materials proceedings

Proceeding Paper

Effect of Fungi Removal Method on the Mechanical Properties of Polymer Composites Reinforced with Oat and Millet Husks †

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† Presented at the 10th MATBUD'2023 Scientific-Technical Conference "Building Materials Engineering and Innovative Sustainable Materials", Cracow, Poland, 19–21 April 2023.

Abstract: Polymer composites reinforced with crop husks show susceptibility to fungi of the *Agaricomycotina* subdivision, particularly *Coniophora puteana*. The material's resistance to the fungi is evaluated by exposing specimens to the fungus for 16 weeks, and then determining the mass loss and changes in mechanical properties. An important pre-testing step is cleaning the samples of mycelium. The study compares the effects of the manual cleaning method with a soft brush and with water under pressure. The aim of the study is to select a method that is non-destructive to the material. The results will be evaluated by mass loss, bending strength and modulus of elasticity.

Keywords: polymer composites; husks; oat; millet; fungi; mycelium removal

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Citation: Goron, M.; Sudoł, E. Effect of Fungi Removal Method on the Mechanical Properties of Polymer Composites Reinforced with Oat and Millet Husks. *Mater. Proc.* **2023**, *13*, 33. https://doi.org/10.3390/materproc2023013033

Academic Editors: Katarzyna Mróz, Tomasz Tracz, Tomasz Zdeb and Izabela Hager

Published: 16 February 2023

1. Introduction

Recently, in the construction industry, great attention has been paid to various ecological aspects. As a result, materials that are easily biodegradable and durable are in demand [1,2]. In some applications such as terraces and façades, wood has been replaced by composites. Composite materials are made of polymers (PP, PVC or HDPE) acting as matrices and lignocellulosic particles as fillers. Natural fillers in the form of flour, fibers or shavings, as well as fibers from crop husks, for example, rice or oat [3–5], are used in composite materials. Natural fibers have a low density, are biodegradable, widely available and inexpensive. Therefore, they are viewed as more attractive materials than commonly used synthetic fibers. However, they also have various disadvantages, such as low thermal stability and susceptibility to moisture absorption or microorganisms [6–8]. It was thought that a thermoplastic material completely encapsulates the natural fiber component of the composite, protecting it from moisture and fungal decay. However, some publications show that fully encapsulating natural fibers with a polymer matrix is not possible [9,10].

Some publications prove that biodeterioration has a negative impact on the mechanical and physical properties of natural fiber composites [7,9,11–13]. This is determined by weight loss and changes in material strength [1,3,12,14]. Composite materials used in outdoor conditions are exposed to many factors, such as UV radiation, freezing, leaching, temperature changes and biological agents, so it is difficult to determine which factor has a decisive effect on degradation, since they all act simultaneously [3,15].

One of the most important steps in preparing composite specimens for testing after exposure to fungi is mycelium removal. Following exposure to fungi, specimens usually show significant mycelium overgrowth, which can interfere with the testing equipment and impair measurements. In addition, mycelium can cause damage to human health, so it should be removed before further tests. Often, this step is skipped in method descriptions in composite-related publications [6,13,16,17]. Some authors shortly describe the cleaning process. Composites can be cleaned with a soft sponge [8] or by sanding [18].

As composite materials are now widely used, this study aims to explore the resistance to fungal decay of the *Agaricomycotina* subdivision [19] and the effect of the method of mycelium removal after microbiological exposure on their mechanical properties. The subject of the study is composites reinforced with natural fibers, including oat and millet husks.

2. Materials

2.1. Composite Materials

The subject of the study is two types of composite construction profiles made of the following materials:

1. A composite with a PVC matrix and pulverized oat husk filler;
2. A composite with a PVC matrix and pulverized millet husk filler.

The profiles are designed for outdoor flooring, produced from the mixtures described in Table 1.

Table 1. Composition of composites used in the production of profiles.

Marking	Matrix	Mineral Filler (Type/Share [1])	Natural Filler (Type/Share [1])	Other [2]
Oat	PVC	$CaCO_3$/ 50 phr	Pulverized oat husks/ 30 phr	Physical modifiers, stabilizers; polyethylene wax
Millet			Pulverized millet husks/ 30 phr	

[1] The proportion of mixture components is given in parts per 100 phr (parts per hundred rubber). [2] Detailed data are not available due to company secrecy.

The profiles were manufactured by extrusion under industrial conditions. The usable surfaces were mechanically treated by brushing, which is standard for construction profiles.

2.2. Testing Specimens

Test specimens (80 × 10 × 5 mm) were cut from the central part of the profiles. The following series of specimens were used in the tests:

1. Reference specimens;
2. Test specimens (exposed to water and fungi);
3. Control specimens for the determination of dry mass.

Specimens were cut from each composite in the following quantities:

- Ten specimens for series 1 and 3;
- Forty specimens for series 2, including two methods of cleaning (by brushing and with water under pressure) and two methods of testing (loss in mass and flexural strength), which required 10 specimens for each version.

3. Methods

The resistance of the composites to a fungus of the *Agaricomycotina* subdivision was assessed using two methods. After exposure to the *Coniophora puteana* fungus and the application of two methods of mycelium removal, the mass loss and changes in flexural strength of the specimens were determined.

3.1. Mass Loss of Composites after Exposure to Coniophora Puteana

3.1.1. Pre-Conditioning by Soaking in Water

Series 2 specimens cut from both types of composite profiles were subjected to the aging cycle by soaking. The specimens were soaked in distilled water for 14 days. The water was changed 9 times during the cycle. The specimens were later dried in an environment-controlled room with a temperature of 20 °C and a humidity of 65%.

3.1.2. Determination of Initial Dry Mass

Series 3 specimens were also soaked in distilled water for 14 days, which was changed 9 times. Ten specimens were dried in an oven at 103 °C to constant weight. A moisture coefficient of each material was determined using these measurements. The initial dry mass for the remaining series was calculated by multiplying the moisture coefficient by their weight after soaking in water for 14 days.

3.1.3. Preparing Fungi for the Test

A medium of 40 g of malt extract, 35 g of agar and 1000 mL of distilled water was prepared in Kolle flasks. The medium was sterilized in an autoclave at 121 °C and 1 atm pressure. After solidifying, the medium was inoculated with hyphae of the fungus *Coniophora puteana*. The flasks were stored in an incubating chamber at 22 °C and 70 % humidity until the surface of the medium was completely covered by the mycelium.

3.1.4. Assessment of Fungal Activity

Using previously prepared test cultures of Coniophora puteana, the fungal activity of the specimens was verified. Specimens of Scots pine wood (15 × 25 × 50 mm) were placed in Kolle flasks on medium covered by the mycelium. The flasks were stored in an incubating chamber at 22 °C and 70% humidity for 16 weeks. After removal of the mycelium, the wood specimens were weighed and the mass loss was determined. It was confirmed that they achieved the required loss in mass over 20%.

3.1.5. Mass Loss Test

The composite specimens of series 2 were placed on the mycelium with the usable surface in direct contact with it. The flasks with specimens were transferred to the incubating chamber for 4 months. After the incubation period, the specimens were removed from the flasks. Half of the specimens underwent manual mycelium removal with a soft brush. The other half underwent mycelium removal using pressurized water and were dried for 7 days at 40 °C. Furthermore, the specimens were dried at 103 °C to determine the final dry mass after their exposure to fungus. Loss in mass was calculated according to (1). The result is expressed in %.

$$\Delta m_d = (m_i - m_c)/m_c \cdot 100, \quad (1)$$

where m_i—initial dry mass of the specimen, in grams; m_c—final dry mass of the specimen, in grams.

3.2. Bending Strength and Modulus of Elasticity after Exposure to Coniophora Puteana

The preparation and exposure to fungus of the specimens used to determine the flexural strength of the composites after fungal decay were carried out in the same way for the mass loss specimens (3.1.) After the incubation period of 4 months, the specimens were removed from the flasks, cleaned of mycelium by two methods and dried in an environment-controlled room at 20 °C and 65% humidity.

The specimens were tested for flexural strength and modulus elasticity. The specimens were placed with the usable side down to apply tension to the fungus-exposed surface. The load was applied at a rate of 2 mm/min until destruction.

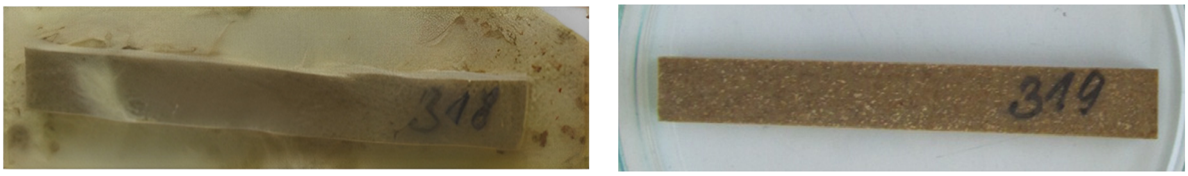
The results obtained for the specimens exposed to fungi were related to the results obtained for the specimens in the initial state. The initial state results were obtained from previous tests for the same materials [20]. This made it possible to determine, according to (2), the effect of this exposure on the tested materials. The result is expressed in %.

$$\Delta\sigma_d = (\sigma_d - \sigma_i)/\sigma_i \cdot 100, \Delta E_d = (E_d - E_i)/E_i \cdot 100, \quad (2)$$

where σ_d, E_d—the flexural strength and modulus of elasticity after fungi exposure, in MPa; σ_i, E_i—the initial flexural strength and modulus of elasticity, in MPa.

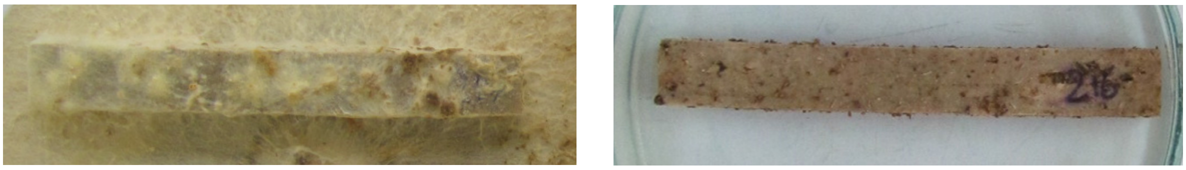
4. Results and Discussion

Figures 1a and 2a illustrate the specimens of composites after exposure to *Coniophora puteana* over 16 weeks. The oat husk-reinforced composites demonstrated higher resistance to fungal overgrowth. The millet husk-composites showed significant susceptibility to *Coniophora puteana*. The mycelium was removed manually from both types of specimens using a soft brush (Figures 1b and 2b). The specimens, which were cleaned by water, looked the same, but had to be dried for 7 days at 40 °C to remove excess moisture.



(**a**) (**b**)

Figure 1. Oat husk-reinforced composite specimens after (**a**) exposure to *Coniophora puteana*; (**b**) mycelium removal.



(**a**) (**b**)

Figure 2. Millet husk-reinforced composite specimens after (**a**) exposure to *Coniophora puteana*; (**b**) mycelium removal.

The mass loss test results of the composites with oat filler obtained after 14 days of leaching in water, followed by 4 months of exposure to fungi at 22 °C and at 70% humidity, are identical for both methods of mycelium removal (Figure 3). The mass loss for this material is 0.3% compared to the initial state, demonstrating stable results regardless of the cleaning method. The composites reinforced with millet husks show a higher loss in mass and a larger difference between the two methods of mycelium removal (0.8% following cleaning with a brush and 2.3% following cleaning with water). The microstructure of the millet filler composite is irregular with large pores [20] that may cause fragments of filler to be pushed out by water under pressure. Soft brushing protects the specimens from this process, but also may affect the mass loss.

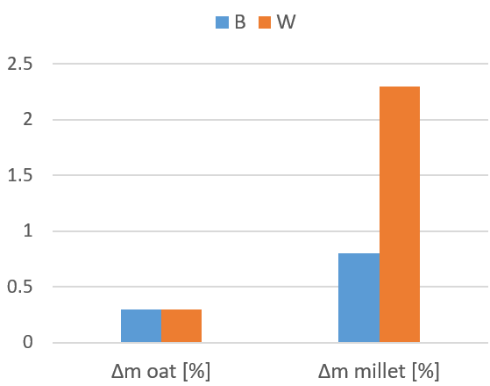
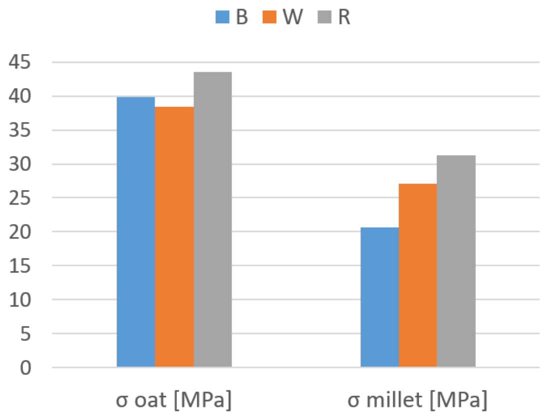


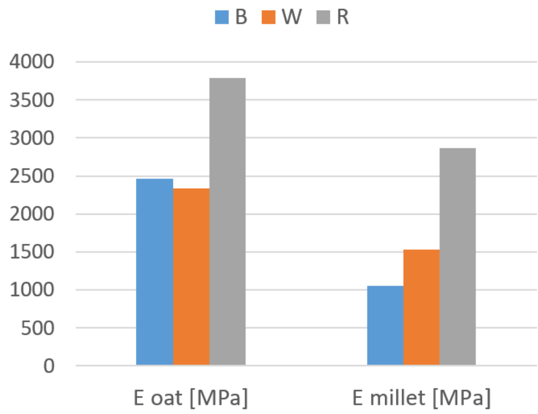
Figure 3. Mass loss test results of oat and millet husk-reinforced composites after exposure to Coniophora puteana and manual mycelium removal with a soft brush (B) and with water under pressure (W). The results are expressed in %.

Bending Strength and Modulus of Elasticity after Exposure to Coniophora Puteana

The test results of the specimens' mechanical properties show that in the initial state, the composites reinforced with oat and millet husk had a bending strength of 44 MPa and 31 MPa, respectively, while the specimens' modulus of elasticity was 3790 MPa and 2870 MPa, respectively (Figure 4). It can be noticed that the properties of the composite with oat husks are more useful than those of the composite with millet husks in the initial state. The reason for this may be the influence of the shape and wetting degree of the filler. Oat fillers have an elongated shape, which is advantageous for their mechanical properties. In general, millet fillers have irregular shapes [20]. In addition, fillers naturally have different moisture absorption capacities.



(**a**)



(**b**)

Figure 4. Test results of (**a**) bending strength and (**b**) modulus of elasticity of oat and millet husk-reinforced composites after exposure to *Coniophora puteana* and manual mycelium removal with a soft brush (B) and with water under pressure (W). Test results of reference specimens in initial state (R) have been also presented. The results are expressed in MPa.

The test results of the composites obtained after 14 days of soaking in water, followed by 4 months of exposure to fungi at 22 °C and at 70% humidity, show that these conditions reduce their flexural strength and modulus of elasticity, compared to the reference material. This was also reported in other papers [8]. The method of cleaning had a small effect on the oat husk composite. After exposure to fungus and cleaning with a brush, the oat husk composite had a bending strength of 40 MPa and a modulus of elasticity of 2460 MPa, while after water cleaning, the bending strength of this composite was 38 MPa and a modulus of elasticity was 2340 MPa (Figure 4).

On the other hand, the effect of the mycelium removal method on the composite with millet filler was significant in comparison to the oat filler composite. After exposure to *Coniophora puteana* and cleaning with a brush, the composite had a bending strength of 21 MPa and a modulus of elasticity of 1050 MPa, while the composite cleaned with water had a bending strength of 27 MPa and a modulus of elasticity of 1530 MPa (Figure 4). In relation to the mass loss test of the composite with millet husks, the reason for the difference may be the lack of fillers in the pores, as well as the higher efficiency of the mycelium removal method with water.

5. Conclusions

Based on the results of the oat husk-reinforced composite tests, the following conclusions can be drawn:

- The mass loss regardless of the mycelium removal method was 0.3% compared to the initial state.
- Brushed specimens showed slightly higher flexural strength and a higher modulus of elasticity than the specimens cleaned with water under pressure.

- The method of mycelium removal does not significantly affect the physical and mechanical properties of the oat filler composite.

The conclusions based on the results of the millet husk-reinforced composite are as follows:

- The mass loss of the specimens varies depending on the method of mycelium removal. Cleaning with water under pressure resulted in the highest mass loss at 2.3%, while the brush-cleaned specimens had a mass loss of 0.8%.
- The results of the flexural strength and modulus of elasticity tests also confirm the loss in mass of the specimens. The millet husk-composite after water cleaning showed higher results, including after brushing, compared to the oat husk composite.
- The differences in the results with different cleaning methods may be caused by the microstructure of millet husk fillers.

It was concluded that the effects of mycelium removal methods depend on the type of filler. It is still difficult to determine which was the most effective method of cleaning the specimens due to the constrained conditions and few composite fillers used. Further research including extended parameters that affect the mechanical and physical properties of the natural filler-reinforced composites is planned.

Author Contributions: Conceptualisation, M.G.; methodology, M.G.; formal analysis, E.S.; investigation, M.G.; writing—original draft preparation, M.G.; writing—review and editing, E.S.; visualization, M.G. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Ministry of Education and Science as part of the project LN-002.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data presented in this study are available on request from the corresponding author.

Acknowledgments: The publication cost of this paper was covered with funds provided by the Polish National Agency for Academic Exchange (NAWA) ("MATBUD'2023—Developing international scientific cooperation in the field of building materials engineering" BPI/WTP/2021/1/00002, MATBUD'2023).

Conflicts of Interest: The authors declare no conflict of interest. The funders had no role in the study's design, in the collection, analyses, or interpretation of data, in the writing of the manuscript, or in the decision to publish the results.

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