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О. Садова, В. Кашицький, Д. Гусачук, М. Вишинський, Є. Чернота

Луцький національний технічний університет

ДОСЛІДЖЕННЯ МЕХАНІЧНИХ ВЛАСТИВОСТЕЙ ТА СТРУКТУРИ БІОКОМПОЗИТІВ, НАПОВНЕНИХ КАВОВОЮ ГУЩЕЮ

В статті досліджено міцність на стискання біокомпозитних матеріалів на основі глютинової матриці та кавової гущі залежно від вмісту наповнювача та ступеня підсушування композиції. Визначено міцність на стискання біокомпозитів залежно від концентрації біополімерної матриці, вмісту композиції в прес-формі, температури основної термічної обробки та додаткової термічної обробки. Досліджено, що максимальну міцність на стискання 79,6 МПа мають біокомпозитні матеріали із ступенем підсушування 20%. Також досліджено мікроструктуру біокомпозитів оптимального складу (вміст кавової гущі в кількості 200 мас. ч.) зі ступенем підсушування композиції 20%.

Ключові слова: *розчин глютину, кавова гуща, міцність на стискання, термічна обробка, ступінь підсушування, структура.*

O. Sadova, V. Kashytskyi, D. Husachuk, M. Vyshynskyi, Ye. Chernota

STUDY OF MECHANICAL PROPERTIES AND STRUCTURE OF BIOMPOSITS FILLED WITH COFFEE GROUNDS

The article investigates the compressive strength of biocomposite materials based on glutin matrix and coffee grounds depending on the filler content and the degree of drying of the composition. The compressive strength of biocomposites is determined depending on the concentration of the biopolymer matrix, the composition content in the mold, the temperature of the main heat treatment and additional heat treatment. It is investigated that biocomposite materials with a degree of drying of 20% have a maximum compressive strength of 79.6 MPa. The microstructure of the biocomposite sample of the optimal composition (coffee grounds content in the amount of 200 wt. parts) with a degree of drying of the composition of 20% is also investigated.

Key words*: glutin solution, coffee grounds, compressive strength, heat treatment, degree of drying, structure.*

Problem statement. Nowadays Today, plastic products based on synthetic binders are very common in the world due to their properties, in particular, the combination of low weight with high strength and corrosion resistance. However, the excessive accumulation of plastic waste creates a serious environmental problem due to the long-term decomposition during disposal and their toxicity. Millions of tons of plastic waste, which is difficult to recycle or utilise, are generated every year. Plastic waste decomposes for hundreds of years, polluting soils and water bodies. Toxic substances are released during the combustion of plastic waste. They harm the atmosphere, polluting the air [1]. This necessitates the development of new alternative materials that are safer for the environment. Biocomposite materials based on natural fibers and natural polymers can partially replace products made of traditional plastic. They decompose faster and have a smaller negative impact on the environment, reducing the carbon footprint. The relevance of the development of biocomposites is due to the strategic priorities of sustainable development, since such materials are environmentally friendly, are made from renewable sources and reduce energy costs [2].

Analysis of recent research and publications. Construction and automotive industries are currently the main areas of introduction of biocomposite materials . However, new promising markets and applications of biocomposite products are emerging as a result of improvements, innovative solutions and increased productivity [3]. The direction of wood composites is of significant economic importance. The direction is rapidly developing in the concept of creating new composites and more in the development of new types of binders for them. There is significant progress in the development of biobased adhesives, caused by external restrictions, in particular, strict conditions of regulatory documents on the reduction and even elimination of formaldehyde and other materials that are toxic. Consumer awareness is no less important, that is the desire of industry to give preference to environmentally friendly materials and reduce or even eliminate its dependence on exhaustible petrochemical products with a subsequent increase in the price of raw materials [4].

The properties of reinforced natural biocomposites are determined by both the characteristics of the filler and the polymer matrix. The properties of biocomposites depend on the type of natural fiber and its modification, the use of modifiers, the ratio of the mass of the filler to the polymer, the manufacturing and processing technology, as well as the efficiency of the interfacial connection between the reinforcing materials and the matrix [5].

Petinakis E et al. [6] investigated composites based on polylactic acid (PLA) and wood flour. They observed that the tensile strength was not significantly improved by increasing the number of wood flour

particles. With the addition of the modifier methylene diphenyl diisocyanate, the mechanical properties of the composites, in particular the tensile strength and Young's modulus, increased by 10% and 135% with increasing wood flour content.

The properties of epoxy composites [7] filled with crushed walnut shells, which are organic waste of agricultural production, were investigated. Composite samples containing 20, 30, 40 and 50 wt. % crushed walnut shells have increased stiffness and hardness compared to epoxy polymers. The introduction of this filler leads to a decrease in the tensile strength and Charpy impact resistance of the composite material. Thermogravimetric analysis has shown that the introduction of crushed walnut shells improves the thermal stability of composite materials.

The authors of [8] developed an environmentally friendly frame for glasses made of recycled coffee grounds, natural oils and a biopolymer based on vegetable oils, which are used as a binder. The glasses are waterproof due to a special type of hydrophobic coating. The biomaterial of the frames decomposes in about 10 years under natural conditions.

In [9], biocomposite samples without delaminations and structural defects were obtained by introducing the optimal content of filler (wood flour), formed by pressing. Biocomposite materials based on starch binder were modified with an aqueous solution of glutin. It was found that the most homogeneous and dense structure of biocomposites is formed at the optimal content of the modifier in the amount of 70 wt. parts, which ensures an increase in the hardness of biocomposite materials.

Compositions of biocomposite materials based on glutin and crushed straw have been developed. It was found that higher degrees of drying of the composition (25%) are advisable to use for biocomposites with lower filler content (140 wt. parts) and, conversely, lower degrees of drying of the composition (20%) are sufficient for biocomposites with higher filler content (150 wt. parts). This is economically advantageous, since the duration of the technological process is reduced, which will lead to a decrease in energy costs. The highest compressive strength of 82.8 MPa is possessed by biocomposites with a filling degree of 150 wt. parts of crushed cereal stalks and a drying degree of 20% of the composition with a glutin solution concentration of 50% [10].

Biocomposite materials with a filler content of 200 wt. parts have a higher compressive strength (75.8 MPa) compared to materials with a lower filler content of 190 wt. parts. The strength of biocomposites increases with a higher filler particle content (200 wt. parts), which is determined by the ability of coffee grounds particles to deform as a result of compression during molding. The optimal density (1.17 g/cm^3) provides an additional increase in strength due to the physical and chemical bonds between coffee grounds particles and glutin macromolecules. Coffee bean particles have low mechanical characteristics and are less deformed during pressing under the influence of a specific load of 8 MPa compared to pressing under a pressure of 11 MPa. Therefore, biocomposite materials formed under a pressing pressure of 8 MPa have a higher impact strength of 3.55 kJ/m^2 [11].

Statement of tasks. The aim of the work is to study the mechanical properties and structure of biocomposite materials based on a glutin solution of various concentrations with coffee grounds.

Presentation of the main material. Coffee grounds in the amount of 180-210 wt. parts were used as a filler for biocomposites. An aqueous solution based on bone glue with a concentration of 43% was used as a polymer binder. The pre-dried filler was added to the glutin solution and mixed to obtain a homogeneous composition. The composition was pressed at a temperature of 50-60 °C to a mass loss of 5%, 10%, 15%, 20% and 25%. The composition was placed in a mold and pressed. Next, the samples in the mold were heat treated for 2 hours at a temperature of 150 °C. It is necessary to press the composition after 1 hour of holding time to ensure the density of the material. The biocomposite samples were cooled in the mold in still air at room temperature.

Biocomposite samples filled with coffee grounds in an amount of 180 wt. parts with a drying degree of 10% have a low compressive strength of 9.6 MPa (Fig. 1). This indicates insufficient removal of moisture from the composition during its drying, which prevents the complete structuring of the material. The compressive strength of a biocomposite of this composition increases with an increase in the degree of drying of the composition from 10% to 25%. The compressive strength of biocomposites with a degree of drying of the composition of 15% increases by 4.2 times (40.4 MPa). The compressive strength of biocomposites with a degree of drying of the composition of 20% increases by 5.2 times (50.3 MPa). The compressive strength of biocomposites with a degree of drying of the composition of 25% increases by 6.9 times (66.8 MPa). This indicates an increase in the structuring of biocomposite materials due to the removal of excess moisture that enters the material during its formation.

Biocomposite samples containing 180 wt. parts of coffee grounds contain a large number of small pores that are evenly distributed over the surface. Presumably, these pores are formed during heat treatment due to the removal of excess moisture from the volume of the material. The surface of samples with a degree of drying of the composition of 10% and 15% is uneven at the ends with brittle burrs. Biocomposite samples with a higher degree of drying of the composition contain a smaller number of pores on the surface. The surface of the ends and the side surface of biocomposite samples with a degree of drying of the composition of 20% are even, which indicates a high degree of structuring of the high-density biocomposite due to the removal of a larger amount of moisture from the material, which confirms the obtained high strength values. The degree of drying of the composition of 25% is too high, since the edges of the ends have burrs.

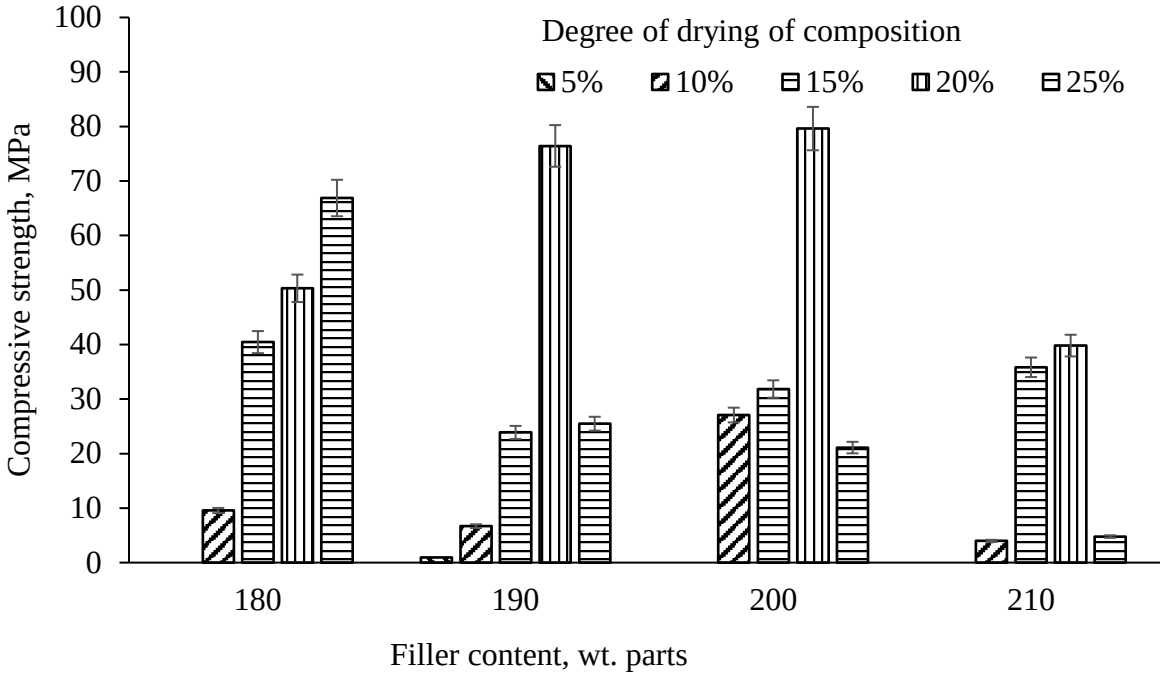


Fig. 1. **Compressive strength of biocomposites filled with coffee grounds, depending on the filler content and the degree of drying of the composition**

Biocomposites with a content of 190 wt. parts of coffee grounds and a degree of drying of the composition of 5% have the lowest compressive strength of about 1 MPa. These biocomposite samples contain a significant amount of moisture, which does not allow to obtain a material with high strength. The edges on the end of the sample are brittle and uneven, which confirms the low compressive strength of these biocomposites. The compressive strength of biocomposites with a higher degree of drying of the composition of 10% is 6.7 MPa. Higher degrees of drying of the composition up to 15% and 20% allow to increase the compressive strength of biocomposites by 3.5-11.4 times, which is 23.9 MPa and 76.4 MPa, respectively. The compressive strength of biocomposites increases due to the formation of an additional number of physical and chemical bonds in biocomposite materials because excess moisture is removed from the material. The surface of the samples becomes smoother and contains fewer pores as the degree of drying of the composition increases from 5% to 20%.

The biocomposite samples with a composition drying degree of 20% have the smoothest surface. This biocomposite has the highest compressive strength of 76.4 MPa among samples with a content of 190 wt. parts of coffee grounds. The biocomposite sample with a composition drying degree of 25% contains small pores on the side surface. The end surface is uneven. The light brown color of the sample indicates an insufficient content of the biopolymer matrix, as a result of which the biocomposites are brittle. The compressive strength of these biocomposites (25.5 MPa) is 3 times lower compared to the compressive strength of the biocomposite with a composition drying degree of 20% (76.4 MPa). Therefore, drying the composition to a degree of 25% with a high filler content is impractical.

The compressive strength of biocomposites filled with 200 wt. parts of coffee grounds with a degree of drying of the composition of 10% is 27.1 MPa. The compressive strength increases with an increase in the degree of drying of the composition from 10% to 20% and decreases with a degree of drying of the composition of 25%. The compressive strength of biocomposites with a degree of drying of 15% is higher by 15% compared to a degree of drying of 10% and is 31.8 MPa. Biocomposites with a degree of drying of

20% have a maximum compressive strength of 79.6 MPa. Increasing the degree of drying of a highly filled composition is not advisable, since the compressive strength of biocomposites decrease by 2 times (39.8 MPa).

Biocomposite samples with a higher filler content (200 wt. parts) have high strength (79.6 MPa). The surface of these samples with a higher degree of drying of the composition (20%) is smooth without visible structural defects in the form of pores. The degree of drying of the composition of 25% is too high and does not allow obtaining high-strength biocomposites. In this case, the binder content in the composition is insufficient, which causes the formation of microcracks and complicates the structuring of the biocomposite material.

The lowest compressive strength of 4.0 MPa is possessed by biocomposites filled with 210 wt. parts of coffee grounds with a low degree of drying of 10%. The lateral surface of the sample is uneven, since there is a burr on the end of the sample due to an excess of binder. The compressive strength of biocomposites increases by 9 times (35.8 MPa) with an increase in the degree of drying of the composition to 15%. Increasing the degree of drying of the composition to 20% allows obtaining biocomposites with a compressive strength of 39.8 MPa, which is 12% higher compared to the compressive strength of biocomposite materials with a degree of drying of the composition of 15%. The surfaces of biocomposite samples with degrees of drying of the composition of 15% and 20% are smooth, which indicates a high hardness of the material.

Biocomposites with a composition drying degree of 25% have a compressive strength lower by 8.3 times (4.8 MPa). These results of compressive strength at a given composition drying degree (25%) are significantly lower compared to the compressive strength of biocomposites filled with 190 wt. parts and 200 wt. parts. The side surface of the biocomposite sample is smooth, without macroscopic defects. A developed surface relief formed by coffee grounds particles was recorded on the end of the sample. This indicates an insufficient amount of binder for a given filler content in the composition and a lower degree of structuring.

Therefore, the next stage of optimizing the technology for forming biocomposites was to establish the optimal concentration of the glutin solution. Biocomposite samples were formed with different concentrations of the solution: 50%, 46%, 43%. The content of coffee grounds was chosen to be 200 wt. parts, since biocomposite materials with a given filler content have the highest values of compressive strength. The density of biocomposite materials was 1.17 g/cm^3 and 1.38 g/cm^3. It was found that the compressive strength of the developed biocomposites decreases with a decrease in the concentration of bone glue in the aqueous solution of the matrix (Fig. 2). Among biocomposites with a composition density in the mold of 1.17 g/cm^3, biocomposites based on a glutin solution with a concentration of 50% have the highest compressive strength of 73.2 MPa. Compressive strength of biocomposites decreases by 9% with a decrease in glutin concentration to 46% (66.9 MPa). Reducing glutin concentration to 43% leads to an additional decrease in the compressive strength of biocomposites (58.9 MPa) by 24% and 14% compared to biocomposites based on glutin solution with a concentration of 50% and 46%, respectively. The decrease in compressive strength of biocomposites is associated with a decrease in the quantitative content of bone glue and an increase in the quantitative content of water, the excess of which in the material prevents the structuring of the material.

Among the developed biocomposites with a density of 1.38 g/cm^3, the highest compressive strength of 74.8 MPa is possessed by biocomposite materials also with a glutin solution concentration of 50%. With a decrease in the glutin solution concentration to 46%, the compressive strength of biocomposites decreases by 17% (63.7 MPa). A further decrease in the glutin solution concentration to 43% leads to an additional decrease in the compressive strength (63.1 MPa) of biocomposites by 19% and 1% compared to biocomposites based on glutin solution with a concentration of 50% and 46%, respectively. Biocomposites filled with 200 wt. parts of coffee grounds with a glutin solution concentration of 50% and a composition density of 1.38 g/cm^3 have a 2% higher compressive strength of 74.8 MPa compared to biocomposites with a glutin solution concentration of 50% and a composition density of 1.17 g/cm^3.

It is proposed to reduce the temperature to 100 °C or 120 °C in order to optimize the heat treatment. It was found that the compressive strength of biocomposites increases with a decrease in the glutin concentration from 50% to 43%. Biocomposites with a glutin solution concentration of 50%, for which heat treatment was carried out at temperatures of 100 °C and 120 °C, have the lowest compressive strength values of 5.4 MPa and 9.6 MPa (Fig. 3), respectively.

Biocomposites with a glutin concentration of 46%, which were heat treated at temperatures of 100 °C and 120 °C, have 2.2 times higher values of compressive strength of 11.9 MPa and 20.7 MPa,

respectively. Biocomposites with a glutin concentration of 43%, which were heat treated at temperatures of 100 °C and 120 °C, have the highest values of compressive strength (2.3-3.6 times higher than the previous ones) of 42.9 MPa and 47.8 MPa, respectively. However, these values of compressive strength are 1.6-1.7 times lower compared to the maximum value of compressive strength (74.8 MPa) of biocomposites with a glutin concentration of 50% and a composition density of 1.38 g/m^3, which were subjected to heat treatment at a temperature of 150 °C for 2 hours. Therefore, reducing the heat treatment temperature to 100 °C and 120 °C is not advisable, since at a higher temperature of 150 °C, a greater number of physical and chemical bonds are formed between the biopolymer matrix and the filler.

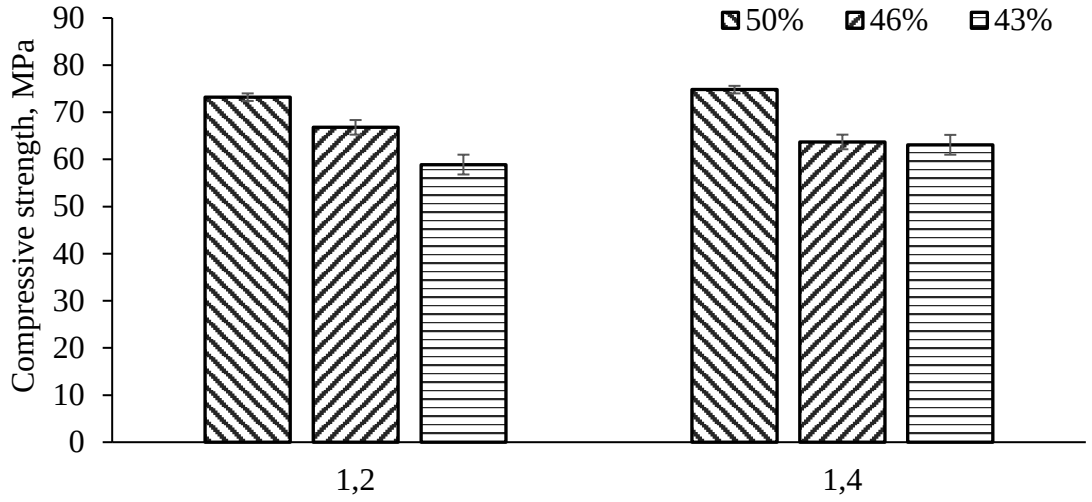


Fig. 2. **Compressive strength of biocomposites depending on the concentration of glutin solution and the density of the composition (1.2 g/cm^3 and 1.4 g/cm^3)**

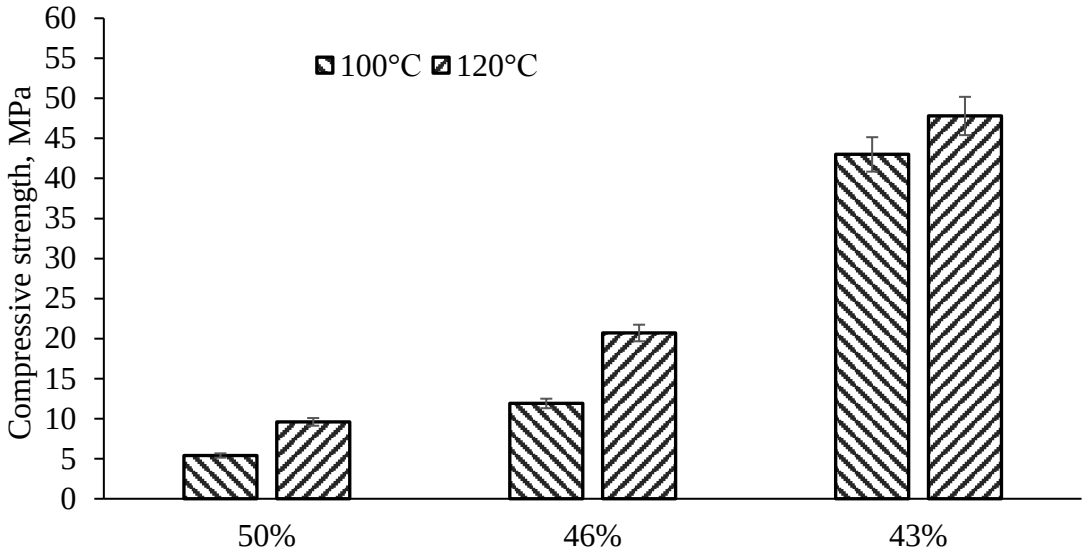


Fig. 3. **Compressive strength of biocomposites depending on the concentration of glutin solution and the temperature of the main heat treatment**

Structural defects in the form of small cracks are present on the side surfaces of the biocomposites (Fig. 4, a, b, d, e). These cracks are formed as a result of the elastic aftereffect after removing the samples from the molds. The presence of cracks facilitates the destruction of the materials and explains the obtained low values of the compressive strength of the biocomposites with a glutin concentration of 50% and 46%, which were subjected to heat treatment at temperatures of 120 °C and 150 °C with a holding time of 2 hours.

No cracks were detected on the surfaces of biocomposites with a glutin concentration of 43% (Fig. 4, c, f). This explains the higher values of compressive strength obtained for these biocomposites compared to biocomposite samples with a glutin concentration of 50% and 46%. The higher valuesof compressive strength of biocomposites with a lower glutin concentration of 43% can be explained by the better interaction of the components with each other during their mixing due to the lower viscosity of the

biopolymer binder. As a result, a greater number of bonds are formed between the components. Also, cracks are not formed due to the elastic aftereffect of the material.

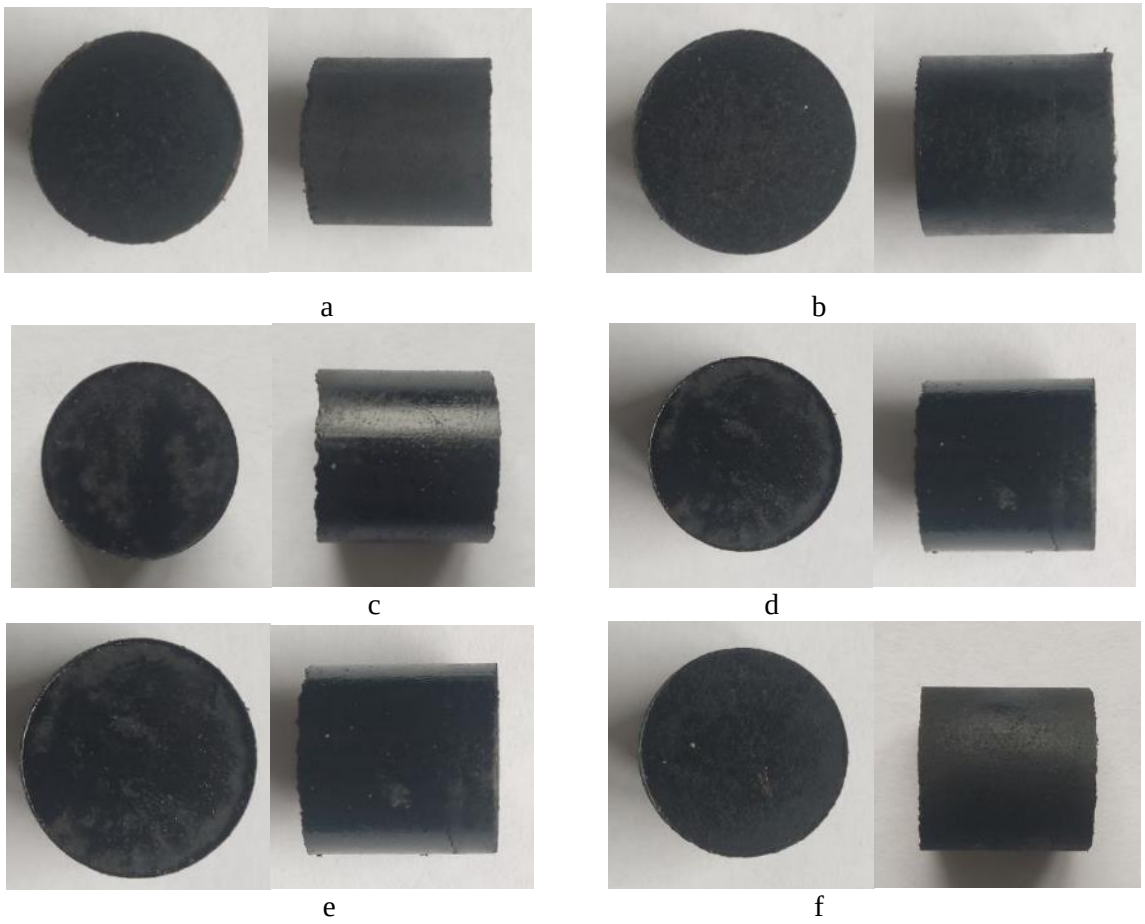


Fig. 4. **General appearance of biocomposite samples with a composition density of 1.17 g/m^3 (a-c) and 1.38 g/m^3 (d-f) and different glutin concentrations: a, d – 50%; b, e – 46%; c, f – 43%**

The next stage was to determine the feasibility of additional heat treatment. Biocomposite materials with a composition density of 1.38 g/m^3 and a glutin solution concentration of 43% were formed. The content of coffee grounds in the biocomposites was 190 wt. parts and 200 wt. parts. The biocomposite samples were subjected to the main heat treatment at a temperature of 150 °C with a holding time of 2 hours for biocomposites. The sample was cooled in a mold in still air at room temperature. Then the biocomposite samples were subjected to additional heat treatment at a temperature of 50 °C.

It was found that the compressive strength of biocomposites with a coffee grounds content of 190 wt. parts and 200 wt. parts is 23.9 MPa and 62.1 MPa (Fig. 5), respectively. The compressive strength of biocomposites increases by 1.5-21% with an increase in the holding time of the samples from 1 hour to 2 hours. The compressive strength of biocomposites with a filler content of 190 wt. parts and 200 wt. parts is 30.3 MPa and 63.0 MPa, respectively. The subsequent increase in the holding time of biocomposites to 3 hours additionally increases the compressive strength of the side composites by 9-24%, which is 40.6 MPa (190 wt. parts) and 70.1 MPa (200 wt. parts).

The increase in the strength of biocomposites can be explained by the higher structuring of biocomposite materials due to the additional removal of excess moisture. Exposure in a thermal field for 4 hours is inappropriate, since it leads to a slight decrease of 1-5% in the compressive strength of biocomposites with a content of 190 wt. parts and 200 wt. parts (39.8 MPa and 66.9 MPa, respectively). The decrease in the compressive strength of biocomposites can be explained by too high removal of moisture from the materials, resulting in the formation of fewer physical and chemical bonds between the components of the biocomposite material. There are no visible structural defects in the form of pores or cracks on the surface of the biocomposites (Fig. 6). The surfaces are smooth, without pronounced relief.

The microstructure of the biocomposite material based on a 43% glutin solution with a content of 200 wt. parts of coffee grounds, a degree of drying of the composition of 20% and a density of the composition of 1.38 g/cm^3 has a pronounced surface relief (Fig. 7, a).

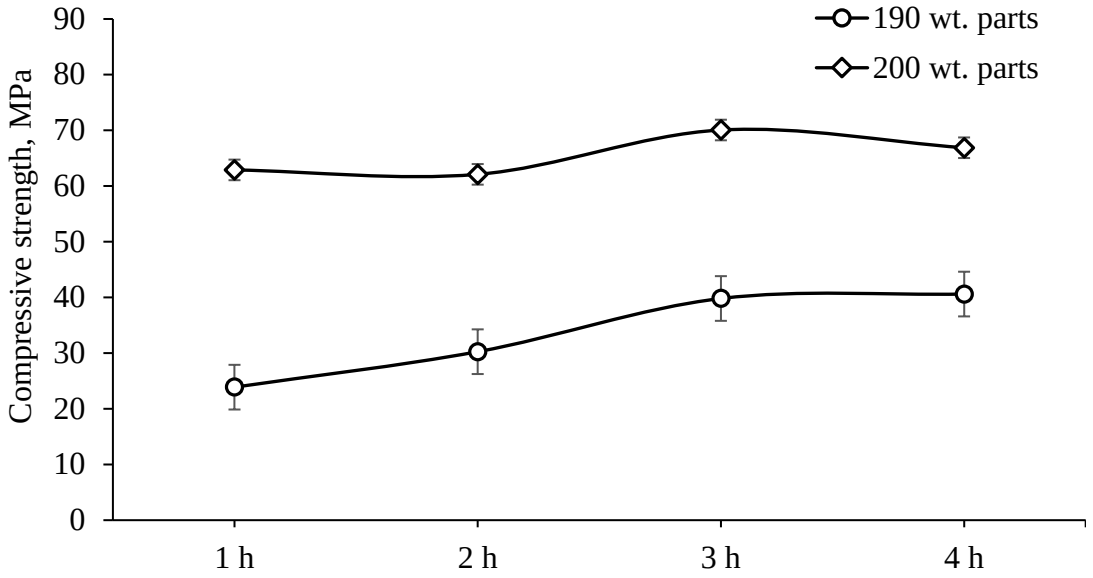


Fig. 5. **The effect of additional heat treatment at a temperature of 50 °C on the compressive strength of biocomposites**

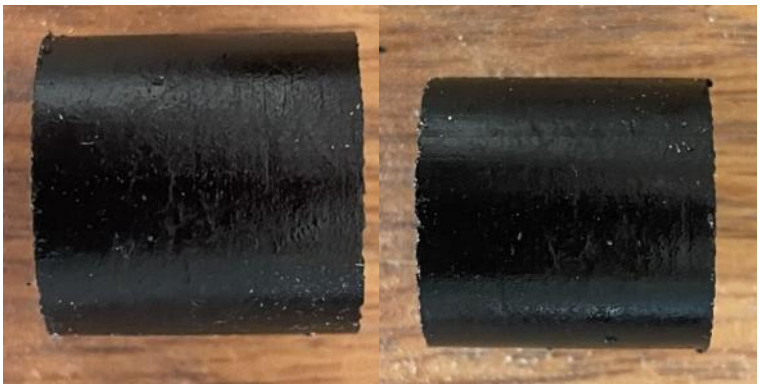


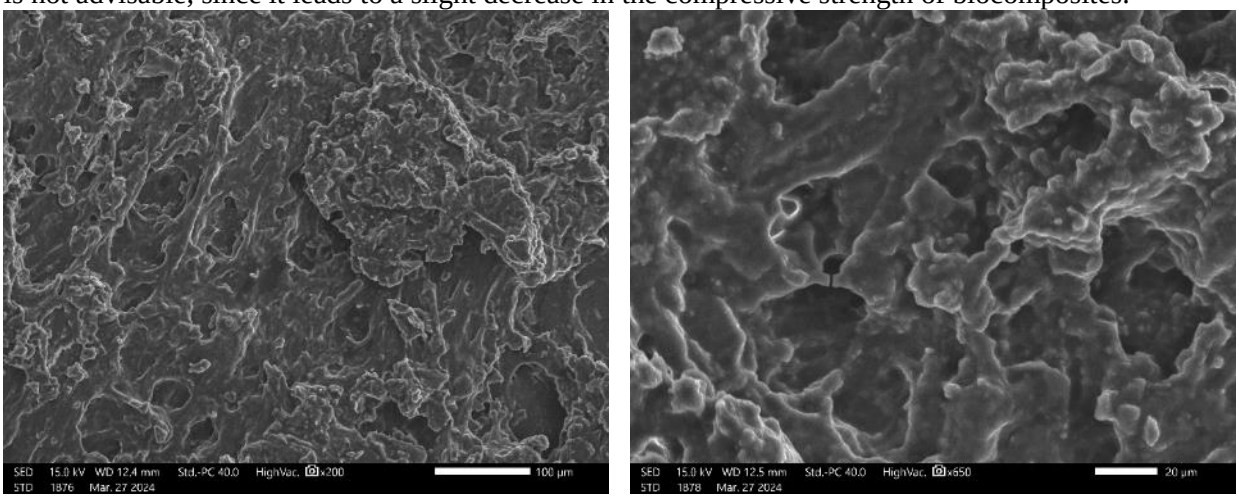
Fig. 6. **General appearance of biocomposite samples with an additional heat treatment**

The microstructure of the biocomposite material is homogeneous, which indicates high strength and hardness of the material. The coffee ground particles are covered with a biopolymer matrix (Fig. 7, b), which indicates about a high degree of structuring of the biocomposite. Open porosity is observed throughout the entire volume of the material.

Conclusions. It was found that the biocomposite material with a content of 200 wt. parts of coffee grounds and with a degree of drying of the composition of 20% has the highest compressive strength of 79.6 MPa. Biocomposites with a glutin solution concentration of 50% and a composition density of 1.17 g/cm^3 and 1.38 g/cm^3 have the highest values of compressive strength of 73.2 MPa and 74.8 MPa, respectively.

Among the biocomposites that were heat treated at lower temperatures of 100 °C and 120 °C, the highest values of compressive strength of 42.9 MPa and 47.8 MPa, respectively, are obtained for biocomposites with a glutin concentration of 43% compared to biocomposites with a glutin solution concentration of 46% and 43%. However, these values of compressive strength are 1.6-1.7 times lower compared to the maximum compressive strength (74.8 MPa) of biocomposites with a glutin concentration of 50%, which were subjected to heat treatment at 150 °C for 2 hours. Therefore, reducing the heat treatment temperature to 100 °C and 120 °C is not advisable, since a greater number of physicochemical bonds are formed between the biopolymer matrix and the filler at a higher temperature of 150 °C, which indicates a higher degree of structuring of these biocomposite materials. Carrying out additional heat treatment with holding the biocomposites in a thermal field for 3 hours additionally increases their compressive strength

by 9-24%, which is 40.6 MPa and 70.1 MPa for a coffee grounds content of 190 wt. parts and 200 wt. parts, respectively. The increase in the strength of biocomposites can be explained by the higher structuring of biocomposite materials due to additional removal of excess moisture. Increasing the holding time to 4 hours is not advisable, since it leads to a slight decrease in the compressive strength of biocomposites.



a, ×200 b, ×650

Fig. 7. **Microstructure of a biocomposite material filled with coffee grounds in an amount of 200 wt. parts with a degree of drying of the composition of 20%**

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