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The development of glutin-based biocomposite materials with advanced hydrophobicity

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Abstract. Biocomposite materials, which contain components of natural origin, have low resistance to water absorbtion due to high lyophilicity of components. It leads to quick destruction of biocomposite products under the conditions of high humidity exploitation that defines the necessity and importance of conducting scientific research in this sphere. The aim of the article is to research the hydrophobic additives influence on the compressive strength and hygroscopicity of biocomposite materials consisting of glutin matrix (100 parts by weight) and wood flour (100 parts by weight). The technology of forming biocomposite materials includes a step-by-step pressing the composition at a pressure of 10-15 MPa and a heat treatment of the product in a press mold at a temperature of 140°C. Experimental methods of investigating the biocomposite materials compressive strength and hygroscopicity were used in the work. It was experimentally determined that the modifying additive (paraffin) optimal content is 4 parts by weight per 100 parts by weight of biopolymer matrix. At this content, there is an increase compressive strength by 2.8-4.6 times of the biocomposite materials compared to biocomposites with different paraffin content. As a result of the state analysis of biocomposite samples which were destroyed under the static loading influence, the features of the cracks occurrence and propagation in biocomposites depending on different modifying additive content, the conduct of the composition preliminary processing or biocomposites additional processing in a thermal field were determined. The use of protective hydrophobic coatings on the biocomposite samples surface provided an increase in the biocomposites resistance to the moisture negative influence. The most effective were coatings based on a paraffin solution, which provided a reduction in water absorption by 45-50% compared to biocomposites coated with wax, drying oil Oksol, and sunflower oil. The complex use of hydrophobic substances as modifying additives and protective coatings has practical significance, as they positively affect the processes of forming the structure, increasing the strength and hydrophobicity of biocomposite materials used in a humid environment

Keywords: wood flour; sunflower oil; drying oil Oksol; paraffin solution; wax melt; compressive strength; thermal field

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Introduction

A biocomposite materials significant drawback is their high hydrophilicity, which is caused by the plant-based fillers ability to absorb moisture and water in significant quantities. This leads to the proliferation of microorganisms and biocomposite materials intensive degradation over a short period of product use. Therefore, it is relevant to research and determine the modifying additives impact on the physical, mechanical and operational properties of biocomposite materials, as the use of hydrophobic substances results in the interaction deterioration between the polymer matrix and the filler surface.

Polymer composite products contain a polymer matrix obtained through the petroleum products processing. Petroleum is a finite raw material source. This poses a problem related to the search for alternative raw material sources, the solution to which lies in the use of renewable resources. R.A. Ilyas & S.M. Sapuan (2020) noted that such raw stuff sources include materials of plant or animal origin which are able to regenerate under favourable conditions of the natural environment.

The use of natural materials as fillers in polymer composites has a history of decades, but their exploitation is limited to the use of synthetic polymer matrices. In the works of S.A. Albab *et al.* (2019), K.R. Sumesh *et al.* (2024), the research results on biocomposite materials based on polyether, polyurethane, epoxy, phenol-formaldehyde, and urea-formaldehyde resins were analysed, focusing on the feasibility of developing biocomposites with high physical, mechanical and operational characteristics. These resins have a high adhesive ability to form strong physical and chemical bonds with the plant-based fiber surface.

In the N. Uppal *et al.* (2022) study, the feasibility of forming biocomposite materials based on epoxy resins and fillers in the form of discrete flax fibers, industrial hemp, ramie and kenaf, as well as the sisal, pineapple, and banana leaves, was determined. Such composites have high economic profitability and satisfactory mechanical characteristics. The proposed fillers, after special treatment, are environmentally safe and potentially suitable raw stuff for new biocomposite materials.

Polymer composite materials based on synthetic polymers are widely used in the automobile industry as they provide high specific strength to constructions. Such polymer composites contain carbon and glass fibers, which are resistant to atmospheric factors, hence their disposal through natural means is complicated. In the M. Zwawi (2021) work, a high risk of waste accumulation in the form of discarded polymer composite parts and environmental pollution is identified. Consequently, there arises a need for the development of new biocomposite products containing environmentally safe fillers of natural origin that can be utilised in natural conditions without harming the environment.

The process of forming polymer composite materials using molten binders poses a significant danger to workers and the environment, as harmful substances emitted under the heating influence can evaporate. To mitigate the synthetic resins harmful impact is possible while using another binder type that allows the formation of polymer composite products without heating. M.A. Shamsudin *et al.* (2020) have developed a technology for forming such materials based on silicone rubber or epoxy resins containing kenaf fibers.

In the M.A. Paglicawan *et al.* (2021) study, the hybrid-type polymer composites production is practiced with the combined use of synthetic and natural origin fibers. Such fibers include aramid fibers, glass fibers, abaca fibers, and kenaf fibers. Synthetic fibers possess higher strength, resulting in biocomposites exhibiting better mechanical properties, as glass fibers withstand higher loads and ensure the integrity of the polymer matrix. This approach allows to replace a part of the synthetic polymer with another phase, represented by some material of natural origin, thereby improving the polymer composites mechanical properties. In the work of R. Chaturvedi *et al.* (2022), a biocomposite material containing granite particles and jute fibers was developed. This reduced the biocomposite products water absorption to 0.1% and enabled the mineral waste reuse, which poses significant environmental hazards. As a result of organic and mineral fillers hybrid use, an optimal balance between economic indicators and operational characteristics is achieved.

From 2004 to 2024, research on using materials of plant origin as matrices has been actively conducted. Polylactic acid (PLA) is widely used in global science as a biopolymer matrix for forming biocomposite materials. Particularly, in the work of R. Scaffaro *et al.* (2021), fine-dispersed fillers obtained from marine and agricultural waste were used for forming biocomposite materials based on PLA. Plant-derived powder fillers allow replacing up to 20% of the biopolymer matrix without significant changes in technological and operational properties. The interaction between the biocomposite material components can be improved with the use of modifying additives and coupling agents, thereby enhancing the resistance to biocomposite materials thermomechanical degradation and expanding the application scope of products based on natural components.

F. Ortega *et al.* (2021) established that biocomposite materials consisting solely of natural origin components exhibit high biodegradability. This determines their high susceptibility to degradation under the influence of the atmospheric factors and microorganisms. Such an approach to forming biocomposites ensures their ability for processing through composting or controlled application of fertilisers, positively affecting environmental safety and addressing the waste accumulation problem.

The aim of the study was to determine the influence of the hydrophobic additive content paraffin on the compressive strength and the influence of the

hydrophobic coating type on the water absorption resistance of biocomposite materials based on natural components.

Materials and methods

Biocomposite materials samples based on a glutin matrix and a wood flour were prepared. A 60% concentration glutin solution was obtained by dissolving bone glue granules in water, the physicochemical properties of which corresponded to the technical data specified in the Official website of Kremer Pigmente. 63000 Bone glue granules (2016). Preparation of the glutin solution began with soaking a measured amount of bone glue granules in an airtight container for 12-15 hours. The next stage involved soaking water-saturated bone glue granules in a drying oven at 50°C for 2.5-3.0 hours to intensify the dissolution process. To achieve high solution uniformity, it was periodically stirred. Wood flour was obtained by further mechanical grinding of fine-dispersed wood particles, which are waste products from lumber production. After grinding, the raw material was dried at 80-90°C for 1-2 hours with periodic mixture mixing. The prepared mixture was separated into fractions using a set of sieves, and the fraction with particle sizes of 0.5-0.7 mm was separated. The paraffin solution was obtained by dissolving paraffin flakes in drying oil, the physicochemical properties of which corresponded to DSTU 4153-2003 (2004).

The composition was prepared by mechanically mixing components in a ratio of 100 parts by weight of wood flour to 4 parts by weight of paraffin solution per 100 parts by weight of glutin solution. Mixing was carried out using a laboratory mill with a high-frequency rotation (20 kHz) of the hammer-type working element. A measured amount of the composition was placed into the matrix cavity of a cylindrical mold press form. The press form end holes were closed, and uniaxial pressing was performed with a 50 kN force. The punch was fixed in the press form to prevent elastic aftereffects. The composition in the press form was subjected to heat treatment at 140°C for 90 minutes with additional composition pressing. After cooling, the samples were removed from the press form and coated using hydrophobic environmentally friendly materials of natural origin: paraffin solution, beeswax, refined sunflower oil, drying oil Oksol. The coating was applied thinly to the cylindrical samples surface with subsequent stepped holding of the samples in a drying oven at 100°C for 20 minutes.

The compressive strength was determined according to ASTM D695-23 (2023) on cylindrical samples with a 30 mm height and a 20 mm diameter. The compression of the biocomposite samples was carried out under conditions of the approach of the lower traverse at a 2 mm/min speed with the fixation of the maximum load at which the biocomposite samples were destroyed. Hygroscopicity was determined by the weight method according to ASTM D3201/D3201M-20 (2020) on cylindrical samples with a holding time of 24 hours in a desiccator. Weighing was performed on WPS 110/C/1 3rd class laboratory scales with a measurement accuracy of up to 0.5 mg.

Results and discussion

Experimental results indicate that the biocomposite materials compressive strength limit without the modifying additive (paraffin) is 22-24 MPa (Fig. 1). The modifying additive introduction in an amount of 2 parts by weight does not lead to a change in the biocomposites compressive strength limit, indicating insufficient additive content. The paraffin uses in amounts ranging from 4 parts by weight to 12 parts by weight results in a decreasing the mechanical characteristics by 26-39%, attributed to the paraffin plasticising effect. Paraffin molecules penetrate the biopolymer matrix, leading to a reduction in the resistance to movement of the glutin amino acids macromolecule segments under static loading. In the of M.H. Mulla *et al.* (2023) work, it is noted that the application of inhibitors (wax, pectin) additionally reduces interfacial interaction, so their quantitative content needs to be limited in the process of developing biocomposite materials.

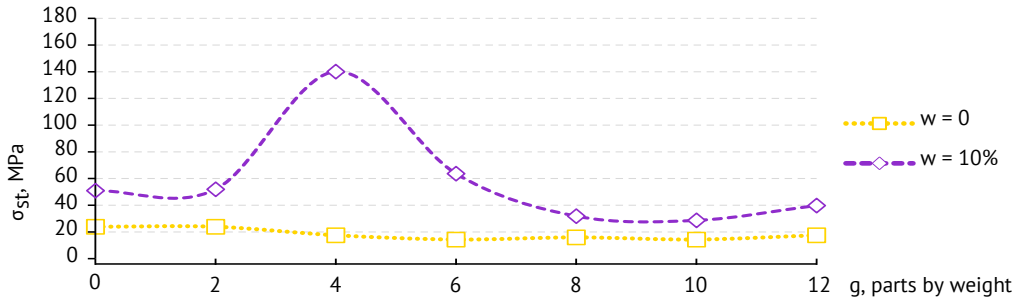


Figure 1. *The relationship between compressive strength limit and the content of the modifying additive (paraffin) at different degrees of composition moisture (W)*

Source: *developed by the authors*

Authors V.P. Kashytskyi *et al.* (2023b) have determined that using a composition without prior heat treatment results in the formation of biocomposite materials with reduced compressive strength due to the

residual moisture content in the composition components. The water is mainly contained in the glutin amino acid chains, as it serves a technological function in preparing the glutin solution. Conducting heat treatment during the biocomposite material formation ensures the removal of a significant moisture amount; however, some water molecules remain in the biopolymer matrix volume and are also adsorbed by wood flour particles.

The composition pre-treatment in a drying cabinet for 20-30 minutes at a temperature of 50-55°C leads to a 10% reduction in the mass of the composition due to moisture removal. The compressive strength limit of biocomposite materials that do not contain paraffin increases by 50-55% (51 MPa) since a rigid structure of the biopolymer matrix is formed. In the case of using paraffin in an amount of 2 parts by weight per 100 parts by weight of glutin, there is no decrease in the biocomposites strength, indicating insufficient additive content. At this content of paraffin, the biocomposite material structure remains unchanged. However, with a paraffin content of 4 parts by weight, the compressive strength limit of biocomposite materials increases by 2.6-2.8 times due to the modifying effect of the additive on structure formation. In this case, there is an even paraffin molecules distribution in the biopolymer matrix volume. It ensures increased packing density of the biocomposite material components by the lubricating additive presence, reducing the resistance to macromolecule segments microdisplacement and wood flour particles. At the level of the biopolymer matrix fine structure, paraffin molecules increase the flexibility of amino acids macromolecule segments, reducing intensive growth of stress in local areas of the biocomposite material. The use of modifying additives improves the properties of materials. For example, authors M. Huda *et al.* (2008) and A. Mohd *et al.* (2021) conducted chemical treatment of the natural fibers surface using silanes or alkali. This provides moisture content regulation and mechanical characteristics improvement of developed biocomposites based on polylactic acid (PLA) matrix compared to biocomposites containing untreated fibers. This is due to the natural fibers surface modification, resulting in improved interphase interaction and adhesion between the biocomposite material components serves a technological function in preparing the glutin solution. Conducting heat treatment during the biocomposite material formation ensures the removal of a significant moisture amount; however, some water molecules remain in the biopolymer matrix volume and they are also adsorbed by wood flour particles.

Increasing the paraffin content to 4-12 parts by weight leads to a decrease in the biocomposite materials compressive strength limit to values of 30-40 MPa, as the excess additive content provides intensive biopolymer matrix plasticisation. As a result, the resistance to static loading decreases due to the reduction in the number of physicochemical bonds between the biocomposite material components.

The biocomposite samples destruction after static loading occurs at different levels of plastic deformation degree. The degree of biocomposite materials deformation that do not contain the modifying additive (paraffin) and have excess moisture in the composition is 8-11%. Cracks are present on the cylindrical sample lateral surface, some of which are located at an angle of 42-47° to the sample end surface, while others are parallel to the end surface (Fig. 2a).

The numerous delaminations presence is associated with the low biocomposite material resistance to static loading under low levels of plastic deformation. The ruptures are related to the water molecules presence within the biopolymer matrix volume, which facilitate the glutin macromolecule segments movement. Consequently, during compression of biocomposite samples with compositions that have lost 10% of their moisture content, rupturing occurs with the fewer cracks formation compared to biocomposite samples containing moisture (Fig. 2b). This indicates a better biocomposites ability to resist static loading due to the formation of a stiffer framework within the biopolymer matrix.

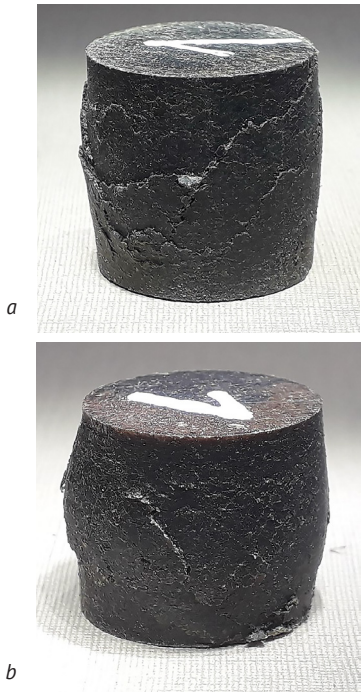
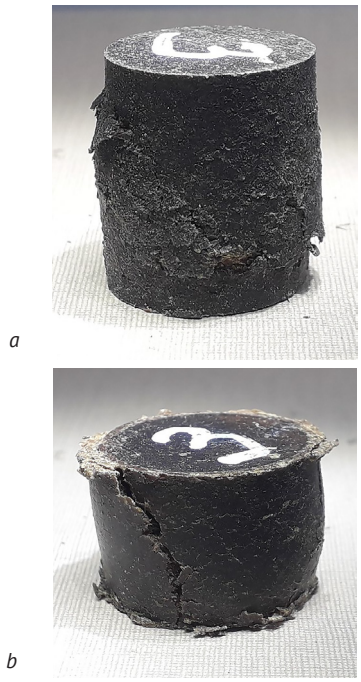


Figure 2. *General view of biocomposite samples without modifying additive after compression*
Note: *a – without heat treatment of the composition; b – loss of 10% of moisture by mass of the composition*
Source: *developed by the authors*

The propagation of a main crack during the biocomposite materials compression with 4 parts by weight of modifying additive fraction occurs at an angle of 43-47° to the sample end face (Fig. 3a). The interface between sample parts is less distinct compared

to the main cracks appearance in biocomposite samples without the modifying additive. This suggests the paraffin plasticising effect on the processes of forming the biopolymer matrix structure, where under the static loading influence, redistribution of normal and tangential stresses occurs. Additionally, local movements of biopolymer matrix macromolecule segments lead to the appearance of stress concentrators followed by material failure. Consequently, during a biocomposite sample compression with 4 parts by weight of paraffin content, whose composition was treated in a thermal field to remove 10% moisture content, a clearly defined main crack is formed, which is located at an angle of 57-62° to the end face surface (Fig. 3b). A single crack presence with a clearly defined shape indicates the predominant influence of normal stresses on crack development. In the biopolymer matrix volume, elastic deformations of local areas prevail, as a structure with amino acid macromolecule segments increased flexibility is formed due to the optimal paraffin molecules content.



a

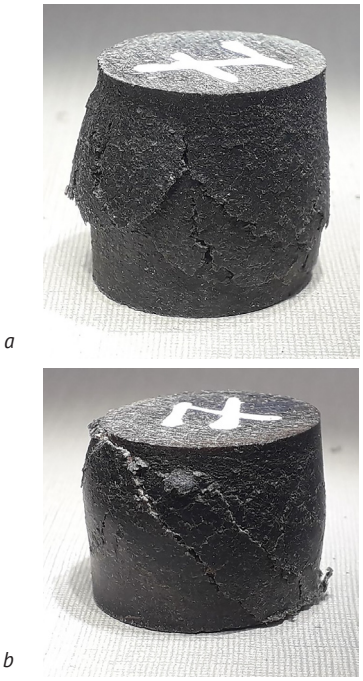
b

Figure 3. *General view of biocomposite samples with 4 parts by weight of the modifying additive (paraffin) content after compression*

Note: *a – without heat treatment of the composition; b – loss of 10% of moisture by mass of the composition*

Source: *photo by the authors*

The paraffin use in an amount of 12 parts by weight per 100 parts by weight of biopolymer matrix leads to the cylindrical samples lateral surface destruction with the numerous flake-like shape areas formation as a result of some biocomposite material local areas displacement in relation to others (Fig. 4a). This indicates the dominant influence of tangential stresses due to significant glutin matrix plasticisation, which contains an excessive moisture and modifying additive amount. In the case of biocomposite samples compression, the compositions of which were treated in a thermal field until the of moisture loss reached 10%, several cracks are formed (Fig. 4b). These cracks are located at an angle of 47-50° to the cylindrical sample end face plane, indicating even normal and tangential stresses distribution. The tangential stresses appearance is caused by the plasticising additive presence, the function of which is performed by paraffin. The appearance of normal stresses is caused by the glutin matrix formation with a lower moisture content, as the composition was treated in a thermal field until the moisture loss reached 10%. The composition contains less moisture, therefore, the ability of the biopolymer matrix macromolecules segments to move without the stress concentrators appearance is reduced.



a

b

Figure 4. *General view of biocomposite samples after compression with 12 parts by weight of the modifying additive (paraffin) content*

Note: *a – without heat treatment of the composition; b – loss of 10% of moisture by mass of the composition*

Source: *photo by the authors*

During the biocomposite materials development, it is necessary to achieve an increase in mechanical properties that ensure the strength and reliability of structural products. In the case of using plant-based fibers, there is a need to consider the fibers' ability to absorb moisture, which leads to deterioration of mechanical properties.

In works by authors E. Munoz & J.A. Garcia-Manrique (2015), L. Chen *et al.* (2022), satisfactory resistance to water absorption of biocomposite materials based on

epoxy or urea-formaldehyde polymers is achieved due to the high degree of the fiber surface wetting by the polymer binder and the hydrophobic barrier formation. However, the problem remains in eliminating the polymer matrix components harmful effects during material formation and disposal of used products. N. Ramli *et al.* (2018) analysed natural fibers used in the automobile industry to achieve ecological efficiency in car manufacturing. They pointed out that the materials used in production do not always meet environmental standards.

In the A. Shahzad (2013) work, a high susceptibility to water absorption of biocomposite materials containing natural hemp fibers was identified. V.A. Alvarez *et al.* (2006) established high biocomposite materials biodegradability during product disposal, but this also leads to rapid deterioration of such products during use. Therefore, the modifying additive (paraffin) usage is justified by the need to improve the biocomposite materials mechanical properties and increase the hydrophobicity of products containing natural components. As a result of experimental studies, it was found that the biocomposite materials ability to absorb moisture, with an optimal paraffin content (4 parts by weight), varies depending on the additional biocomposite materials heat treatment. V.P. Kashytskyi *et al.* (2023a) developed a heat treatment, which is biocomposites exposure in a thermal field at temperature of 50-55°C for 2.0-2.5 hours. This ensures the residual moisture removal from the biocomposite samples surface layers. The highest hygroscopic moisture content (1.4%) was recorded for biocomposite samples without hydrophobic coating and without additional heat treatment (Fig. 5). In the case of the composition pre-heat treatment, which was treated in a thermal field until a 10% moisture loss by mass, the hygroscopic moisture content is 1.27%. With the additional biocomposite samples heat treatment, the hygroscopic moisture content is 0.85-0.86%. This is 35-37% less than in biocomposite samples that were not additionally treated in the thermal field, as the heat treatment in the thermal field ensures a denser structure formation of the biopolymer matrix. At the same time, it is more difficult for water molecules to penetrate into the globules of the glutin amino acid matrix.

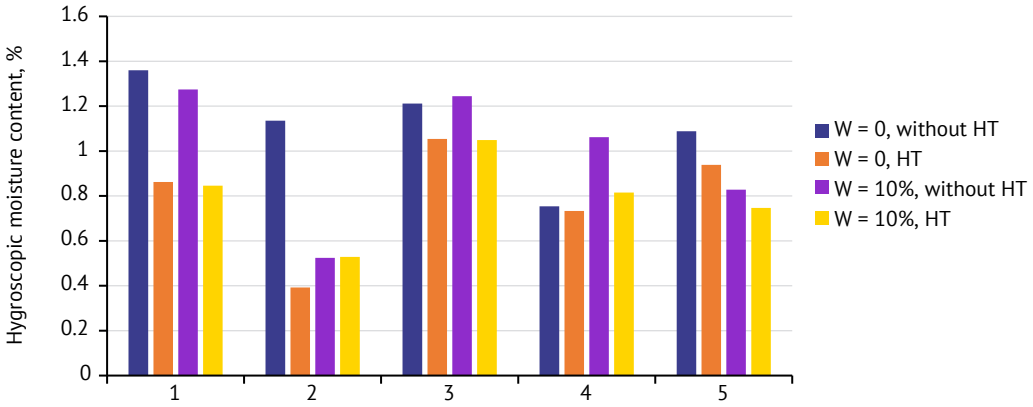


Figure 5. *Hygroscopic moisture content of biocomposite samples with applied hydrophobic coatings*
Note: *1 – without coating; 2 – paraffin solution; 3 – wax melt; 4 – drying oil Oksol; 5 – sunflower oil*
Source: *photo by the authors*

Hydrophobic paraffin coating application on the biocomposite samples external surface allowed achieving a hygroscopic moisture content of 1.13% in the case of using compositions that were not heat-treated. Such biocomposite materials contain excess moisture, which hinders the paraffin molecules adsorption. As a result, the ability to form a quality continuous coating is lost, and water molecules can penetrate into the biocomposite material interior. If additional biocomposite samples heat treatment is performed, the hygroscopic moisture content is minimal (0.39%). This is due to the water molecules removal from the biocomposite samples surface layers and the formation of a solid protective coating. In the case of applying a protective paraffin coating on the samples surfaces whose compositions were heat-treated before losing 10% moisture, the hygroscopic moisture content is 0.52%, that is slightly higher compared to biocomposites whose compositions were additionally heat-treated. However, these values are 52-55% lower compared to biocomposite materials whose compositions were not additionally heat-treated in thermal field. The paraffin coating formation provides the highest degree of biocomposite materials surface hydrophobic protection due to additional surface saturation with paraffin molecules, part of which is inside the material in the form of modifying additive.

Application of hydrophobic coating using melted wax does not provide effective biocomposite material protection from moisture. The hygroscopic moisture content for biocomposites whose compositions were not additionally heat-treated is 1.21-1.25%. This is

0.06-0.15% lower compared to similar samples without protective coating. Biocomposite samples absorb less moisture (1.04-1.05%) when additional heat treatment is carried out. However, this indicator is 0.2% higher compared to the hygroscopic moisture content of biocomposites without protective hydrophobic coating. This can be explained by the wax ability to hydrolyse in a humid environment, forming products capable of allowing water molecules to penetrate.

Biocomposite materials coated with drying oil provide better resistance to moisture absorption. Their hygroscopic moisture content is 0.73-0.82%, which is 0.23-0.32% lower than biocomposites with wax coatings. A similar situation is observed when using sunflower oil to form a hydrophobic coating. In this case, applying drying oil coating provides better hydrophobicity compared to oil coating. This is explained by the ability of drying oil to form a layer on the material surface, which provides better protection against moisture. Compared to drying oil, oil coatings do not always form such a protective barrier, so water molecules penetrate more easily through the oil coating.

Conclusions

Biocomposite materials containing plant-based fillers have a high tendency to absorb moisture. The use of hydrophobic additives allows to increase the biocomposite products hydrophobicity, but in most cases, it leads to the mechanical properties deterioration. The introduction of an optimal paraffin solution amount (4 parts by weight per 100 parts by weight of glutin matrix) allowed to obtain a biocomposite material with high compressive strength values of 138-141 MPa. The modifying additive acts as a lubricant during the composition compression, which contains fine-dispersed wood flour particles coated with a thin layer of biopolymer matrix. Preheating the composition ensures a 10% moisture loss by mass, which further complicates the particles movement during pressing. Therefore, the use of paraffin allows achieving higher biocomposite material density, which improves its resistance to static loads. The paraffin addition ensures the biopolymer matrix equilibrium structure formation as a result of residual stresses relaxation that occurs during the composition pressing and heat treatment in the process of forming biocomposite products.

Biocomposite materials with an optimal modifying additives content are destroyed without visible plastic deformation signs, forming a main crack located at an angle of 57-62° to the cylindrical sample end surface. This indicates the material's resistance to tangential stresses that cause plastic deformation. Consequently, the material is capable of withstanding higher compressive loads compared to biocomposites with a different composition.

Applying a paraffin solution to the biocomposite samples surface ensures the protective layer formation. In this case, biocomposite materials that underwent heat treatment to remove 10% moisture have the lowest hygroscopic moisture content (0.39%). Increasing the biocomposites hydrophobicity is associated with the paraffin molecules adsorption on the wood flour particles surface, which have minimal moisture content. The excess moisture presence in the composition or the use of other hydrophobic additives does not effectively increase the biocomposite materials hydrophobicity.

Further research is planned to investigate the effect of other hydrophobic additives on the formation processes, structure and mechanical properties of biocomposite materials based on natural components.

Conflict of Interest

None.

Acknowledgements

None.

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Розробка глютинових біокомпозитних матеріалів з підвищеною гідрофобністю

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Анотація. Біокомпозитні матеріали, що містять компоненти природного походження, мають низьку стійкість до водопоглинання через високу ліофільність компонентів. Це призводить до швидкого руйнування біокомпозитних виробів в умовах експлуатації підвищеної вологості, що визначає необхідність та важливість проведення прикладних досліджень в даній галузі. Метою роботи є дослідження впливу гідрофобних добавок на міцність при стисненні та гігроскопічність біокомпозитних матеріалів, що складаються з глютинової матриці (100 мас. ч.) та деревного борошна (100 мас. ч.). Технологія формування біокомпозитних матеріалів полягає у поетапному проведенні операції пресування композиції за тиску 10-15 МПа та термічної обробки виробу у прес-формі за температури 140°С. У роботі використано експериментальні методи дослідження міцності при стисненні та гігроскопічності біокомпозитних матеріалів. Експериментально встановлено оптимальний вміст модифікуючої добавки (парафін) в кількості 4 мас. ч. на 100 мас. ч. біополімерної матриці. За такого вмісту відбувається підвищення межі міцності при стисненні біокомпозитних матеріалів у 2,8-4,6 разів порівняно з біокомпозитами з іншим вмістом парафіну. В результаті аналізу стану зруйнованих під дією статичного навантаження біокомпозитних зразків визначено особливості виникнення та поширення тріщин в біокомпозитах з різним вмістом модифікуючої добавки, а також залежно від проведення попередньої обробки композиції або додаткової обробки біокомпозитів у тепловому полі. Використання захисних гідрофобних покриттів на поверхні біокомпозитних зразків забезпечило підвищення стійкості біокомпозитів до негативного впливу вологи. Найбільш ефективним виявилися покриття на основі розчину парафіну, які забезпечили зниження водопоглинання на 45-50 % порівняно з біокомпозитами, що покриті розплавом воску, оліфою та олією соняшниковою. Комплексне використання гідрофобних речовин як модифікуючих добавок та захисних покриттів мають практичне значення, оскільки позитивно впливають на процеси формування структури, підвищення міцності та гідрофобності біокомпозитних матеріалів, які експлуатуються у вологому середовищі

Ключові слова: деревне борошно; олія соняшникова; оліфа оксоль; розчин парафіну; розплав воску; міцність при стисненні; теплове поле