

Influence of Repeated Injection Molding Processing on Some Mechanical and Thermal Properties of Wood Plastic Composites

Alperen Kaymakci ^{a,*} and Nadir Ayırlımış ^b

The influence of repeated cycles of injection molding on some mechanical and thermal properties of wood plastic composites (WPCs) was investigated. Pine wood flour (50 wt%) was mixed with HDPE (50 wt%) containing compatibilizing agent (MAPE, 3 wt%) in a co-rotating twin-screw extruder. The test specimens were produced by injection molding from the pellets dried to the moisture content (MC) of 1%. After the mechanical properties of the WPC test specimens were determined, the failed specimens were subsequently pelletized. These pellets were stored and dried for 4 to 6 h until the repeated injection was molded. These processes were repeated seven times. The results revealed that the flexural strength of WPCs increased by about 5.26% from the original granules to the third injection cycle, but further increments in the number of cycles decreased the flexural strength. The tensile strength and tensile modulus of the WPC specimens increased until the third cycle of injection molding while they tended to decrease after the third injection cycle.

Key words: Repeated injection molding; WPC; Flexural properties; Tensile properties; Thermal properties

Contact information: a: Department of Wood Mechanics and Technology, Forestry Faculty, Kastamonu University, Kastamonu, Turkey; b: Department of Wood Mechanics and Technology, Istanbul University, Bahçekoy, Istanbul, Turkey; *Corresponding author: akaymakci@kastamonu.edu.tr

INTRODUCTION

The production of plastic-based materials has attracted increasing attention due to their relatively low price, low density, easy processing conditions, durability, and good mechanical characteristics. Plastics are used in a number of applications such as coating, wiring, packaging, and in the automotive and construction industries (Kazemi 2013). Also, plastics usually have low mechanical properties, modulus, and strength compared with other materials. Considering the short service life of most plastics, especially in packaging applications, re-using is an option to extend their life cycle. Re-using consumes less energy than other recycling techniques, which makes it a more preferable approach (Martins and De Paoli 2000; Kazemi *et al.* 2007; Jakubowicz and Enebro 2012; Kazemi 2013).

Worldwide technological advancement and industrialization is increasing the consequences of human activities on the environment. It is necessary to reduce the environmental impact of waste. Wood plastic composite is a composite material made of thermoplastics including polypropylene or polyethylene and wood fiber or flour. The thermoplastics and wood can be taken from both virgin and recycled sources. Because of their low density, low cost, non-abrasive, non-toxic, and eco-friendly properties, wood plastic composites have received much attention. Several researchers have investigated the potential use of waste or recycled plastics in WPCs. Some reports suggested that the composites produced from waste or recycled plastics exhibited better mechanical properties than the virgin ones, although recycling by melt reprocessing may cause thermal, oxidative, or mechanical degradation that can change the structural properties of plastics

(Krishnan and Debes 2004; Kazemi *et al.* 2006; Ashori and Nourbakhsh 2009).

The mechanical, physical, thermal, and morphological properties of WPCs have been comprehensively examined in previous papers (Kazemi *et al.* 2006; Adhikary 2008; Ghasemi and Kord 2009; Ayrilmis and Kaymakci 2013; Kaymakci and Ayrilmis 2014). Most reports focus on determining the reinforcing filler types and loading levels, modified fillers, or coupling mechanism on the technological properties of WPCs. Recycling is a broad field and has to be divided into different topics: the use of recycled materials for the production of WPC, the use of process waste (in-plant recycling), and the use of the WPC after their lifetime. This study focused on the second recycling topic, which was the use of process waste in the plant. Extensive research did not reveal any reports concerning the effect of repeated injection molding on the properties of WPCs. The majority of this work was focused on the effect of the number of extrusion cycles. The goal of this research was to investigate the effect of number of injection molding cycles on the some mechanical and thermal properties of WPC.

EXPERIMENTAL

Materials

The high-density polyethylene (HDPE) was used as the matrix material. Petkim (Izmir, Turkey) provided HDPE with an MFI of 5 g per 10 min. Pluss Polymers Pvt. Ltd. (Gurgaon, India) provided maleic anhydride polyethylene, Optim-E156, with a 4.5 g per 10 min MFI and a density of 0.95 g/cm³, which was used as the compatibilizing agent.

Pine wood flour (WF) with a mesh size of 40 was supplied by the WPC Decking Factory in Tekirdag, Turkey. The wood flour was dried in a laboratory scale furnace at 100 °C for 24 h to a MC of 1 to 2%.

Production of WPCs

The WF (50 wt%) and the HDPE (50 wt.%) with MAPE (3 wt.%) granulates were processed in a twin-screw co-rotating extruder. The temperatures of the zones and the die were set at 165 °C, 170 °C, 180 °C, and 190 °C. The screw speed was kept at 4 rpm to avoid high pressure due to the excessive amount wood flour content. The extruded strand was then pelletized, and the pellets were stored in a polyethylene bag and dried (1 to 2%) for 4 to 6 h. The specimens were prepared using an injection molding machine (model TSPX 60, TSP, Zhejiang, China). The temperatures of the injection molding machine were set at 170 to 190 °C. The samples were injected at a pressure of 5 N/mm² with a chilling time of about 25 s. To examine the effect of the number of cycles on the flexural and tensile properties of recycled WPC, the injection molding process was repeated seven times without adding any raw materials or other additives.

After each cycle of injection molding, the WPC test specimens were ground using a granulator to obtain small pellets. These pellets were again injection molded into specimens, and the flexural and tensile properties of the specimens acclimatized at a temperature of 23 ± 2 °C and RH of 50 ± 5% ISO 291(2008) were determined.

Determination of Flexural and Bending Properties

Flexural modulus and strength of the injected molded WPC specimens were measured accordance to ISO 178 (2010) standard. The tensile properties of the samples were examined with ISO 527-1 (2012). Ten specimens were measured for the both the tensile and flexural properties.

Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC)

The injected pellets were ground with a Willey mill prior to analysis. Thermogravimetric analysis of the samples was done by using the Perkin Elmer STA 6000 thermal analyzer. For the TGA test, samples (3 to 5 mg) were heated in an aluminum pan up to 600 °C with the heating rate of 10 °C/min and kept at this temperature for 2 min to monitor thermal history. Additionally, melting and crystallization behavior of the WPCs were studied in a heat-flux type differential scanning calorimeter (DSC, Perkin Elmer DSC 4000) according to ASTM D3418. The test samples weighing about 9 to 10 mg in an aluminum crucible were heated to 250 °C with the rate of 10 °C/min and kept at this temperature for 2 min to remove thermal history. Then the samples were cooled to 0 °C with the rate of 10 °C/min by an electrical cooling device. All heating-cooling runs in melting and crystallization studies were carried out under nitrogen (N₂) atmosphere at a flow rate of 30 mL/min to prevent oxidation of the samples.

Statistical Analysis

An analysis of variance was conducted to examine the effect of repeated injection molding processes on the tensile and flexural properties of WPCs by utilizing the SPSS statistical package program (SPSS 17.0, Chicago, USA). Significant ($p < 0.05$) differences between mean values of the WPC cycles were found using Duncan's multiple range test.

RESULTS AND DISCUSSION

Mechanical Properties

The effect of the number of injection molding cycles on the flexural and tensile properties of recycled WPC is demonstrated in Figs. 1 through 4. The flexural strength of WPCs increased by 5.26% from the original granules to the third injection cycle, but further increment decreased the flexural strength of WPC samples (Fig. 1). The flexural strength of the specimens that were reinjected four times did not change significantly from the control group.

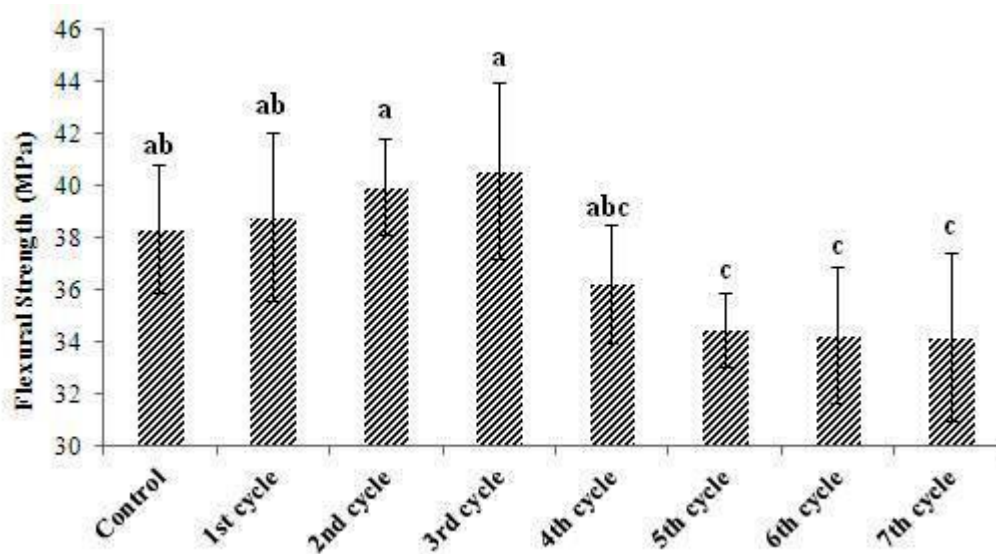


Fig. 1. Flexural strength of test specimens after repeated injection molding cycles (the different letters on the bars of the standard deviations show the significant differences ($p < 0.05$) among the WPC groups)

A similar tendency was observed for the flexural modulus (Fig. 2). The flexural modulus of the samples increased by 2.0% from the original granules to the third injection molding cycle, but then decreased by 5.3% as the number of cycles reached seven. As the cycles increased from the third to the fourth injection, the flexural strength and modulus were adversely affected, decreasing from 40 to 36 MPa and 4763 to 4589 MPa, respectively. The flexural strength and flexural modulus decreased to 34 MPa and 4567 MPa, respectively, as the number of reinjection cycles increased from four to five. There was no significant difference in the flexural properties between the fourth and seventh cycle in the injection molding.

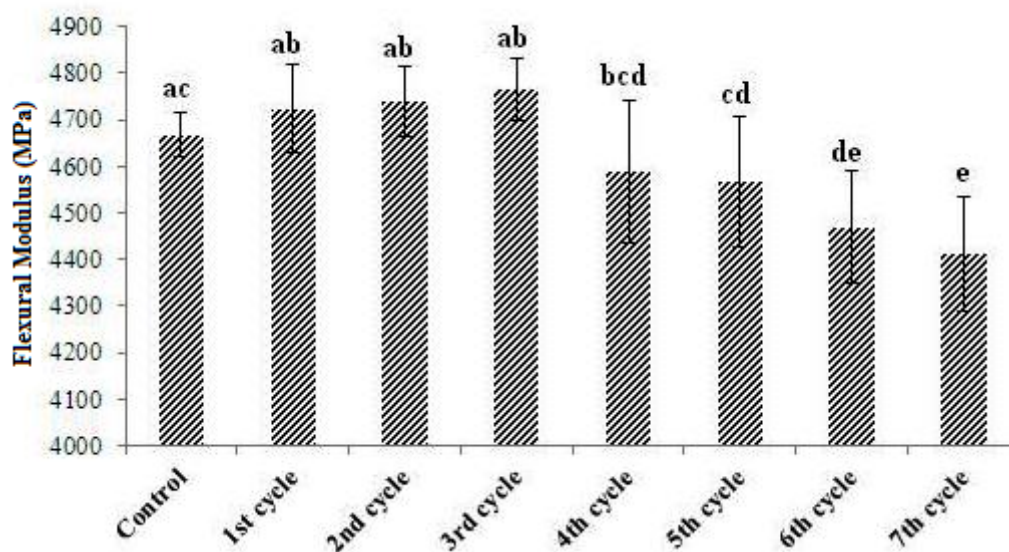


Fig. 2. Flexural modulus of test specimens depending on the number of injection molding cycles (the different letters on the bars of the standard deviations show the significant differences ($p < 0.05$) among the WPC groups)

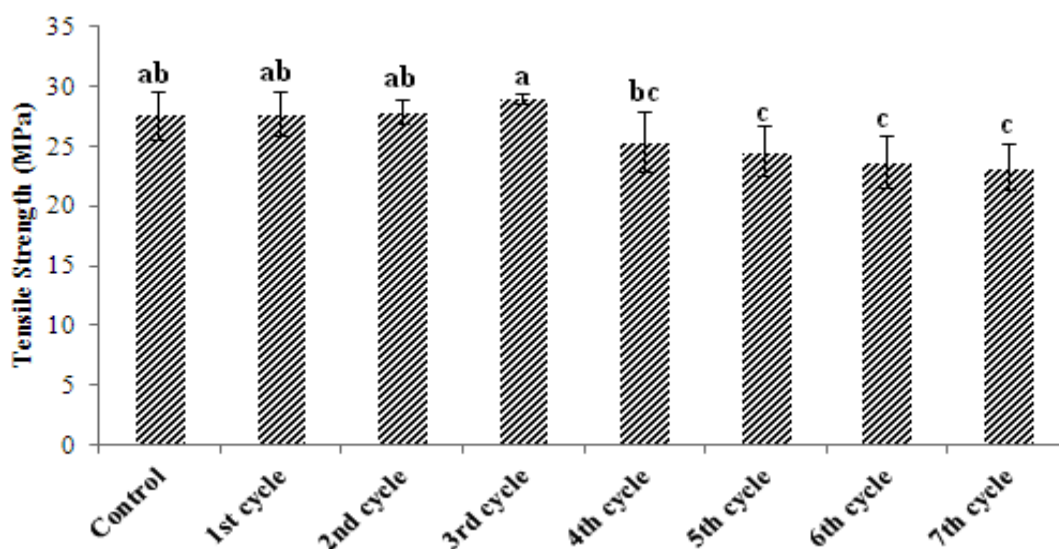


Fig. 3. Tensile strength of the test specimens depending on the number of injection molding cycles (the different letters on the bars of the standard deviations show the significant differences ($p < 0.05$) among the WPC groups)

The tensile strength and modulus of the specimens increased until the third cycle of injection molding. The tensile strength and modulus showed a tendency to decrease after the third injection cycle. The tensile strength of specimens increased by about 3.70% from the original granules to the third injection cycle (Fig. 3). However, the tensile strength decreased by 14.3% after the third cycle to the seventh.

The tensile modulus test results were similar to tensile strength (Fig. 4). The tensile modulus increased by 1.05% from the original granules to the third injection cycle, but then decreased by 6.61% from the third cycle to the seventh.

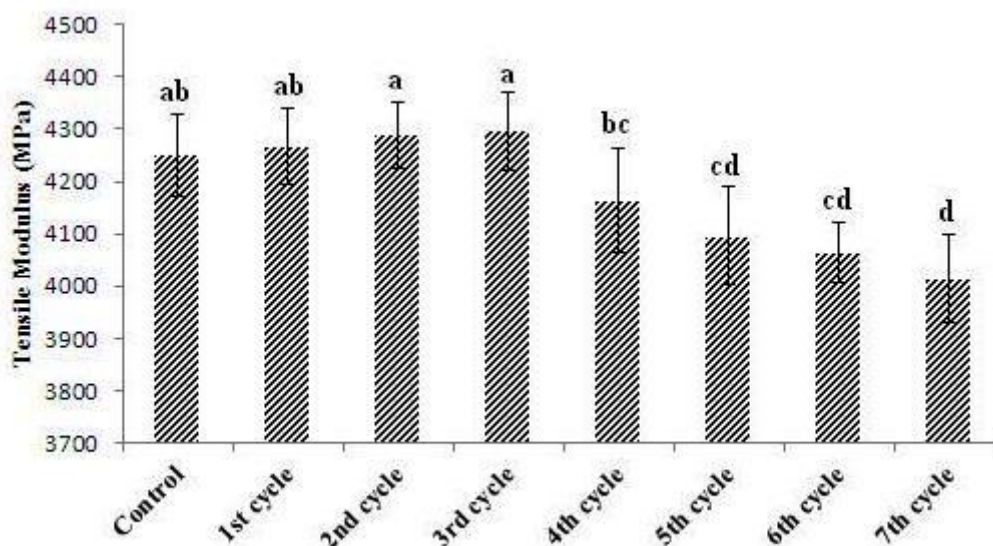


Fig. 4. Tensile modulus of test specimens depending on the number of injection molding cycles (the different letters on the bars of the standard deviations show the significant differences ($p < 0.05$) among the WPC groups)

The increase in tensile properties of the specimens in the early steps of the re-injection stage was partly explained by the re-polymerization process. In the early stages of re-injection, the tensile properties of the samples improved, which was attributed to the generated crosslinking between the chains of polymer matrix. This increased the crystallinity of the polymer matrix, which improved the tensile strength and modulus of the WPCs (Cornier-Rios 2003). Oikonomidou *et al.* (2012) reported that the polymer macromolecules were exposed to high temperatures and intensive shearing during extrusion reprocessing, which promoted mechanical properties of the WPCs. The decreased mechanical properties of the WPCs above the third cycle can be attributed to the thermo-oxidative or thermo-mechanical degradation of the polymer matrix.

The reduced mechanical properties of specimens manufactured after the third injection molding cycle also could be related to the degradation mechanism of wood particles under high pressure and temperature; wood components are exposed to thermal degradation during WPC reprocessing (Liu *et al.* 2009). The thermal degradation of wood resulting from high operating temperatures and reprocessing (re-extrusion and re-injection) may lead to adverse properties, such as decreased mechanical properties.

The flexural and tensile properties of the WPCs increased until the third cycle of injection molding. This result is explained by better wood flour dispersion and wetting (TabkhPaz *et al.* 2013). Enhanced wood flour dispersion and wetting of the HDPE resulted in superior mechanical properties. Interfacial adhesion was improved by adding coupling agent (MAPE) between wood flour and HDPE, which lead to less micro-voids and wood

flour-HDPE debondings in the interphase region. This behavior is attributed to the reaction of the hydrophilic hydroxyl groups from the wood flour and the acid anhydride groups from MAPE, leading to the formation of ester linkages (San *et al.* 2008).

The thermal and mechanical degradation of the components affected the ultimate properties of the specimens. Thermal properties and shear histories of polymer and wood particles change depending on the number of injection molding cycles (TabkhPaz *et al.* 2013). The improvement of the mechanical properties of the samples after the third injection molding cycle reflects that the repeated shear histories enhanced the dispersion of wood filler in the matrix. However, the reduction in the mechanical properties of the specimens beyond the third cycle was mainly attributed to the thermal degradation of wood flour. Wood flour contains cellulose, hemicellulose, lignin, and extractive materials. Each component contributes to the total properties of the wood flour or fillers. The holocellulose, α -cellulose, and lignin contents of pine (*Pinus nigra*) are 64.7%, 35.5%, and 33.0%, respectively (Akgul *et al.* 2010). The chemical structure of wood can change, and elevated temperatures affect its properties. Cellulose content is primarily responsible for the tensile and flexural properties of wood fiber; therefore, decreasing the degree of polymerization reduces the tensile and flexural properties of wood (Sweet and Winandy 1999). Also, many extractives such as alcohols, resins, terpenes, formic acid, and acidic acid are released from the wood (Manninen *et al.* 2002; Graf *et al.* 2003).

Because there was no comparable research found in the search of the literature, the findings of this study were compared with studies of WPCs reproduced by re-extrusion (Yarahmadi *et al.* 2001; Delva *et al.* 2014). In these studies, the properties of WPCs manufactured with the re-extrusion process were reduced with an increase in the number of re-extrusion cycles. For example, in a previous study, the tensile modulus of PP composites was 21.9 N/mm², while the tensile modulus of PP composites after 7 extrusion cycles was 21.1 N/mm². Yarahmadi *et al.* (2001) reported that the elongation at break increased by 13% from the first to the third extrusion cycle, which showed a clear correlation between the optimum level of elongation and the maximum mechanical properties obtained. The findings in this study were consistent with the results of the WPCs manufactured with repeated extrusion processing.

Thermal Properties

The effect of the number of injection molding cycles on the TGA of recycled WPC is presented in Table 1. The onset temperatures of WPCs increased by 1.33% from the original granules to the third injection cycle, but further increment in the injection cycle decreased the onset temperatures of WPC samples (Table 1). The same tendency was found in the peak temperatures, weight loss, and residue after 500 °C. These results were mainly attributed to the drop in the molecular weight induced by reinjection processing (La Mantia *et al.* 2002). The weight loss (3rd peak) of the specimens increased to 62.5% in the first three cycles of cyclic loading. Further increment in the reinjection cycle decreased the weight loss of the WPCs. The increment in the weight loss until the third cycle could be related to the thermal degradation of the wood flour and polymer matrix. The residue contents of the WPCs showed a similar trend to the results of the weight loss. The residue content of the WPC increased from 11.6% to 13.4% at the end of the third reinjection cycle. This was found to be 9.8% in the WPC specimens reinjected seven times. The peak temperatures decreased as the number of reinjection cycles increased.

Heat treatment of wood in the barrel of an injection molding machine produced intensively degraded hemicellulose. In addition, heat treatment either removes or changes the structure of the most sensitive components of wood (Kaboarani 2009; Xing and Li 2014).

Thermal degradation of the HDPE started at a temperature greater than 400 °C and degraded with much higher speed than wood. The first decomposition peak temperature of the wood was around 200 °C. The initial degradation temperature of wood was much lower than HDPE, but the speed was much slower. It was expected that heating wood at high temperatures decomposed hemicelluloses to a large extent, and cellulose and lignin to a lesser extent. As a result, the decomposition rate of the WPCs decreased after the third cycle, which should give wood more thermal stability (Table 1).

Table 1. Results of Thermogravimetric Analysis (TGA)

WPC groups	Onset Temperature (°C)	Peak Temperature (°C)			Weight Loss (%)			Residue after 500 °C (%)
		1 st peak	2 nd peak	3 rd peak	1 st peak	2 nd peak	3 rd peak	
Control	420.0	270.0	350.0	470.0	9.8	18.6	58.7	11.6
1st Cycle	421.1	270.7	351.4	470.9	9.9	19.2	59.2	12.1
2nd Cycle	422.8	272.3	352.7	473.4	10.2	20.7	60.6	12.8
3rd Cycle	425.6	274.4	354.4	475.3	11.4	22.2	62.5	13.4
4th Cycle	419.8	269.7	349.7	469.5	9.7	18.5	58.5	11.4
5th Cycle	418.4	268.4	348.5	467.7	9.5	18.1	57.6	10.9
6th Cycle	416.5	266.6	346.7	466.1	9.4	17.4	56.1	10.7
7th Cycle	414.7	262.7	344.1	463.2	9.1	16.3	54.8	9.8

The DSC results of the WPCs are given in Table 2. The second melting peak temperature (T_m) values of the WPCs increased from 128.4 to 131.5 °C as the WPCs were reinjected three times. However, it decreased to 125.6 °C after the third cycle to the seventh. The increase in the T_m in the reinjected WPCs until the third cycle may be due to disruption of the HDPE crystal lattice network by the presence of wood particles. According to the results of the second melting temperature, the crystalline structure of the polymer remained unaltered for the first injection cycle.

Crystallization peak temperature and crystallization enthalpy of the WPCs increased with increasing reinjection cycle up to the third cycle, but further increment in the cycles decreased the crystallization peak temperature and crystallization enthalpy (Table 2).

The degree of crystallinity in HDPE is typically in the range of 60 to 80% (Klyosov 2007). The degree of crystallinity of the specimens increased from 42.5% to 49.6% up to third cycle, but it decreased to 38.6% from the third to the seventh cycle. High crystallization temperature and crystallinity are interesting properties for thermoplastic processors since they reduce the production time, saving costs (Taflick *et al.* 2015). As the WPCs were injected three times, the second melting enthalpy increased from 59.8 to 70.6 J/g, but it decreased to 56.2 J/g as the number of the reinjection cycles was increased from three to seven.

Table 2. Results of Differential Scanning Calorimetry (DSC) Analysis

WPC groups	Second melting enthalpy (ΔH_m) (J/g)	Second melting peak temperature ($^{\circ}\text{C}$) (T_m)	Crystallization peak temperature T_c ($^{\circ}\text{C}$)	Crystallization enthalpy (ΔH_c)	X_c degree of crystallinity (%)
Control	59.8	128.4	117.8	58.7	42.5
1st Cycle	64.5	128.6	118.5	59.8	44.6
2nd Cycle	67.7	129.2	119.7	60.9	47.9
3rd Cycle	70.6	131.5	122.5	62.1	49.6
4th Cycle	59.3	127.6	117.1	57.6	41.2
5th Cycle	58.5	127.2	116.4	57.1	40.6
6th Cycle	57.4	126.4	115.8	56.4	39.5
7th Cycle	56.2	125.6	114.7	55.7	38.6

CONCLUSIONS

1. The flexural strength and modulus of wood-plastic composites (WPCs) increased by approximately 5.26% and 2.0% from the first to the third injection cycle, respectively, but a further increase in the number of cycles decreased the flexural strength and modulus.
2. The tensile strength and modulus properties of the WPCs increased by 3.70% and 1.05% until the third cycle of injection molding, respectively. The tensile properties showed a tendency to decrease after the third injection cycle.
3. Based on the results obtained from this research, the flexural and tensile properties of the WPCs increased until three cycles of injection molding. In particular, the results of this study are important for WPC manufacturers because each product has by-products from sprue and runner in the mold during the injection molding process. For this reason, WPC product and its by-products can be processed three times without deterioration during in-plant recycling.
4. The second melting enthalpy, second melting peak temperature, crystallization peak temperature, crystallization enthalpy, and degree of crystallinity of the WPCs increased with repeated injection up to three cycles, but they decreased from the third to seventh injection cycle.

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