Investigation of Mechanical Properties of FDM Materials Produced with SiO₂ Reinforced PLA Filaments

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ABSTRACT

Recently, FDM printers have taken their place in many places from workplaces to homes. Polymerbased filaments are used in these printers. The wide variety of polymers also increases the variety of products that can be used in FDM printers. For this reason, many studies are carried out in this field to ensure the production of parts with better mechanical properties, better surface quality and to use 3D printing capabilities in specific applications. In this study, the effect of SiO2 composition on PLAbased filaments on the mechanical properties of the materials produced using these filaments was investigated. SiO2 reinforced PLA filaments are produced with 10, 20 and 30 wt% SiO2 reinforcement. Tensile specimens with 100% fill rate and grid fill shape were produced from this composite filament. The test results showed that SiO2 reinforcement decreased the yield and tensile strength values and elastic modulus of the materials, but increased the toughness values.

Keywords: Silicon Dioxide (SiO₂), PLA (Polylactic Acid) Filament, FDM, 3D Printer, Tensile Test, Mechanical Characterization

1. INTRODUCTION

Traditional manufacturing methods are not suitable for prototype production and replacement part production in terms of time and cost. The main reason for this is that traditional methods are very expensive in boutique production and the initial investment costs are very high. However, additive manufacturing methods, which have an increasing usage rate today, are one of the most up-to-date manufacturing methods that allow to solve this problem (Attaran, 2017). 3D printing techniques, which are becoming more and more widespread today, are the most up-to-date method that allows solving these problems. Additive manufacturing has many different types in terms of the method applied and the material used. FDM (fused deposition modeling), which works with the logic of melting polymer-based materials and stacking them in layers, is the most common method among them (Evlen vd., 2019). The material used in this method is the filament form of many different polymer-based materials (Kristiawan vd., 2021). In the FDM method, these filaments, which can be produced in different diameters, are melted by heating in an extruder, they are passed through a nozzle of different diameters and stacked on top of each other to produce parts (Uzun & Erdoğdu, 2020). PLA is one of the most preferred filaments in FDM 3D printers. PLA, namely polylactic acid, is produced from dextrose obtained from bio-based materials (Jacobsen vd., 1999). Although PLA raw material production is made in a center in the world, the conversion of raw material into filament is carried out by many different companies.

There are many studies based on FDM production using PLA filaments. The basis of these studies is the effects of 3D writing parameters on mechanical properties (Günay vd., 2020)(Thiago vd., 2017). In 3D printers, many parameters such as nozzle diameter, filament diameter, layer height, printing





speed, table temperature, nozzle temperature, scanning angle and filling density can be changed (Bacak vd., 2021). It is known that especially the scanning angle, fill rate, layer height and writing speed are the factors that affect the mechanical properties the most (Letcher, 2015)(Popescu vd., 2018)(Öz & Öztürk, 2022). Another important factor affecting the mechanical properties is the quality and content of the filament used. Filaments using high quality raw materials give better results in 3D printing (Wenjie Liu vd., 2017). In addition, different materials are added to the filaments to improve the mechanical properties of the manufactured parts (Bedi vd., 2018). There are different types of filaments produced as ceramic, metal or fiber reinforced (Wenbo Liu vd., 2018)(Sodeifian vd., 2019), and the parts produced with these filaments also have the feature of composite structure (Dudek, 2013). It is aimed to improve the basic mechanical properties of the parts produced with reinforced filaments, as well as to improve other properties such as abrasion and thermal resistance (Kumar vd., 2012)(Ivanov vd., 2019). In this study, it was aimed to produce SiO2 reinforced PLA filaments and to examine the effects of SiO2 reinforcement and reinforcement ratio on the basic mechanical properties of the parts produced in 3D printers. For this purpose, PLA filament containing 3 different ratios of SiO2 was produced. The produced materials were tested in standard 3D printers and the changes in mechanical properties were observed with the tests made with the produced samples.

2. MATERIAL AND METHOD

The production of filaments used in 3D printers is carried out in single screw extruders. In the production of additive-free filaments, PLA granules and other necessary materials (colorant, binder, etc.) are mixed and filled into the raw material inlet silo (Figure 1). The material, which is melted and mixed by heating in the extruder, is passed through the die. The filament coming out of the mold is pulled with a speed-controlled puller and the diameter is adjusted with 5% precision. The filament that comes out is cooled and wound on the reel. The production of reinforced filaments is carried out in two stages. First of all, PLA granules and SiO2 powders, which are brought together at an appropriate rate in order to mix PLA granules with SiO2 particles homogeneously, as in the filament production logic, are melted and mixed in a twin-screw extruder and then regranulated with the help of a crusher (Figure 2). In the second stage, these granules are passed through the filament production line and turned into filaments. SiO2 reinforced PLA filaments used in the study were produced by FILAMEON Company.



Figure 1. Working principle of extruder used in filament production.





Figure 2. Extruder and crusher used in the production of PLA-SiO2 mixed pellets

SiO2 is used as a reinforcement material in polymer materials because it reduces the size of the dispersed phase particles and homogenizes the structure and is economical (Odent vd., 2017). This is the reason why SiO2 was chosen as a reinforcement element in this study. SiO2 reinforced PLA composite filaments are produced to contain 10, 20 and 30% SiO2 by weight. SiO2 was used at an average single powder size of 44 μ m (Table 1). In the produced filaments, in order to ensure the homogeneous distribution of the SiO2 reinforcement in the product and to create the interfacial bond with PLA, 60% of the TPU was added to the SiO2. The 3D printing performances of the produced reinforced PLA filaments were tested on standard printers and appropriate printing parameters were determined. The best results were obtained with these filaments at 200 °C temperature and 80 mm/min printing speed. With the specified parameters, tensile test specimens were produced with a single filling type and filling density. The fill type used in the production of the samples is the grid fill, as shown in Figure 3. The samples were produced on the ENDER 5 Pro brand 3D printer (Figure 4). The tensile samples were produced in accordance with the ASTM D638 standard (Figure 5) and were carried out on the MTS Criterion device at a tensile speed of 0.002 mm/s (Popescu vd., 2018).

	PLA wt%	SiO2 wt%	TPU wt%	SA wt%
PLA	99,5	-	-	0,5
PLA-SiO2-10%	83,75	10	6	0,25
PLA-SiO2-20%	67,5	20	12	0,5
PLA-SiO2-30%	51,5	30	18	0,75

 Table 1. Components and component amounts of manufactured filaments.





Figure 3. Grid fill type used in the production of tensile specimens.



Figure 4. Ender 5 3D printer used for the production of tensile specimens.



Figure 5. Tensile specimen dimensions according to ASTM D638



3. RESULTS

Test samples were produced with PLA filaments produced by adding 10%, 20 and 30% SiO2. For comparison, production was also made with filaments that did not contain SiO2. The tensile test results of the materials printed in 3D printers are shown in Figure 6. The test results show that the material produced with pure PLA has a higher strength value than the others. However, it has been observed that the material also has a low level of toughness that is not expected from a polymeric material. In the tensile curves of the materials produced with filaments containing SiO2, it is seen that the yield and tensile strength values have decreased, but the toughness values have increased at a high rate.



Figure 6. Tensile curves of materials produced with filaments that do not contain SiO2 and contain 10, 20 and 30 %.

The yield, tensile, modulus of elasticity, resilience and toughness values obtained from the tensile curves of the materials (Table 2) were evaluated in Figure 7 according to the SiO2 ratio. The yield strength values are the stress value corresponding to the 0.2% plastic deformation of the materials. It is seen that the yield strength values decrease linearly with SiO2 reinforcement (Figure 7-a). The tensile strength values, which show the maximum stress values of the materials, also decreased with the increase in the SiO2 ratio, as in the yield strength values (Figure 7-b). With the increase of SiO2 reinforcement and reinforcement ratio, the elastic modulus of the materials decreased (Figure 7-c). The change in yield and tensile strength and modulus of elasticity of the materials with increasing strain amount with SiO2 reinforcement was as expected. The change in the resilience values of the materials differed according to the strength values. As expected, these values decreased with SiO2 supplementation (Figure 7-d). However, with the increase of SiO2 ratio above 20%, the elastic deformation ability of the materials was lost at a high rate. The toughness values of the materials reached their maximum value with 10% SiO2 supplementation. With the reinforcement ratio exceeding 10%, the toughness values started to decrease (Figure 7-e). As seen in the tensile curves, the situation where the SiO2 reinforcement ratio is 30% is the lowest in terms of strength values and deformation capabilities of the material. In addition, the linear decrease in the properties went out of the linear at this rate and showed sharp decreases. The results showed that increasing the SiO2 reinforcement ratio above 10% started to lose the obtained toughness gains.



	σ _y (N/mm ²)	σ _y (N/mm ²)	E (GPa)	Resilience (J/m ³)	Toughness (J/m ³)
PLA	53,28	55,12	9,58	1,396	1,914
PLA-SiO2-10%	41,5	45,17	8,99	0,906	19,414
PLA-SiO2-20%	36,58	38,76	8,5	0,475	13,776
PLA-SiO2-30%	25,29	31,48	7,01	0,058	6,094

ou(N/mm2)

Table 2. Mechanical property values obtained from materials by tensile test.



a)

8,99

9,58

10











Figure 7. Variation of a) yield strength values, b) tensile strength values, c) elastic modulus, d) resistance values, e) toughness values depending on SiO2 ratio.



The fracture behavior of materials during tensile tests reflects their mechanical properties. Images taken from the fracture areas are shown in Figure 8. In all reinforced and non-reinforced materials, fracture occurred in a brittle type and no necking was observed (Figure 8-c). As a result of the toughness gained with SiO2 reinforcement, the plastic deformation ability of the material has increased. However, the deformation that occurred during the tensile tests was evenly distributed throughout the measuring region of the samples (Fig. 8 c-d). In the images taken from the material cross-section, the elongation along the fiber lines were seen. It has been observed that deformations occur perpendicular to the fiber lines in materials containing high SiO2 (Figure 8-d). High deformation occurring in materials with 30% SiO2 ratio also caused deterioration in stacked fiber structures. This is seen in the images taken from the material section (Figure 8 a-b).



a)

b)



Figure 8. a) Tensile sample section view of PLA material b) tensile sample section view of PLA-SiO2-30% material, c) vertical view of tensile sample of PLA material, d) vertical view of tensile sample of PLA-SiO2-30% material

4. DISCUSSION

In this study, the effect of post-printing mechanical properties of SiO2 supplementation on PLA filaments, which are used extensively in 3D printers, was investigated. Tensile samples were produced with PLA filaments obtained by adding 10%, 20% and 30% SiO2 and these samples were subjected to tensile tests. According to the test results;

- SiO2 reinforcement made to PLA filament decreased the yield and tensile strengths after printing and increased the toughness values at high rates.



- The material with 10% SiO2 ratio has the highest toughness value, and the toughness values decreased with the increase of this ratio.

- The most optimum material in terms of yield and tensile strengths, modulus of elasticity and resilience values, apart from the toughness value, was the material containing 10% SiO2.

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