Annals of Warsaw University of Life Sciences – SGGW
Forestry and Wood Technology № 121. 2023: 11-20
(Ann. WULS - SGGW, For. and Wood Technol. 121, 2023: 11-20)
Received: 07.09.22 / Accepted: 21.03.23 / Published: 29.03.23

Selected flexural and hygroscopic properties of waste wood dust - polylactic acid biocomposite for 3D printing.

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Abstract: *Chosen flexural and hygroscopic properties of waste wood dust - polylactic acid biocomposite for 3D printing.* The study shows chosen flexural and hygroscopic properties of PLA-waste wood dust biocomposite intended for use in 3D printing. Materials were mixed in 3 variants, differentiated by weight content of waste wood dust – 0%, 10%, 20% and 30% and extruded in a two-stage process using an extruder of original design. Variant without waste wood dusts was prepared as well. That filaments were then used to create samples using a 3D printer. For the printed samples chosen properties were tested i.e. MOE, MOR, water absorption and thickness swelling. It was found that waste wood dust does not significantly affect MOR and MOE. It was shown that it's content negatively impacts the water absorption and thickness swelling. It was demonstrated that the tested material can successfully be used in 3D printing.

Keywords: WPC, Waste wood dust, 3D printing, PLA

INTRODUCTION

Wood industry in Poland annually generates 8 million m^3 of wood waste (Szostak et al. 2004). Larger fractions, such as chunk wood, wood chips and shavings are used in production of paper and wood-based materials like particleboards or fiberboards (Wach and Kołacz 2003). Bark is widely used in plant cultivation (Gorzelak 1998). Waste wood dust constitutes 5% of industrial wood waste (Kajda-Szcześniak 2013), which equals to 400 thousand m^3 annually. Constant development of wood technology, aimed at higher productivity and surface quality in pair with wider use of fiberboards is expected to increase the amount of waste wood dust generated during industrial wood processing (Wieloch and Wilkowski 2015). High specific surface of wood dust resulting in a need of higher amounts of glue and glue conglomeration (Drouet 1992), as well as surface aging resulting in lower glue adhesion (Zenkteler 1984) both contribute to difficulties in the use of waste wood dust in the wood industry. Topic of waste wood dust is widely depicted in scientific publications, but mainly in terms of health and safety concerns (Fangrat et al. 1986; Ogiołda 1998; Wolny 2010) or its use as a fuel (Golec et al. 2007; Werle 2021). Outside the wood industry possibilities of waste wood dust use were researched in plant cultivation (Domoradzki and Korpal 2005; Domoradzki, Kaniewska and Weiner 2012) and in water treatment (Mashkoor et al. 2018). Despite positive results this use cases have not yet been implemented on industrial scale. Growing interest in use of waste wood dust in WPCs can also be noticed (Żelaziński et al. 2019). Gozdecki et al. (2010) indicates, that properties of these composites are not significantly worse than those of WPCs made with wood flour created specifically for this purpose.

Market share of WPC filaments intended for 3D printing, both in professional and hobby spaces is steadily growing. Most of these materials contain about 30% of wood, not seldom recycled. Usually content of wood and selected physio-mechanical properties are the only information available regarding the commercially available WPC filaments. Manufacturers rarely share information regarding additives and processes used during manufacturing of these materials (colorFabb 2022). The most popular polymer both in WPC filaments and 3D printer filaments in general is PLA (Stříteský 2020). PLA is manufactured from renewable resources such as corn, beets or sugarcane. It's mechanical properties are similar to those of petrol-derived polymers such as PS or PE (Balart et al. 2018). It's also possible to manufacture PLA from food waste, limiting competition between polymer and food manufacturers (Kwan 2018). PLA is fully biodegradable in specific conditions, but currently there is no infrastructure nor legislation allowing management of waste PLA (Tokiwa and Calabia 2006). Biodegradability and sustainability of PLA are important factors contributing to this polymer's extensive use, but the most important factor is its ease of use. Due to low thermal expansion PLA doesn't require an enclosed 3D printer. Lower printing temperatures reduce degradation and VOC emissions compared to ABS and lower the operating costs (Wojtyła, Klama and Baran 2017).

Research of waste and recycled wood (Pringle et al. 2018), as well as waste wood dust and petrol-derived polymers based WPC filament (Löschke et al. 2019) have been performed, yet, there is lack of publications regarding waste wood dust's impact on properties of PLA based WPCs intended for 3D printing.

MATERIALS

Materials used during the study were PLA granulate intended for extrusion molding – Ingeo Biopolymer 2003D, and waste wood dust obtained from the filter of a production facility, where 90% of lignocellulosic materials, such as plywood, raw and finished particleboards, fibreboards, and 10% of solid wood were processed. This processing consisted in 60% of sawing, in 30% of routing and in 10% of sanding.

Waste wood dust was sorted to particles passing through a 0.16mm sieve and mixed with PLA granulate in the following weight ratios:

- for variant 1 – 10:90,
- for variant 2 – 20:80,
- for variant 3 – 30:70.

Variant 0, consisting of solely PLA granulate was also prepared. These mixes were then extruded using an extruder of original design (Figure 1.) in a two stage process. First stage consisted of extruding the prepared materials through a 3mm diameter nozzle and cutting them into a WPC granulate. The second stage consisted of extruding the granulate obtained during first stage through a 1.5mm diameter nozzle. The extruder was fitted with a single, 20mm diameter screw and it's length to diameter ratio (L/D) was 13.5:1. Extruder's temperature during both stages was kept at 145^{o}C. Filament obtained this way was then used to print test samples measuring 80mm x 10mm x 4mm using a Voron 2.4 3D printer equipped with Phaetus Voron Dragon HF hotend and Clockwork extruder. Samples' models were prepared in Autodesk Fusion 360 and then converted to g-code using Ultimaker Cura 4.13. Print settings are presented in Table 1. Not mentioned settings were left default, as provided by software manufacturer.

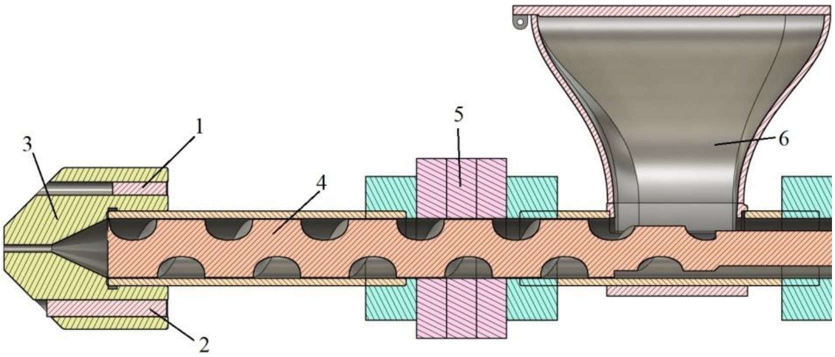

Figure 1. Extruder diagram, where: 1 – thermocouple, 2 – heating element, 3 – head, 4 – screw, 5 – radiator, 6 – hopper

Table 1. Print settings used in the study

Parameter	Value
Nozzle material	Steel
Nozzle diameter	0.6mm
Layer height	0.3mm
Wall line width	1.2mm
Top and bottom thickness	1.2mm
Infill	30%
Nozzle temperature	200°C
Bed temperature	60°C
Print cooling	50%

MOR and MOE tests were performed according to PN-EN ISO 178:2019. Water absorption and thickness swelling tests were performed according to PN-EN 317:1999. These methods were chosen according to PN-EN 15534-1+A1:2017-12. 7 samples of each variant were tested for each of the aforementioned tests. Due to unequal variances between variants Kruskal-Wallis H test with post-hoc Dunn test were used to determine significance of differences between results (Kruskal and Wallis 1952; Dunn 1964). Confidence level of 95% was chosen for these tests.

RESULTS

No significant differences were found between variants in both MOR (Figure 2.) and MOE (Figure 3.) tests. For MOR test the Kruskal – Wallis p test value equals 0.546 and for MOE test it equals 0.065. This lack of significant differences between variants of different wood content is uncharacteristic for WPC. According to Jian et al.(2022) both MOR and MOE are higher for WPCs with a higher wood content, up to 50% - 65% depending on their composition. Vastly different breakage of samples from variant 0 and variants containing waste wood dust were also observed (Figure 4.). Samples consisting of solely PLA broke between layers while maintaining integrity of each layer, while the samples containing waste wood dust broke in the middle with rupture of the layers in a single cross-section. Both this behavior and lack of significant differences in MOR and MOE tests might be a result of weak adhesion between PLA and waste wood dust. Use of a coupling agent might improve adhesion and, as a result, improve flexural properties of tested composite (Włodarczyk-Fligier, Polok-Rubiniec and Chmielnicki 2018).

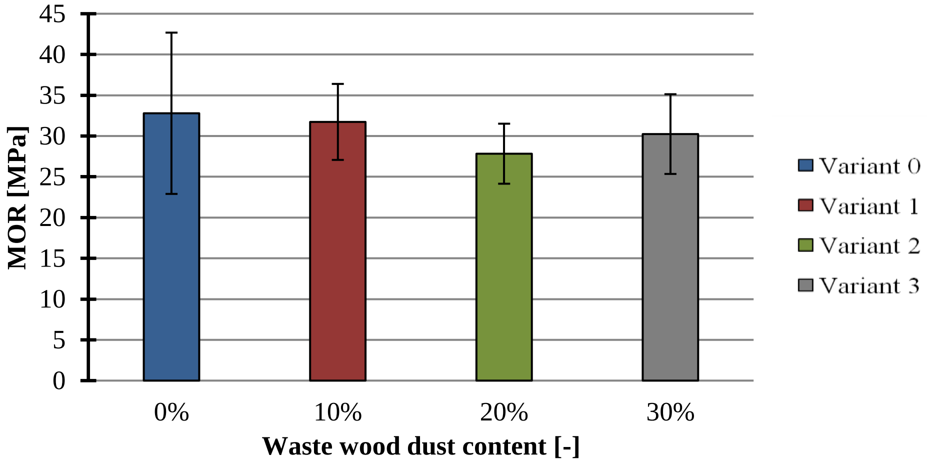

Figure 2. Average modulus of rupture for each tested variant

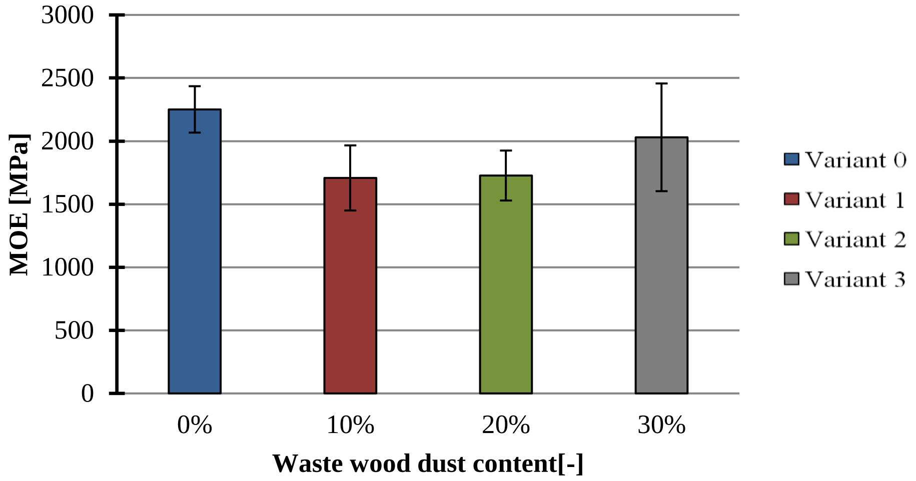

Figure 3. Average modulus of elasticity for each tested variant

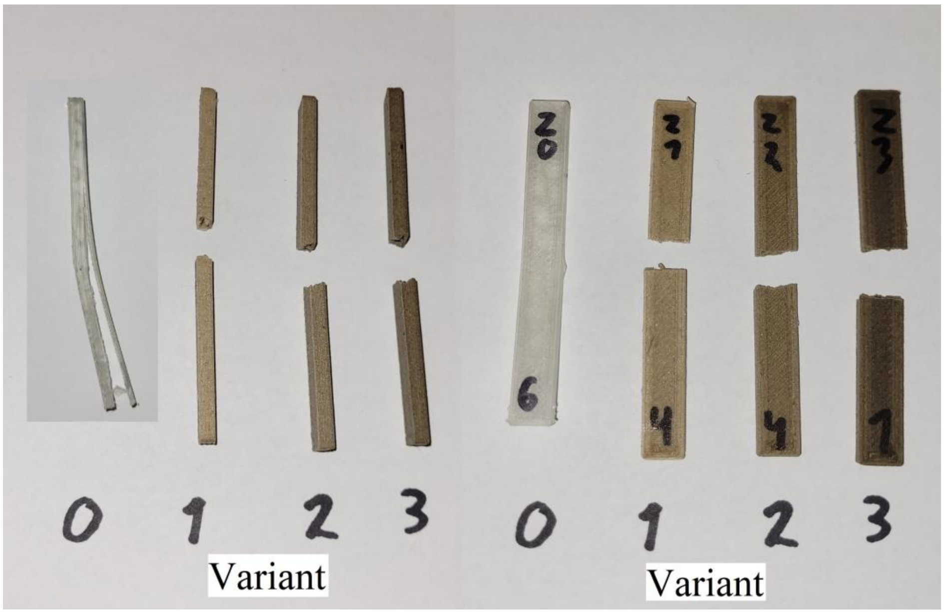

Figure 4. Samples characterisic for each variant after MOR test

Presented in Figure 5. are average results for water absorption test after 2h of immersion. Average for variant 0 (0.57%) was significantly lower than averages for variant 1 (2.39%), variant 2 (4.16) and variant 3 (3.58%). Differences between variants containing waste wood dust were found to not be statistically significant. Water absorption after 24h (Figure 6.) for variant 0 averaged 1.78% and was 3.3 times lower than average for variant 1 (5.92%) and 5.8 times lower than average for variant 2 (10.32%). Average for variant 3 is 9.49%, and is over 5.3 times higher than average for variant 0, but doesn't differ significantly from averages for variants 1 and 2. Results suggest that after short-time immersion in water waste wood content doesn't impact water absorption quantitatively, just qualitatively. Results obtained both after 2h and 24h of immersion are uncharacteristically high for WPCs. Usually WPCs reach water absorption of about 10% after 10 - 12 weeks, not 2 or 24 hours of immersion (Radoor et al. 2021, Gnatowski 2005). Uncharacteristically high water absorption might be a result of using a hygroscopic polymer, such as PLA and/or weak adhesion between waste wood dust and PLA resulting in small gaps in the composite.

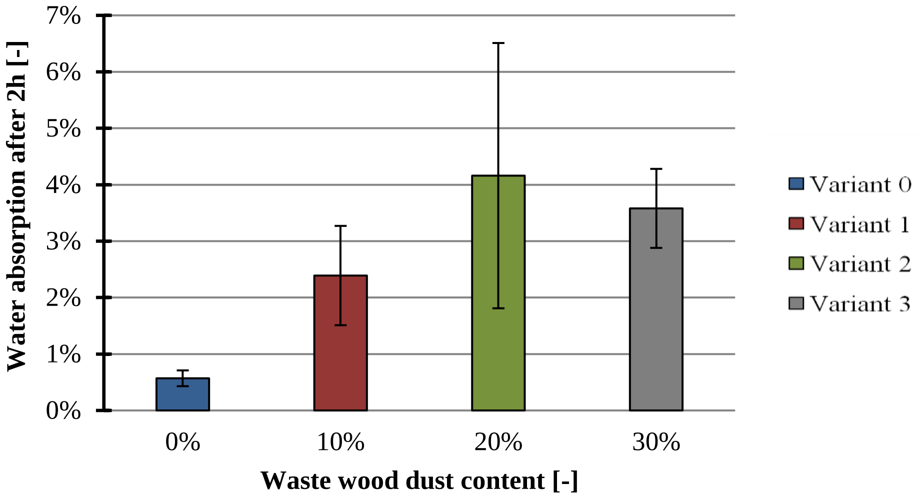

Figure 5. Average water absorption for each tested variant after 2h of immersion

Table 2. Statistical test's p value for water absorption after 2h of immersion

Kruskal-Wallis H test			0.001
Dunn test			
1	**2**	**3**	**Variant**
0.027	0.000	0.000	**0**
Differences statistically significant for p < 0.05	0.162	0.162	**1**
		1	**2**

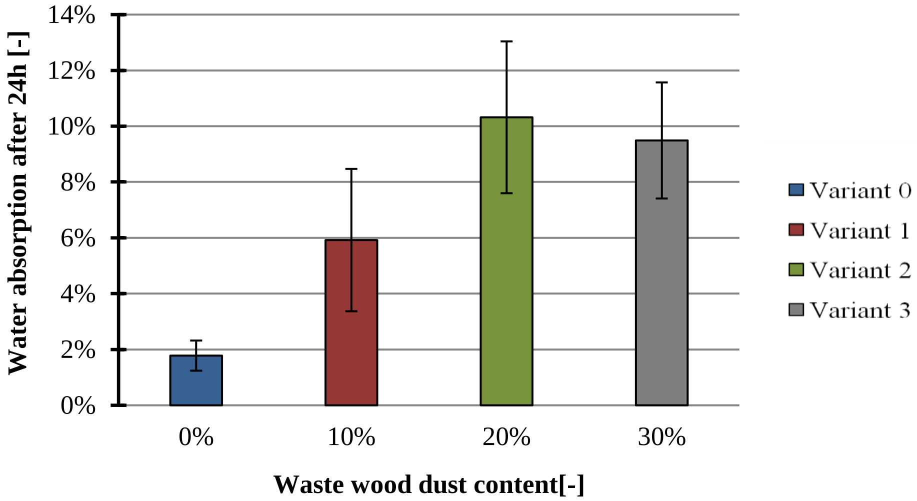

Figure 6. Average water absorption for each tested variant after 24h of immersion

Table 3. Statistical test's p value for water absorption after 24h of immersion

Kruskal-Wallis H test			0.001
Dunn test			
1	**2**	**3**	**Variant**
0.041	0.000	0.001	**0**
Differences statistically significant for p < 0.05	0.047	0.119	**1**
		0.673	**2**

In both thickness swelling after 2h (Figure 7.) and 24h (Figure 8.) of immersion tests average for variant 2 doesn't differ significantly from averages for variants 1 and 3, average for variant 0 isn't significantly different from 0. All other differences are statistically significant. Average thickness swelling after 2h of immersion for variant 1 equals 1.48% and for variants 2 and 3 respectively 2.11% and 3.07%. After 24h of immersion average for variant 1 equals 1.73%, for variant 2 2.62% and for variant 3 4.22%. Results obtained from thickness swelling test suggest linear relationship between waste wood content and thickness swelling. According to Radoor et al. (2021) linear relationship between wood content and thickness swelling is unusual for WPCs without a coupling agent. Similarly to water absorption thickness swelling is uncharacteristically high, but considering thickness swelling to water absorption ratio shown in Table 6. (Percentage of thickness change per 1% of mass increase) these values are in line with data presented by Tomec et al. (2021). This suggests, that high thickness swelling of tested composite is a result of it's high water absorption.

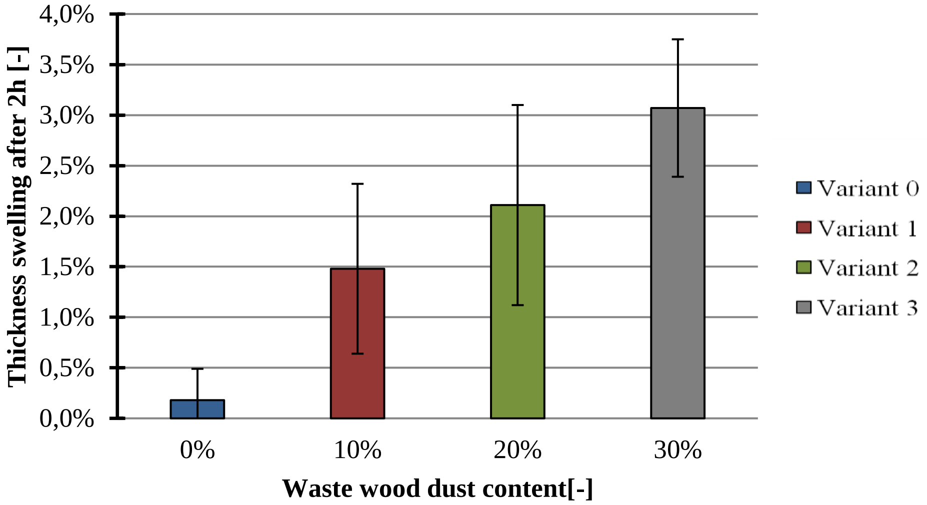

Figure 7. Average thickness swelling for each tested variant after 2h of immersion

Table 4. Statistical test's p value for thickness swelling after 2h of immersion

Kruskal-Wallis H test			0.000
Dunn test			
1	**2**	**3**	**Variant**
0.025	0.004	0.000	**0**
Differences statistically significant for $p < 0.05$	0.516	0.041	**1**
		0.162	**2**

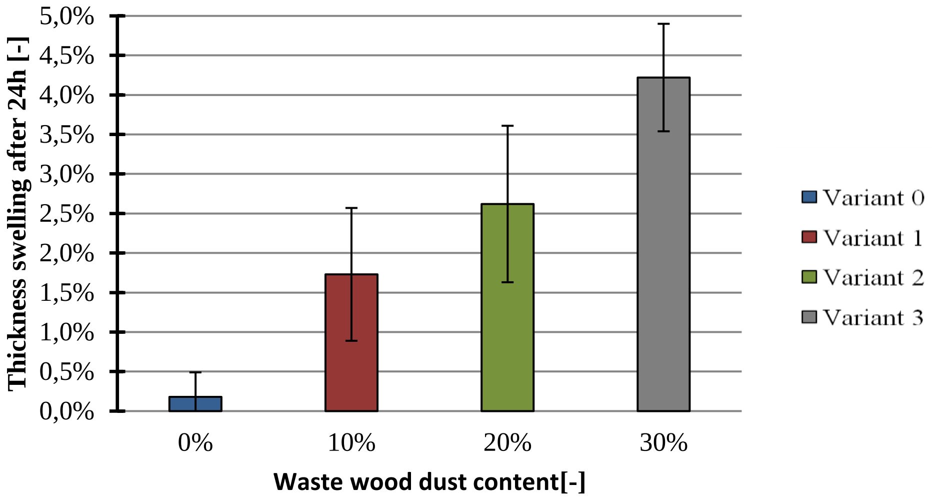

Figure 8. Average thickness swelling for each tested variant after 24h of immersion

Table 5. Statistical test's p value for thickness swelling after 24h of immersion

Kruskal-Wallis H test			0.000
Dunn test			
1	**2**	**3**	**Variant**
0.044	0.003	0.000	**0**
Differences statistically significant for $p < 0.05$	0.269	0.004	**1**
		0.074	**2**

Table 6. Thickness swelling after 24h, water absorption after 24h and their ratio for variants 1, 2 and 3

Variant	Thickness swelling	Water absorption	Thickness swelling to water absorption ratio
1	1.73%	5.92%	0.29%
2	2.62%	10.32%	0.25%
3	4.22%	9.49%	0.45%

CONCLUSIONS

Based on performed tests of waste wood dust – PLA biocomposite the following conclusions can be drawn:

- Tested composite's MOR and MOE are not significantly worse than those of PLA,
- Tested composite's water absorption and, as a result, thickness swelling is uncharacteristically high for a WPC composite, but linear relationship between waste wood dust content and thickness swelling allows adjustment of the properties of this material to the expected conditions of use,
- Both flexural and hygroscopic properties of tested composite might be improved by the use of a coupling agent,
- Waste wood dust – PLA biocomposite can be successfully used in 3D printing, when manufactured elements will be used in low humidity conditions.

ACKNOWLEDGMENT

The presented research was performed within the activities of Wood Technologists Student Research Group, Faculty of Wood Technology, Warsaw University of Life Sciences -

SGGW and financed under “Student Research Groups create innovation” program (Grant no. SKN/SP/534481/2022).

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Streszczenie: *Wybrane właściwości higroskopijne oraz mechaniczne przy rozciąganiu biokompozytu odpadowe pyły drzewne – polilaktyd przeznaczonego do druku 3D*. Opracowanie przedstawia wybrane właściwości biokompozytu odpadowe pyły drzewne – polilaktyd przeznaczonego do druku 3D. Materiały zostały zmieszane w 3 wariantach, zróżnicowanych pod względem wagowego udziału odpadowych pyłów drzewnych – 0%, 10%, 20% i 30%. Następnie zostały one wyekstrudowane w dwuetapowym procesie przy pomocy autorskiego ekstrudera. Tak przygotowany filament został wykorzystany do wytworzenia próbek przy pomocy drukarki 3D. Dla wydrukowanych próbek zbadano MOE, MOR, nasiąkliwość oraz spęcznienie na grubość. Stwierdzono, że zawartość odpadowych pyłów drzewnych nie wpływa w istotny sposób na MOE i MOR. Wykazano negatywny wpływ odpadowych pyłów drzewnych na nasiąkliwość i spęcznienie na grubość badanego materiału. Stwierdzono, że badany kompozyt może być skutecznie wykorzystywany w druku 3D.

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