Effects of Filler Type and Content on the Mechanical, Morphological, and Thermal Properties of Waste Casting Polyamide 6 (W-PA6G)-based Wood Plastic Composites

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Cast polyamide 6 (PA6G), trade name Castamide, is a semi-crystalline polymer widely used in the engineering plastics industry. There is a need to recycle valuable waste (W)-PA6G generated during part manufacturing of this polymer (approximately 30%). This study attempts to utilize W-PA6G in the manufacture of wood-plastic composites as a polymeric matrix. The effect of lignocellulosic filler type (FT) and filler content (FC) on the mechanical, morphological, and thermal properties of W-PA6G-based composites were investigated. During manufacturing, N-butyl benzene sulfonamide (N-BBSA) and lithium chloride (LiCl) were utilized as a plasticizer and a melt temperature-lowering salt, respectively. The rice husk (RH) and Uludağ fir wood flour (WF) filled W-PA6G-based composites were successfully manufactured using a combination of extrusion and injection molding. Compared to RH filled composites, WF filled composites provided better tensile and flexural properties (both strength and modulus) at 20% and 30% filler contents. Morphological study showed the nonhomogeneous distribution of fillers in the polymeric matrix. Lignocellulosic filler resulted in reduced melting temperature and crystallinity of W-PA6G-based composites. This reduction was more pronounced in RH filled composites.

Keywords: Cast polyamide 6; Composite; Mechanical; Thermal; Morphological

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INTRODUCTION

In recent years, the application of lignocellulosic material filled thermoplastic composites, or wood-plastic composites (WPCs), has increased in numerous areas such as wall coverings, floor coverings, balustrades, window profiles, docks, door frames, furniture, boat hulls, pallets, façade coverings, architectural profiles, and automotive parts (Lei and Wu 2010; Xu *et al.* 2018; Kabakci and Kesik 2020). Compared to unfilled ones, lignocellulosic material filled thermoplastics have increased bending strength, stiffness, high elasticity (both flexural and tensile modulus), low thermal expansion, and low cost (Lei and Wu 2010). Extensive research has been conducted to assess the utilization of commodity thermoplastic, such as polyethylene (Mengeloglu *et al.* 2007; Adhikary *et al.* 2008; Çavdar *et al.* 2019), polypropylene (Espert *et al.* 2004; Mengeloglu *et al.* 2007), polyvinyl chloride (Jiang and Kamdem 2004), and polystyrene (PS) (Poletto *et al.* 2011) in WPC manufacturing. Addition of wood fibers has been found to enhance the mechanical properties of the commodity polymers, but their mechanical properties might not be enough for most engineering applications (Gardner *et al.* 2015). This directed attention to WPC produced from engineering thermoplastics in the last decade is due to their superior properties (excellent chemical resistance, high thermal stability, and high tensile, bending, and impact strengths) (Kiziltas *et al*. 2014). Several studies have been conducted in an attempt to produce WPCs with engineering thermoplastics. Yörür *et al.* (2013) reported that mechanical properties were improved due to the decrease in thickness swelling values with the increase of PA6G in wood composites. Chen and Gardner (2008) produced WPCs utilizing PA66 as a polymer matrix and reported composites with improved storage modulus, thermal stability, and reduced glass transition temperature. In some studies, engineering plastics are incorporated into general-purpose plastics to form polymer blends. In a two-step extrusion process, Lei and Wu (2010) produced wood flour filled polyethylene terephthalate (PET)/high-density polyethylene (HDPE) composites. In the first step, polymer blends were extruded at high temperature (allowing PET to melt). In the second step, wood flour was added into the previous polymer mixture (pellets) at low temperature to be extruded (allowing only HDPE to melt). Increased mechanical properties, storage modulus, and matrix crystallinity with wood flour addition were reported. Similarly, Liu *et al*. (2009) produced HDPE/Nylon 6/Banana fiber composites using various compatibilizers.

Cast polyamide 6 (PA6G) is one of the most widely used thermoplastics in the engineering plastic industry (Bozdemir 2011; Yörür and Şeker 2019). During production of PA6G parts, approximately 30% wastes are generated and can annually sum up to 5,000 tons in the European Union (Formisano *et al.* 2016; Formisano and Bonten 2019). Waste (W)-PA6G, a component of global solid wastes, is a promising raw material for WPCs due to its large volume and low cost. The PA6G has excellent mechanical properties with a melting temperature (T_m) higher than 200 °C. Because wood filler starts decomposing around approximately 200 °C, high T_m of polymers limits their utilization in WPC manufacturing (Sears *et al*. 2001; Lei and Wu 2010; Zierdt *et al*. 2015; Zierdt *et al*. 2016; Xu *et al.* 2018). It was reported that lithium chloride (LiCI), a halogenic salt, can remarkably reduce the melting temperature of polyamide 6 (PA6) (Amintowlieh *et al*. 2012; Xu *et al.* 2018). Recently, Yörür and Şeker (2019) reported that with the utilization LiCI and N-butyl benzene sulfonamide (N-BBSA) as an additive, melting temperatures of W-PA6G and W-PA6G composites were reduced approximately 3.6% and 7.2%, respectively. The mechanical, thermal, and morphology of properties of waste-cast polyamide 6 (W-PA6G)-based WPCs are not reported in the literature. The aim of this study was to investigate the effect of filler type (FT: rice husk [RH] and fir wood flour [WF]) and filler content (FC: 0, 10, 20, and 30%) on the mechanical, morphological, and thermal properties of W-PA6G-based composites.

EXPERIMENTAL

Materials

Rice husk (RH) and Uludağ fir (*Abies bornmülleriana* (Mattf.)) wood flour (WF) were used as fillers. The RH (density 0.90 to 0.150 g/cm^3) was provided by a paddy factory (Karabük, Turkey). Uludağ fir wood chips were supplied by a timber factory in Karabük, Turkey. Waste cast polyamide 6 was kindly donated by Plamek Plastic Industry and Commerce Plant (Bursa, Turkey). N-butyl benzene sulfonamide (N-BBSA) was purchased from Sigma Aldrich (St. Louis, MO, USA) as a plasticizer (density of 1.15 g/mL and molecular weight of 213.30 g/mol at 25 °C). Lithium chloride (LiCl) salt (purity of 99%, weight of 42.39 g/mol and density of 2.068 g/cm³ at 20 °C) was used to lower the melting temperature of W-PA6G (Carlo Erba 2018). Mold release agent with 31% boron nitride (BN) was used (Alfa Aesar 2019).

Methods

The rice husk particles (RH) and Uludağ fir wood (WF) were turned into flour form using a Wiley mill (Altundal, Kahramanmaraş, Turkey) and further classified. Particles that passed through a 40-mesh (400 µm) screen and that stayed on a 60-mesh (250 µm) screen were used in this study. Then, the classified lignocellulosic filler materials and W-PA6G were dried for 24 h at a temperature of 103 ± 2 °C in an oven.

The W-PA6G-based composites were manufactured based on the manufacturing recipe presented in Table 1. All ingredients were homogeneously mixed in a high intensity mixer (900 to 1000 rpm in 2 min). Mixtures were fed into a single screw extruder (TTB 30, Teknomatik, Istanbul, Turkey) with five heating zones. The extruder screw speed was set as 50 rpm and the temperature ranged from 230 to 250 °C from barrel to die

Table 1. Production Recipe of W-PA6G Added WPCs

ID	Filler (%)	W-PA6G (%)	LiCl (%)	N-BBSA (%)
W-PA6G	-	96.0	2	2
PA-10RH	10	86.0	2	2
PA-20RH	20	76.0	2	2
PA-30RH	30	66.0	2	2
PA-10WF	10	86.0	2	2
PA-20WF	20	76.0	2	2
PA-30WF	30	66.0	2	2

The extruded compounds were first cooled in a water pool (23 ± 2 °C) and were granulated into pellets using a Wiley mill. The pellets were dried at 103 ± 2 °C in an oven (24 h) to reduce moisture content below 1% before the injection molding using HAIDA HDX-88 (Haida Plastic Machinery Co., Ltd., Ningbo, China). Injection pressure and temperatures (from feed zone to die zone) were set as 5 to 6 MPa and 190 to 220 °C, respectively. Before testing, the specimens were conditioned in a climate cabinet at 23 ± 2 °C and 65 ± 2% relative humidity. Tensile, flexural, and (notched) impact strength (IS) properties were determined based on the procedures of ASTM D638 (2001), ASTM D790 (2003), and ASTM D256 (2000), respectively, and at least five samples from each composition were tested. Tensile and flexural property tests were implemented on a Zwick 10 KN (Zwick/Roell, Ulm, Germany), while a HIT5, 5P (Zwick) was used for IS testing on notched specimens. The notch was added using a RayRan™ Polytest notching cutter (London, UK).

The morphological properties of the fractured surfaces were studied using scanning electron microscopy (SEM) (EVO LS10; Carl Zeiss, Jena, Germany). All samples were plated with gold before microscopic observations (Sputter Coater 108 Auto; Cressington, London, England). The differential scanning calorimeter (DSC) analysis was performed on a HITACHI-DSC-STA7300 machine (Hitachi, Tokyo, Japonya) using a heating rate of 10 °C/min under nitrogen atmosphere from room temperature to 300 °C. Thermo-gravimetric analysis (TGA) of the test samples was performed on the HITACHI-TGA-STA7300 machine using a heating rate of 10 °C/min under nitrogen atmosphere from room temperature to 800 °C. For statistical analysis, the statistical software Design-Expert®, version 7.0.3 (Stat-Ease Inc., Minneapolis, MN, USA) was used.

RESULTS AND DISCUSSION

Mechanical Properties

The effect of filler type (FT) and filler content (FC) on tensile properties (tensile strength (TS), tensile modulus (TM), and elongation at break (EatB)), flexural properties (flexural strength (FS), flexural modulus (FM)), and IS of W-PA6G-based composites were determined at room temperature. The mean values of measured mechanical properties are reported in Table 2.

Table 2. Mechanical Properties of W-PA6G-based Wood-plastic Composites

ID	TS (MPa)	TM (MPa)	EatB (%)	FS (MPa)	FM (MPa)	IS (kJ/m²)
W-PA6G	65.2 (2.46)	709 (58)	182 (6.7)	63.5 (2.7)	1237 (58)	18.7 (8.3)
PA-10RH	62.4 (3.3)	931 (46)	8.5 (2.4)	72.0 (12)	1482 (114)	3.1 (0.7)
PA-20RH	30.1 (1.5)	893 (24)	3.80 (0.2)	63.6 (5.5)	1529 (155)	1.9 (0.3)
PA-30RH	25.7 (3.7)	674 (116)	4.8 (0.3)	40.4 (1.8)	965 (40)	1.3 (0.3)
PA-10WF	38.0 (13.0)	819 (81)	4.9 (0.5)	54.2 (13.0)	1569 (101)	10.2 (6.1)
PA-20WF	42.9 (2.6)	1036 (42.2)	4.2 (0.3)	77.3 (5.5)	2189 (494)	2.7 (1.6)
PA-30WF	38.5 (3.2)	1039 (80.7)	3.6 (0.3)	63.7 (6.6)	2472 (161)	3.3 (3.3)

*: Values in parentheses are standard deviation

ANOVA table and interaction graphs of the tensile properties are resented in Table 3 and Fig. 1a, respectively. TS values of W-PA6G and W-PA6G-based composites were in the range from 25.7 to 65.2 MPa. The statistical analysis showed that lignocellulosic filler type did not have a significant effect on TS ($P = 0.8663$). However, filler content had a significant effect on TS ($P < 0.0001$). Both the RH and WF fillers reduced the TS of W-PA6G-based composites, and this reduction was affected by filler. The WF filled W-PA6G-based composites provided better tensile and flexural properties (both strength and modulus) than the RH filled ones at 20% and 30% filler contents. Elsabbagh *et al.* (2017) produced PA6 composites using kenaf and flax as lignocellulosic fillers. It was reported that the addition of filler reduced TS values, and increased filler. The WF filled W-PA6G-based composites provided better tensile and flexural properties (both strength and modulus) than the RH filled ones at 20% and 30% filler contents, which was caused additional property reduction in composites. Reduced TS values in beech fiber filled PA6- and PA11-based composites were also reported by Zierdt *et al.* (2016). Similar results for various thermoplastic composites filled with lignocellulosic material were reported by others (Xu 2008; Malkapuram *et al*. 2009; Ku *et al*. 2011). Significant effect of two factors' interaction (FTxFC) suggested that the influence of filler content was not the same and showed some variety among filler types. For this study, RH filler performed better at low filler content (10%) but WF outperformed it at higher filler contents.

Table 3. Analysis of Variance (ANOVA) Table of Tensile Strength

Source	Sum of Squares	df	Mean Square	F-Value	p-value Prob > F
Model	9023.66	7	1289.09	46.17	<0.0001
A-Filler Type	0.80	1	0.80	0.029	0.8663
B-Filler Content (%)	6708.36	3	2236.12	80.09	<0.0001
AB	2314.50	3	771.50	27.63	<0.0001
Pure Error	893.46	32	27.92		
Cor Total	9917.12	39			

The TM values of the W-PA6G and W-PA6G-based composites were in the range of 709 to 1039 MPa (Table 2). The interaction graph of TM is presented in Fig. 1b.

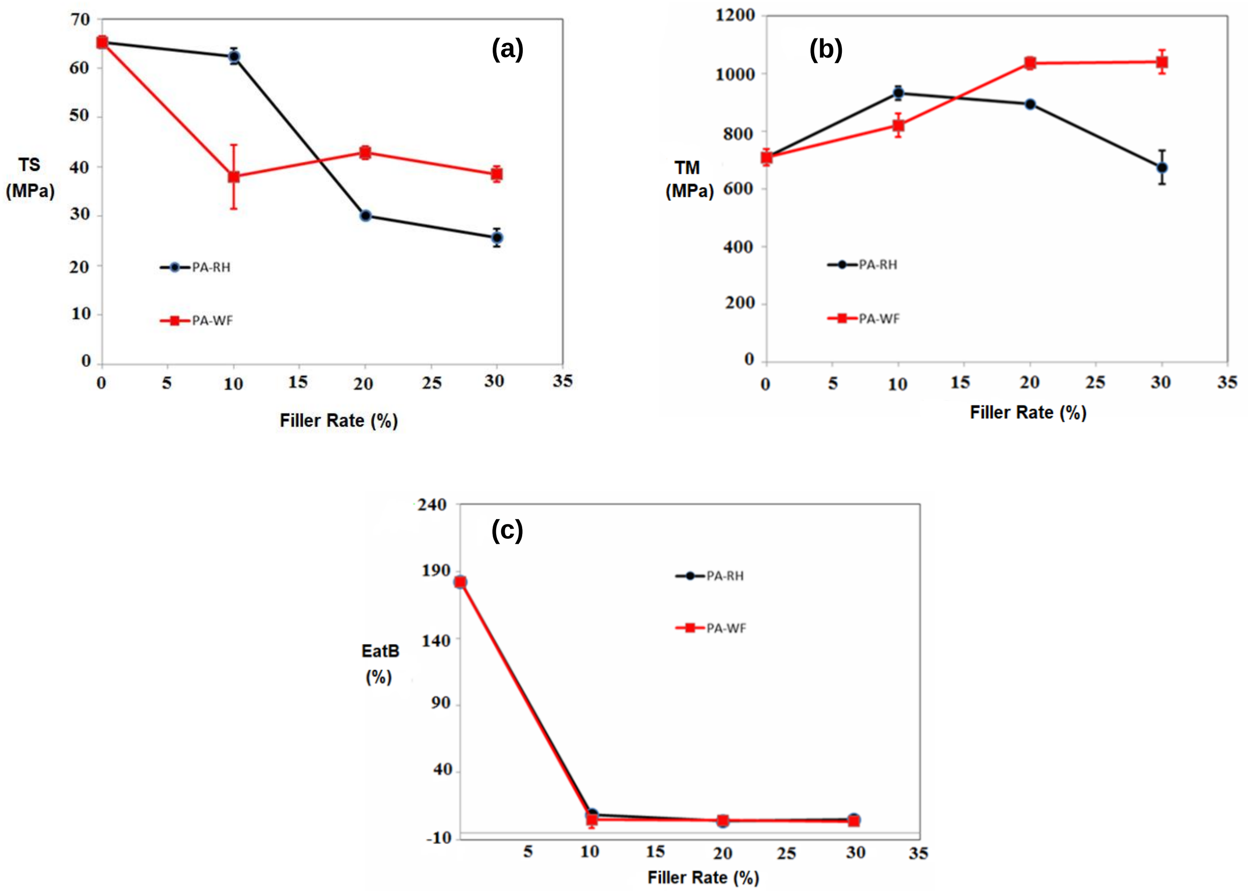


Fig. 1. Interaction graphs of tensile properties: (a): TS; (b): TM; and (c): EatB

The FT, FC, and their interaction (FTxFC) had a significant effect on TM ($P < 0.0001$). Initially, as expected, both RH and WF improved TM values of the composites. At higher filler contents, WF continued to improve TM values but surprisingly, over 10%

utilization, RH addition significantly reduced the TM values. The RH is reported to be more thermal sensitive and has lower cellulose content compared to WF. It is believed that chemicals evaporating at high processing temperatures might cause chain scission of the polymer molecules. Improved TM values with lignocellulosic filler, in addition to the thermoplastic matrices, were also reported by other researchers (Klyosov 2007; Xu 2008; Mengeloglu and Karakuş 2012; Kiziltas *et al.* 2014; Yörür 2016; Çavuş 2020). It is explained that a mixture of high modulus filler and low modulus polymer results in a composite with higher modulus compared to the polymer itself (rule of mixture).

For EatB, values in the range of 3.6 to 182% were measured. The interaction graph of EatB is presented in Fig. 1c. Filler type did not have significant effect on EatB ($P = 0.295$). In contrast, filler content had a significant effect on EatB ($P < 0.0001$). Nuñez *et al*. (2003), La Mantia *et al*. (2005), and Dönmez Çavdar *et al*. (2011) stated that there is a decrease in elongation at break values due to increased brittleness of the resulting composites with the addition of lignocellulosic filler.

The interaction graphs of the flexural properties are presented in Fig. 2. Flexural strength values of W-PA6G and W-PA6G-based composites were in the range from 40.4 to 63.5 MPa (Table 2 and Fig 2a). The statistical analysis showed that FT had a significant effect on FS ($P = 0.045$). The effects of FC and FTxFC interactions on FS were also statistically significant ($P < 0.0001$). The WF filled W-PA6G composites provided overall better FS values than the RH filled ones. Because wood plastic composites are generally considered as an alternative to plastic lumber, it is useful to compare them with the ASTM D6662 (2007) polyolefin plastic lumber standard. According to this standard, flexural strength values are required to be at least 6.9 MPa. All manufactured composites provided results that were well over this standard.

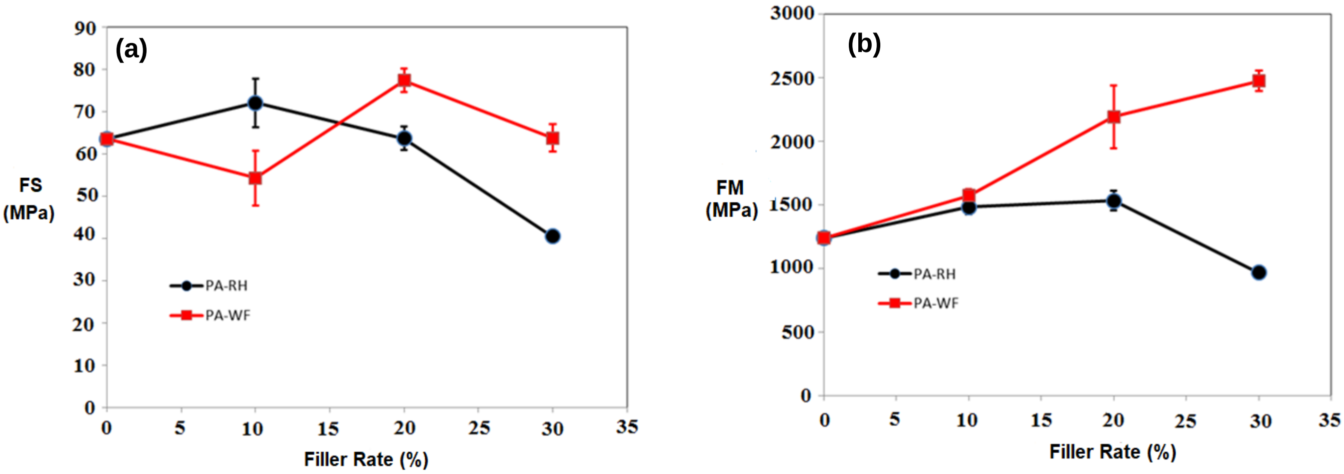


Fig. 2. Interaction graphs of flexural properties: (a): FS and (b) FM

The FM was significantly affected by FT, FC, and FTxFC interaction ($P < 0.0001$). The FM values of W-PA6G and W-PA6G-based composites were in the range from 965 to 2472 MPa (Table 2 and Fig 2b). The overall performance of WF was better than RH as fillers. Once again at higher filler contents, performance of composites filled with RH were remarkable reduced. The 30% WF filled composites provided FM values of almost double of the W-PA6G. Kiziltas *et al*. (2014) reported an approximately 47% increase in FM of PA6-based composites when 20% lignocellulosic material was used as filler. Jin and

Matuana (2008) manufactured PVC-based composites using maple wood flour as lignocellulosic filler and reported an approximately 18.4% increase in FM of PVC-based composites when 20% lignocellulosic material was used as filler.

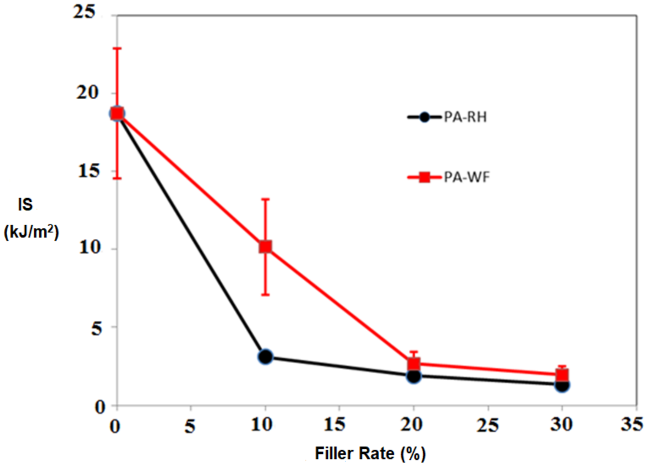


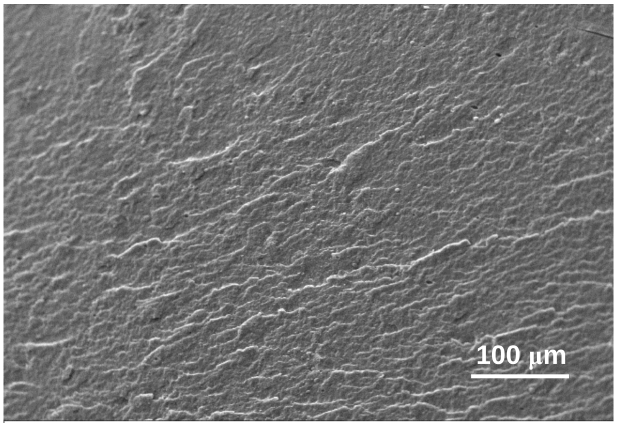
Fig. 3. Interaction graph of impact strength (IS)

For IS, values were measured in the range of 1.3 to 18.7 kJ/m^2. The interaction graph of IS was presented in Fig. 3. Filler type did not have a significant effect on IS (P = 0.174). In contrast, filler content had a significant effect on IS (P < 0.0001). Regardless of filler type, IS values were significantly reduced with the rising percentage of filler. Addition of filler in the polymer matrix increased the brittleness of composite and limited their energy absorption potentials. Hajji *et al.* (2008) produced PVC composites using pine as lignocellulosic fillers. It was reported that the addition of filler reduced IS values. Reduced IS values with lignocellulosic filler presence was also reported by others (Liu *et al*. 2009; Çavdar *et al.* 2011; Kiziltas *et al*. 2014; Aydemir *et al*. 2015; Zierdt *et al*. 2015; Elsabbagh *et al.* 2017; Oliver-Ortega *et al*. 2018; Çavus 2020).

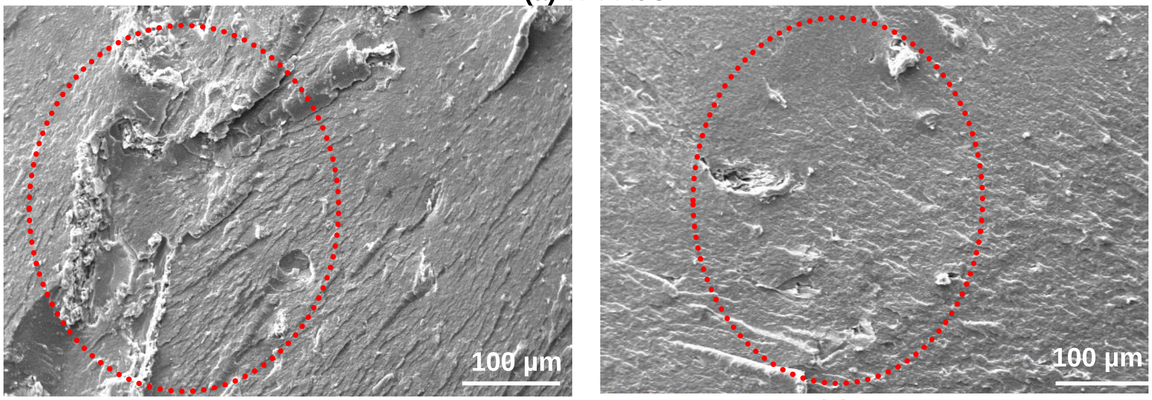
Morphology of the W-PA6G and W-PA6G-based composites (with 20% filler) was studied using SEM. The SEM images of the selected samples are presented in Fig. 4. Nonhomogeneous distribution of the RH and WF fillers in W-PA6G matrix can be seen in Figs. 4b and 4c, respectively. At a certain part of the polymer matrix, fillers were grouped in close distance. This situation might cause to the heterogeneous mechanical properties based on the location samples.

Thermal Properties

Thermal properties are important characteristics for polymers and polymer composites. They play a key role in selecting processing parameters and final product application areas. In this study, DSC and TGA of W-PA6G and their 30% RH and WF filled composites were evaluated. The DSC data of the studied samples are presented in Table 3.



(a) W-PA6G



(b) PA-20RH **(c)** PA-20WF

Fig. 4. SEM images of selected samples at x500 magnification: (a) W-PA6G, (b) PA-20RH, and (c) PA-20WF

Table 3. DSC Data of Recycled W-PA6G-based WPCs

ID	T_m (°C)	T_c (°C)	ΔH (J/g)	X_C (%)
W-PA6G	211.3	176.3	108.5	47.2
PA-30RH	181.8	-[a]	29.14	13.5
PA-30WF	190.5	-[a]	21.8	18.1

[a]: Not detectable in DSC curves; X_c: the crystallinity

Changes in the melting curve after lignocellulosic filler addition are presented in Fig. 5. The lignocellulosic filled composites had broader melting curves compared to the W-PA6G peak. Improved crystal growth provides a narrower peak (Xu *et al.* 2013), but restricting the formation of polymer crystals results in broader peaks (Ying *et al.* 2018). Results showed that the addition of both RH and WF lignocellulosic fillers in W-PA6G matrix reduced the melting temperatures (T_m) of W-PA6G from 211 °C to 182 °C and 191 °C (Table 3). The crystallinity values of the PA30RH and PA-30WF composites (approximately 14% and 18%) were lower than the W-PA6G samples (approximately 47%). Klata *et al.* (2003) reported that glass filament reinforcement reduced crystallinity of PA6 composites. Other researchers reported increased crystallinity in PP-based lignocellulosic composites due to nucleating ability of the lignocellulosic filler (Joseph *et al.* 2003; Arbelaiz *et al.* 2006; Lei and Wu 2010). Joseph *et al.* (2003) produced polypropylene-based composites with 10, 20, and 30% sisal fiber and observed an increase in crystallinity without changing T_m. Similar findings were reported by Arbelaiz *et al.* (2006) for PP-based 30% flax fiber composite (produced samples at crystallization temperature (T_c) of 135 °C and 140 °C). However, almost 9% lower crystallinity compared to PP (X_{cPP}: 57.4% and $X_{cPP+Flax}$: 52.5%) was measured in composites produced with a T_c of 145 °C. It is obvious that material choice and composition, manufacturing parameters, and preferred manufacturing process might affect the crystallinity of the composites. There is a need for further study to investigate the effect of lignocellulosic fiber and their possible synergistic effect on the thermal properties of W-PA6G-based composites.

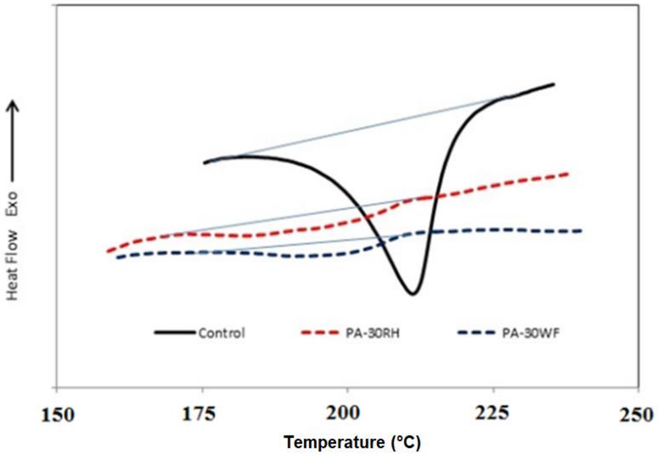


Fig. 5. DSC melting curves of the samples

Thermogravimetric analysis is one of the key thermal analysis techniques to investigate the thermal behavior of carbonaceous materials. It measures the rate of weight loss of the sample as a function of temperature and time, which is utilized to predict the thermal behavior of the material (Mansaray and Ghaly 1998). The TGA and DTGA (derivative thermogravimetric analysis) curves and TGA data of the samples are presented in Figs. 6a and 6b, and Table 4, respectively. The TGA of both RH and WF revealed an initial slight weight loss between ambient temperature and approximately 100 °C. This could be due to the gradual evaporation of absorbed moisture. The onset, endset, and major decomposition temperatures of the RH were 173, 493, and 329 °C, respectively. The temperatures for the WF were 182, 461, and 356 °C. This mass loss was due to the decomposition of the three major constituents of the lignocellulosic fillers (cellulose, hemicellulose, and lignin). Hemicellulose, cellulose, and lignin decomposes in the range of 150 to 350 °C, 275 to 350 °C, and 250 to 500 °C, respectively (Mansaray and Ghaly 1998; Kim *et. al* 2004). At 750 °C, the ash content of RHF (39%) was much higher than that of WF (18.9%). Ash in the RHF is mainly composed of silica (96%) (Kim *et al.* 2004). The mass loss steps of W-PA6G occurred rapidly above 400 °C. Its mass loss started at around 296 °C and was completed at 466 °C with the maximum degradation rate temperature at 432 °C (Fig 8b). A similar result for PA6 was reported by Elsabbagh *et al.* (2017). The results showed that the thermal stability of RHF and WF was lower than that of W-PA6G.

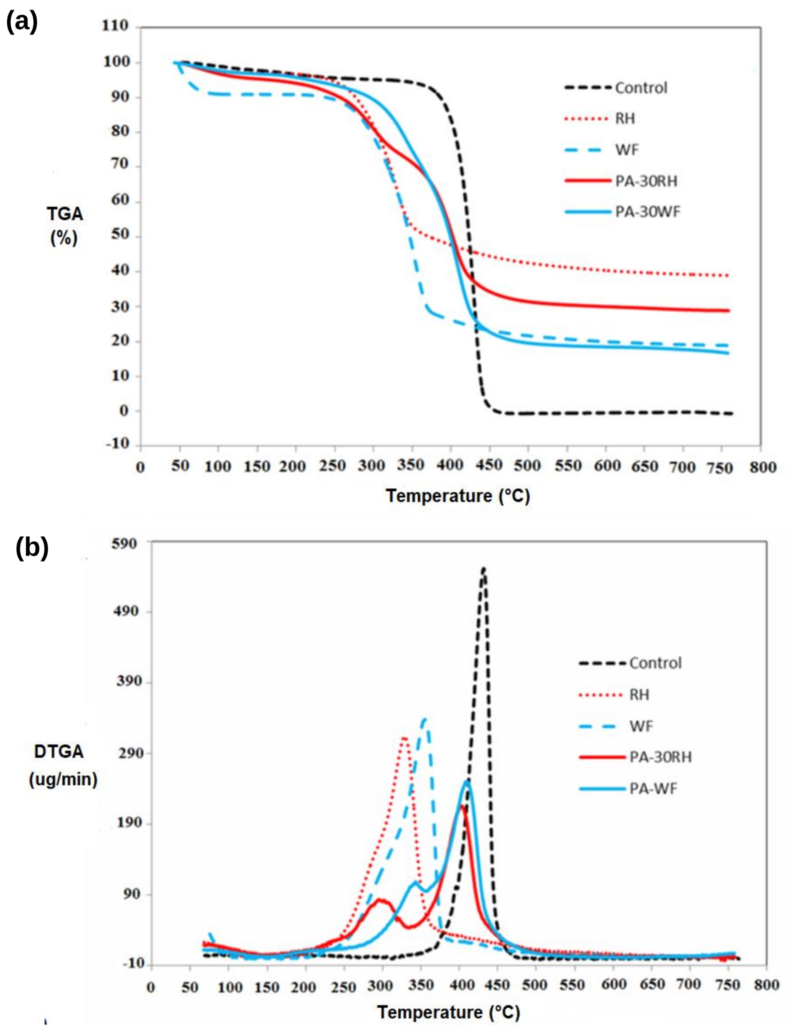


Fig. 6. (a) TGA mass loss and (b) derivative DTGA curves of the samples

Table 4. TGA Data of Recycled W-PA6G-based WPCs

ID		T_{Onset} (°C)	T_{endset} (°C)	T_{decomp} (°C)	Weight Loss at T_{onset} - T_{endset} (%)	Residue at 750 °C (%)
W-PA6G		296.4	465.6	431.6	95.2	-
PA-30RH	*1st Peak*	171.7	327.6	295.0	21.8	28.9
	2nd Peak	327.6	492.0	404.0	43.1	
PA-30WF	*1st Peak*	204.9	348.8	338.6	20.4	16.9
	2nd Peak	348.8	447.5	406.9	52.1	
RH		172.8	492.7	328.6	54.2	39.0
WF		182.3	461.0	355.6	68.2	18.9

In the RH and WF filled W-PA6G composites, two distinct DTGA peaks were detected. Through the addition of RH and WF, it was found that the onset temperature for degradation decreased to 172 °C and 182 °C, respectively. This decrease is related to the thermal degradation of hemicelluloses followed by alpha cellulose and finally by lignin (Elsabbagh *et al.* 2017). The onset-endset temperatures of the second decomposition peaks of RH and WF filled composites were 328 to 492 °C and 348 to 447 °C, respectively. Residue amounts at 750 °C for RH and WF filled composites were around 29% and 17%, respectively.

CONCLUSIONS

In this study, the effect of filler type (FT) and filler content (FC) on the mechanical, morphological, and thermal properties of waste-casting polyamide 6 (W-PA6G)-based composites were investigated. During manufacturing, N-butyl benzene sulfonamide (N-BBSA) and LiCl were utilized as a plasticizer and as melt temperature-lowering salts. The following conclusions were derived:

1. The rice husk (RH) and wood fiber (WF) filled W-PA6G-based composites were successfully manufactured using a combination of extrusion and injection molding.
2. The WF filled W-PA6G-based composites provided better tensile and flexural properties (both strength and modulus) than the RH filled ones at 20% and 30% filler contents. Impact strength values with lignocellulosic filler presence reduced.
3. Lignocellulosic filler presence resulted in reduced melting temperature and crystallinity of the W-PA6G-based composites. This reduction was more pronounced in the RH filled ones.

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