Effect of Infill Value on Decay Resistance, Thermal, and Mechanical Properties of 3D Printed Polylactic Acid Composites Filled with Wood Fibers

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Polylactic acid (PLA)-based composites with wood filler were 3D-printed using fused deposition modeling (FDM) at different infill settings (*i.e.,* 10 to 100%) to evaluate their strength and biodegradation properties. Microvoids were present in the commercial wood-filled PLA filaments. Wood-PLA filament had reduced thermal stability compared with mineral-filled PLA filament due to the presence of thermally degradable wood flour. The printed composites had a denser internal structure with increased infill. The flexural modulus of elasticity and modulus of rupture also increased with infill value. Sixteen-week fungi test performed using a brown rot, *Postia placenta*, and a white rot, *Irpex lacteus*, did not lead to significant sample weight loss and strength reduction for composites at various infill values. Therefore, 3D printed composites with PLA-based filament containing 30 wt.% wood fiber were shown to be resistant to biodegradation by common decay fungi.

Keywords: Wood-filled PLA; 3D printing; Infill rate; White rot; Brown rot

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INTRODUCTION

Polylactic acid (PLA) is a 'green' sustainable plastic; it is polymerized *via* the ring-opening polymerization of the lactic acid monomer that can be prepared from annually renewable agricultural resources such as corn, sugar cane, cassava, and beet (Vink *et al.* 2003; John *et al.* 2007; Auras *et al.* 2010; Groot and Borén 2010; Sin *et al.* 2012). PLA has the potential to partly substitute for common commercial plastics made from petroleum-based resources, thereby reducing the dependence on non-renewable crude oil (Vink *et al.* 2007; Vink and Davies 2015; Cosate de Andrade *et al.* 2016). PLA is known to be biodegraded into carbon dioxide and water in a period of over two years, while fossil-based polymers need hundreds of years to break down into environmentally friendly components (Garlotta 2001; Kale *et al.* 2007; Auras *et al.* 2010; Sin *et al.* 2012).

Wood-filled PLA composite has been developed and used to reduce the high cost and improve the properties of PLA (Farsi 2012; Mukherjee and Kao 2011; Kutnar and Muthu 2016; Picketing *et al.* 2016; Teuber *et al.* 2016). The organic wood filler component, in particle or flour form, acts as a discontinuous reinforcing agent in a continuous matrix of PLA. The added characteristics for the wood-filled PLA include improved biodegradability, high stiffness, and low density compared with pure PLA polymer (La Mantia and Morreale 2011; Porebska *et al.* 2015; Kutnar and Muthu 2016).

Among the many methods used to make wood-filled PLA composites, 3D printing is being widely adopted to explore a variety of material designs (Hull 1986; Berman 2012). Fused deposition modeling (FDM) is the most popular 3D printing technology due to the machine's low cost and easy operation (Crump *et al*. 1996; Mohamed 2015).

Wood-filled PLA composites, like other durable wood-plastic composites (WPCs), are desired for long-term multiuse applications as an exterior product, especially in harsh outdoor environmental conditions. Biological durability is an essential feature for these applications. Morris and Cooper (1998) found brown-rot and white-rot fungi attack upon recycled WPC lumber after only four years in outdoor service. The wood materials in WPCs serve as a nutrient source for microbial decay and have the potential to become degraded, depending on preconditioning, wood loading, wood particle geometry, and additives. Mankowski and Morell (2000) demonstrated the patterns of white and brown rot fungi attack in a mixture of wood and high-density polyethylene (HDPE) through a soil block test for 12 weeks. It was observed that approximately 30% weight loss occurred in the 70/30 (wt%) wood-HDPE mixture, while the weight loss of the 50/50 mixture was little. Verhey *et al.* (2001) reported that wood particle size in 50/50 (wt%) wood-polypropylene composite affects the fungal decay resistance of WPCs, and the decay susceptibility decreases significantly with a reduction of particle mesh size from 20 to 40 (Verhey *et al.* 2001; Verhey and Laks 2002). Cavdar *et al.* (2018) analyzed the effect of long-term leaching on the decay resistance of wood-filled HDPE composites treated with boron compounds. The fungal decay susceptibility of wood-filled HDPE composites was affected by the leaching test for 60 days. Water absorption and weight loss increased due to the addition of high content of wood flour (Cicala *et al.* 2018).

There have been several studies that examined the properties of wood-filled plastic composites (Pickering *et al.* 2016). However, there are relatively few studies on the decay properties of wood-filled PLA composites made with 3D printing, which have a relatively low wood fiber content (less than 50 wt%). The objective of this study was to investigate morphological properties, decay resistance, and mechanical properties of 3D printed wood-filled PLA composite after 16 weeks of fungal exposure.

EXPERIMENTAL

Materials and Methods

Materials

Wood-filled PLA filament with 30 wt% wood fiber (WOOD-QBX-US) and PLA filament (PLA-QBX-Gold) for 3D printing was purchased from Foshan QIBAIXI trade Co., LTD (Foshan, Guangdong, China). The PLA filament was colored gold and contained a small portion of mineral filler (~2% talc). The mean filament diameter was 1.75 mm.

3D printing procedure for preparing composites

Table 1 lists the 3D printing parameters for preparing the 3D printed composites. A CREALITY CR-10 3D printer (CR-10, Creality, Shenzhen, Guangdong, China) was used to produce the composites. These composites were printed with two types of filament at various infill settings. Four samples were manufactured for each different printing condition.

Table 1. 3D Printing Parameters Used to Manufacture Test Samples for the Study

Design software	AutoCAD 2018
Slicer software	Simplify3D
Rectangular dimensions (L*W*T in mm)	60.0*12.8*3.2
Relative humidity (%)	55 ± 2.0
Temperature (°C)	22 ± 3.0
Build direction	Y-direction (flat)
Build plate surface material	glass
Nozzle material	copper
Nozzle diameter (mm)	0.4
Cooling fan speed (%)	60
Filament diameter (mm)	1.75 ± 0.03
Infill angle offsets	rectilinear (45°/-45°)
Layer height (mm)	0.2
Retraction speed (mm/min)	1800
Build plate temperature (°C)	60
Print speed (mm/min)	2100
Print temperature (°C)	210(PLA), 220 (wood-filled PLA)
Infill settings (%)	10,50,100 (PLA Filament) 10,30,50,75,100 (wood-filled PLA Filament)

Characterization of Filaments and Printed Composites

Properties of filaments

The internal microstructure of the wood-filled PLA filament was observed by scanning electron microscopy (SEM). The filament samples were dipped in liquid nitrogen and then impact-fractured. The fractured surface was sputter-coated with Pt under vacuum at 20 mA for 4 min prior to taking SEM images on a Quanta 3D DualBeam FEG FIB-SEM (FEI Company, Eindhoven, Netherlands) at an acceleration voltage of 5.0 kV. Thermogravimetric analysis (TGA) was conducted using a Q50 TGA analyzer (TA Instruments Inc., New Castle, DE, USA) under the nitrogen atmosphere to analyze the thermal properties of the filaments. The heating rate, temperature range, and sample weight were 10 °C, 30 to 600 °C, and 20 mg, respectively.

Fungal biodegradation properties of printed composite

All types of composites were oven-dried at 50 °C for 24 h, and their initial weight was measured using a top-loading balance. All samples were then sterilized by gamma irradiation. The treated samples were submerged in water using vacuum-assisted impregnation prior to decay testing. Decay tests were conducted inside autoclavable plastic food containers (80 mm deep by 110 mm in diameter). A 10-mm hole was made in the plastic lid of each container and plugged with cotton to provide some air during testing. Each container had 200 g soil and 60 mL distilled water. Pine feeder strips were used in containers with no-fungi (control) and brown-rots. Cotton wood feeder strips were used in the containers with white-rot. Each container had two feeder strips for two separate samples. All prepared containers were autoclaved using a Sterilmatic Autoclave (95-6233, Market Forge Industries, Burlington, VT, USA) for 20 min at 121 °C for steam sterilization and then cooled for 24 h at room temperature.

The fungal test was carried out according to American Wood Protection Association E22-16 (AWPA, 2017). For brown-rot and white-rot groups, inoculation was first done for containers using cultured brown-rot fungi - *Postia placenta* (PP) and white-rot fungi - *Irpex lacteus* (IL). After allowing the fungi to grow for two weeks in the test containers, test samples were placed on the inoculated feeder strips (one sample on each strip). Then the prepared culture containers with the test samples were placed inside a conditioning chamber along with prepared containers of No Fungi group at 25 °C and 85% relative humidity for 16 weeks. After 16 weeks, the specimens were cleaned, dried at 50 °C, for 24 h, and weighed again. The degree of degradation of the specimens was determined by the weight loss percentage according to Eq. 1, each exposed group (no-fungi control,

$$WL\ (\%) = \frac{W_1 - W_2}{W_1} \text{ x } 100 \quad (1)$$

where WL represents the weight loss percentage of each specimen (%), and W_1 (g) and W_2 (g) are the weight of the specimen before and after the decay test, respectively. Each group had 4 specimens, and the average data was calculated. Untreated southern pine samples were used as positive control to test vigorousness of the test fungi.

Mechanical properties of printed composites

The mechanical properties of the 3D printed composites in each group before and after the decay tests were assessed. The flexural test was conducted according to ASTM D790-17(2017) using the universal testing machine (5582, Instron, Norwood, MA, USA) with loading speed 5 mm/min. The modulus of elasticity (MOE) and flexural modulus of rupture (MOR) were calculated by this method.

RESULTS AND DISCUSSION

Morphology Properties

Figure 1 shows the cryo-fractured surface of wood-filled PLA filament. Many micron-size voids were observed on the fracture surface as shown in Fig. 1 (a and b). These voids can be explained by the fact that the presence of wood fiber could hinder the crystal growth of PLA when the wood-filled PLA filament was manufactured. This could be due to water vapor presented in wood fiber and evaporation of the water led to voids in the polymer matrix.

Figure 1 (c) shows that wood fillers marked by red arrows existed in the filament. SEM analysis also showed some portion of mineral fillers in the gold-colored PLA filament, which was further verified through the thermal degradation analysis shown in later sections.

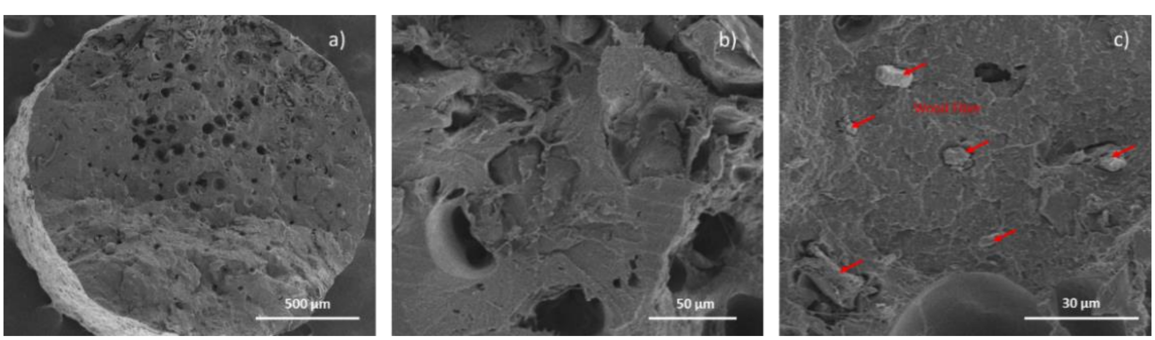

Fig. 1. Microimages of wood-PLA filament with three different magnifications.

The internal structures of the 3D printed composites made by wood-filled PLA and PLA are shown in Fig. 2. These bar-shape composites had different internal structures with different infill settings. The 3D printed composites made with a 10% infill had a loose internal structure with significant empty spaces, whereas a solid internal structure was observed in the composite printed with the 100% infill setting. Thus, an increase in the infill of the printed composites was reflected in the tight internal structure of both the wood-filled PLA and PLA composites. The difference in the composite internal structure could affect the strength, water absorption and decay resistance properties of the composites.

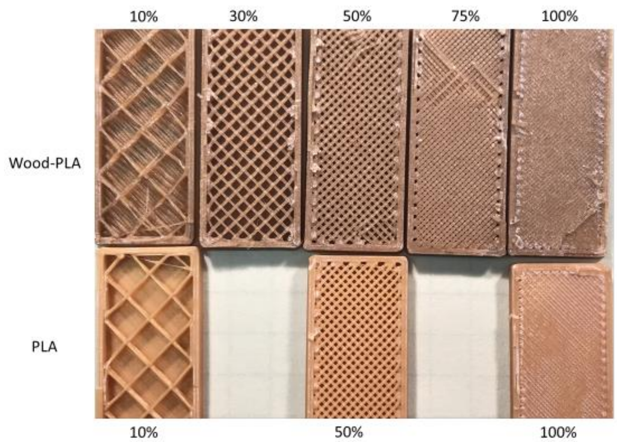

Fig. 2. Internal structure of 3D printed bar-shape composites and wood-PLA and PLA filaments at various infill settings.

Thermal properties

The TGA and differential thermogravimetric (DTG) curves of the wood-filled PLA and PLA filaments are presented in Fig. 3 to compare the thermal degradation properties of both filaments.

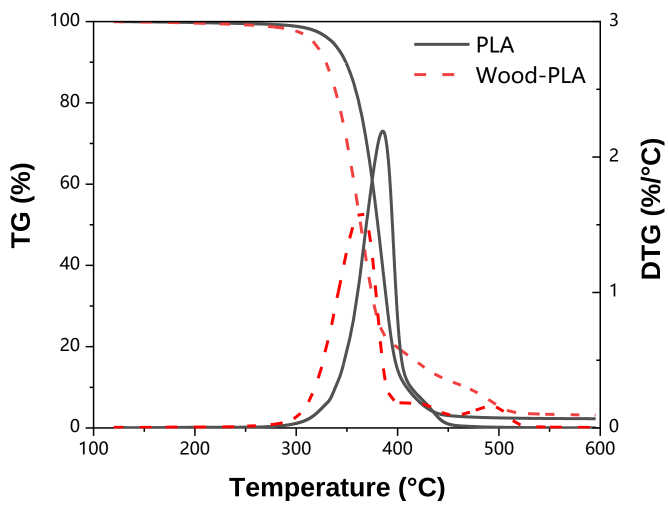

Fig. 3. TG and DTG curves of wood-filled PLA and PLA filaments at 10 °C/min heating rate

The onset temperature, peak temperature, and residue of the PLA filament were 302.6 °C, 385.2 °C, and 2.26%, respectively. The residue weight (*i.e.*, 2.26%) for PLA filament, indicates the presence of some mineral-based fillers in the PLA filament as shown in the SEM analysis. The corresponding values for the wood-filled PLA filament were 281.9 °C, 364.1 °C, and 3.15%, respectively. These results suggested that the wood-filled PLA filament had lower thermal stability than the PLA filament due to the addition of wood, which has a relatively low thermal degradation temperature and a high carbonization property compared with those of PLA (Tao *et al.* 2017) and mineral filler. The use of wood in the composite also led to more char formation, resulting in a higher residue weight.

Decay resistance properties

Figure 4 shows photos of the wood-filled PLA and PLA composites (100% infill as an example) after the fungal test. Fungal-hyphae appeared on the feeder strips and test samples for the brown-rot and the white-rot groups, while the no-fungi control group was clean from any obvious fungal growth.

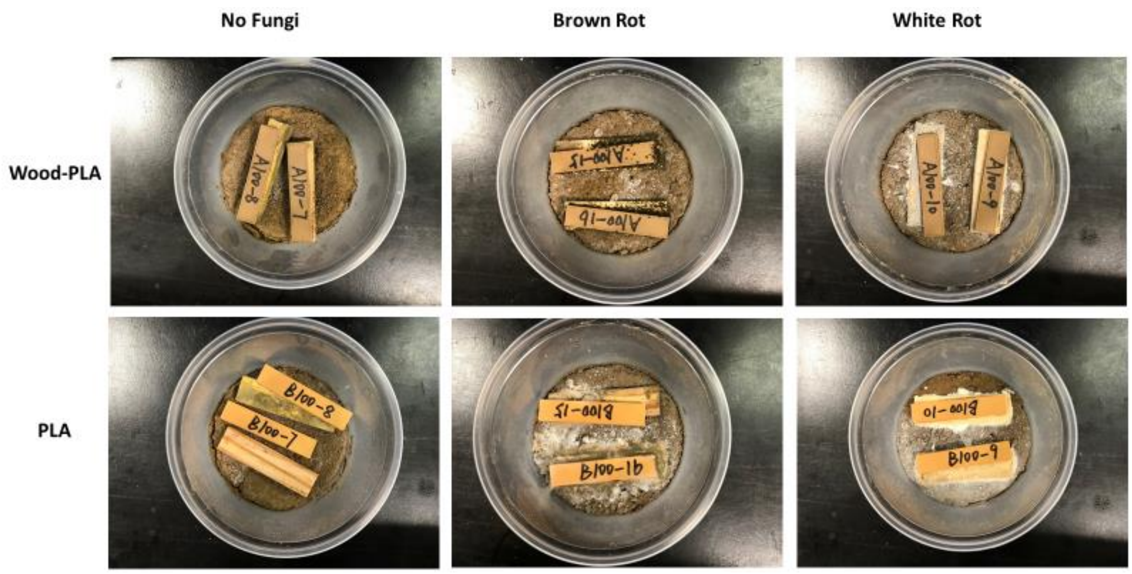

Fig. 4. Photos of wood-filled PLA and PLA samples after the fungal test

The weight loss results of Southern pine wood control, wood-filled PLA, and PLA composites after the fungal test are listed in Table 2. Southern pine wood control samples had 55.21±3.10% weight loss for brown-rot - *Postia placenta (PP)* and 20.41±2.30% for white-rot *Irpex lacteus (IL)*. The data for the positive wood control showed the strong vigorousness of the test fungi. A small amount of weight loss was exhibited in the wood-filled PLA composites, but the PLA composite showed no weight loss after all types of fungal tests. There were no noticeable differences in weight loss according to different infill rates and fungal test conditions for PLA-based composites. It can be inferred that the infill rate and fungi type did not significantly affect the decay resistance of 3D printed composites with both types of filaments. The small or no weight loss of wood-filled polymer composite (at low wood fiber loading level) has been reported in the published literature such as Mankowski and Morrell (2000). Wood particles are encapsulated by polymers in the composites and fungi often could not break through the polymer to get to the wood during the 16-week test period. For PLA based composites, our test data shows that they cannot be significantly biodegraded as well, similar to other polymer-wood composites.

Table 2. Weight Loss Percentages of 3D Printed Composites after the Fungal Test

Infill (%)	No-fungi		Brown-rot *Postia placenta* (PP)		White-rot *Irpex lacteus* (IL)	
	Wood-PLA	PLA	Wood-PLA	PLA	Wood-PLA	PLA
10	0.06%	-0.07%	0.04%	-0.16%	-0.08%	-0.22%
30	-0.05%		0.05%		-0.05%	
50	0.01%	-0.02%	0.01%	-0.02%	0.04%	-0.08%
75	-0.05%		0.00%		-0.02%	
100	0.00%	-0.09%	0.05%	-0.08%	0.03%	-0.03%
Southern pine wood control			55.21±3.10%		20.41±2.30%	

Note: Values represented means of four specimens per group. Negative values meant weight including residues of soil and mold. All standard deviations were 0.00.

Mechanical properties

The MOE and MOR of the 3D printed wood-filled PLA and PLA composites with various infill values and fungal types were measured and the associated data is shown in Table 3 and Fig. 5. The MOE and MOR values of the PLA composite groups were significantly higher than those of wood-filled PLA at all infill values (Table 3). The results show the effect of filler type and amount on the 3D printed composite properties for commercial filaments. Both MOR and MOE values increased with increased infill, as the composites had less internal voids at higher infill rates.

Table 3. Summary Table of Bending Properties of 3D Printing Samples

Infill	Unexposed Group		Exposed Group: No Fungi		Exposed Group: Brown Rot (PP)		Exposed Group: White Rot (IL)	
	Wood-PLA	PLA	Wood-PLA	PLA	Wood-PLA	PLA	Wood-PLA	PLA
	MOE (MPa)							
10	1018.49 (68.86)	1467.03 (41.66)	881.21 (48.15)	1340.77 (169.65)	945.11 (40.35)	1268.76 (330.84)	937.18 (25.86)	983.62 (308.36)
30	1162.96 (194.89)		1185.41 (160.87)		1178.70 (202.17)		1122.67 (162.41)	
50	1325.01 (184.93)	1732.57 (48.15)	1236.18 (191.76)	1680.27 (21.75)	1225.07 (231.04)	1756.75 (51.20)	1205.51 (240.44)	1739.39 (23.84)
75	1377.02 (153.75)		1300.66 (158.79)		1399.19 (208.58)		1202.23 (61.44)	
100	1774.67 (39.12)	2169.55 (89.70)	1711.84 (76.40)	2065.78 (89.18)	1689.80 (68.29)	2147.06 (95.90)	1699.54 (43.72)	2054.73 (49.52)
	MOR(MPa)							
10	21.37 (0.59)	31.97 (0.75)	20.80 (0.92)	29.25 (4.02)	21.89 (1.08)	27.91 (6.67)	21.15 (0.67)	20.96 (7.49)
30	29.49 (3.45)		30.13 (3.17)		28.42 (2.83)		28.44 (2.66)	
50	32.63 (2.25)	43.11 (1.30)	32.61 (2.66)	41.49 (1.47)	32.30 (3.00)	44.80 (4.91)	32.53 (3.10)	42.63 (0.75)
75	37.90 (2.10)		37.51 (2.23)		37.46 (2.11)		34.84 (0.34)	
100	50.64 (0.91)	62.81 (1.16)	48.42 (1.27)	59.47 (0.96)	48.52 (1.00)	60.31 (1.68)	47.99 (0.90)	59.47 (0.80)

*Values were the average numbers of four replicates;
*Values in parentheses were the average standard deviations of four replicates;

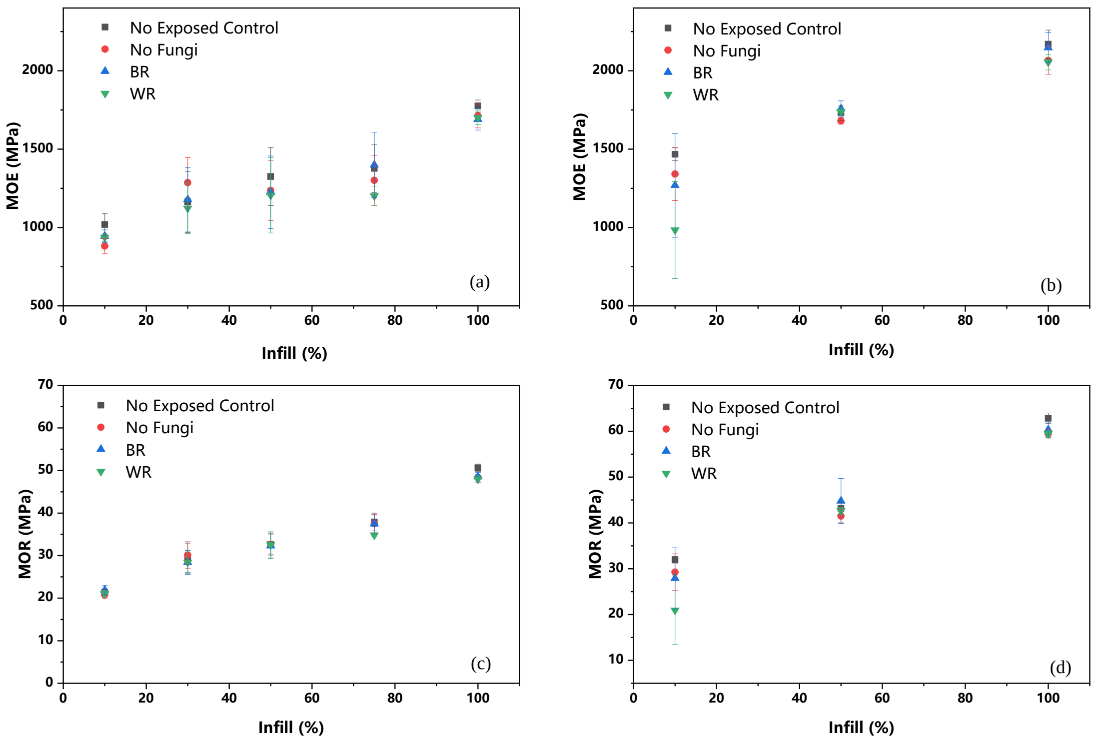

Fig. 5. MOE (a and b) and MOR (c and d) data as a function of infill rate. (a and c): Wood-PLA; (b and d): PLA

A comparison of the MOE and MOR properties of the unexposed group and exposed group showed that 3D printed composites in the unexposed group had slightly higher or similar data values than these of the exposed group. In the exposed group, the MOE/MOR values of exposed composites without fungi were slightly higher than those of exposed composites with brown and white rots. The two exposed composites with fungal groups had similar MOE/MOR values. Since there was little or no sample weight loss, the small loss in the MOE/MOR value due to high humidity and fungus exposure was probably due to a moisture softening effect. For the composite under high humidity condition during the 16-week testing period, the swelling of wood fibers in the composite weakened the fiber-composite interface (Liu *et al.* 2013), leading to reduced MOE. The internal void structure of the composite could also affect their MOE/MOR values under high humidity conditions due to difference in their swelling potentials (Lv *et al.* 2016).

Overall, the fungus-damage (biodegradation) to the composite seemed to be very small under the test conditions. This implies that the composite can be applied for exterior uses. At the same time, the composite cannot be easily biodegraded after their uses to reduce their pollutions to the environments.

CONCLUSIONS

In this work, specimens were 3D printed from commercial poly(lactic acids) (PLA) and wood-filled PLA filaments by the fused deposition modeling (FDM) method at different infill settings, and decay resistance, thermal and mechanical properties of the samples were measured. The following conclusions can be made from the study:

1. The commercial wood-filled PLA filaments (30 wt.% wood infill) had many micro internal voids, and some inorganic fillers were observed in the PLA filament. For both materials, the internal structures of 3D printed composites became increasingly denser with an increase in the printing infill value.
2. Wood-filled PLA filament had lower thermal stability compared with PLA filament due to the addition of wood filler.
3. An exposure of decay fungi over a period of 16 weeks for 3D printed wood-filled PLA composites with 30 wt.% wood content led to very little sample weight loss, indicating that the printed composites are fungal decay resistant with less biodegradability for potential exterior applications.
4. Measured MOE and MOR values for 3D printed composites increased about linearly with printing infill settings before and after fungal decay exposures. The exposure to the high humidity and decay fungi during the 16 weeks showed only slight negative effect of the strength/modulus properties of the composites.

ACKNOWLEDGMENTS

This study was supported from China Scholarship Council (Grant No. 201808410102), Henan Scientific and Technological Research Program (Grant No. 182102110027), National Institute of Forest Science (Seoul, Korea), and the USDA NIFA McIntire Stennis project (Project No. 1000017).

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Article submitted: May 3, 2020; Peer review completed: June 28, 2020; Revised version received and accepted: July 6, 2020; Published: July 13, 2020.
DOI: 10.15376/biores.15.3.6724-6734