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Properties of fiber-gypsum composite formed on the basis of hemp (*Cannabis sativa* L.) fibers grown in Poland and natural gypsum

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Abstract: *Properties of fiber-gypsum composite formed on the basis of hemp (Cannabis sativa L.) fibers grown in Poland and natural gypsum.* The popularity of composites reinforced with natural fibers is constantly growing and therefore, they are a subject of many scientific works as well. An example of interesting concept is the use of hemp fibers to reinforce a gypsum matrix and therefore, presented study was aimed to determine the effect of their content on the properties of resultant composites. Moreover, the influence of setting temperature was also investigated. The scope of the research included determination of properties such as: density, setting time, bending strength, modulus of elasticity and thermal conductivity coefficient. Studies have shown that as the amount introduced fibers increases, the density of manufactured composites decreases. Furthermore, increase in the content of hemp causes a significant extension in setting time of the gypsum matrix. Based on the outcomes of mechanical properties, it was found that the optimal content of fibers is 4% and further increase in their share results in a deterioration of flexural strength characteristics. The increase in a setting temperature leads to the reduction in their bending strength and modulus of elasticity. Composites reinforced with hemp fibers demonstrate significantly improved thermal insulation properties.

Keywords: hemp fibers, natural gypsum, hemp fibers-gypsum composites

INTRODUCTION

Gypsum and concrete, which are considered as mineral binders, are popular due to the numerous advantages such as, for example, ease of processing, high availability and low price (Podawca 2007). In Poland, calcium sulphate is obtained mainly from following two types of sources: from the natural resources (national resources are estimated at nearly 254 million Mg) (Szuflicki et al. 2020) and from flue gas desulphurization process using the wet lime method in a coal-fired power plants (approx. 2.9 million Mg is produced this way annually) (Pichniarczyk 2000, Kania 2019). Regardless of the source, gypsum is characterized by the same chemical composition (Czernik et al. 2021). However, it can demonstrate different water-gypsum ratio, shape and size of the grains and strength characteristics of produced bonds. The biggest disadvantage of mineral binders is their brittleness (Musielak et al. 2001; Gontarz and Podgórski 2015) and the tendency to develop cracks within the structure (Hoštálková et al. 2019). In order to improve properties of the resultant boards and minimize the negative features, various reinforcement methods, such as introduction of natural fibers to the structure, have been widely studied for years.

An interesting example of a natural fibers source, which can be used as a reinforcement of mineral matrix is hemp. It has been used by mankind for thousands of years. Many factors contributed to the wide range of possibilities of hemp fibers application such as ease of cultivation, high adaptability to environmental conditions and the ability to use literally every part of the plant. The area of domestic crops is growing very dynamically. As stated by Czapaluk and Czerniak (2020) the total crop area in Poland is reaching 3027 ha. Despite the diversity of chemical composition, dimensional characteristic and mechanical properties of hemp fibers, which depend e.g. on habitat and weather conditions, they are constantly gaining popularity in many applications. Nowadays, it is estimated that about 25 000 products based on it are known and available on the market (Strzelczyk and Kaniewski 2021). What is interesting, besides the commercial cultivation, hemp is also used for phytoremediation of soils polluted by mining and metallurgical industries. Moreover, hemp fibers were also used as a reinforcement of various types of polymer matrixes, such as: polypropylene (Hargitai et al. 2008), polyethylene (Lu and Oza 2013), polystyrene (Bhowmik et al. 2022), poly (lactic acid) (Song et al. 2012), and the obtained outcomes are very promising.

Therefore, the aim of the conducted research is to expand the knowledge in the field of manufacturing gypsum composites reinforced with hemp fibers grown in Poland on the basic properties of the obtained materials.

MATERIALS AND METHODS

Following materials were used for production of fibers-gypsum composites. Hemp fibers (*Cannabis Sativa* L.) of the Białobrzeska variety. The introduced fibers were characterized by a diameter of approx. 117 μ m and average length of 51.4 mm. Furthermore, building gypsum of natural origin was applied as a matrix for composites. It was used due to a relatively quick setting time and a good strength. The gypsum grains with spherical shape had an average size of 48 μ m.

Both reference gypsum boards and experimental fibers-gypsum composites with assumed density of 1100 kg/m³ were produced in laboratory conditions. All variants were manufactured by implementing the same technological procedure regarding water-gypsum ratio which was kept at the same level of 0.6:1. The fibers-gypsum composites differed in the content of introduced hemp fibers (weight ratio of gypsum dry mass to fiber dry mass). Following amounts of lignocellulosic reinforcement were applied to the gypsum matrix: 2%, 4%, 6% and 8%. Prior to mixing fibers were dried in laboratory oven until the moisture content of approx. 6% was achieved. The composites were manufactured with the use of form having a dimensions of 255 mm × 355 mm. After formation process was done, they remained in the form for 12 hours. In order to determine the effect of setting temperature on the properties of composites, they were left for hardening in 20°C, 40°C and 60°C. Then, obtained materials were cut to obtain the samples with the dimensions depending on the test they were intended for.

Density of manufactured composites was determined according to EN 323 (1999). Measurements of linear dimensions were performed with an accuracy of 0.01 mm using the electronic calliper. Thickness of boards was measured with the use of thickness gauge with an accuracy of 0.01 mm.

Measurements of gypsum matrix setting time was carried out according to EN 13279-2 (2014). It was measured from the moment of mixing water with gypsum, with an accuracy of 30 seconds. Five repetitions were performed for each variant.

The determinations of static bending strength (f_m) and modulus of elasticity (E_m) were conducted in accordance with EN 310 (1999). Samples used for tests were characterized by a linear dimensions of 255 mm × 50 mm and thickness depending on the variant. Ten samples from each variant were used for investigations.

Determinations of the thermal conductivity coefficient of manufactured composites were carried out with the use of device presented in Figure 1. It was equipped with two chambers: heating chamber (target temperature of $60 \pm 1^{\circ}$ C) and receiving chamber (maximum temperature of $40 \pm 1^{\circ}$ C). Test sample with dimensions of 350 mm × 255 mm was placed between the chambers. The APAR controller was responsible for both controlling the temperature inside the chambers and collecting results of measurements in a regular 1 second intervals.



Figure 1. Schematic presentation of the device used for thermal conductivity coefficient determination (1 - heat source, 2 – tested sample, 3 - thermocouples, 4 – external fan, 5 – internal fan, 6 – barrier dissipating the heat flux, 7 - heating chamber thermocouple, 8 - heat flux sensor, 9 – receiving chamber thermocouple, 10 – APAR controller)

After stabilizing the conditions inside the chambers, the experiment lasted for 2 hours minimum. On the basis of the maximum surface voltage determined by the sensor, the value of heat flux density going through the sample was calculated using formula 1:

$$q = U_t \times C \tag{1}$$

where: q – heat flux density (W/m²), C – calibration coefficient of 35.8 (W/m² mV), U_t – voltage (mV)

The heat transfer coefficient was calculated according to formula 2. Temperatures used for calculations were the average values of about 100 stabilized measurements.

$$\lambda = \frac{q \times d}{t_i - t_e} \tag{2}$$

where: λ – heat transfer coefficient (W/m·K), q – heat flux density (W/m²), t_i – upper chamber temperature (°C), t_e - lower chamber temperature (°C), d – thickness (mm).

RESULTS AND DISCUSSION

Figure 2 shows the effect of hemp fibers content introduced into the gypsum matrix on density of the resultant panels. As the fibers content increased, the density of the manufactured fibers-gypsum composites decreased which was evidenced by the coefficient of determination R^2 . The highest values of density were found in the case of gypsum board without the addition of fibers, while the lowest for the fibers-gypsum board containing 8% of hemp fibers. Observed effect can be explained by a significant difference in the volume of the mixture of gypsum with fibers which indicates that it is not possible to increase the content of fibers to more than 8% (the ratio of dry mass of fibers to the dry mass of gypsum).



Figure 2. The effect of hemp fibers content on density of composites.

The effect of hemp fibers content on the setting time of fibers-gypsum composites is shown in Figure 3. Hemp fibers as a hygroscopic material have the natural ability to absorb and release the moisture. When they were introduced into a gypsum matrix, the fibers got soaked in mixture of water with gypsum. However, the soaked fibers released water for a longer time than the gypsum matrix that surrounded them. It probably extended the setting time of the matrix because gypsum as a hydrophilic material absorbed water from fibers at the further stages of setting process which is shown by the significant extension in setting time with the increasing content of hemp fibers. The difference between the extreme variants, i.e. gypsum board and the composite containing 8% of fibers was over 42 minutes, which means that the setting time was more than four times longer. It is especially important because some studies indicated that hindrance in gypsum setting process can noticeably affect the mechanical properties of produced composites (Chłądzyński and Pichniarczyk 2006).



Figure 3. The effect of hemp fibers content on the setting time of composites.

Results of the strength properties of gypsum boards and fibers-gypsum composites are crucial for establishing their potential application. The first stage of research was aimed to determine the effect of the content of hemp fibers introduced to the gypsum matrix (the only variable) on bending strength (f_m) of manufactured composites in the case of which the setting process was conducted at 20°C (Figure 4). The lowest value of 3.1 MPa was observed for non-reinforced gypsum board. The introduction of hemp fibers in the amount ranging from 2% to

4% significantly increased bending strength results. However, further increase in the content of introduced fibers caused a significant deterioration of strength. Fibers-gypsum composite containing 8% of hemp has not been tested due to the inability to provide samples with dimensions required by the standard. The reason for observed deterioration in the case of variants assuming the addition exceeding 4% was too low share of gypsum providing the overall strength of the resultant composite.

The second stage of research concerned determination of the effect of temperature during the setting process on the bending strength outcomes (Figure 4). The highest strength was observed for the composites whose setting process took place at 20°C. In the case of composites with 2% fiber content, increasing the temperature to 40°C during the gypsum setting process resulted in a deterioration of strength values by 11.2% compared to the variant produced at 20°C. Further increase in setting temperature to 60°C resulted in even more noticeable decrease in strength by approx. 26%. The observed negative effect of increased setting temperature probably resulted from too rapid evaporation of water, which consequently led to creation of void spaces and development of micro-cracks within the board structure.



Figure 4. The effect of setting temperature on bending strength of composites containing various contents of fibers.

Figure 5 presents the effect of setting temperature on the modulus of elasticity (E_m) of fibersgypsum composites containing various amounts of fibers.



Figure 5. The effect of setting temperature on modulus of elasticity of composites containing various contents of fibers.

Regardless of the content of fibers introduced to the gypsum matrix, the increase in setting temperature led to decrease in the values of modulus of elasticity. Overall, it can be concluded that increasing the temperature during the setting process, which was intended to accelerate the evaporation of water from fibers-gypsum mixture, had a negative effect on flexural modulus of elasticity, similarly as in the case of bending strength results.

The effect of fibers content on the thermal conductivity coefficient of the manufactured fibers-gypsum composites is summarized in Table 1. The increase in the amount of introduced fibers led to the improvement in insulating characteristics of resultant boards. The addition of 2% of fibers reduced thermal conductivity by nearly 20% comparing to reference variant. The lowest thermal conductivity was found in the case of composite reinforced with 8% of fibers and the improvement was 36%. Such large differences resulted from the presence of air in void spaces created in the case of larger amounts of fibers.

Fibers content (%)	λ [W/mK]	q [W/m ²]	Density [kg/m ³]
0	0.215	88.81	1175
2	0.173	78.87	1089
4	0.151	57.28	966
6	0.148	64.44	921
8	0.138	55.13	833

Table 1. Thermal conductivity coefficient of manufactured fibers-gypsum composites.

CONCLUSIONS

- 1. Board manufactured without the addition of hemp fibers was characterized by the highest density. As the content of introduced fibers increased, the density of obtained panels significantly decreased.
- 2. The increase in the amount of added fibers results in extension of setting time. Composites containing 8% of hemp fibers are characterized by four time longer setting time than non-reinforced boards.
- 3. The introduction of hemp fibers in the amount ranging from 2% to 4% leads to the improvement in flexural strength characteristics of manufactured composites. However, increasing the amount of added fibers to 6% and more causes deterioration in both bending strength and modulus of elasticity.
- 4. The increase of temperature during setting negatively affects bending strength and modulus of elasticity of resultant composites regardless of the content of hemp fibers within their structure.
- 5. As the amount of introduced fibers increases, the value of thermal conductivity coefficient decreases. Therefore, reinforcement of composites with hemp fibers improves their insulating properties.

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Streszczenie: *Właściwości kompozytów włóknisto-gipsowych powstałych na bazie włókien konopi siewnej* (*Cannabies sativa* L.) *uprawianych w Polsce i gipsu naturalnego*. Popularność kompozytów wzmacnianych włóknami naturalnymi nieprzerwanie rośnie, co sprawia, że są one przedmiotem wielu badań naukowych. Przykładem interesującej koncepcji jest wykorzystanie włókien konopi siewnej w celu wzmocnienia płyt gipsowych. A zatem, przeprowadzone zostały badania dotyczące wpływu ilości dodawanych włókien konopnych oraz temperatury sieciowania wzmocnionej matrycy na właściwości wytworzonych kompozytów gipsowych. Zakres przeprowadzonych eksperymentów obejmował określenie gęstości, czasu sieciowania, wytrzymałości na zginanie, modułu sprężystości oraz współczynnika przewodzenia ciepła. Badania wykazały, iż wraz ze wzrostem

udziału włókien czas sieciowania matrycy znacznie się wydłuża, a gęstość otrzymywanych kompozytów maleje. Wykazano również, iż optymalną ilością wzmocnienia konopnego zapewniającą najlepsze właściwości wytrzymałościowe jest 4%, a dalsze zwiększenie jego udziału powoduje znaczne pogorszenie wytrzymałości. Ponadto zwiększenie temperatury podczas sieciowania wpływa negatywnie na charakterystykę wytrzymałościową wyprodukowanych płyt. Właściwości izolacyjne ulegały natomiast znacznej poprawie wraz z rosnącym udziałem włókien konopnych w płycie gipsowej.

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