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Comparison of Wood Fiber Reinforced PLA Matrix Bio-Composites Produced By Injection Molding and Fused Filament Fabrication (Fff) Methods

Enjeksiyon Kalıplama ve Erimiş Filament Ekstrüzyonu (Efe) Yöntemleri Ile Üretilen Ahşap Elyaf Takviyeli PLA Matris Biyo-Kompozitlerin Karşılaştırılması

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ABSTRACT

The Fused Filament Fabrication (FFF) method is the most popular method preferred for shaping polymers with AM. FFF is known to have low cost and high printing speeds compared to other AM techniques. PLA material, which is a completely bio-based thermoplastic polymer with many desirable properties, including easy processing ability, strength, hardness, and biodegradability, is widely used in material processing by the FFF method. In this study, the PLA matrix was reinforced with natural fibers to increase the mechanical properties and contribute to recycling. Bio-composite compounds with 15 wt.% wood fiber reinforced PLA matrix was prepared using a twin-screw extruder. Test specimens were produced the FFF method and injection molding method. Thermal analysis of the prepared compounds, filaments, and produced specimens was carried out. A decrease in the Tg value of the compound reinforced with natural fiber was observed, while an increase in the Tm value was observed. The Tg value of the specimens produced by the FFF method increased compared to the injection specimens. It was determined that the stress at break values of the specimens produced by injection were 2 times higher than the specimens produced by FFF. The impact strength of the specimens produced with injection molding is 51.75% higher than the specimens produced with FFF. The bio-composite materials produced in the study were examined under scanning electron microscopy (SEM). Surface interactions and homogeneous fiber distribution between matrix and fiber were investigated.

Key Words

PLA, biomaterials, additive manufacturing, wood fibers.

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ÖZ

Polimerlerin eklemeli imalat (Eİ) ile şekillendirilmesinde tercih edilen en popüler yöntem ise erimiş filament ekstrüzyonu (EFE) yöntemidir. EFE, diğer 3D baskı tekniklerine kıyasla düşük maliyetli ve yüksek baskı hızlarına sahip olduğu bilinmektedir. Kolay işleme kabiliyeti, mukavemeti, sertliği ve biyolojik olarak bozunabilirliği dahil olmak üzere birçok arzu edilen özelliğe sahip tamamen biyo-bazlı bir termoplastik polimer olan PLA malzemesi EFE yöntemi ile malzeme işlemede yaygın olarak kullanılır. Bu çalışmada mekanik özellikleri arttırmak ve geri dönüşüme katkı sağlamak amacıyla PLA matris doğal fiberler ile güçlendirilmiştir. Ağırlıkça %15 ahşap fiber takviyeli PLA matrisli biyo kompozit bileşimler hazırlanmıştır. Test numuneleri EFE yöntemi ve enjeksiyon kalıplama yöntemleri kullanılarak üretilmiştir. Hazırlanan bileşimlerin, filamentlerin ve üretilen numunelerin termal analizleri gerçekleştirilmiştir. Doğal fiber ile güçlendirilen bileşimlerin Tg değerinde azalma Tm değerinde ise artış gözlemlenmiştir. EFE yöntemi ile üretilen numunelerin Tg değeri ise enjeksiyon numunelere göre artmıştır. Enjeksiyon ile üretilen numunelerin kopma gerilmesi değerlerinin EFE ile üretilen numunelere göre 2 kat yüksek olduğu belirlenmiştir. Enjeksiyon kalıplama ile üretilen numunelerin darbe dayanımları ise EFE ile üretilen numunelere göre %51,75 daha yüksektir. Çalışmada üretilen biyo-kompozit malzemeler taramalı elektron mikroskobu (SEM) altında incelenmiştir. Matris ve lif arasındaki yüzey etkileşimleri ve homojen lif dağılımı incelenmiştir.

Anahtar Kelimeler
PLA, biyomalzemeler, eklemeli imalat, ahşap lifleri.

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INTRODUCTION

New legal regulations developing in the world create a demand for new sustainable materials [1]. The destruction of non-biodegradable petroleum-based polymers in the environment, the depletion of non-renewable petroleum resources, and increasing concerns about dependence on petroleum cause this demand [2].

Increasing trends in this area highlight green bio-composites with low environmental impact. For this reason, research is carried out on the development of biodegradable polymers and thus, petroleum-based polymers are replaced by biodegradable polymers [3,4]. Bio-composites, a combination of bioplastic matrix and natural fibers reinforcements, have emerged as promising alternatives to traditional petroleum-based polymer and synthetic fiber-containing composites. They offer a wide range of advantages such as lower density, renewability, biodegradability, and a better cost per requirement [2,5–7].

There has been a lot of recent research on biodegradable polylactic acid (PLA), produced from renewable resources. PLA is mostly produced from crops, usually corn, wheat, or sugarcane, by fermentation into lactic acid followed by polymerization. In addition, natural fibers as an alternative reinforcement to synthetic fibers such as glass and carbon fiber in polymer matrix composites have attracted the attention of many researchers and scientists due to several advantages [8]. These natural fibers include flax, hemp, kenaf, jute, coconut, bamboo, and the like [9]. Natural fibers have many advantages when compared to synthetic fibers. These are low in cost and density, comparable specific tensile strength, non-abrasive, non-irritating to the skin, production consuming less energy, less health risk, renewability, recyclability, and biodegradability [10]. In addition, thermoplastic polymers are combined with natural fibers to reduce production costs while maintaining their properties [11]. Studies use reinforcing materials such as wood fibers, wood chips, cellulose fibers, and cellulose nano by using PLA matrix, a biodegradable polymer [11–16]. In addition, plant-derived fibers such as flax, hemp, kenaf, and bamboo fibers have been used in various studies to strengthen PLA [2,7,12,17–21].

It is known that natural and artificial cellulose fibers positively affect the mechanical properties of PLA matrix composites [6]. Many factors can such as the hydrophilic properties of natural fibers, fiber reinforcement ratio, fiber content, surface treatment, and production method affect the performance of natural fiber reinforced polymer composites [5].

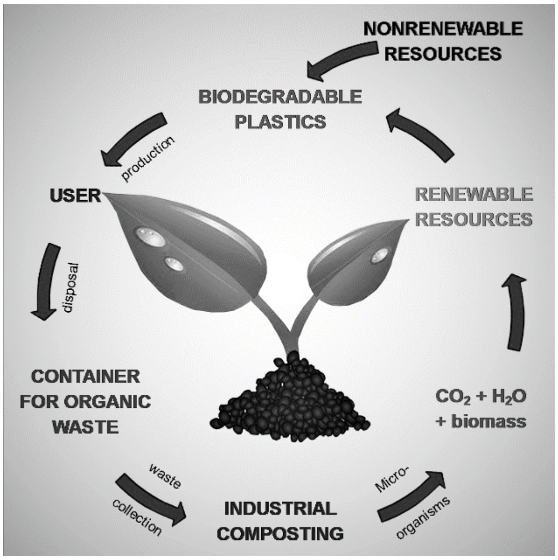

Figure 1. The life cycle of PLA.

One of the recent promising methods of processing polymer materials is additive manufacturing technologies. Fused Filament production (FFF), also known as one of the 3D printing techniques, is the most widely used additive manufacturing method. The FFF method is a production process that produces computer-aided drawing data layer by layer in slices with the polymer that it extrudes from a heated nozzle in a melted state. Additive manufacturing technologies can provide numerous benefits such as rapid production, assembly-free production, low labor costs, the ability to produce complex geometries, and limitless design options [22].

Increasing the fiber ratio in composite materials ensures high performance. It is known that an increase in fiber ratio leads to an increase in tensile strength properties [5]. In the production with the FFF method, the increase in the fiber ratio causes deterioration in the structure of the filament raw material used in the production after a certain level. In addition, some testing values can remain constant above a certain rate [23,24]. For the extrusion of the filament raw material to be used in the FFF method, the fiber ratio should be optimized depending on the properties of the matrix and the reinforcement element.

For parts produced by additive manufacturing methods to be used as final products, their mechanical properties must be similar to those produced by conventional manufacturing methods (for example, injection molding) [25]. One of the most used methods of polymer production in the industry is injection molding [26]. It is expected that the FFF method will provide similar properties to alternative methods. With the FFF method, it is possible to produce short fiber reinforced polymer matrix filaments.

Wood fiber is the most easily accessible fiber among natural fiber reinforcements. It has also been the subject of studies in many different fields. For example, in the study of Xu et al, the effect of wood fiber reinforcement produced from pinewood waste on autoclaved aerated concrete was investigated. The comparison was made with autoclaved aerated concrete reinforced with polyester fiber. It has been observed that wood fiber reinforcement has a positive effect on flexural strength and increases thermal conductivity [27].

Csizmadia et al worked on wood-reinforced PLA matrix composites. Chemical treatment was performed on wood fibers with phenolic resin. They observed a significant increase in the strength of the composites produced by extrusion and a decrease in water absorption [19]. The results of the addition of alkenyl succinic anhydride (ASA) surface-treated calcium carbonate to a 40 wt.% wood fiber reinforced PLA matrix were investigated by Ozyhar et al. The benefits of surface-treated minerals compared to untreated minerals, thermomechanical properties of PLA composites, weight variation was investigated. Surface-treated mineral reinforcement was found to improve wood fiber construction with PLA[20]. Migneault et al, on the other hand, investigated the effects of different fiber surface chemical properties on mechanical properties of 40 wt.% wood fiber reinforced PLA matrix bio-composites. The specimens were produced by injection molding. They determined that fibers with a carbohydrate-rich surface have better interfacial bonding [28].

Guo et al tested the biodegradability of modified wood fiber, unmodified wood fiber reinforced PLA matrix composites, and pure PLA products produced by hot press. The mechanical strengths, molecular weights, thermal properties, and microstructures of the specimens, which they kept buried in the soil for 6 months, were examined. They concluded that wood-containing specimens deteriorated faster [29]. Neighborhood et al also conducted a life cycle assessment (LCA) of PLA bio-composites with wood fiber reinforcement [13].

Ashori et al examined the effect of wood fiber reinforcements in a different matrix. They used as a matrix recycled high-density polyethylene (rHDPE) and polypropylene (rPP) matrix and as a reinforcement recycled (rONP) fibers from old newspapers. They measured the mechanical properties and water absorption capacity of the specimens produced by hot press [30].

In addition to all these studies, studies have been carried out to produce PLA matrix wood fiber reinforced bio-composites by additive manufacturing method [12,16,22,31]. Ayrilmis et al produced lattice structures of wood fiber reinforced PLA matrix bio-composites by the FFF method in their study. They investigated the changes in mechanical properties of these lattice structures produced in different surface thicknesses. It was stated that Brinell hardness increased in specimens with a surface thickness greater than 2 mm, and all mechanical properties were significantly improved with an increase in surface thickness [22]. Freedom of design, which is one of the most important features of additive manufacturing methods, allows the geometry of the internal structure of the part to be shaped during production. Kain et al investigated the effect of different angled filling patterns on mechanical properties in the production of wood fiber reinforced PLA bio-composites by the FFF method. Kain et al. to observe the effect of different fiber ratios, two different specimens were produced as 15 wt.% and 25 wt.% by weight. It was determined that a higher fiber ratio exhibited better mechanical properties. While the highest tensile strength was determined as 15, 30 degrees in different angled infill patterns, high strength was observed in 0-degree specimens in the impact test comparison [31]. In a similar study, Liu et al. PLA matrices reinforced with wood, ceramic, copper, aluminum, and carbon fibers separately and their mechanical properties were compared. The effects of different PLA composites, structure orientations, and angled infill patterns (raster angles) on mechanical properties were analyzed [16]. Antonio et al. fatigue analyses of the specimens produced by the FFF method in different parameters using a commercially available wood-reinforced PLA matrix filament were investigated. Optimal parameters required for production in the FFF method, layer thickness, filling ratio, nozzle diameter, and extrusion speed were determined [12]. This study was used to determine the production parameters.

Most of the research has focused on the effects of fiber ratio on mechanical properties, chemical modifications to improve fiber/polymer compatibility, and the effects of FFF production parameters. In this study, the mechanical properties of the specimens that used wood-reinforced PLA matrix bio-composite compounds produced with FFF and injection molding were compared. Specimens were produced using the FFF method and injection molding method with the compounds prepared with 15 wt.% wood reinforced PLA matrix by weight. Impact strength, tensile strength and thermal analysis results, morphological characterization data, and changes in physical properties of the produced specimens were investigated.

MATERIALS and METHODS

Materials

In this study, 15 wt.% wood fiber reinforced PLA matrix material (WR15 PLA) was used. These bio-composite compounds were produced by the Eurotec company. The density of the WR15 PLA is 1.26 gr/m^3 and the melting temperature is 170-190 °C. 15 wt.% of beechwood fibers are added to the PLA matrix.

Preparation of WR15 PLA compound

Wood-reinforced PLA matrix bio-composite compounds were produced in an 18mm diameter twin screw compounder. Pre-drying was carried out at 85°C for 4 hours. The feed zone is 25°C, melt zone is 170°C, mixing & conveying is 190°C and die head temperature is 200°C.

Preparation of WR15 PLA filament

Filament production was carried out in a single screw extruder using WR15 PLA compounds. The diameter of the filaments is 2.85mm (-/+0.15). As seen in Figure 3, the extruded material was wound onto the reel without any problems. In the extrusion process, pre-drying was performed at 85°C for 2 hours. Feed-zone temperature is 25°C. The Melt zone is 180°C and died head temperature is 200°C.

Figure 2. WR15 PLA compound.

Figure 3. WR15 PLA filament.

Production of WR15 PLA by Injection Molding

Test specimens were produced using an Arburg injection molding device and WR15 PLA compounds. To compare the mechanical properties, Charpy test specimens by the ISO 179 standard shown in figure 4 and 1A type tensile test specimens by the ISO 527 standard appearing in figure 5 were produced. In the injection molding process, the drying process was carried out until the humidity of 200ppm was achieved and lasted for 5 hours at 85°C. Mold temperature was 30°C, melt temperature was 180°C, holding pressure time was 15s, holding pressure was 900 bar and cooling time was 20 hours.

Figure 4. Charpy test specimens.

Figure 5. Tensile test specimens.

Figure 6. Tensile test specimens.

Figure 6. Tensile test specimens.

Production of WR15 PLA by FFF

The specimens were produced by the FFF method using WR15 PLA filaments. Tensile test specimens of type 1A by the ISO 527 standard seen in figure 6 in two different orientations and Charpy test specimens by the ISO 179 standard appearing in figure 7 in two different orientations were produced. The specimens were produced with infill patterns at two different angles. The first of them is -/+45 and the other is 0/90. The production parameters in the FFF method are as follows: Layer height 0.2mm, wall thickness 0.8mm, nozzle diameter 0.8mm, infill density 100%, nozzle temperature 210°C, flow 105%, and print speed 50mm/s.

Thermal Characterization

The glass transition temperature (Tg) and melting temperature (Tm) of WR15 PLA specimens, that were produced in different production methods, were measured by Differential Scanning Calorimetry (DSC) analysis. In addition, thermogravimetric analysis (TGA) was performed to determine the degradation temperatures and additive amounts.

Mechanical Testing

Tensile and Charpy tests were carried out on the specimens produced in the infill pattern at different angles in the FFF method and the others produced in the injection molding method. The Charpy test was performed with Instron Models CEAST Resil Impactor device in figure 8 by ISO179 standard. Tensile tests were carried out with the ZwickRoell Z050 device according to ISO 527 standard. The specimens produced in type 1A were tested.

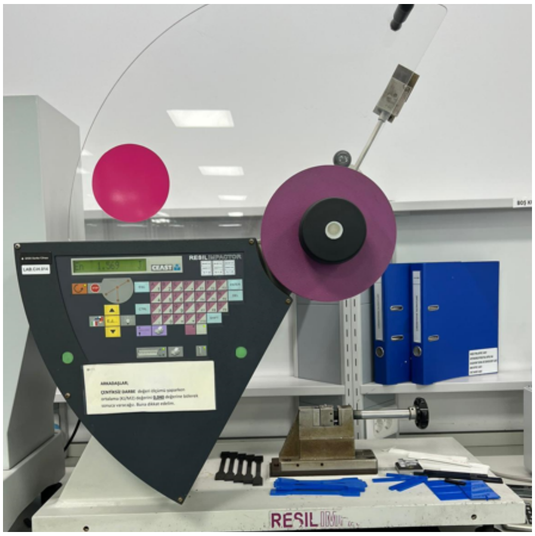

Figure 8. Charpy test device.

Figure 9. Tensile test device.

SEM

Image analyzes were performed using a scanning electron microscope (SEM) with a Carl Zeiss 300VP device. Imaging was done at x-500-700-1000-2000 magnification and 15kV energy. After the mechanical test, the damaged parts of the specimens produced by injection molding and produced by the FFF method, with an infill pattern with an angle of -/+45 degrees and an infill pattern with an angle of 0/90 degrees, were examined.

RESULTS and DISCUSSION

SEM

In SEM analysis, it was observed that wood fibers exhibited homogeneous distribution in WR15 PLA specimens produced by injection molding. However, as can be seen in figure 10, it has been determined that the wood fiber lengths and diameters are in a wide range. Lengths of 372-74 and diameters in the range of 118-53µm were observed. The length of the fiber lengths limits the nozzle diameter, especially in the FFF method[32]. For this reason, a nozzle with a diameter of 0.8mm was used in the FFF method, since there are wood fibers of approximately 400µm in length.

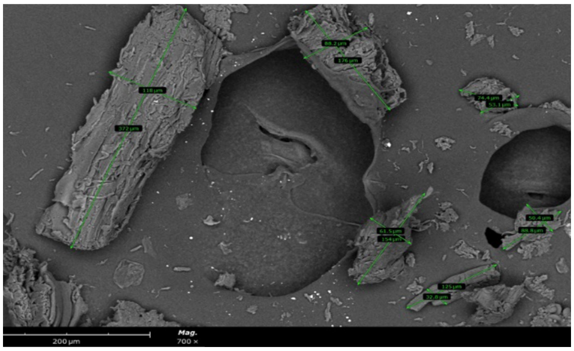

Figure 10. Charpy test device.

It was observed that the fiber orientation in the specimens produced with FFF was parallel to the filling pattern angle. This situation provides an increase in mechanical properties [33,34]. It was observed that the couplings between the fiber and the matrix were good, but there was insufficient bonding between the layers. Insufficient bounding between layers in the FFF method was similar to literature studies [35,36]. To increase the bonding between layers, it is necessary to reduce the layer thickness and use a smaller diameter nozzle [37]. In this study, fiber lengths limited the nozzle diameter.

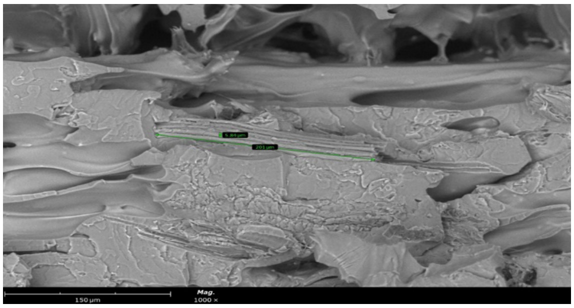

Figure 11. SEM images of FFF Specimen (-/+45)

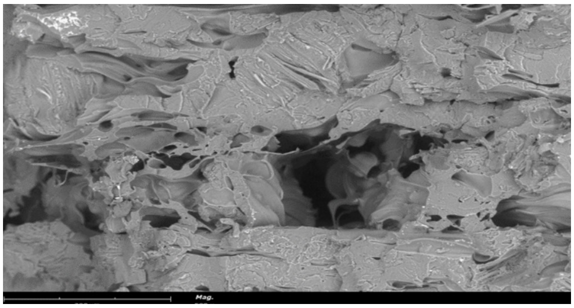

Figure 12. SEM images of FFF Specimen (0/90)

DSC and TGA

DSC analyzes and TGA analyzes of unfilled PLA, PLA compounds reinforced with 15 wt.% wood fiber, and parts of these compounds produced by injection molding and FFF methods were performed. DSC analyses of Unfilled PLA and WR15 PLA specimens are shown in Figure 13. The Tg value, which was 67.37°C in unfilled PLA, decreased to 60.9°C with wood fiber reinforcement. While Tm was 172.27°C in unfilled PLA, it increased to 174.8°C in the WR15 PLA specimen. The high heat transfer value of wood fibers compared to the matrix has created this change. DSC analyzes of the specimens produced by injection molding and FFF methods are given in figure 14. No change was observed in the Tg value of the specimens produced by injection molding, and it was measured as 60.47°C. In the specimens produced with FFF, the Tg value increased to 63.67°C. The low density and porous structure of the specimens produced with FFF caused an increase in the Tg value. Tm values were measured at 174°C, they are the same as the compound values in both production methods. Since the specimens produced in different orientations (-/+45 and 0/90) by the FFF method had undergone the same thermal processes, no difference was observed in the thermal properties of these orientations.

When TGA analyzes are examined, as seen in figure 15, deformation started at lower temperatures in compounds reinforced with wood fiber. The value of 332.71°C in Unfilled PLA is 308.19°C in WR15 PLA compounds. It is an expected situation that natural fiber reinforcement reduces the degradation temperature, as is known from the studies in the literature[38]. It can be said that solid fiber structures accelerate the degradation process. TGA analyzes of the specimens produced by injection molding and FFF are given in figure 16. Specimens produced with FFF change form at lower temperatures because they pass through the filament production stage. The value, which was 314.42°C in the specimens produced by injection molding, was observed as 305°C in the specimens produced with FFF. The fact that the 15 wt.% fiber reinforcement is wood has caused decomposition and mass loss at high temperatures. The void content of the specimens produced with FFF affected the amount of mass measured after 305°C. The density values of the specimens produced in Table 1 confirm this

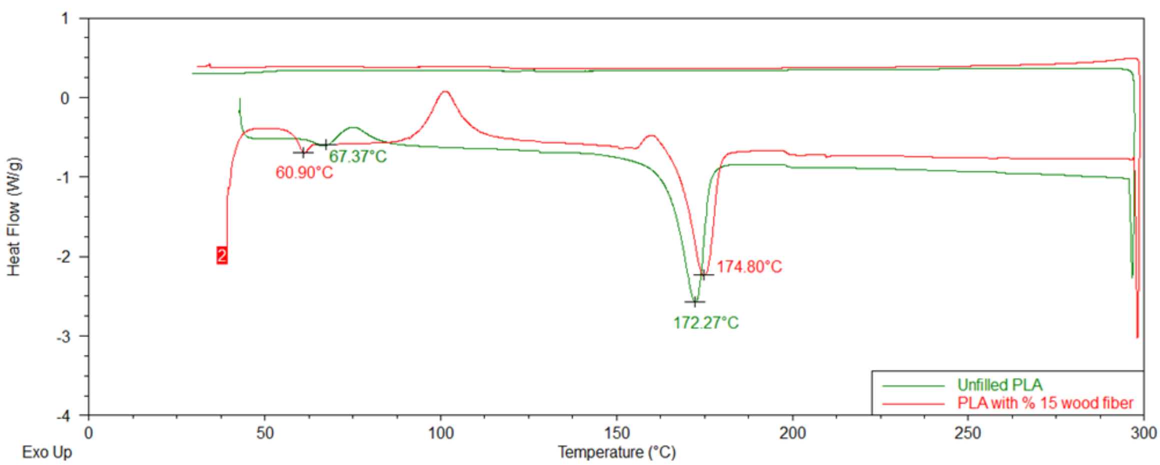

Figure 13. DSC analysis of Unfilled PLA and WR15 PLA compound.

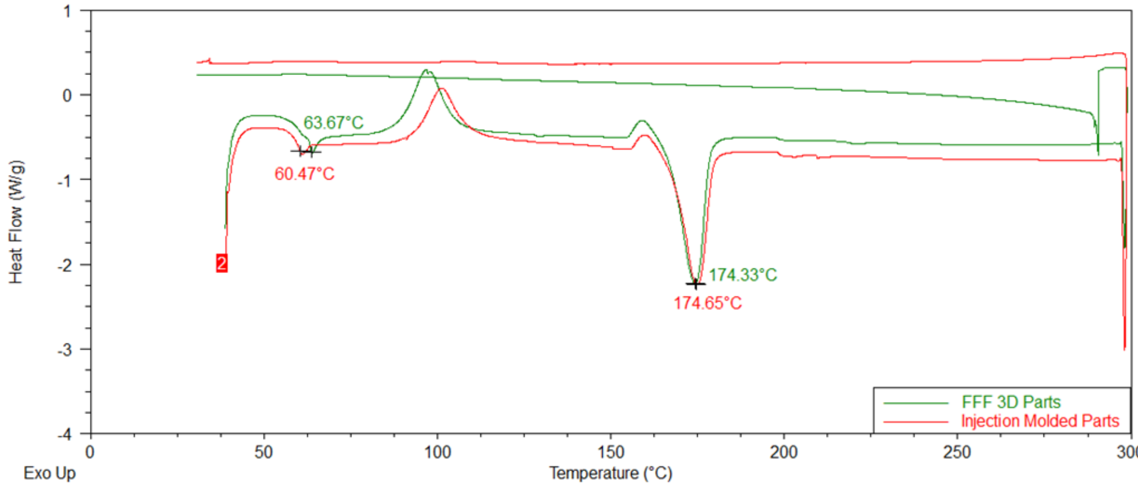

Figure 14. DSC analysis of FFF Part and Injection Molded Part.

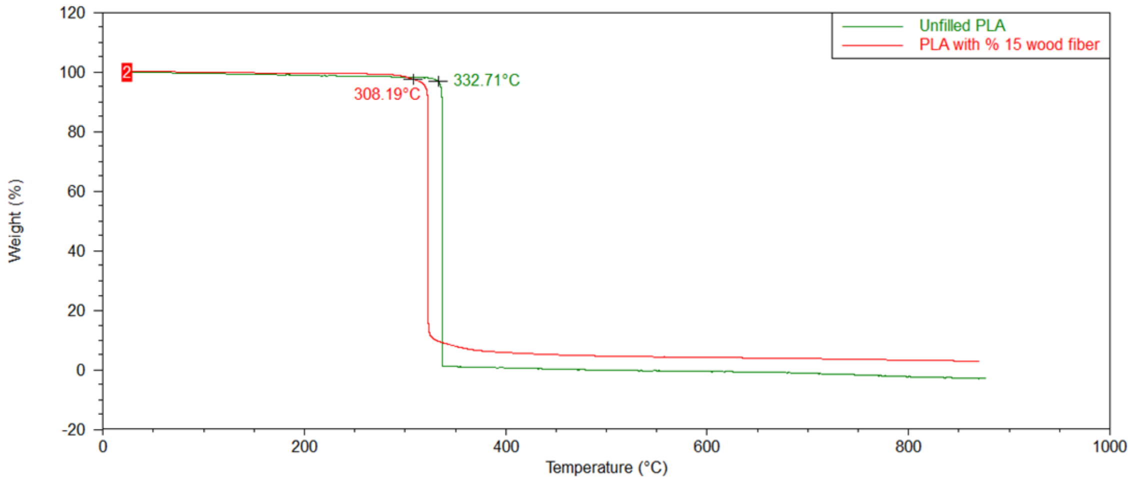

Figure 15. TGA analysis of Unfilled PLA and WR15 PLA compound.

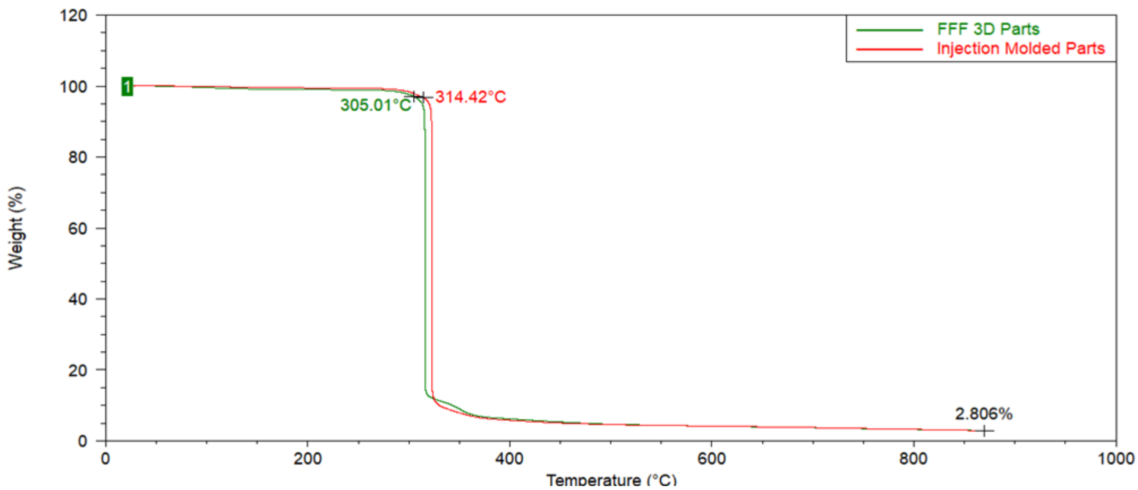

Figure 16. TGA analysis of FFF Part and Injection Molded Part.

Mechanical Tests

Table 1 shows the results of the mechanical tests. The stress at break, strain at break, tensile modulus, and Charpy impact values in the table are the average values of the five specimens produced. The FFF method, by its nature, creates hollow structures. It is known that the production parameters affect the density in the FFF method. In addition, the density values of the specimens are given in Table 1. While there was no change in the density of the specimens produced by injection molding, it was observed that the density of the specimens produced by the FFF method was lower. The gap structures observed in the SEM analysis are the reason for this. The density of the specimens produced in the -/+45 orientation with the FFF method is higher than those produced in the 0/90 orientation.

After analyzing tensile test results, it was determined that the stress at break value of the specimens produced with injection molding was 2 times higher than the specimens produced with FFF. The specimens produced by injection molding have higher stress at break values due to the high density of the specimens produced by injection molding, the fact that the specimens produced with FFF do not have strength in the z-axis, and the shear stress between the layers in the specimens produced with FFF. In the specimens produced with FFF, it was observed that the specimens with the -/+45 orientation had slightly higher stress at break value than the specimens with the 0/90 orientation.

The high density of the specimens produced in -/+45 orientation indicates that the gaps between the layers are lower. SEM images also support this situation. In addition, in the FFF production technique, the nozzle movement in the -/+45 direction in the cartesian coordinate system provides higher extrusion than the 0/90 orientation. Tensile modulus and Charpy impact values are higher in 0/90 oriented specimens. The position of the fiber orientations increases the impact strength in specimens with 0/90 orientation. While the Charpy impact values of the specimens produced by injection molding were 13.87kJ/m2, the closest value was observed as 9.14kJ/m2 in the specimens with 0/90 orientation. The Charpy impact values of the samples produced by injection molding were observed to be 51.75% higher. Since they have a more rigid and solid structure compared to additive manufacturing, the impact resistance of the specimens produced by injection molding is higher.

While the strain at break value was 2.62% in the specimens produced by injection molding, it was 1.61% in the -/+45 oriented specimens and 1.65% in the 0/90 oriented specimens produced by the FFF method. To achieve the best possible mechanical performance in the structural part, careful selection of the fill direction is recommended for structural components subjected to mechanical stress. [31]. Due to the different characteristics observed in the specimens produced with FFF depending on the orientation, the orientations in which the structural parts will be produced according to the loads they will be exposed to should be determined.

Table 1. Mechanical Tests of unfilled PLA and WR15 PLA compound

PROPERTY	Condition	Unit	Standard	WR15PLA		
				Injection Molded Results	FFF Results	
					-/+45	0/90
Stress at Break	5 mm/min	Mpa	ISO 527	67,73	33,31	32,44
Strain at Break	5 mm/min	%	ISO 527	2,62	1,61	1,65
Tensile Modulus	1 mm/min	MPa	ISO 527	3796	2477	2627
Charpy Impact, unnotched	+ 23°C	kJ/m²	ISO 179/1U	13,87	8,29	9,14
Filler Content	-	%	-	0	0	0
Density	+ 23°C	g/cm3	ISO 1183	1,2632	1,1277	1,1031

CONCLUSION

The manufacturing method has a significant influence on mechanical properties. Specimens produced with injection molding have 44% higher tensile modulus than specimens produced with FFF.

The fact that the production is made with filament in the FFF method includes an extra process. This causes the polymer matrix to degrade at lower temperatures.

It has been observed that orientation influences the mechanical properties in specimens produced with FFF. It was observed that the stress at break value was as low as 2.6% in the -/+45 oriented specimen, and the Charpy impact value was 10% higher in the 0/90 oriented specimen. Tensile modulus value was also found to be 6% higher in the 0/90 oriented specimen compared to the -/+45 oriented specimen.

In SEM analysis, it was observed that wood fiber length increased up to approximately 400micron. This has limited the nozzle diameter to be used in production with FFF. A nozzle with a diameter of 0.8mm was used. In future studies, comparisons can be made with the FFF production parameter to increase interlayer coupling. With lower fiber reinforcement, production can be performed with lower nozzle diameters.

In this study, the properties of PLA matrix bio-composite materials with 15 wt.% wood fiber reinforcement in different production methods were investigated. In future studies, the effect of different wood fiber ratios on the mechanical properties of PLA matrix can also be examined. It is also possible to compare specimens at different orientation angles.

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