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Resistance of construction profiles made of polymer composites reinforced with cereal husks to the fungi

Odporność profili budowlanych z kompozytów polimerowych zbrojonych łuskami zbóż na grzyby domowe

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Abstract. Resistance of profiles made of PVC composites with pulverised oat, millet, and rice husk filler to fungi was analysed. Products containing oat and rice husks revealed similar susceptibility to the action of *Coniophora puteana, Gloeophyllum trabeum* and *Coriolus versicolor*, which was lower than the susceptibility of the composite containing millet husks. *Coniophora puteana* demonstrated the greatest mycelium development extent, changing the profiles' surface morphology. Exposure to fungi in a wet environment decreased the flexural strength and flexural modulus, which was the highest for the composite reinforced with millet husks. The influence of a wet environment was of crucial significance. Microorganisms changed the flexural properties only slightly.

Keywords: polymer composites; oat; millet; fungi; microstructure; flexural properties.

Streszczenie. Analizowano odporność profili z kompozytów PVC z napełniaczem z pulweryzowanych łusek owsa, prosa i ryżu na działanie na grzybów domowych. Wyroby z łuskami owsa i ryżu wykazały porównywalną podatność na działanie *Coniophora puteana, Gloeophyllum trabeum* oraz *Coriolus versicolor*, ale mniejszą niż kompozyt z łuskami prosa. *Coniophora puteana* wykazał największy stopień rozwoju grzybni i zmienił morfologię powierzchni profili. Ekspozycja na działanie grzybów w środowisku mokrym skutkowała zmniejszeniem wytrzymałości na zginanie i modułu sprężystości, największym w przypadku kompozytu zbrojonego łuskami prosa. Kluczowy był wpływ samego środowiska mokrego. Mikroorganizmy nieznacznie zmieniły właściwości przy zginaniu.

Słowa kluczowe: kompozyty polimerowe; łuski; owies; proso; grzyby domowe; mikrostruktura; właściwości przy zginaniu.

Natural fibre polymer composites (NFPC) have been used in construction for many years, mainly for producing facade profiles, outdoor floors, window, and door joinery, platforms and landscape architecture element [1]. The dominant type of NFPCs is that with a thermoplastic polymer matrix filled with wood fibres or pulverised cultivated plants husks, stems and leaves [2]. The most popular construction products include those with PVC matrix and wood flour or rice husk fillers [3], but attempts are made to introduce composites with other cereal husk fillers. Because plant fibres are lignocellulose structures whose chemical composition differs depending on the plant species, using another filler may change the composite properties [2, 4]. The fitness for construction applications of profiles made with the new filler shall be evaluated according to the rules applicable for construction products, according to the usability criterion, referring to a set of critical features for the given application [5], from the point of view of the product's impact on the building structure's fulfilling the seven essential requirements set out in Regulation No. 305/2011 [6].

The functional properties of products made of polymer composites depend primarily on the interactions at the matrix and filler phase border [2,3]. Ensuring proper interaction is a challenge in the conditions of construction profiles' use. The hydrophilic nature of plant fibres makes them swell easily once exposed to water, leading to the cracking of the hydrophobic polymer matrix [3, 7]. The interaction between the fibres and the polymer deteriorates, reducing the stress transfer capacity at the phase border, which is accompanied by a decrease in the mechanical parameters [8].

This paper focuses on the issue of profile degradation under the influence of fungi in a wet environment, which is indispensable for their development. The results of previous studies indicate that NFPCs are particularly susceptible to the *Basidiomycetes* class [7, 8] belonging to the *Agaricomycotina* subtype [9] in the current taxonomy. The polymer matrix is characterised by negligible susceptibility to biodecomposition [10, 11]. The composite's resistance to fungi depends on the filler type and quantity, the size of particles, and the extent of their dispersion [12, 13]. Composites reinforced with low-absorbability fibres are less sensitive to fungi than those containing highly absorptive fibres [14]. The fibres in NFPC were found to reach up to 70% humidity, which makes optimum conditions for microorganisms' growth at adequate

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temperature and pH [15]. Lignin and cellulose are the food for fungi. White--rot fungi, e.g. *Coriolus versicolor*, attack lignin, while brown-rot fungi, e.g. *Coniophora puteana*, decompose cellulose [16]. Proper dispersion of fibres in the matrix promotes bioresistance. It fosters good interphase adhesion and reduces voids [1, 7] which play the role of channels for fungi migration through the material, facilitating the transport of enzymes [17]. NFPC's resistance to microorganisms decreases as the filler's amount increases [18]. Previous studies on NFPC biodegradation reported diversified susceptibility to the action of different fungi strains [14, 15, 19]. The most intensive growth was observed for *Trametes versicolor* and *Coniophora puteana*, at a simultaneous weight loss not exceeding 5% and with no significant change in the flexural modulus [8]. Nonetheless, cases are known of flexural strength reduced to 30%, elasticity to 40% [20 – 23], and impact strength to 16% [20].

This paper aimed to determine the susceptibility of construction profiles made of composites with PVC matrix and pulverised Polish cereal – oat and millet – husks filler to fungi, which had not been analysed before. A standard composite reinforced with rice husks was tested for comparison. All solutions were exposed to *Coniophora puteana, Gloeophyllum trabeum*, and *Coriolus versicolor*. Mycelium growth, composite surface morphology, flexural strength, and modulus were evaluated.

Testing methods

The tests were carried out on multi-chambered profiles intended for outdoor floors, extruded in industrial conditions from a PVC matrix composite with an addition of $CaCO_3$ (50 phr – 50 parts per 100 parts per weight of the mix) and a filler made of pulverised oat (30 phr), millet (30 phr) or rice (60 phr) husks. The profiles' surfaces were mechanically brushed, which is a standard procedure for construction products. No corrugation was made.

From the central part of the chambers, five series of samples were cut out, sized 80 x 10 x 5 mm, each containing five pieces. The first series of samples were seasoned in laboratory conditions (temp. 23±2°C, relative humidity 50±5%). They were the reference samples.

The second series was subject to washing out according to EN 84 [24], which involved immersing the samples in water for fourteen days and replacing the water nine times. A nutrient medium was prepared in Kolle-type culture flasks consisting of 40 g of malt extract, 35 g of agar, and 1,000 ml of water. The nutrient medium was sterilised in an autoclave at 121°C and 1 atm pressure. The samples were placed in the culture flasks and kept for four months in a culture chamber (temperature: 22±1°C, relative humidity: 70±5%). The samples were used to determine the impact of a wet environment on the tested material.

The samples from the other three series were exposed to fungi after washing out, as described above. The medium was inoculated with *Coniophora puteana* (Schumacher ex Fries) BAM Ebw. 15, *Gloeophyllum trabeum* (Persoon ex Fries) BAM Ebw. 109 and *Coriolus versicolor* (Linnaeus) hyphae. The culture flasks were incubated until entirely covered by mycelium. The composite samples were placed on the mycelium so that their usable surface remained in direct contact with it; then, they were taken to a cultivation chamber (temperature: 22±1°C, relative humidity: 70±5%) for four months. The activity of fungi was verified according to ENV 12038 [25], reaching a weight loss of much over 20%. After the exposure, the samples were manually cleaned of mycelium with a soft brush.

The extent of mycelium growth on the samples was visually assessed right after the exposure. The samples with the most significant mycelium growth were selected for SEM examination. They were cleaned of mycelium using water under pressure and dried for seven days at 40±2°C. The microstructure analysis was carried out with Sigma 500 VP scanning electron microscope with a cold cathode field emission, which enables reaching a high resolution at a low accelerating voltage. The tests were carried out at a 10 KeV accelerating voltage of the excitation electron bundle, using the SE detector for gold-sprayed samples. The applied magnification was 200x.

The samples from all series were also subject to flexural strength σ and flexural modulus E testing. The test was done with a strength testing machine class 1, according to EN ISO 178 [26]. The supports were spaced at 64 mm. The samples were bent with the usable side towards the supports to subject the space exposed to fungi to the impact of tensile stress. The load was applied at 2 mm/min. until damage. During bending, the load-deflection curve was recorded in the linear-elastic range, including the values of force and deflection corresponding to $\varepsilon_1 = 0.0005$ and $\varepsilon_2 = 0.0025$ strain. Based on the force values recorded at ε_1 and ε_2, typical stress values were determined, and the flexural modulus was calculated.

The test results for the samples exposed to a wet environment were compared to those obtained for the original samples to determine, according to (1), the environment's impact on the tested material. The result was expressed in %.

$$\Delta\sigma_m = [(\sigma_m - \sigma_i)/\sigma_i)] \bullet 100$$
$$\Delta E_m = [(E_m - E_i)/E_i)] \bullet 100 \quad (1)$$

where:

σ_m, E_m – flexural strength and flexural modulus in wet condition [MPa];

σ_i, E_i – flexural strength and flexural modulus in the original condition [MPa].

The test results obtained for the samples exposed to fungi in a wet environment were compared to those obtained for the original samples, which enabled determining, according to (2), the exposure's impact on the tested material. The result was expressed in %.

$$\Delta\sigma_d = [(\sigma_d - \sigma_i)/\sigma_i)] \bullet 100$$
$$\Delta E_d = [(E_d - E_i)/E_i)] \bullet 100 \quad (2)$$

where:

σ_d, E_d – flexural strength and flexural modulus after exposure to fungi in a wet environment [MPa];

σ_i, E_i – flexural strength and flexural modulus in the original condition [MPa].

In order to determine, according to the guidelines [27], the impact of fungi on the tested material, the difference was calculated – according to (3) – between the change caused by exposure to fungi in a wet environment and the change caused only by the wet environment.

$$\Delta\sigma_f = \Delta\sigma_d - \Delta\sigma_m \quad \Delta E_f = \Delta E_d - \Delta E_m \qquad (3)$$

where:
$\Delta\sigma_m$, ΔE_m – according to (1);
$\Delta\sigma_d$, ΔE_d – according to (2).

Results

The visual assessment helped determine the diversified development extent of each strain's mycelium on the given composite and the diversified susceptibility of the tested composites to the given strain. No growth was observed of either *Coriolus versicolor* or *Gloeophyllum trabeumon* the composite samples with oat husks (Photos 1a, 1b). *Coniophora puteana* developed to a medium extent (Photo 1c), similarly to *Coriolus versicolor* on the composite samples with millet husks (Photo 2a). The composite with millet husk filler revealed low susceptibility to *Gloeophyllum trabeum* growth whose mycelium developed only slightly (Photo 2b), and high susceptibility to *Coniophora puteana* whose mycelium developed well (Photo 2c). The samples of composite reinforced with rice husks did not reveal the growth of either *Coriolus versicolor* or *Gloeophyllum trabeum* (Photos 3a, 3b), while *Coniophora puteana* developed in a moderate extent (Photo 3c). Therefore, from the mycelium development extent perspective it can be concluded that *Coniophora puteana* exerted the most significant impact on the tested products, which corresponds to the results of studies on biodegradation of composites containing wood flour [11, 28]. The composite with oat husks was characterised by resistance tofungi similar to that of the composite containing rice husks. The

Photo 1. Samples of composite reinforced with oat husks after exposure to: a) *Coriolus versicolor*; b) *Gloeophyllum trabeum*; c) *Coniophora puteana*
Fot. 1. Próbki kompozytu zbrojonego łuskami owsa po ekspozycji na: a) Coriolus versicolor; b) Gloeophyllum trabeum; c) Coniophora puteana

Photo 2. Samples of composite reinforced with millet husks after exposure to: a) *Coriolus versicolor*; b) *Gloeophyllum trabeum*; c) *Coniophora puteana*
Fot. 2. Próbki kompozytu zbrojonego łuskami prosa po ekspozycji na: a) Coriolus versicolor; b) Gloeophyllum trabeum; c) Coniophora puteana

Photo 3. Samples of composite reinforced with rice husks after exposure to: a) *Coriolus versicolor*; b) *Gloeophyllum trabeum*; c) *Coniophora puteana*
Fot. 3. Próbki kompozytu zbrojonego łuskami ryżu po ekspozycji na: a) Coriolus versicolor; b) Gloeophyllum trabeum; c) Coniophora puteana

product reinforced with millet husks revealed higher susceptibility to the set exposures than all other tested solutions.

The surface morphology of the samples exposed to *Coniophora puteana* was analysed with the SEM method. Marked elongated shallow pitches with quite a regular pattern, resulting from machining, were observed on all tested composites (Photos 4a-4c). The composite sample with oat husks revealed an exposed natural fibre fragment, which is well wetted by the matrix, and an apparent elongated crater in the matrix, suggesting the loss of its remaining part (Photo 4a). The analysis of the surface morphology of the composite with millet husks revealed degradation in a form of matrix voids (Photo 4b). Their shapes suggest that before the exposure to *Coniophora puteana* the matrix surrounded the plant filler particles in these places [2]. The microscopic image of the surface of the composite with rice husks was similar to the one for the composite with oat husks. Moreover, a higher number of smaller plant particles well dispersed in the matrix were observed (Photo 4c), adding to the composite's advantage [3, 13]. In this composite, some particles were subject to biodegradation, which is demonstrated by small craters with fairly regular shapes [11]. Rice husk particles exposed during brushing were observed; they were well-surrounded by the matrix after the exposure.

The analysis of mechanical characteristics test results reveals that in their original condition, the composites containing oat, millet, and rice husks were characterised by the flexural strength of 43 MPa, 31 MPa, and 44 MPa, respectively (Fig. 1), while the flexural modulus amounted to 3780 MPa, 2860 MPa, and 3490 MPa, respectively (Fig. 2). The characteristics are similar to those obtained for construction profiles made of composites reinforced with rice husks [23, 29, 30], but lower than those reported for other NFPCs [31]. It shall be pointed out that although the properties of the composite reinforced with oat husks were similar to those obtained for the reference product containing risk husks, the parameters of the

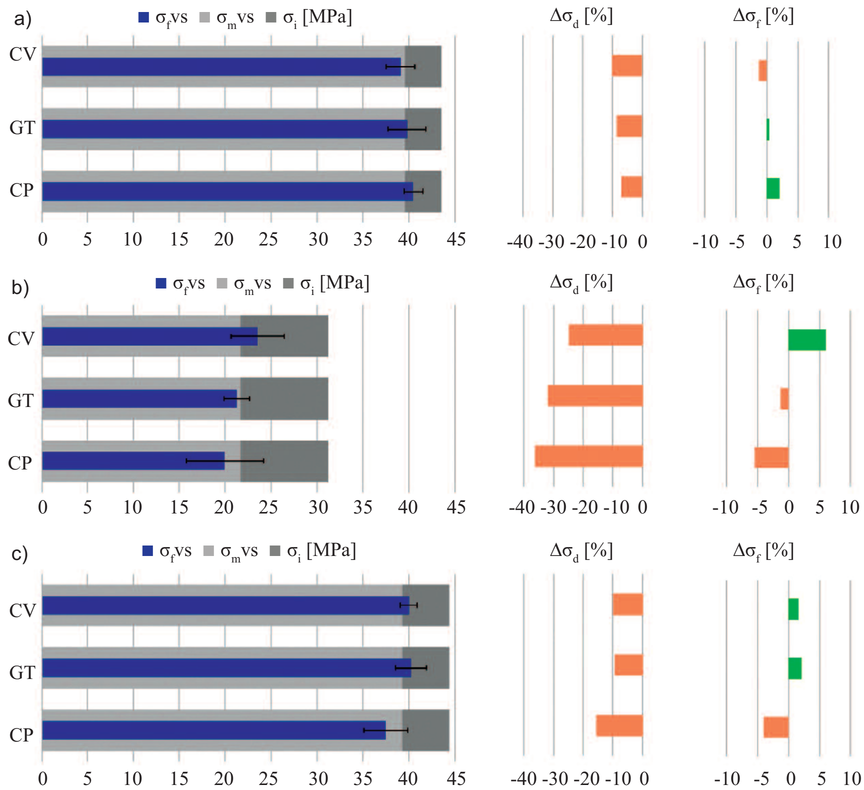

Fig. 1. Results of flexibility strength tests on composites reinforced with: a) oat; b) millet; c) rice husks after exposure to *Coriolus versicolor* (CV), *Gloeophyllum trabeum* (GT) and *Coniophora puteana* (CP) fungi strains in wet environment (σ_f) against the flexibility strength results after exposure to wet environment (σ_m) and in the original condition (σ_i). The extent is presented of the changes caused by exposure to fungi in a wet environment ($\Delta\sigma_d$) and fungi after considering the environment's impact ($\Delta\sigma_f$). The error bars illustrate the standard deviation (n = 10)

Rys. 1. Wyniki badań wytrzymałości na zginanie kompozytów zbrojonych łuskami: a) owsa; b) prosa; c) ryżu po ekspozycji na działanie w środowisku mokrym grzybów (σ_f) szczepu Coriolus versicolor (CV), Gloeophyllum trabeum (GT) oraz Coniophora puteana (CP), na tle wytrzymałości na zginanie uzyskanej po działaniu środowiska mokrego (σ_m) oraz w stanie wyjściowym (σ_i). Przedstawiono także wielkość zmian wywołanych ekspozycją na działanie grzybów w środowisku mokrym ($\Delta\sigma_d$) oraz samych grzybów, po uwzględnieniu poprawki z tytułu wpływu tego środowiska ($\Delta\sigma_f$). Słupki błędów obrazują odchylenie standardowe (n = 10)

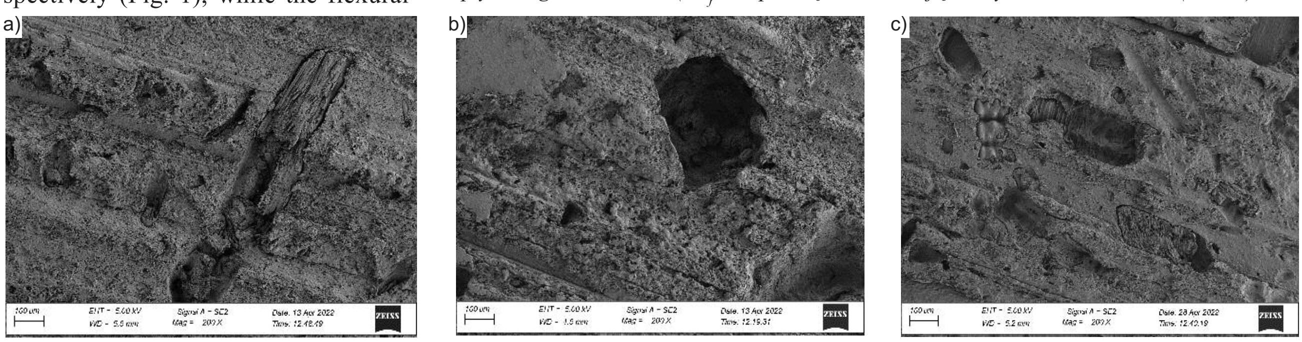

Photo 4. Microstructure of the husk-reinforced composite's surface: a) oat; b) millet; c) rice following the exposure to *Coniophora puteana*, 200*x* magnification

Fot. 4. Mikrostruktura powierzchni kompozytu zbrojonego łuskami: a) owsa; b) prosa; c) ryżu po ekspozycji na Coniophora puteana, powiększenie 200x first composite were much lower. The above can result from the filler's shape and wetting extent [4]. The SEM analysis revealed that millet particles were elongated (Photo 4a), which is beneficial for mechanical properties [22]. The craters in the matrix of the millet composite were slenderer (Photo 4b).

The tested composites revealed diversified susceptibility to wet environments. After fourteen days of washing out in the water and four months of exposure on a clean medium at 22±1°C and 70±5% RH, the flexural strength decreased to 40 MPa, 22 MPa, and 39 MPa (Fig. 1), respectively for the composites with oat, millet and rice husks, and the flexural modulus dropped to 2490 MPa, 1100 MPa and 2810 MPa (Fig. 2). Such significant drops can result from exposing hydrophilic lignocellulose fibres during brushing, which promotes their swelling and deteriorates adhesion on the phase border [1, 22]. The wet environment's impact was particularly evident for the composite with millet husks, where the strength decreased by 30% and the flexural modulus went down by 60%. It suggests that millet husks are more susceptible to water than oat and rice husks [2], which corresponds to the results of composites water absorption tests amounting to 2% for the product reinforced with millet husks, 0.5% for the composite containing millet husks and 1% for that with rice husks [32].

The analysis of the results obtained after fourteen days of washing out in the water and four months of exposure to fungi at 22±1°C and 70±5% RH reveals that the introduction of microorganisms either did not deteriorate or only slightly reduced the flexural strength and flexural modulus compared to the values reported after the exposure in a wet environment. Similar behaviour was observed in other studies [28]. The decrease in the flexural strength and flexural modulus, expressed as $\Delta\sigma_f$ and ΔE_f, calculated according to (3), considering the correction for the wet environment according to [27], did not exceed 5%. The only exception is the ΔE_f value of 12% for the composite with rice husks. The exposure to *Coriolus versicolor* resulted in the flexural strength and flexural modulus reduced

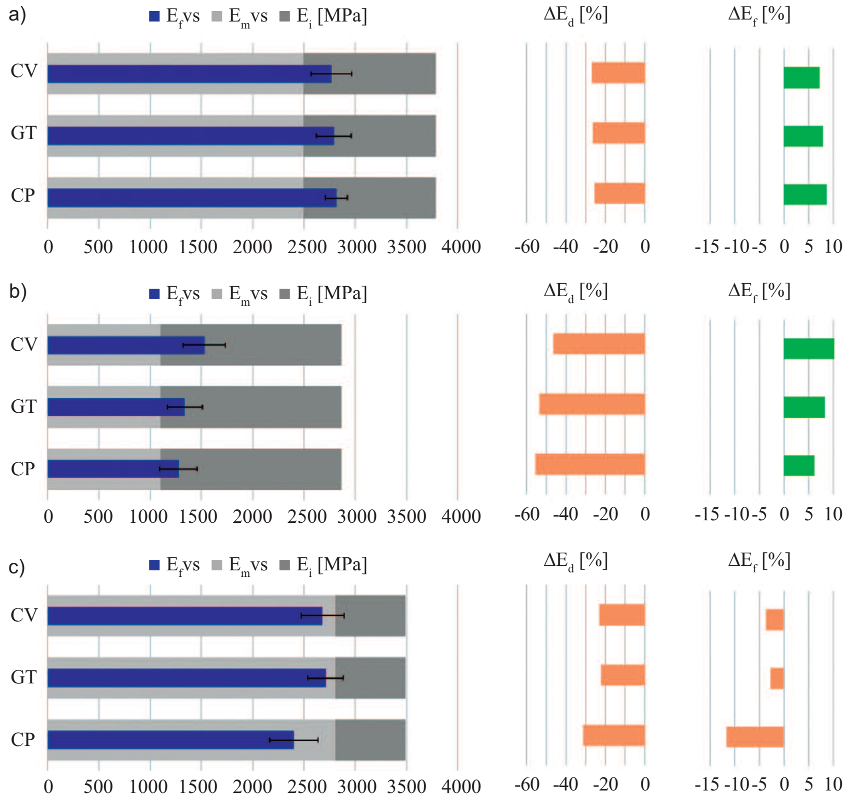

Fig. 2. Results of flexibility modulus tests on composites reinforced with: a) oat; b) millet; c) rice after exposure to the following fungi in a wet environment (E_f): *Coriolus versicolor* (CV), *Gloeophyllum trabeum* (GT) and *Coniophora puteana* (CP) against the results after exposure to the wet environment (E_m) and in the original condition (E_i). The extent is presented of the changes caused by exposure to fungi in a wet environment (ΔE_d) and fungi after considering the environment's impact ΔE_f). The error bars illustrate the standard deviation (n = 10)

Rys. 2. Wyniki badań modułu sprężystości przy zginaniu kompozytów zbrojonych łuskami: a) owsa; b) prosa; c) ryżu po ekspozycji na działanie w środowisku mokrym grzybów (E_f): Coriolus versicolor (CV), Gloeophyllum trabeum (GT) oraz Coniophora puteana (CP), na tle wyników po działaniu środowiska mokrego (E_m) oraz w stanie wyjściowym (E_i). Przedstawiono także wielkość zmian wywołanych ekspozycją na działanie grzybóww środowisku mokrym (ΔE_d) oraz grzybów, po uwzględnieniu poprawki z tytułu wpływu tego środowiska (ΔE_f). Słupki błędów obrazują odchylenie standardowe (n = 10)

only for the composite reinforced with oat husks. Following the exposure to *Gloeophyllum trabeum*, a decrease in the flexural strength was reported for the composite with millet husks and a drop in the flexural modulus for the composite with rice husks. After the exposure to *Coniophora puteana*, a decrease in the flexural strength was observed for the composites with millet and rice husks and a drop in the flexural modulus for the composite reinforced with rice husks.

It shall be highlighted that the total drops in the flexural strength and flexural modulus observed after exposure to fungi in a wet environment, expressed as $\Delta\sigma_d$ and ΔE_d, respectively, and calculated according to (2) without considering the correction for the wet environment, were significantly higher. For the composite with oat husks, the $\Delta\sigma_d$ ranged from 7% to 10%, for the composite with rice husks – from 9% to 16%, and for the one with millet husks – from 24% to 36%. The ΔE_d values amounted to 26% for the composite reinforced with oat husks, and range between 22% and 31% for the composite reinforced with rice husks and from 47% to 55% for the one containing millet husks. The results above are similar to those obtained for other NFPCs [12, 19, 21]. The highest $\Delta\sigma_d$ and ΔE_f values were reported – except for the composite reinforced with oat husks – after the exposure to *Coniophora puteana*, similarly to [28]. The above corresponds to the observation results of mycelium development extent (Fig. 1-3).

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Conclusions

The analysis of the test results reveals that the tested construction profiles made of PVC composite reinforced with pulverised oat husks were characterised by resistance to fungi similar to that of the reference standard PVC composite with rice husks. For both solutions above, a similar extent of *Coniophora puteana, Gloeophyllum trabeum*, and *Coriolus versicolor* growth was observed. The surface morphology of both composites, evaluated after exposure to *Coniophora puteana*, which revealed the highest extent of mycelium growth, can also be considered similar. Exposing the profiles to a wet environment resulted in the flexural strength of the composite reinforced with oat husks being reduced by 9% while the value for the product with rice husks amounted to 12%. The flexural modulus dropped by 34% and 20%, respectively. The introduction of fungi did not result in any further significant reduction in the analysed mechanical properties. As a result of exposure to microorganisms in a wet environment, the flexural strength of the composite with oat husks decreased up to 10% and the flexural modulus dropped up to 26%, while for the composite with rice husks, the values were up to 16% and 31%, respectively.

The profiles reinforced with millet husks turned out more susceptible to fungi. Much significant mycelium growth was observed especially for *Coniophora puteana* and *Coriolus versicolor*. The analysis of surface morphology after exposure to *Coniophora puteana* revealed voids in the matrix in the spaces where the filler had degraded. Following the exposure to fungi in a wet environment, the flexural strength dropped to 36% and the flexural modulus decreased to 55%. It shall be pointed out that the impact of the wet environment was of key significance for the reported drops. The introduction of fungi changed the analysed mechanical properties only slightly.

Further studies are planned on the susceptibility of composites, with oat and millet husks to fungi, considering a longer exposure period than the one implemented in this study.

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