Tensile properties of elastomer process through FFF for biomedical applications

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Abstract: 3D printing technology, specifically the fused filament fabrication technique (FFF), is growing in both, industrial and private sector. Due to the large number of possible applications for parts built through this technique, the need to study new materials increases. This paper aims to characterize a thermoplastic elastomer material, TPE 96A. TPE is a flexible material that, among others, can have applications in the field of biomedicine thanks to its flexibility and strength. In order to study the material, two controlled printing parameters (layer height and fill density) are related with its mechanical properties defining the responses obtained in a tensile test. A factorial design of experiments is applied to optimize the process. The specimens are manufactured according to the ASTM D638 standard. Finally, the results will be analyzed by means of an analysis of variance test (ANOVA). Results show that the highest Young’s modulus achieved experimentally is 129 MPa if a combination of 75% of fill density and a 0.2 mm layer height is used for manufacturing the samples.

Keywords: 3D printing, Fused Filament Fabrication, Thermoplastic elastomer, Tensile test.

1. Introduction

The current development of the use of 3D printing is growing exponentially, so new materials are being tested. The investigation of the behavior of materials that can be manufactured using the process of material extrusion (MEX), also known as Fused Filament Fabrication (FFF), focuses not only to know the mechanical response of the material, but also to work with sustainable materials, reducing the amount of waste.

There are different families of materials used in additive manufacturing (AM) depending on the final use of the pieces manufactured. Materials with elastomeric properties can be used in the field of biomedical applications, as the parts printed with them will have a suitable final appearance. There are some researches that have studied this kind of materials. For example, Bachtiar et al. (2020) [1] studied a biocompatible elastomer appropriate for vascular implant applications, defining the mechanical properties and optimizing FFF parameters to take advantage of its high flexibility and strength. Abdullah et al. (2017) [2] analyzed composite materials for craniofacial reconstruction applications manufactured through FFF technique, thus, studying the roughness and strength of the polyamide compound used in the work. Dohmen et al [3] and Lee et al [4] studied the effect of adding magnetic particles to thermoplastic elastomers materials for applications of soft robotics. Jun Yin et al [5] studied a bi-material
structure made of thermoplastic polyurethane (TPU) and acrylonitrile butadiene styrene (ABS), concluding that the increase of the bed temperature significantly improves the interfacial bonding strength.

As observed in the reviewed papers, the exploration of the properties of thermoplastic elastomers used in AM is growing in the field of manufacturing engineering. The aim of this research is to characterize and evaluate the mechanical properties of a thermoplastic elastomer so it can be used in biomedical applications. Therefore, this paper focuses on the mechanical behavior of TPE 96 A processed through FFF. The influence of the printing parameters selected for manufacturing the samples is studied through a fractional factorial experimental design as a means of an optimization of all the process of manufacturing, testing and analyzing the results of the samples.

2. Materials and methods

In this paper, the tensile properties of TPE 96A, a semi-flexible filament based on polyolefin with both thermoplastic and elastomeric properties, are studied and analyzed. This material is manufactured and sold by Fillamentum, a company from Czech Republic. Therefore, this paper contains an experimental part that comprises tensile tests performed on specimens of TPE, following the American Society for Testing and Materials (ASTM) D638 standard [6]. Some basic physical and chemical properties of the raw material are described in table 1, as provided by the manufacturer.

| Property              | Typical Value                                      |
|-----------------------|----------------------------------------------------|
| Material density      | 1.15 g/cm³ (test method: ISO 1183)                 |
| Melt flow index       | 25 g/10 min                                        |
| Polymer base          | Polyolefin                                         |
| Tensile strength      | 5 MPa                                              |

To better understand the influence of the manufacturing parameters in these properties once the material has been processed through FFF, an analysis of variance (ANOVA) is made in order to define the significant parameters.

2.1. Study of chemical composition of the material

The mass loss in the material should be studied before starting a 3D printing process because this can lead to printing defects, such as bubbles in fabricated samples that could create stress concentrators.
2.1.1. Thermal gravimetric analysis (TGA). The graph obtained from the TGA (figure 1(a)) shows that the material begins to lose mass at 210 ºC. Loss of mass may be due to loss of additives or may indicate an onset of material degradation. Eventually, this graph shows how the behavior is the same for extruded and non-extruded material.

In addition, the behavior of the material was studied at the extrusion temperature (245 ºC). TPE 96A loses 1.2% in mass at 245 ºC. This is not a significant loss, so it means that TPE can be printed at this temperature, as recommended the manufacturer. The molecules degrade at 410 ºC, a fact that must be taken into account for the filament manufacturing process and the printing process. Therefore, these temperatures should never be reached.

The second graph obtained (figure 1(b)) shows that from 400 ºC to 600 ºC the remaining charge is 30%, which could indicate the existence of inorganic elements within the raw material.

2.2. Differential scanning calorimetry (DSC)

For this analysis, two cooling-heating cycles were performed. From the first heating cycle (figure 2(a)) it is observed that the extruded and non-extruded material have the same behavior. The graph also shows that energy absorption is detected at 155 ºC, this may imply a phase change of the material (crystalline phase). From the second cooling-heating cycle (figure 2(b)), the glass transition temperatures are observed: the glass transition temperature is located below 0ºC.

![Figure 2. Representative DSC curves for TPE 96A.](image)

2.3. Geometry

The design of the specimens is according to the ASTM D638 standard [6], which regulates the test method for tensile properties of plastics. This standard indicates a range of values in order to define the dimensions of the samples, figure 3 shows the dimensions adopted.

![Figure 3. Dimensions and shape of the samples: 7 mm thickness.](image)
The manufacturing process followed four steps (figure 4). The test samples were designed using SolidWorks and sliced using the software Simplify3D, where the different variable parameters were set according to the DoE defined. Finally, the parts were manufactured from the g-code generated with an Ender Pro-3 printer.

Figure 4. Additive manufacturing process.

2.4. Design of experiments (DoE)

To perform the tensile test, a design of experiments (DOE) technique was used. In this case, two printing parameters (figure 5) considered the most influential in mechanical behavior were included in the study. Two levels with a center point were also defined (table 2) resulting a full factorial design. These two parameters are the layer height (describes the thickness of each layer deposited on the bed by the nozzle) and the fill density (defines the amount of material deposited in the inner part of each sample). Both were selected considering the bibliography studied as well as the experience of previous work of the research group. Table 3 contains the values of the seven parameters set as constant.

Table 2. Variable parameters TPE 96A.

| Variable parameters | Level   |
|---------------------|---------|
|                     | Low     | Medium | High   |
| Layer height        | 0.20    | 0.25   | 0.30   |
| Fill density        | 25      | 50     | 75     |

Table 3. Constant parameters TPE 96A.

| Constant parameters | Fill pattern | Nozzle diameter [mm] | Printing velocity [mm/s] | Fan velocity [%] | Bed temperature [ºC] | Nozzle temperature [ºC] | Number of outline perimeters |
|---------------------|--------------|----------------------|--------------------------|-----------------|-----------------------|--------------------------|-------------------------------|
| Value               | Honeycomb    | 0.4                  | 25                       | 60              | 60                    | 245                      | 4                             |
data from the cell that has built-in. An additional load cell of 2kN was connected to DAQ (Spider) software and attached to the machine. The testing machine operated at a speed of 20 mm/min.

All the samples were measured using digital micrometer before the test in order to achieve the average area by calculating it from the width and thickness of four different sections of samples.

Also, a full high definition (FHD) camera was used in this setup equipped with a lighting system, which supplies a uniform light intensity so that it does not interfere with the video obtained. The camera, which was also connected to the DAQ, recorded a video of the test at 59,94006 Hz sampling frequency (figure 6).

![Universal testing machine equipped with the FHD camera.](image)

2.6. Analyzing process

Data acquisition was through an HD camera and the universal machine. On one hand, the data acquired by the load cell was displayed in a time sequence of the force data. This file also contained the recorded voltage versus time. On the other hand, the camera obtained a sequence of frames that contained the displacement of the samples during the test. These two documents defined the whole deformation process of each sample tested.

The data obtained from the tensile test was processed with a variant of Matlab routines already used in previous studies \[8\] in order to generate the stress-strain curve for TPE 96A. To carry out this process, the routines create a matrix of points that defines the position of the material at every stage of the test. Therefore, the difference between the initial and final position of the points were calculated. After this step, the displacements of the matrix’ points were analyzed and converted into engineering deformations. Finally, from the deformations obtained in the last step, the stress-strain curve, which provides the behavior of the material tested in tension, was generated. The mechanical characteristic parameters of TPE 96A extracted from the curve were eventually used as response variables for the ANOVA model.

3. Results and discussion

Five different configurations and three samples of each configuration were printed and tested. Table 4 contains the average of the mechanical responses obtained from the three samples for each configuration, including the standard deviation of all the repetitions, which shows the associated error of the statistical data set.

Once all the tests were performed, the results were processed with statistical models by applying an analysis of variance with a significance level of 95%, where all the mechanical responses as well as the influence and interaction of the parameters studied were evaluated.
### Table 4. Mechanical responses TPE 96A.

| Configuration | Layer height (mm) | Fill density (%) | Young’s Modulus, E (MPa) | Elastic limit, Rp0.2 (MPa) | Maximum strength, σ_{max} (MPa) | Maximum deformation, ε_{max} (%) |
|---------------|-----------------|-----------------|--------------------------|---------------------------|-------------------|-----------------------------|
| 1             | 0.20            | 25              | 63.61 ± 2.44             | 0.42 ± 0.01               | 2.40 ± 0.06       | 370.70 ± 14.34             |
| 2             | 0.30            | 25              | 77.60 ± 2.17             | 0.50 ± 0.02               | 3.61 ± 0.19       | 510.72 ± 21.97             |
| 3             | 0.20            | 75              | 128.96 ± 0.49            | 0.47 ± 0.07               | 3.62 ± 0.11       | 446.81 ± 5.55              |
| 4             | 0.30            | 75              | 119.86 ± 8.43            | 0.57 ± 0.01               | 4.01 ± 0.23       | 558.76 ± 37.83             |
| 5             | 0.25            | 50              | 103.49 ± 7.57            | 0.52 ± 0.01               | 3.37 ± 0.21       | 571.28 ± 21.35             |

**3.1.1. Young’s Modulus.** From figure 7(a), which contains the p-values of each of the parameters analyzed, it can be concluded that layer height variations do not affect the results on Young’s modulus response, as a very small range of values have been studied. In contrast, to maximize the value of the Young’s modulus for TPE, the value of its fill percentage must be increased.

In addition, the interaction between the two parameters is also significant (p-value = 0.029). These two parameters are directly associated with the amount of material used; thus, the fill percentage will have a strong correlation with the layer height due to the forces that are created between the number of layers of the sample (determined by the layer height) and the contact surface (determined by the percentage of filling).

![Figure 7. Main effects for means calculated through ANOVA. (a) Response: Young’s modulus. (b) Response: Elastic limit.](image)

**3.1.2. Elastic limit.** The results obtained with the analysis of variance show that the two parameters are statistically influential on the elastic limit response (figure 7(b)). Therefore, in order to increase the elastic limit, the values of both the layer height and the fill density within the studied range must be increased. However, the interaction between the two variables is not influential to evaluate the elastic limit, since its p-value is much higher than α (0.830 > α).

**3.1.3. Maximum strength.** When studying the maximum strength, in figure 8(a) it is observed that both layer height and fill density, are statistically significant parameters, since the two functions contained in the graph show high slopes, which indicates a strong dependence on the parameter that represent.

In addition, the interaction (p-value = 0.035) between these two parameters is analyzed. The interaction declares that for layer height values of 0.2 mm, the change of the value of fill density is more
sensitive. But for layer height values of 0.3 mm, the effect of increasing or decreasing the value of fill density is lower.

![Graph](image)

**Figure 8.** Main effects for means calculated through ANOVA. (a) Response: Maximum strength. (b) Response: Maximum deformation.

### 3.1.4. Maximum deformation

Figure 8(b) contains the statistical significance with numerical values and graphical functions. The layer height and the fill density are statistically significant parameters as their p-values are lower than 0.05. The effect of deformation is shown when changing from a layer height of 0.20 mm to 0.25 mm, as it increases. In contrast, no significant change is observed in the range of 0.25 mm to 0.30 mm. That is, the largest significant effect is between 0.20 mm and 0.25 mm layer height. In the case of the fill density, there is a positive increase of the values of deformation in the interval between 25% and 50%, but when the density changes between 50% and 75%, this value decreases. However, the interaction between these parameters (p-value = 0.121) is not statically significant for the maximum deformation response of the samples within the range of values studied.

### 3.1.5. Optimal printing parameters

Based on the results of this study, the combination that allows to obtain a higher value of Young's modulus is the one that implies 75% fill density and a 0.2 mm layer height. Thus, the TPE 96A has a Young's modulus of 129 MPa, and an elastic limit of 0.47 MPa. The maximum stress of the material once processed is 3.62 MPa and it can deform 447% of its initial position. Table 5 contains a summary of the influences resulting from the study.

From the ANOVA study, the following behavior can be observed:

- The value of Young’s modulus, elastic limit and maximum strength increase with the fill density due to the fact that a higher percentage means that there are more fibers that support the applied force and less porosity in the piece. These results have also been found by other researchers in similar studies such as M. Damous Zandi *et al* [9]. However, maximum deformation increases its value with 50% of fill density. This may be due to the fact that the deposition of more material makes the samples stiffer but it makes them break sooner as they cannot be deformed due to the forces created between the layers. This effect of the fill density has already been demonstrated in other researches [10].
- A layer height of 0.30 mm results in better results in all mechanical responses, except in the Young’s modulus, in which this parameter has no statistically significant effect.
Table 5. Mechanical responses for TPE 96A.

| Layer height (mm) | Fill density (%) | Young’s Modulus (E) | Elastic limit (R\text{p}0.2) | Maximum strength (\sigma_{\text{max}}) | Maximum deformation (\epsilon_{\text{max}}) |
|------------------|------------------|---------------------|-----------------------------|----------------------------------------|----------------------------------------|
| n.i.             | 75               | 0.30 ↑              | 0.30 ↑                      | 0.30 ↑                                 | 50 ↑                                   |

↑↑ influential parameters
n.i. non influential parameters

4. Conclusions

The mechanical behavior of the polyolefin-based elastomer has been studied in this research, achieving a better understanding of the influence of the printing parameters on tensile tests studied with a full factorial design. At sight of the results, the recommended design rules are the following ones:

- A layer height of 0.30 mm should be selected if the user intends to obtain high results in all mechanical responses, regardless the Young’s modulus in which this parameter has no statistical influence.
- If the final application of the printed pieces is expected to reach high deformations, a density of 50% should be used as a manufacturing parameter. However, a fill density of 75 % evolves better results for Young’s Modulus, elastic limit and maximum strength.

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