Furmanski, P., Cieslikiewicz, L., Sinka, M., & Bajare, D. (2023). Method for Prediction of Thermal Conductivity of Bio-Based Building Composites Enhanced With Microencapsulated PCM. *AIP Conference Proceedings*, 2801, 030020. doi: 10.1063/5.0146677

[18] Madsen, B., Thygesen, A., & Lilholt, H. (2009). Plant fibre composites – porosity and stiffness. *Composites Science and Technology*, 69(7–8), 1057–1069. doi: 10.1016/J.COMPSCITECH.2009.01.016

[19] Degrave-Lemeurs, M., Glé, P., & Hellouin de Menibus, A. (2018). Acoustical properties of hemp concretes for buildings thermal insulation: Application to clay and lime binders. *Construction and Building Materials*, 160, 462–474. doi: 10.1016/J.CONBUILDMAT.2017.11.064

[20] Chikhi, M. (2020). Effective thermal conductivity of porous biomaterials: Numerical investigation. *Journal of Building Engineering*, 32, 101763. doi: 10.1016/J.JOBE.2020.101763

[21] Li, Z.J., Huang, Z.W., Dai, H.L., Yao, Y., & Li, Y.S. (2024). Hygrothermal coupled modeling and behavior analysis of natural fiber-reinforced tubular composites. *Construction and Building Materials*, 411, 134384. doi: 10.1016/J.CONBUILDMAT.2023.134384

[22] Chikhi, M., Agoudjil, B., Boudenne, A., & Gherabli, A. (2013). Experimental investigation of new biocomposite with low cost for thermal insulation. *Energy and Buildings*, 66, 267–273. doi: 10.1016/J.ENBUILD.2013.07.019

[23] Frantz, N., Dutra, L.F., Nguyen, D.M., Almeida, G., & Perré, P. (2024). Effects of phase ratios, density and particle shapes on directional thermal conductivity of vegetable concrete: A predictive model. *Construction and Building Materials*, 410, 134238. doi: 10.1016/J.CONBUILDMAT.2023.134238

[24] Glé, P., Lecompte, T., Hellouin de Ménibus, A., Lenormand, H., Arufe, S., Chateau, C., Fierro, V., & Celzard, A. (2021). Densities of hemp shiv for building: From multiscale characterisation to application. *Industrial Crops and Products*, 164, 113390. doi: 10.1016/J.INDCROP.2021.113390

[25] Collet, F., Bart, M., Serres, L., & Miriel, J. (2008). Porous structure and water vapour sorption of hemp-based materials. *Construction and Building Materials*, 22(6), 1271–1280. doi: 10.1016/J.CONBUILDMAT.2007.01.018

[26] Abbas, M.S., Gourdon, E., Glé, P., McGregor, F., Ferroukhi, M. Y., & Fabbri, A. (2021). Relationship between hygrothermal and acoustical behavior of hemp and sunflower composites. *Building and Environment*, 188, 107462. doi: 10.1016/J.BUILDENV.2020.107462

[27] Lawrence, M., & Jiang, Y. (2017). Porosity, pore size distribution, micro-structure. *RILEM State-of-the-Art Reports*, 23, 39–71. doi: 10.1007/978-94-024-1031-0_2

[28] Wei, Y., Song, C., Chen, B., & Ahmad, M.R. (2019). Experimental investigation on two new corn stalk biocomposites based on magnesium phosphate cement and ordinary Portland cement. *Construction and Building Materials*, 224, 700–710. doi: 10.1016/J.CONBUILDMAT.2019.07.100

[29] Brewer, C.E., Chuang, V.J., Masiello, C.A., Gonnermann, H., Gao, X., Dugan, B., Driver, L.E., Panzacchi, P., Zygourakis, K., & Davies, C.A. (2014). New approaches to measuring biochar density and porosity. *Biomass and Bioenergy*, 66, 176–185. doi: 10.1016/J.BIOMBIOE.2014.03.059

[30] Jiang, Y., Ansell, M.P., Jia, X., Hussain, A., & Lawrence, M. (2017). Physical characterisation of hemp shiv: cell wall structure and porosity. *Academic Journal of Civil Engineering*, 35(2), 22–28. doi: 10.26168/ICBBM2017.1

[31] Delannoy, G., Marceau, S., Glé, P., Gourlay, E., Guéguen-Minerbe, M., Diafi, D., Nour, I., Amziane, S., & Farcas, F. (2019). Influence of binder on the multiscale properties of hemp concretes. *European Journal of Environmental and Civil Engineering*, 23(5), 609–625. doi: 10.1080/19648189.2018.1457571

[32] Ferroukhi, M. Y., Mesticou, Z., & Si Larbi, A. (2023). Impact of microencapsulated phase change material on a bio-based building composite, hygrothermal and mechanical behavior. *Construction and Building Materials*, 409, 133925. doi: 10.1016/J.CONBUILDMAT.2023.133925

[33] Sahmenko, G., Sinka, M., Paurins, U., & Bajare, D. (2023). Production Technology of Ecological High Performance Fibre Composite Construction Materials. *Journal of Physics: Conference Series*, 2423, 012005. doi: 10.1088/1742-6596/2423/1/012005

[34] Seng, B., Magniont, C., & Lorente, S. (2019). Characterization of a precast hemp concrete. Part I: Physical and thermal properties. *Journal of Building Engineering*, 24, 100540. doi: 10.1016/J.JOBE.2018.07.016

[35] Rahim, M., Douzane, O., Tran Le, A. D., Promis, G., & Langlet, T. (2016). Characterization and comparison of hygric properties of rape straw concrete and hemp concrete. *Construction and Building Materials*, 102, 679–687. doi: 10.1016/J.CONBUILDMAT.2015.11.021

[36] Gourlay, E., Glé, P., Marceau, S., Foy, C., & Moscardelli, S. (2017). Effect of water content on the acoustical and thermal properties of hemp concretes. *Construction and Building Materials*, 139, 513–523. doi: 10.1016/J.CONBUILDMAT.2016.11.018