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EVALUATING THE BIORESISTANCE OF WOOD-POLYMER COMPOSITE BASED ON RECYCLED THERMOPLASTICS WITH A MODIFIED WOOD FILLER AND POLYMER MATRIX

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Abstract. The article investigates the moisture absorption of wood-polymer composite (WPC) and the development of fungal cultures during its operation. The study aims to identify the optimal WPC composition most resistant to fungal growth, examine the effect of fungal development in relation to WPC water absorption, analyse the impact of absorbed moisture on fungal growth, investigate the influence of polymer matrix and wood filler modifications on fungal colony development, assess the fungal resistance of WPC with a modified polymer matrix and wood filler in comparison with industrial and previously proposed samples and provide a description of climatic impacts on WPC as well as data on fungal colony growth and the correlation between material degradation and fungal culture development.

Keywords: composite, wood, recycled raw materials, polyethylene, thermoplastic polyurethane, modification, properties, fungal resistance.

1. Introduction

Wood-polymer composite (WPC) is one of many types of composite materials.[1] WPCs represent a promising material based on natural raw materials, distinguished from wood by their atmospheric, mechanical, and chemical moisture and water resistance.[2, 3] Due to these properties, WPCs are widely used for finishing rooms with high humidity levels and for exterior applications such as terraces and docks.[4, 5] The popularity of products made from these materials is driven by important functional, economic, and environmental factors.[6] A rather relevant direction in the processes of development and research of modern WPCs is the study of the features of legal regulation of waste polymer matrices and organic fillers.[7, 8] It reflects the importance of ensuring effective processes of production and use of WPCs in accordance with the principles of sustainable development.[9, 10] The growing range of raw materials for composite production enables the continuous expansion of their applications. At the same time, new types of adverse side effects arise, affecting the operational characteristics of products. An essential aspect of studying these challenges is the development of new, effective methods for their identification and solutions.

Violating the rules of storage, processing, and raw material preparation leads to the fact that raw materials obtain properties, complicating the production process and resulting in degraded operational characteristics of the final product.[11]

The WPC composition typically includes thermoplastic polymers, wood fillers in the form of sawdust, chips, inorganic additives, and other modifiers. The synthetic polymers used in the production of wood-polymer composites exhibit significant resistance to biological damage.[12, 13] This is confirmed by several decades of plastic products' operation in harsh conditions (machines, household items, pipes, formwork, construction elements, *etc.*).

The use of unmodified WPC in rooms with high humidity or outdoors can lead to the formation of microcracks or dimensional changes in the products, which contribute to a reduction in the material's bioresistance. Various antiseptic agents are used to enhance this resistance, considering they may reduce the material environmental sustainability. The use of

synergistic modification of the wood-polymer composite has been proposed as an alternative.

The main negative factor of WPC products is the presence of moisture both in the raw material and in the final product, leading to the product degradation after a relatively short period of use.

A synergistic modification of the wood-polymer composite has been proposed to improve the physical-mechanical and operational characteristics of the material.

The production of WPC items is one of the most widespread methods in the world for recycling difficult-to-process waste. However, like any material, WPC has both advantages and disadvantages.[14] The main advantage of these types of materials is the environmental aspect, while the primary disadvantage is water absorption. Moisture in the material can be either acquired during operation or residual, which remains in the raw material due to improper storage conditions or errors during production.[15] To determine the moisture content both in the finished product and at intermediate stages of production, methods such as dielectric permeability, the tangent of dielectric loss angle, and electrical resistance are used.[16] Finding these indicators allows for the timely detection of moisture presence in the polymer matrix and wood filler, enabling adjustments to the WPC production process.

Moisture, whether residual or acquired during operation, facilitates the growth of microbes and various fungi both on the surface and within the material. The internal development of microbial and fungal cultures is the primary cause of product degradation.[17] The growth of microbes and fungal colonies inside the material cannot be entirely halted, and consequently, neither can result in its degradation.[18, 19] The use of the synergistic modification method involving the simultaneous modification of both the polymer matrix and the wood filler is a promising and effective approach to preventing water absorption and extending the service life of WPC products.[20, 21]

The research presented in the article aims to develop a WPC composition resistant to water absorption in order to prevent a premature degradation of WPC products. To achieve this goal, the following objectives were necessary:

1. To determine the optimal composition of WPC with a minimal water absorption rate.

2. To investigate the impact of fungal culture development depending on the level of water absorption in WPC.

3. To explore the effect of modification of the polymer matrix and wood filler on the growth of fungal colonies in WPC.

4. To examine the fungal resistance of WPC with a modified polymer matrix and wood filler and compare it with industrial and previously proposed samples.

2. Experimental

2.1. Materials

The research objects included: recycled polyethylene, recycled thermoplastic polyurethane, expanded polystyrene, petroleum-polymer resin, particleboard waste, microcalcite, gasoline, and Candida albicans fungi.

2.2. Methods

The preparation of WPC was carried out as follows: a solvent (gasoline) was added to the petroleum-polymer resin and thoroughly mixed until a homogeneous solution (intermediate modifying mixture) was obtained, which took 10–15 minutes at a speed of 50 rp/m. The next step involved adding expanded polystyrene waste, dissolved in the intermediate modifying mixture through continuous stirring for 10 minutes, resulting in the final modifying mixture. The obtained modifying mixture was then combined with the wood filler for impregnation and mixed for 5–10 minutes until complete homogenization at a speed of 50 rpm. Finally, microcalcite was added to the impregnated and homogenized wood filler and mixed for 5 minutes at a speed of 50 rpm.

The homogenized modifying mixture was dried in a laboratory dryer for 1 hour at a temperature of 70 °C or left to air dry at room temperature for 12 hours.

During the impregnation stage with the liquid phase and subsequent drying, 12–1 % of the excess solvent evaporated. The dried filler was then mixed with recycled polymer raw materials (polyethylene (PE) and a combination of polyethylene and thermoplastic polyurethane (PE + TPU)) and processed into WPC using the extrusion method. The extrusion was carried out on an extruder with a length-to-diameter (L/D) ratio of 40, a screw diameter of 30 mm, and a rotation speed of 90 rpm. The temperature was maintained within the range of 190–195 °C throughout the entire process.

Identifying a sample density was conducted in accordance with ASTM-D-792. Impact strength (ISO 180:2000), flexural strength at break (ISO 178:2010), Brinell hardness (ISO 2039-2), and impact resistance (ISO 6272-1:2002) were evaluated according to international standards. Water absorption was measured following ASTM-D-570, while wear resistance and shrinkage were assessed in accordance with DIN 53 516. Dimensional changes due to heating were determined according to ISO 11501:1995 using an electronic caliper. The measurement accuracy was up to 0.01 %.

The fungal colony development was studied as follows:

Stage 1 – contamination of samples with the museum culture of yeast-like fungi Candida albicans at a concentration of 10^5 CFU/cm³: the culture of the specified concentration was prepared by diluting the museum culture with sterile distilled water until the required concentration was achieved, with concentration measurements performed using a densilameter.

The prepared culture was applied to the surfaces of Sample No. 1 and Sample No. 2, which had been pre-treated with a cotton swab soaked in 70 % alcohol. The surface area subjected to contamination was 50 cm² for each sample, with 25 cm² designated for further incubation during the experiment and 25 cm² used for contamination control by the swab rinse method immediately after inoculation.

Stage 2 – verifying the presence of the specified concentration of Candida albicans yeast-like fungi on the contaminated surfaces of Sample No. 1 and Sample No. 2 using the swab rinse method in accordance with DSTU ISO 18593:2006. A sterile swab was moistened in a sterile physiological solution, and excess liquid was removed by pressing the tip against the wall of a test tube containing the solution. The swab samples were taken from areas of 25 cm² for each sample. During sampling, the swab stick was rolled between the thumb and fourth finger while moving it across the test surface in both perpendicular directions (forming a right-angle pattern). The swab tip was then aseptically broken off or cut into a test tube containing 5 cm³ of physiological solution, which was shaken in a shaker for 30 seconds.

The resulting primary suspension was inoculated into two Petri dishes (for parallel analysis) containing Sabouraud agar, with 1 cm³ of inoculum distributed evenly across the agar surface using a sterile spreader. The Petri dishes were placed horizontally until the surface dried, then incubated in a thermostat at 20–22°C for 5 days. After the incubation period, the number of colonies formed as a result of the swab plating from each sample (Sample No. 1 and Sample No. 2) was counted. The next step involved calculating the number of colony-forming units (CFU) per 1 cm² of the tested surface (*Ns*) using the following formula:

$$Ns = \frac{N \times F}{A} \times D \quad (1)$$

where *Ns* – the number of CFU in 1 cm³ of dilution; *F* – the volume of dilution in the test tube, cm³; *A* – the examined surface area, cm²; *D* – the corresponding value of the applied dilution.

Based on the results of the counting during the control study of surface contamination of Samples No. 1 and No. 2 with the Candida albicans yeast-like fungal culture, it was confirmed that the samples were contaminated with the pathogen at a concentration of 10^5 CFU/cm³.

Stage 3 – finding the presence or absence of increased concentration of Candida albicans on the surface of the samples under conditions favorable for the growth of this pathogen. Creating and maintaining favorable conditions for the formation of new colonies (growth) of Candida albicans yeast-like fungi for 5 days (exposure time) by maintaining a constant temperature of 22 °C and increased humidity (≥78 %) in an incubator, in which Samples No. 1 and No. 2, contaminated with Candida albicans culture at a concentration of 10^5 CFU/cm³, are placed for incubation., Samples No. 1 and No. 2 undergo further examination using the swab method (described in Stage 2) after the incubation period followed by the counting of the grown colonies and the calculation of the pathogen concentration on the surface of each sample (No. 1 and No. 2) using the formula provided in Stage 2.

3. Results and Discussion

The composition of the developed wood-polymer composites with a modified polymer matrix is presented in Table 1.

The composition of the developed wood-polymer composites with modified wood filler is presented in Table 2.

Table 1. The composition of wood-polymer composites with modified polymer matrices

Compo-sition	Recycled PE, %	Recycled TPU, %	Petroleum-based polymer resin, g	Solvent gasoline B-70, g	Polystyrene waste, g	Furniture production waste, g	Microcalcite, g
1	97	3	5	15	5	25	3
2	95	5	5	15	5	25	3
3	93	7	5	15	5	25	3
4	90	10	5	15	5	25	3
5	85	15	5	15	5	25	3
6	80	20	5	15	5	25	3

Table 2. The composition of wood-polymer composites with modified wood filler

Com-position	Recycled PE, %	Recycled PP (polypro-pylene), %	Petroleum-based polymer resin, g	Sol-vent, g	Varnish PF-170, g	Polystyrene waste, g	Furniture production waste, g	Wood waste mixture, g	Micro-calcite, g
1	25	–	20	25	–	–	30	–	–
2	–	25	20	25	–	–	30	–	–
3	25	–	10	20	–	5	30	–	–
4	–	25	10	20	–	5	30	–	–
5	25	–	10	20	–	10	30	–	–
6	25	–	5	15	–	10	20	–	–
7	25	–	5	15	–	10	20	–	10
8	25	–	5	15	–	10	20	–	20
9	25	–	5	15	–	10	–	35	20
10	25	–	5	15	–	8	–	30	15
11	25	–	5	15	15	–	40	–	–
12	25	–	5	15	15	–	30	–	5
13	25	–	5	15	20	–	35	–	5

The water absorption indicators of wood-polymer composites with a modified polymer matrix are presented in Table 3.

The water absorption indicators of wood-polymer composites with modified wood fillers are presented in Table 4.

Table 3. The water absorption indicators of wood-polymer composites with a modified polymer matrix

Composite	Water absorption W, %	Dimensional change in the range of +30 °C to +80 °C	Dimensional change at –15 °C
1	+2.64	Not detected	Not detected
2	+2.51	Not detected	Not detected
3	+2.38	Not detected	Not detected
4	+2.2	Not detected	Not detected
5	+3.6	Not detected	Not detected
6	+5.2	Not detected	Not detected

Table 4. The water absorption indicators of wood-polymer composites with modified wood fillers

Composite	Water absorption, W, %	Wear resistance, m	Wear resistance, V	Dimensional change in the range of +30 °C to +80 °C	Dimensional change at –15 °C
1	+5.7	0.1015	0.2997	Not detected	Not detected
2	+5.8	0.1336	0.320	Not detected	Not detected
3	+6.4	0.112	0.304	Not detected	Not detected
4	+6.6	0.088	0.305	Not detected	Not detected
5	+6.5	0.108	0.308	Not detected	Not detected
6	+6.2	0.142	0.288	Not detected	Not detected
7	+5.4	0.113	0.283	Not detected	Not detected
8	+5.3	0.101	0.270	Not detected	Not detected
9	+5.3	0.102	0.325	Not detected	Not detected
10	+6.3	0.103	0.278	Not detected	Not detected
11	+6.1	0.052	0.325	Not detected	Not detected
12	+5.9	0.072	0.327	Not detected	Not detected
13	+6.2	0.075	0.325	Not detected	Not detected

The water absorption indicators of industrial samples of WPC are presented in Table 5.

The analysis of the data in Tables 3, 4, and 5 indicates the superiority of the proposed samples compared to the industrial samples. The proposed samples demonstrate the effectiveness of the modification process both for the wood filler and the polymer matrix. This is also achieved due to the high homogeneity of the distribution of the wood filler in the polymer matrix, which is proven by the results of microscopic and operational studies in our articles.[22, 23] The advantage of the proposed WPC samples with modified wood filler compared to the industrial samples ranges from 100 %, depending on the type of industrial WPC and the type of the proposed sample.

Table 5. The water absorption indicators of industrial samples of WPC

Sample	Standards for testing methods.	The sample contaminated with Candida albicans yeast-like fungi at a concentration of 10^5 CFU/cm³	The sample contaminated with Candida albicans yeast-like fungi after storing the sample for 5 days in conditions favorable for the growth of the pathogen
Proposed sample No. 4 with synergistic modification	DSTU EN 14126:2008	Candida albicans 10^5	Candida albicans 10^5
Proposed sample No. 8 with modified wood filler	DSTU EN 14126:2008	Candida albicans 10^5	Candida albicans 10^6
Industrial WPC sample	DSTU EN 14126:2008	Candida albicans 10^5	Candida albicans 10^7
MDF sample	DSTU EN 14126:2008	Candida albicans 10^5	Candida albicans 10^9
OSB sample	DSTU EN 14126:2008	Candida albicans 10^5	Candida albicans 10^9

Figs. 1, 2 show the water absorption indicators of the wood-polymer composite with a modified polymer matrix and wood filler. A comparative analysis of Fig. 1 and Fig. 2 demonstrates the effectiveness of the simultaneous modification of both the wood filler and the polymer matrix. The advantage of the synergistic modification over the modification of any single component of the wood-polymer composite is more than five times.

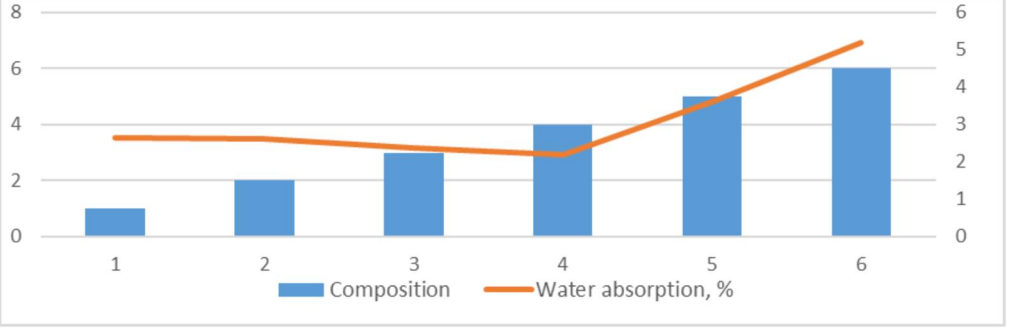

Fig. 1. Water absorption indicators of WPC with a modified polymer matrix and wood filler

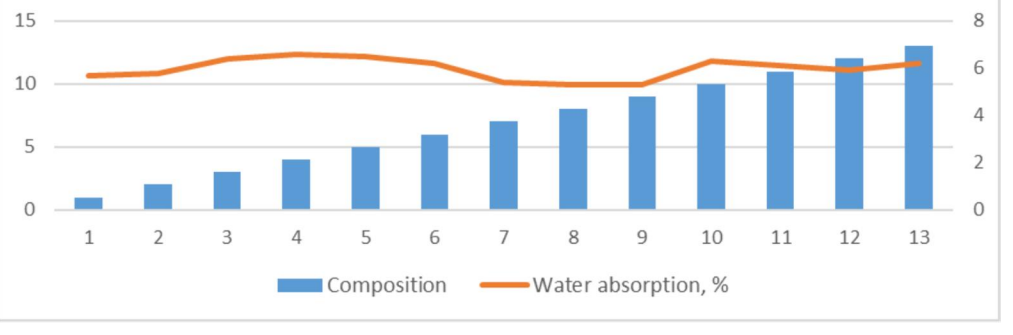

Fig. 2. The water absorption indicators of WPC with modified wood filler

The results of the tests for fungal resistance are presented in Table 6.

Fig. 3 shows the development of the fungal culture over the course of 5 days in the proposed sample with a modified matrix and filler and in commercial samples.

Fig. 3 clearly demonstrate the effectiveness of synergistic modification in the production of wood-polymer composites.

4. Conclusions

Thus, the analysis of accumulated scientific and industrial experience indicates the advantages of the proposed method of synergistic modification in the production of wood-polymer composites. Analysis of the obtained results indicates the superiority of the developed WPC samples based on a matrix in the form of secondary polyethylene (25 and 90 wt. %) and thermoplastic polyurethane (10 wt. %) compared to industrial samples. The proposed samples demonstrate the efficiency of the modification process of both the wood filler and the polymer matrix. The advantage of the proposed WPC samples with a modified wood filler compared to industrial samples ranges from 100 % depending on the type of industrial WPC and the type of the proposed sample. The efficiency of simultaneous modification of both the wood filler and the polymer matrix of WPC samples based on a matrix in the form of secondary polyethylene (25 and 90 wt. %) and thermoplastic polyurethane (10 wt. %) was also established. The development of fungal culture within 5 days in the proposed WPC samples based on a matrix in the form of secondary polyethylene (25 and 90 wt. %) and thermoplastic polyurethane (10 wt. %) with a modified matrix and a filler has been proven to be slower than in industrial samples.

Table 6. Testing of samples for fungal resistance

Sample	Standards for testing methods	The sample contaminated with Candida albicans yeast-like fungi at a concentration of 10^5 CFU/cm³	The sample contaminated with Candida albicans yeast-like fungi after storing the sample for 5 days in conditions favorable for the growth of the pathogen
Proposed sample No. 4 with synergistic modification	DSTU EN 14126:2008	Candida albicans 10^5	Candida albicans 10^5
Proposed sample No. 8 with modified wood filler	DSTU EN 14126:2008	Candida albicans 10^5	Candida albicans 10^6
Industrial WPC sample	DSTU EN 14126:2008	Candida albicans 10^5	Candida albicans 10^7
MDF sample	DSTU EN 14126:2008	Candida albicans 10^5	Candida albicans 10^9
OSB sample	DSTU EN 14126:2008	Candida albicans 10^5	Candida albicans 10^9

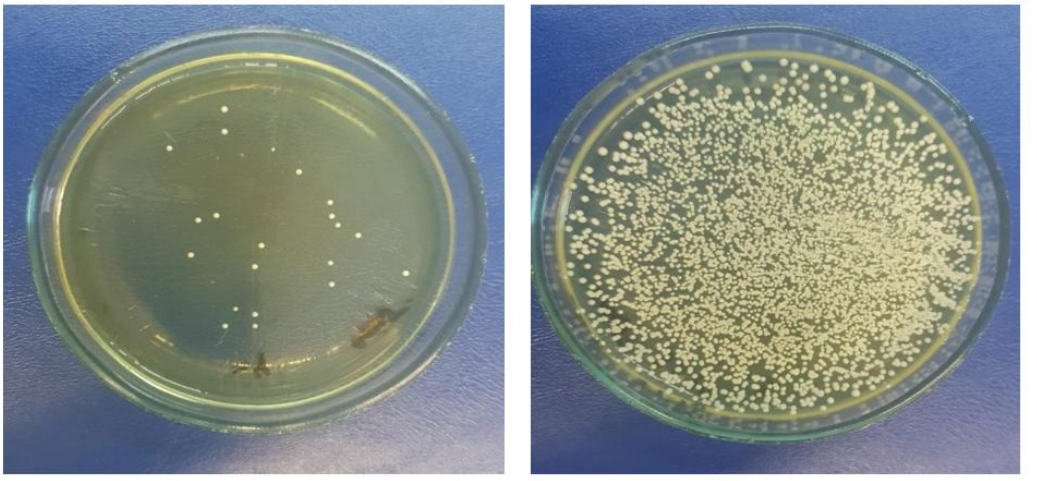

Fig. 3. Development of the fungal colony: A – proposed sample; B – commercial sample.Water absorption indicators of WPC with a modified polymer matrix and wood filler

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ВИЗНАЧЕННЯ БІОСТІЙКОСТІ ДЕРЕВИНО-ПОЛІМЕРНОГО КОМПОЗИТУ З ВТОРИННИХ ТЕРМОПЛАСТІВ З МОДИФІКОВАНИМИ ДЕРЕВИННИМ НАПОВНЮВАЧЕМ І ПОЛІМЕРНОЮ МАТРИЦЕЮ

Анотація. *У статті досліджено результат поглинання вологи деревино-полімерним композитом (ДПК) та розвиток грибної культури під час його експлуатації. Аналіз одержаних результатів свідчить про перевагу розроблених зразків ДПК на основі матриці у вигляді вторинних поліетилену (25 та 90 мас. %) та термопластичного поліуретану (10 мас. %) порівняно із промисловими зразками. Запропоновані зразки демонструють ефективність процесу модифікації як деревного наповнювача, так і полімерної матриці. Перевага запропонованих зразків ДПК із модифікованим деревним наповнювачем порівняно з промисловими зразками коливається від 100 % залежно від типу промислового ДПК та типу запропонованого зразка. Також встановлено ефективність одночасної модифікації як деревного наповнювача, так і полімерної матриці зразків ДПК на основі матриці у вигляді вторинних поліетилену (25 та 90 мас. %) та термопластичного поліуретану (10 мас. %). Перевага синергетичної модифікації над модифікацією будь-якого окремого компонента деревно-полімерного композиту – більше ніж у п'ять разів. Доведено розвиток культури грибів упродовж 5 діб у запропонованих зразках ДПК на основі матриці у вигляді вторинних поліетилену (25 та 90 мас. %) та термопластичного поліуретану (10 мас. %) з модифікованою матрицею та наповнювачем та у промислових зразках.*

Ключові слова*: композит, деревина, вторинна сировина, поліетилен, термопластичний поліуретан, модифікація, властивості, грибостійкість.*