Environmentally friendly poly(butylene succinate) composites with hemp shives

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Abstract: Effect of hemp shives (30–90 v/v %; size 0.5 and 0.8 mm) on the structure (SEM), mechanical properties (tensile properties, impact strength) and thermal properties (thermal conductivity, Vicat softening temperature) and water absorption of PBS composites was investigated. With the increase in filler content, the mechanical and insulating properties of the composites deteriorated due to weak interactions at the filler-polymer matrix interface. Water absorption also increased. Better properties were obtained with a smaller filler size.

Keywords: hemp shives, PBS, polymer composites, mechanical properties, thermal conductivity.

Przyjazne dla środowiska kompozyty poli(bursztynianu butylenu) z paździerzami konopnymi

Streszczenie: Zbadano wpływ paździerzy konopnych (30–90% v/v; wielkość 0,5 i 0,8 mm) na strukturę (SEM), właściwości mechaniczne (wytrzymałość na rozciąganie, udarność) i termiczne (przewodność cieplna, temperatura mięknienia Vicat'a) oraz absorpcję wody kompozytów PBS. Wraz ze wzrostem zawartości napełniacza pogarszały się właściwości mechaniczne i izolacyjne kompozytów, ze względu na słabe oddziaływania na granicy faz napełniacz - osnowa polimerowa. Zwiększała się również chłonność wody. Lepsze właściwości uzyskano w przypadku mniejszej wielkości napełniacza.

Słowa klucze: paździerze konopne, PBS, kompozyty polimerowe, właciwości mechaniczne, przewodnictwo cieplne.

Biodegradable polymers and their composites are materials that will play a key role in the coming future in many industries. Some of them are even colloquially called "gamechangers", despite their lack of mechanical properties, UV resistance or water absorption properties. The market for commercially available biopolymers has been growing steadily for years. The global market for biopolymers is expected to grow from 2.47 million tons in 2024 to 5.73 million tons by 2029 [1], but it must be admitted that their amount in the global production scale is still marginal compared to traditional petrochemical-based plastics, the production of which amounted to 374.2 million tons in 2023 [2].

Based on market data, the packaging industry is the market with the highest absorption rate of new bio-based materials, especially when it comes to single-use packaging. This situation is due to the attitude of policy makers and evolving legal acts, to name just the "Packaging Directive" [3] and secondary documents developed by the European Commission. The parent document - Directive 94/62/EC of the European Parliament and of the Council on packaging and packaging waste – was corrected in 2018 by adding Extended Producer Responsibility for Single-Use Plastics. Also initiatives organizing a huge number of enterprises, public entities and of course countries, where programs such as the "EuGreen Deal" and the "Fit for 55" package [4] are milestone actions indicating an increased need to start the workflow on biopolymer composites and plant materials to be transformed into valuable products. This need is partly due to the high price of biodegradable polymers compared to popular petrochemical polymers such as polypropylene or polyethylene, which are widely used as packaging materials. One of these polymers is poly(butylene succinate) also known as PBS, which is an aliphatic type of polyester. This polymer is widely used in the packaging industry [5] and for agricultural purposes [6, 7] due to its good pro-

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cessability and biodegradability [8, 9]. The wider application of this polymer is problematic due to its low tensile strength and high price compared to commonly used polymeric materials. In recent years, many works have appeared in which attempts have been made to create composite materials based on the PBS matrix [10–14] or its blend with other polymers, such as PLA [15, 16] to obtain better mechanical properties.

Polymer composites filled with natural fibers are well known engineering materials. These types of materials are increasingly used in the automotive industry, successfully replacing their synthetic counterparts [10]. Low price and broad abundance make natural fibers a viable alternative to synthetic ones such as glass or aramid fibers, not only for economic reasons but also for comparable mechanical properties. An additional benefit of using natural origin materials in composites with biodegradable plastics is their complete biodegradability. By directing these materials for composting processes, they can be easily decomposed into simple organic compounds. Naturally, the use of a plant-derived filler does not always lead to an improvement in composite tensile properties (although they are welcome) but it can, for example, improve the thermal conductivity of the material. At least they can be used to reduce the cost of the polymer matrix material by decreasing its mass ratio in the composite and making it more sustainable. An example of such a filler is the hemp shiv, which is the woody part of the hemp stem and has been widely used as an insulating material for many years in the form of hemp concrete, due to its specific physical properties. The shives consist of the following: cellulose (around 44%), hemicellulose (18–27%), lignin (22–28%) and other components like extractives (1–6%) and ash (1–2%) [17]. The reason for the improved thermal properties of the hemp shiv are low density and high porosity (76–78%) [18]. Because of the material composition and presence of hydrophilic hydroxyl groups, hemp shives are very susceptible to variations in humidity and tend to absorb water. Over the past years, many research teams have improved its natural properties by creating new composite materials or/and chemically changing the natural structure of the shiv [17–23]. The results of these works are materials which insulation properties do not differ from the values obtained by commercially available solutions or even exceed them. Despite the numerous advantages of hemp shives, their utilization remains quite narrow and is used as a component in building industry as thermal-insulating materials. However, given the high amount of absorbed CO_2 along with the short growth time and the high crop of hemp, the shiv is a potentially excellent source of a widely available plant filler with a low or even negative carbon footprint for applications in biodegradable composites [20, 21]. The use of plastics as a binder can potentially eliminate the biggest disadvantage of the shiv, which is its high water absorption, by closing its porous structure with a biopolymer. It may lead to creating economically beneficial composite material with a low environmental impact and additional advantage which could be its insulating properties. Moreover, these materials can be utilized in the plastics industry (as dry mixes, semi-finished products ready for processing: extrusion, injection, etc.), construction (insulation materials) according to the properties of the final product.

The aim of this work was to obtain biodegradable PBS/hemp shive composites with filler content of 30, 50, 70 and 90 v/v% and to investigate their structure (SEM), mechanical properties (tensile properties and impact strength) and thermal properties (thermal conductivity, Vicat softening temperature). Water absorption was also studied, and the obtained results were compared with pure PBS.

EXPERIMENTAL PART

Materials

Poly(butylene succinate) (PBS) was supplied by Mitsubishi Chemical (Tokyo, Japan) under the trade name BioPBS FZ71PM. Selected properties of PBS are shown in Table 1. Dustless hemp shives (Fig. 1) with dimensions ranging from 5 to 25 mm were purchased from Hemp Combinate Ltd. (Gronowo Górne, Poland) and used after preparation.

T a b l e 1. BioPBS FZ 71 selected properties

Property	Value
Melt flow rate (190°C/2.16 kg), g/10 min	22
Density, g/cm^3	1.26
Tensile strength, MPa	30
Flexural modulus, MPa	630
Elongation at break, %	120
Melting point, °C	115

Materials preparation

The filler in the form of raw sticks was pre-ground with a kitchen mixer (Zepter VG-022 MixSy, Wollerau, Switzerland) to reduce the uneven distribution of the average stick size fractions. After grinding, the materials were sieved on a vibrating shaker (EKO-LAB LAB-11-200, Jasień, Poland). Two fractions were obtained: 0.5–0.8 mm (assigned as 0.8 mm in the rest of the work) and smaller than 0.5 mm (assigned as 0.5 mm). Then, the filler was dried for 8 h at 90°C. PBS was also dried at 60°C for 5 h before processing (according to the manufacturer's safety data sheet).

Composites preparation

Dried PBS and filler were mixed in the assumed volume ratios in an internal mixer (PolyLab QC + Haake Rheomex, Karlsruhe, Germany) for 8 min at 195°C (all three zones). The filler content was 30–90% (v/v). Then, the composites were ground in a laboratory mill (Waner,

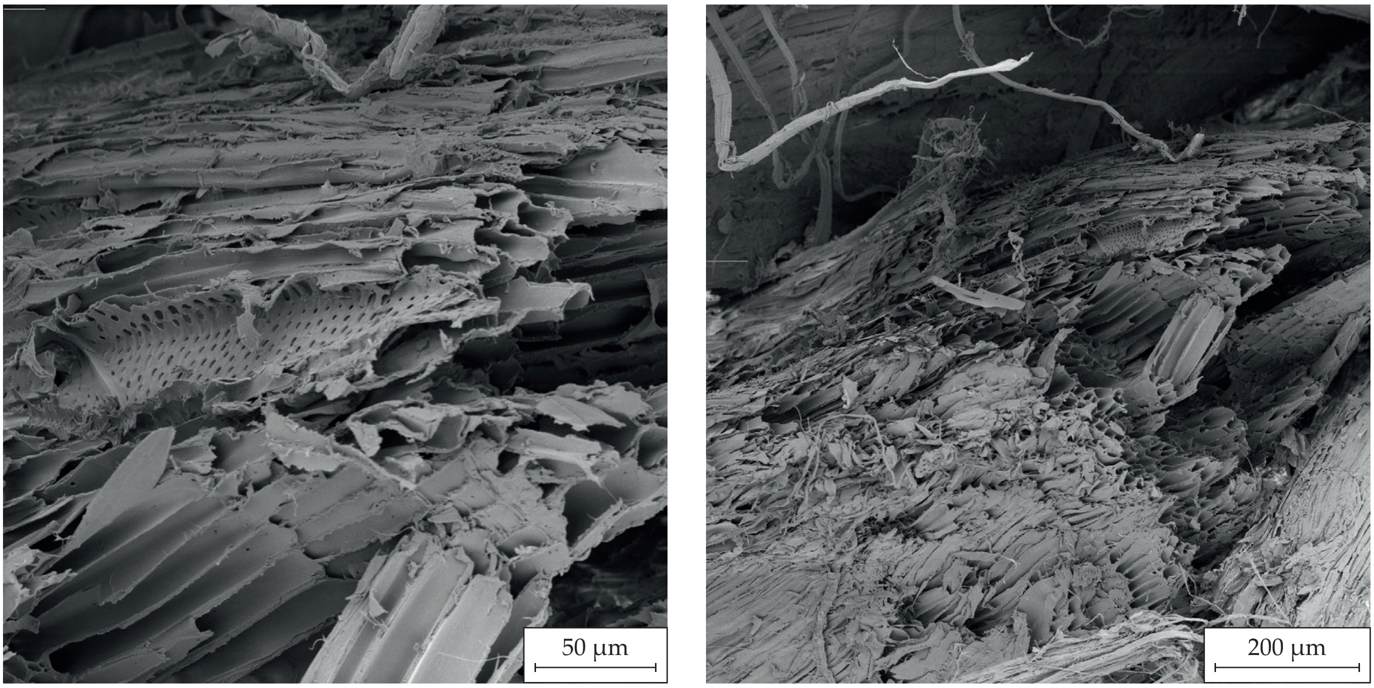

Fig. 1. SEM images of hemp shives

Wertheim, Germany) equipped with a 5 mm sieve. The ground composites were pressed at 200°C and 55 bar for 1 min into 10 × 10 cm plates (thickness 1.0 mm) using a hydraulic press (LP30-B, Labtech Engineering Co. Ltd., Thailand). For tensile strength measurements, the samples were cut using a pneumatic cutter (Ceast). To evaluate the impact strength of composites, rectangular bars (80 × 10 × 4 mm) were made. A pneumatic-piston injection molding machine (Proma) was used for this purpose. The plasticization time of the material was 2 min, and the working pressure was 5 bar.

SEM analysis

The morphology of the obtained composites was studied using a scanning electron microscope SEM Vega Tescan 3 (Kohutovice, Czech Republic) on samples in cross-section after non-plastic fracture. Before testing, the samples were sputtered with gold (Cressington 108 sputtering machine, Watford, UK) for 60 s at 40 mA.

Tensile properties

Tensile properties were characterized using a Lloyd LR 10K tensile machine (Ametek, Meerbush, Germany) according to PN-EN ISO 527-3. Measurements were performed under the following conditions: head 10 kN, crosshead speed 10 mm/min. At least 5 repetitions were performed for each batch of composite.

Impact strength

Impact tests were carried out using a Charpy impact hammer (Resil 5.5, CEAST Italy). The pendulum speed was 3.5 m/s, and the impact energy was 4 J (according to ISO 179-2). The dimensions of the samples (without notches) were as follows: 80 × 10 × 4 mm. Each measurement series (batch) consisted of 4 samples.

Thermal conductivity

The thermal conductivity of the composites was measured using a Hukseflux THASYS THA01 (Delft, The Netherlands) according to ASTM 1114. Measurements were made by placing two 1.0 mm thick plates in the apparatus chamber between a centrally mounted thin radiator and the heat sinks.

Vicat softening temperature

The Vicat softening temperature (VST) of pure PBS and composites was measured according to method A of ISO 306 using a static load of 10 N and a heating rate of 120°C/h. The test was performed three times, and the dimensions of the tested samples were 30 × 10 × 4 mm.

Water absorption

Water absorption (WA) was determined in accordance with ASTM D570. Samples measuring 10 × 10 × 1 mm were cut from 1 mm thick sheets. Before testing, the samples were dried at 60°C to a constant weight. The mass of each dried composite material (W_d) was measured using a weighing scale. The composite materials were immersed in distilled water for 24 h at 25°C. Then they were removed from the water and wiped to remove excess of water. The final mass (W_f) was measured immediately after drying. Tests were performed in triplicate for

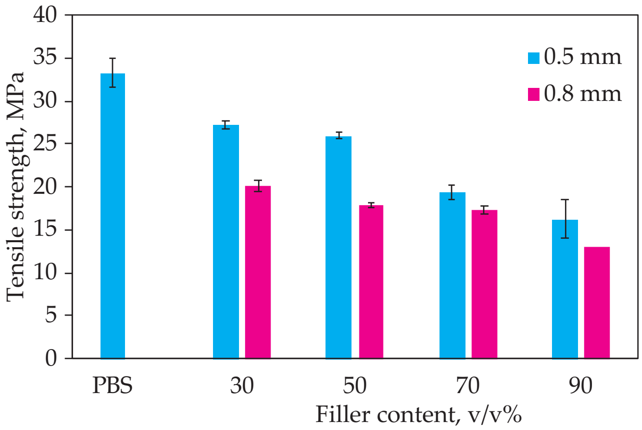

Fig. 2. Tensile strength of composites

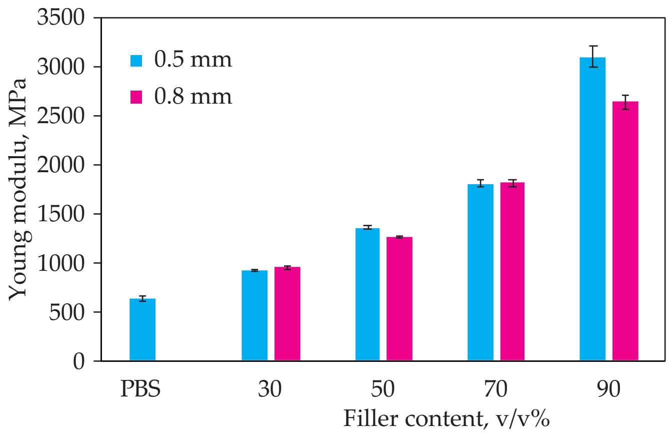

Fig. 3. Young modulus of composites

each composite. The composites were weighed, and the mean value and standard deviation of water absorption were calculated using Equation 1.

$$\%WA = \frac{W_f - W_d}{W_d} \cdot 100 \quad (1)$$

Where W_f is the mass of the sample after drying and W_d is the mass after immersion in water.

RESULTS AND DISCUSSION

Tensile properties

Figure 2 illustrates the typical behaviour of fiber-reinforced materials. For composites filled with a smaller fraction (0.5 mm), the addition of 30 v/v% filler leads to a 25% decrease in tensile strength. Increasing the filler content to 50 v/v% causes a further decrease in tensile strength, but it is not as pronounced as before. For the composite containing 70% filler, a 78% decrease in tensile strength was observed compared to pure PBS, while the composite with the maximum filler content (90 v/v%) still retains about 50% of the initial strength. The average decrease in tensile strength with increasing filler content is 11% compared to pure PBS.

Compared to the composite filled with larger hemp shives (0.8 mm), the observed decreases in tensile strength are much bigger, but qualitatively the relationship remains unchanged. This difference may be related to the distinct effect of free tubular spaces in the shives tested (Fig. 1). The addition of 30% filler leads to a decrease in tensile strength by 40%, while at 50% filler the strength drops to 54% of the initial value and then to 69% at 90% fiber content. SEM observations (Fig. 1) were supported by density tests, showing that the density of the shives before grinding was only 0.255 g/cm^3, which confirms their highly porous nature.

In both filler sizes (0.5 mm and 0.8 mm) none of the tubes are filled with a polymer matrix (due to the high polymer viscosity and small tube diameter), which leads to poor load transfer. However, it should be emphasized that because the filler size of 0.8 mm is 60% larger compared to 0.5 mm, their distribution is more prone to nonuniformity, which leads to the formation of local stress concentrations. Table 2 shows the effect of filler dimension on elongation of the composites. Analysis of the results shows a negative effect of filler addition on the elongation of composite materials, regardless of the size of the fiber used. Moreover, the composites are characterized by significantly bigger stiffness compared to pure polymer (Fig. 3). The addition of 30 v/v% of filler causes an increase in the tensile modulus by about 30%. As expected, the highest stiffness was obtained (5-fold increase) in the case of a composite containing 90 v/v% of the smaller filler.

This phenomenon can be explained by physical constraints occurring in a highly loaded fiber composite, where some fibers can create areas with unevenly distributed fillers (Fig. 4). It is necessary to consider not only the total amount of filler, but also its dimensions - assuming a tubular shape, the 0.8 mm filler is about 60% larger on the longitudinal axis compared to the 0.5 mm fraction.

T a b l e 2. Effect of filler dimension on composites elongation

Filler content, v/v %	Elongation at maximum load, %		Elongation at break, %	
	0.5 mm	0.8 mm	0.5 mm	0.8 mm
0	13.05	13.05	247.45	247.45
30	5.21	3.31	5.54	3.42
50	3.12	2.12	3.14	2.20
70	1.41	1.28	1.41	1.29
90	0.72	0.72	0.72	0.72

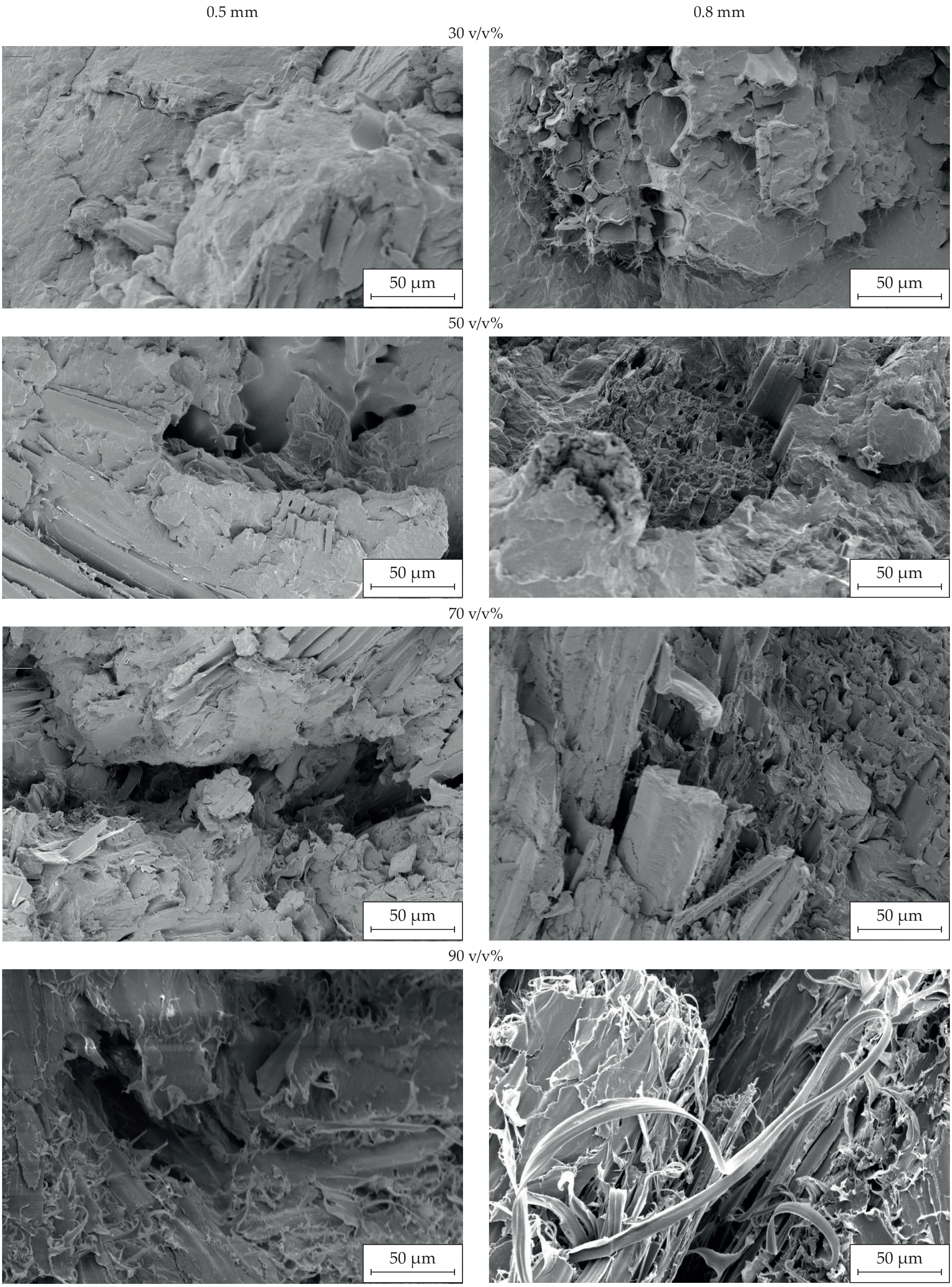

Fig. 4. SEM images of composites with different filler contents (30–90 v/v%) and dimensions (0.5 and 0.8 mm)

Another approach that can help in understanding this phenomenon is the decreasing effect of the filler amount on the mechanical properties of the material. It has been verified, especially regarding fibrous fillers, that highly filled composites show a disproportionate gain compared to composites with lower filler loading [24]. Filler efficiency has been introduced as a factor to predict the behavior in highly loaded composites, where the remaining free volume in the matrix must be considered when discussing reinforcement effects [25].

Charpy impact strength

The effect of filler length and content on Charpy unnotched impact strength is shown in Fig. 5. A significant decrease in impact strength was observed for all tested composites. The result for PBS was not included in Fig. 5 because it was not destroyed during the test, which indicates impact strength exceeding 100 kJ/m^2. In the case of 50 v/v% filler content, the impact strength decreased by 39%. At a higher level (70 v/v%), only a slight further decrease in impact strength was observed. However, composites with smaller fillers (0.5 mm) showed higher impact strength, while larger fillers (0.8 mm) caused significantly less decrease in impact strength, as a function of content, than smaller ones.

This decrease is due to the low affinity between the matrix and the filler, as confirmed by the SEM images (Fig. 4). This fact is widely known in literature and concerns plant-derived materials that are hydrophilic in nature and require physical or chemical modification to improve the adhesion between the filler and the polymer matrix. It should also be mentioned that in the case of the 0.8 mm filler, it was observed that the filler tends to form agglomerates of pure hemp sticks and voids, which can also cause a reduction in the impact strength of the composite materials. With a higher filling, the downward trend is noticeably smaller. It should be remembered that the composites were prepared in volumetric ratios so that from a certain degree of filling, most of the mechanical properties were determined by the hemp shives themselves, which in this case do not play a reinforcing role. Samples with 90% filling were not included in the measurement series due to the high viscosity of the material, which made it impossible to obtain shapes by injection molding.

Thermal conductivity

Fig. 6 shows that the thermal conductivity of the composites increases slightly with the increase in filler content, which may indicate a lack of improvement in insulating properties. This fact may be related to the filler granulometric fraction and free spaces inside the composites, which can be observed in SEM images (Fig. 4). Hemp shives are known for their insulating properties and, as renewable raw material, are used as a natural alternative to commercially available counterparts. These properties result from the fact that the highly porous internal structure of the ground shives is destroyed, which leads to a drastic reduction in their insulating properties.

The biggest differences can be observed in the case of composites filled with a filler fraction of 0.5 mm. The addition of 30 v/v% and 50 v/v% of filler increases thermal conductivity by 15% compared to pure PBS. In turn, composites containing 70 v/v% and 90 v/v% of fillers have higher thermal conductivity by 25% and 42%, respectively. Interestingly, in the case of composites with a larger filler (0.8 mm), an increase in this parameter was found at the level of statistical error. Composites with 30, 50 and 70 v/v% filler showed the same thermal conductivity [approximately 0.216 W/(m · K)], which is 10% higher than that of pure PBS. Interestingly, a sharp decrease was observed in the highly loaded composite (90 v/v% shive content), giving a value equal to pure PBS [0.190 W/(m · K)]. This phenomenon may be caused by the highly porous structure of the composites, which consist of low-density hemp shives. In the case of fiber-based insulation materials, heat

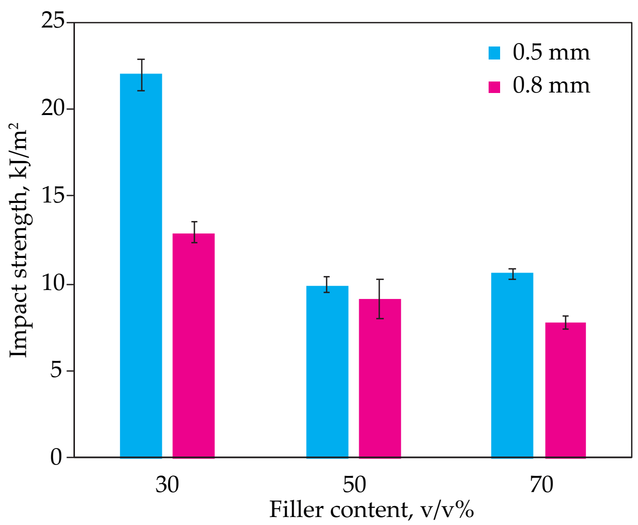

Fig. 5. Charpy unnotched impact strength of composites

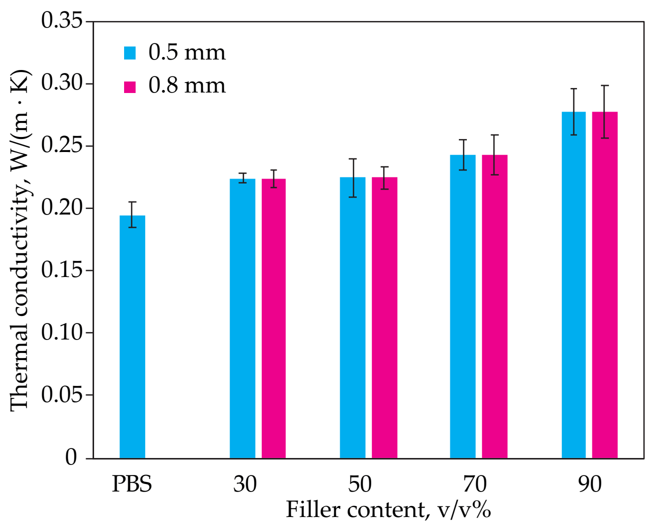

Fig. 6. Thermal conductivity of composites

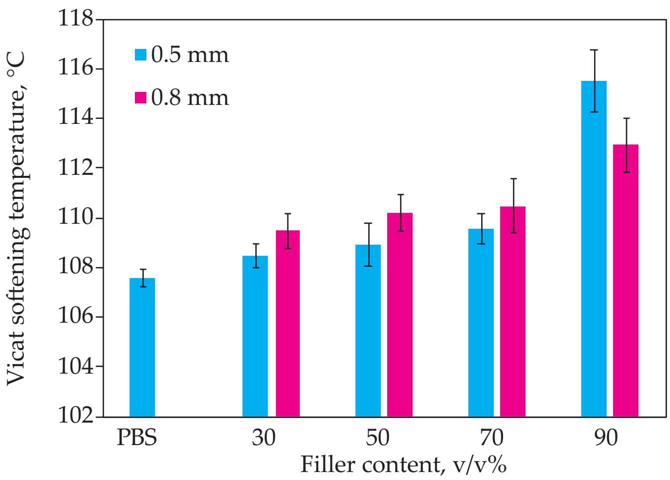

Fig. 7. Vicat softening temperature of composites

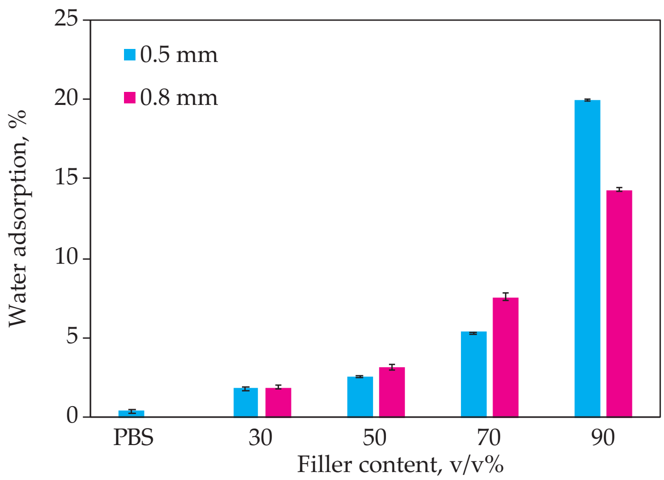

Fig. 8. Water adsorption of composites

T a b l e 3. Thermal conductivity of various materials

Material	Thermal conductivity, W/m·K
Rock wool	0.033–0.045 [26]
Cork	0.037–0.050 [26]
Low density polyethylene (LDPE)	0.28-0.32 [27]
Polypropylene	0.14 [27]
Polystyrene	0.04-0.14 [27]
PBS	0.19

transfer is related to the density of the filler and its orientation [26]. To better present the range of thermal conductivity of the obtained composite materials, Table 3 provides values of this parameter for several different materials.

Vicat softening temperature

A small effect of the content and dimension of hemp shives on the Vicat softening temperature of PBS composites was found (Fig. 7). The largest increase in this parameter was noted for composites containing 90 v/v% filler. The composite obtained with smaller fiber had a Vicat softening temperature higher by 8°C, and with larger fiber by 5°C.

Water adsorption

Water absorption tests confirmed that water absorption in the obtained composite material is exponentially proportional to the filler content (Fig. 8). The exponential nature is due to the physical nature of the filler discussed earlier. This is due to the incomplete filling of the naturally highly porous shive's structure (Figs. 1 and 3), where the polymer is physically unable to cover and close it. Because of this, water can freely move through the remaining capillaries in the material structure. Fig. 8 also shows that the longer filler length (0.8 mm) absorbs more water than the 0.5 mm filler.

CONCLUSIONS

It was shown that shives do not act as reinforcement in the obtained composite materials, causing a decrease in mechanical properties. One of the reasons is the lack of affinity of hemp shives to the polymer matrix, which is a phenomenon described in the literature in the context of natural fillers. However, the results showed the possibility of producing cost-effective, biodegradable composite material (as a material to produce disposable items such as packaging or tableware) based on hemp shives with an extremely high degree of filling.

The thermal conductivity of the tested composites indicates a deterioration in the insulating properties with the increase in the content of hemp shives, regardless of their dimension. This can be explained by the destruction of the highly porous, closed structure of the shives by the grinding process, which can lead to heat transfer through the free spaces in the material. Additional studies should be conducted on the effect of the filler dimension on the insulation effect on the material. An important issue that requires clarification, and thus an increase in the usability of the material, is the determination of the limiting dimension of the shives, which limits the insulating properties.

Moreover, it was found that with the increase in filler content, the water absorption of composites also increases. This tendency is particularly visible in the case of composites with a high degree of filling (70–90 v/v%). This results from the structure of hemp shives, typical for plant fillers. Moreover (except for the composite with 90 v/v% filling), a greater tendency to water absorption was found for composites obtained with a larger filler (0.8 mm) than with a smaller one (0.5 mm). This could be due to the size of natural capillaries present in the material, which were larger in the case of a larger filler.

Authors contribution

M.B. – conception, data analysis, investigation, writing-original draft, material preparation; K.S. – edition, SEM

microscopy, writing-original draft; K.L – super-vision, edition; J.L. – edition, density measurements
All authors have read and agreed to the published version of the manuscript.

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Conflict of interest
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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