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Reviewed Paper

Thermal and Mechanical Characterization of Lightweight PLA Filaments for Material Extrusion

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ABSTRACT

Additive Manufacturing (AM) has seen tremendous advancements in recent years including advancements in the Material Extrusion (MEX) process. This paper investigates thermal and mechanical properties of lightweight (LW) PLA filaments and attempts to optimize the highest strength-to-weight ratio. MEX is known for its versatility and accessibility due to its low-cost operation and vast capabilities. The characteristics of PLA have been studied extensively, considering PLA has proven to be a great candidate for many applications. Recent studies introduced foaming additives to different polymers, including PLA. The foaming additives are thermally activated with varying degrees of expansion. Three commercially available LW-PLA filaments are tested in this study to identify the lightweighting capability of each material. The flow is calibrated with respect to printing temperature. The degradation and glass transition temperatures of each material are extracted using Thermogravimetric Analysis and Differential Scanning Calorimetry. Taguchi design is employed to examine tensile strength by testing multiple process parameters for each material including print orientation, nozzle diameter, and printing temperature. Three printing temperatures are selected based on the temperature that yields the highest volumetric expansion. Nozzle diameters used are 0.4, 0.6, and 0.8 mm, and the print orientations tested are XY and XZ. Initial results indicate that the strength of LW-PLA, when expanded to the maximum, significantly declines. In addition, results indicate the most influential parameter is the type of LW-PLA, followed by the print orientation, while nozzle diameter and printing temperature have the least influence on tensile strength. This research presents vital material properties of LW-PLA filaments which advances application avenues of MEX.

INTRODUCTION

AM has been ubiquitously employed by a significant number of industries in various fields [1], [2]. One of the most utilized AM techniques is MEX [3], [4]. The MEX technique works though extruding material through a nozzle, whether that material is meltable solids or a slurry [5], [6]. In this context, MEX works by melting a polymer and driving it through a nozzle in a controlled manner [7], [8]. One of the advantages of MEX is that it is capable of customizing the internal structure of a component [9], [10], [11]. This feature can come in handy when needing to customize component properties such as mechanical, thermal, and physical. For example, a component can simply be made LW by having a sparse infill structure, allowing the structure to be dimensionally accurate while having a quasi-hollow structure [12].

Recently, researchers investigated adding expansive microspheres into polymers used in MEX to allow the polymer to volumetrically expand upon extrusion. These microspheres are thermally activated and vary in size, shape, and temperature activation range. This addition of microspheres allows further customization of a component using MEX. Meaning, expansive polymers allow extrusion of expanded beads, such as expanded Polystyrene. Considering the polymer bead expanded volumetrically while maintaining the same amount of polymer material, the density of the bead is now lower. An illustration of the expansive bead is shown in Figure 1.This attribute opens another realm for lightweighting methods for MEX [13]. Polylactic Acid (PLA) is a commonly used polymer in MEX due to its versatility and accessibility [14], [15]. This research investigates three types of commercially available LW-PLA for their mechanical and thermal properties.

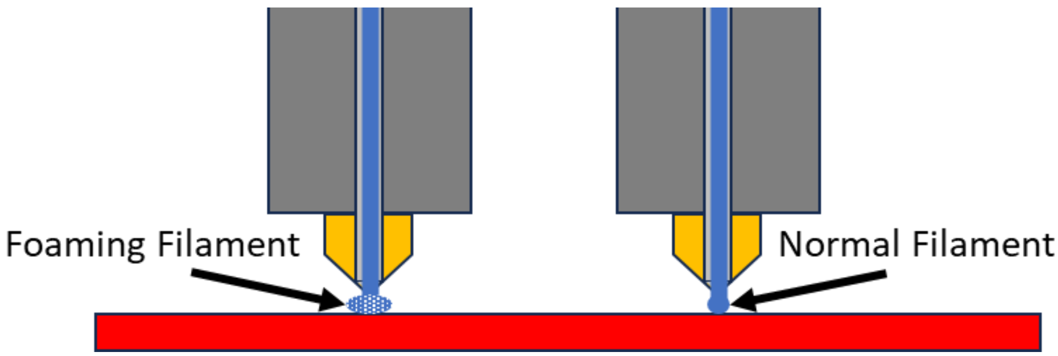

Figure 1: Difference in volume between foaming (expansive) filament and normal filament

MATERIALS & METHODS

Volumetric Expansion

The materials used in this research exhibit volumetric expansion (VE) due to embedded expansive additives within the polymer matrix. These additives are thermally activated and vary in VE with different temperatures. For this reason, multiple temperatures ought to be tested to identify the temperature that yields the maximum VE. Theoretically, maximum VE could yield to the deposition of the lowest density bead. The temperatures are evaluated by fabricating single-walled cubic specimens, shown in Figure 2, with varying temperatures while other parameters are kept constant. The VE is calculated using the following equation:

$$VE = \frac{t_n}{t_{target}} * 100 \quad (1)$$

Where t_n = wall thickness printed at temperature n, and t_{target} = user-defined wall thickness. According to LW-PLA providers, the suggested range of extrusion temperatures to test roughly start with 200 C to 280 C. Once the max VE temperature is identified, a flow calibration is performed to achieve the correct wall thickness.

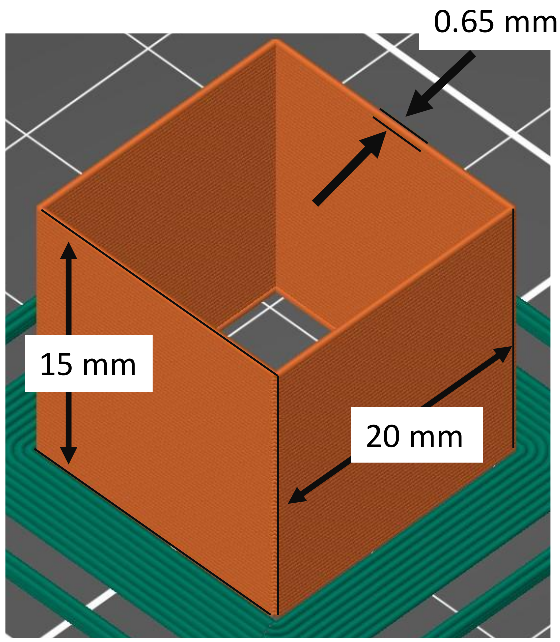

Figure 2: Calibration specimen used for VE with varying temperatures

Thermogravimetric Analysis (TGA)

TGA reveals valuable information about the number of additives within each type of LW-PLA, as well as how the additives affect the degradation temperature of LW-PLA's. TGA operates by exposing a sample to elevated temperatures using a user-defined protocol, while precisely measuring the mass of the specimen. The illustration is shown in Figure 3. TGA is conducted in compliance with ASTM E1131, using the following protocol:

1. Ramp: 10 C/min to 800 C
2. Isothermal: 800 C for 30 minutes

The "Ramp" simply commands the chamber to get up to 800 C at a rate of 10 C per minute, which is where the degradation curve is acquired. The "Isothermal" step commands the chamber to stabilize at 800 C for 30 minutes to clean the tray in case residue is leftover.

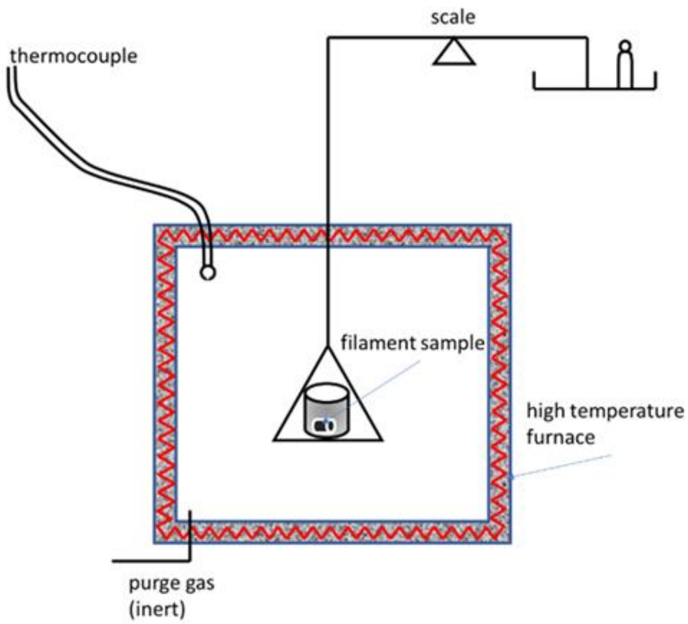

Figure 3: TGA working principle diagram courtesy of [16]

Differential Scanning Calorimetry (DSC)

Another important thermal characterization technique is DSC, which can reveal valuable information such as the glass transition temperature (T_g). The protocol for DSC is typically performed after TGA considering DSC is a non-destructive process while TGA is a destructive process. Thus, the maximum temperature in a DSC protocol should be less than the T deg found in TGA. According to ASTM, the first couple of cycles are used to relief the sample of any residual stresses present that may skew the results. The protocol used for DSC is as follows:

1. Ramp: 10 C/minute to 280 C
2. Isothermal: 5 minutes
3. Ramp: 10 C/minute to room temperature
4. Isothermal: 5 minutes
5. Ramp: 10 C/minute to 280 C
6. Repeat: 2 times (yields 3 cycles)

Tensile Strength

Mechanical characterization is one of the most vital characterization techniques to be conducted for materials. It provides the ultimate and yield strengths of a material, in addition to other properties such as toughness and elasticity. Tensile testing is performed in compliance with ASTM D638 using Type-I specimens with 7 mm thickness. The specimens are fabricated using the ideal extrusion temperature found in the previous VE section, with the flow calibrated to yield the lowest density. The testing machine used is an Instron with a 50 kN load cell, equipped with an extensometer and wedge grips. The specimens are fabricated using a Prusa imk3+, with parameters derived from VE evaluation results.

To understand how different printing parameters affect the mechanical behavior during tensile testing, a Taguchi multi-level orthogonal array is employed. Four parameters are investigated: print orientation (PO), Nozzle Diameter (ND), LW-PLA brand (material), and extrusion temperature. All parameters are to have three levels, except PO with only two levels.

This combination yields to an L18 array. The Design of Experiments (DOE) table, Table 1, shows the different levels of each parameter. To further elaborate on extrusion temperature levels, the "ideal" level is the temperature found to yield maximum VE. The other levels, plus and minus 10 C, are tested to ensure a more encapsulated representation.

Table 1: DOE table for tensile testing

Level	***Print Orientation***	***Nozzle Diameter (mm)***	***Material***	***Temperature***
Level 1	XY	0.4	SainSmart (SS)	Ideal
Level 2	XZ	0.6	Colorfab (CF)	+10 °C
Level 3	--	0.8	eSun	- 10 °C

RESULTS & DISCUSSION

Volumetric Expansion

Figure 4 illustrates the VE with respect to temperature variation for the three materials. Among these, eSun consistently shows the highest VE, followed by SS. eSun reaches its peak VE of 258% at a temperature of 270°C. However, the VE at 250°C is only about 5% less. Additionally, printing at excessively high temperatures leads to stringing and printability issues. Therefore, 250°C, with a VE of 252.7%, is deemed the ideal temperature for eSun. For SS, the VE peaks at 270°C with a value of 246.5%, which is just 3% higher than the VE at 260°C, and 15% higher than at 250°C. Hence, 260°C is selected as the optimal temperature for printing SS. Lastly, CF achieves its peak VE of 205% at 230°C, which is notably lower compared to SS and eSun. It is also observed that the foaming agent in CF is less active at higher temperatures than in the other two filaments.

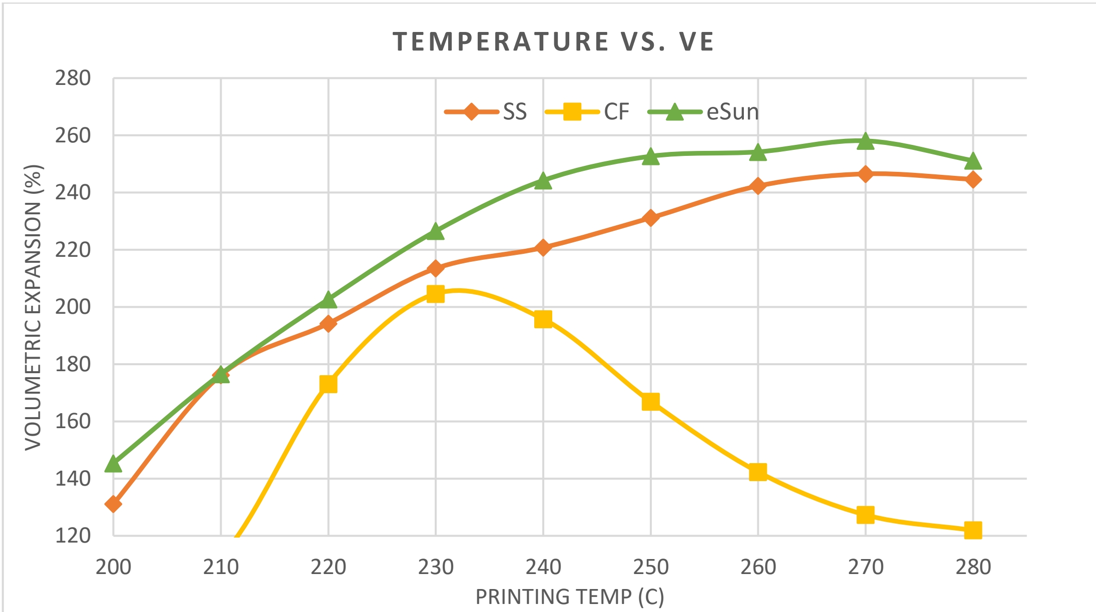

Figure 4: VE results with respect to extrusion temperature for the three LW-PLA's

The next step is to identify the minimum flowrate that yields dimensional accuracy. In this test, an identical hollow cube is printed with varying flow rates ranging from 30% to 50%. This range is determined using the maximum and minimum VE rates previously found across all materials. As mentioned earlier, a 250% VE theoretically leads to a 60% weight reduction (40% of the original weight). This is calculated by dividing the standard flow rate, 100%, by the VE to obtain the possible reduction ratio. The results for the three materials are shown in Figure 5. The findings indicate that eSun can theoretically produce parts with the lowest density among the three materials, achieving a 70% reduction in flow, followed by SS at 65%, and CF at 55%.

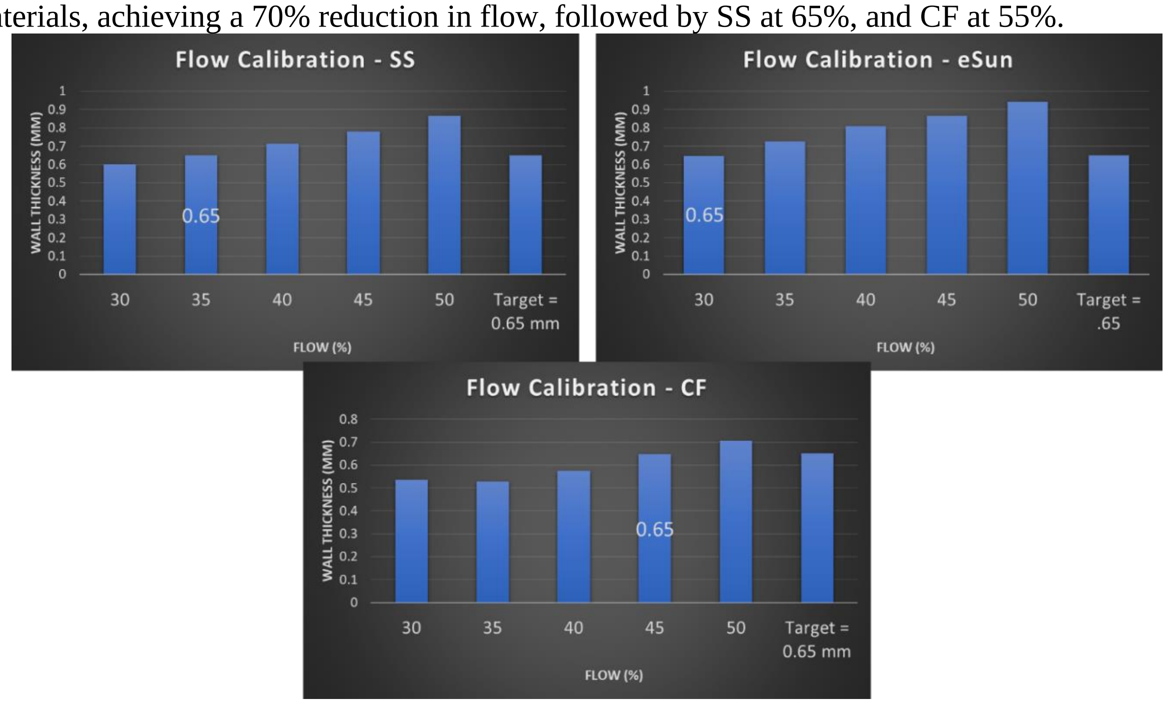

Figure 5: Flow calibration results after identifying ideal extrusion temperature

Thermogravimetric Analysis (TGA)

All three materials underwent a two-step degradation process due to the additives embedded in the matrix material, which may include foaming agents or bond-enhancing additives. According to the TGA curves shown in Figure 6, SS and eSun contain about 20% of additives, marking the end of the first degradation step. In contrast, CF contains only 7% of additives in both sample types. This explains the significant difference in VE observed earlier, with CF having a maximum VE of 205%, compared to around 250% for the other two materials. The overall TGA results are presented in Table 2.

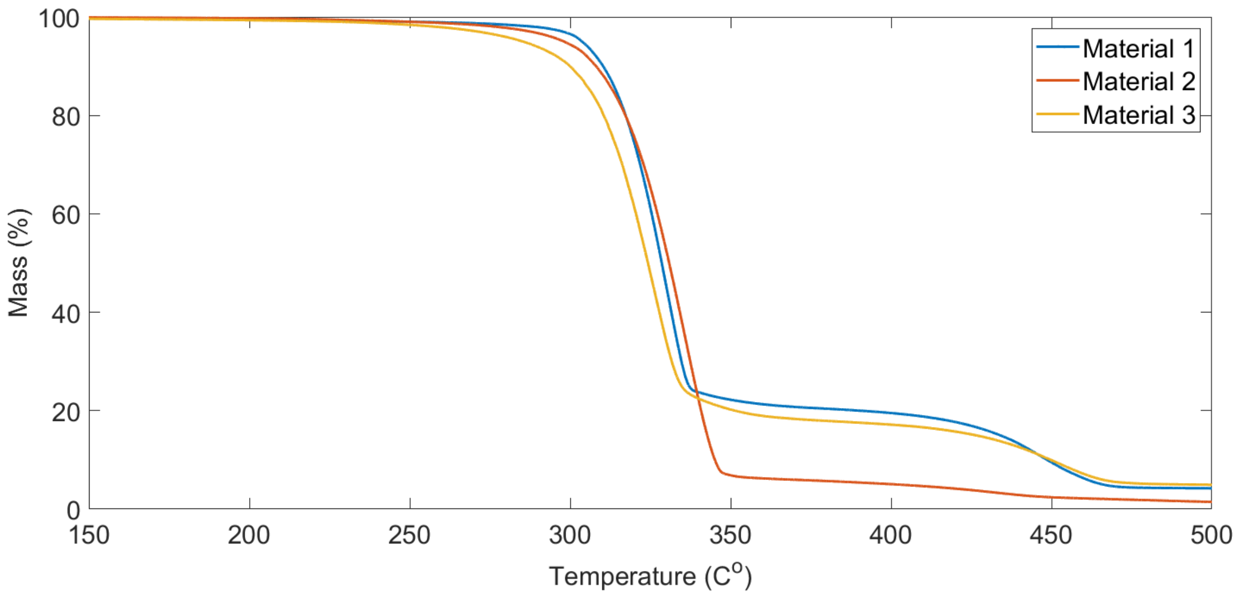

Figure 6: TGA curves for the three LW-PLA's; where materials 1, 2, and 3 are SS, CF, and eSun, respectively.

Table 2: Degradation temperature and additive percentage extracted from TGA curves

Material	*Pre-Print T_{deg} (C)*	*Additives (%)*
SS	307.4	20
CF	314.2	7
eSun	296.6	20

Differential Scanning Calorimetry (DSC)

All three materials experienced a T_g of 45 C to 55 C, which indicates the materials can be operated in very similar thermal environments, not exceeding 50 C. The DSC curves are shown in Figure 7.

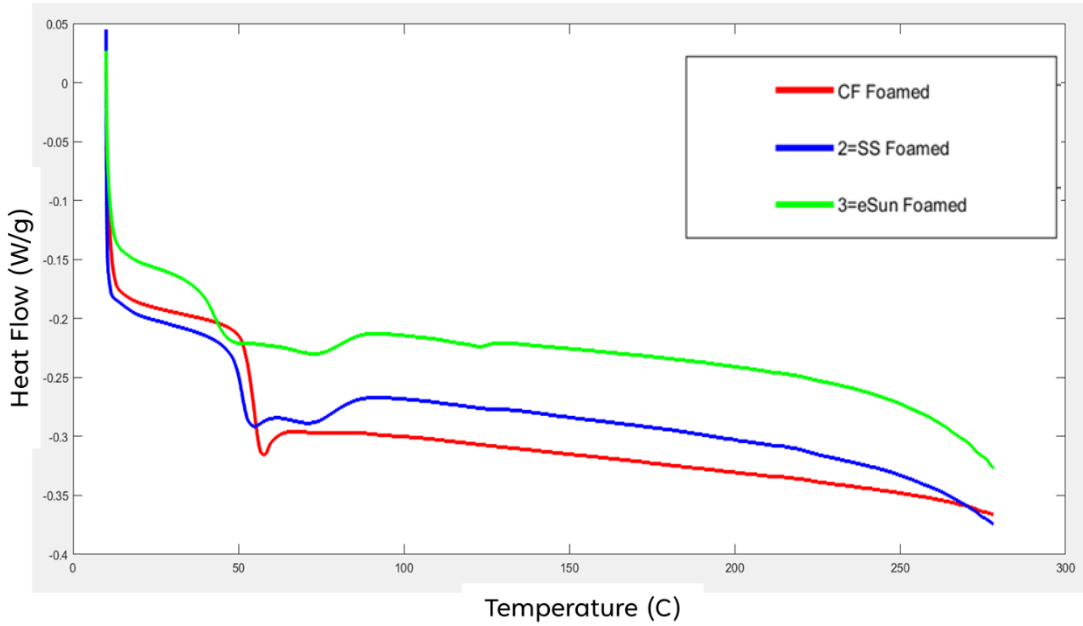

Figure 7: DSC curves for the three LW-PLA's

Tensile Strength

Main effects plot for Signal-Noise (SN) ratios is shown in Figure 8. The results indicate that material type is the most significant factor in affecting tensile strength, followed by PO. Material type is expected to have a significant effect on mechanical results because each material exhibits a different density, since the VE is different for each material. Results indicate that CF consistently has the highest strength. Recall that CF also has the lowest VE, which means that CF samples are the densest (45%), and therefore have more polymer material than its counterparts (30%-35%). In addition, it is a well-known fact that XY specimens have superior tensile strength than XZ specimens [17], [18]. For this reason, PO as a significant factor is expected as well, in favor of XY specimens. Temperature and nozzle diameter ranked in third and fourth, respectively, indicating that these factors do not have as much influence as other parameters.

Table 3 has data that shows the means for each level of each factor. Mainly, it is important to highlight the difference among levels of the material type. In this case, the results indicate that levels 1 and 2 (SS and eSun) have almost identical averages at around 10 MPa. While level 2 (CF) has an average of 17 MPa, which is 70% stronger than its counterparts. With this result, it is clear that while SS and eSun have lower density capability, that capability significantly affects the mechanical strength of the component.

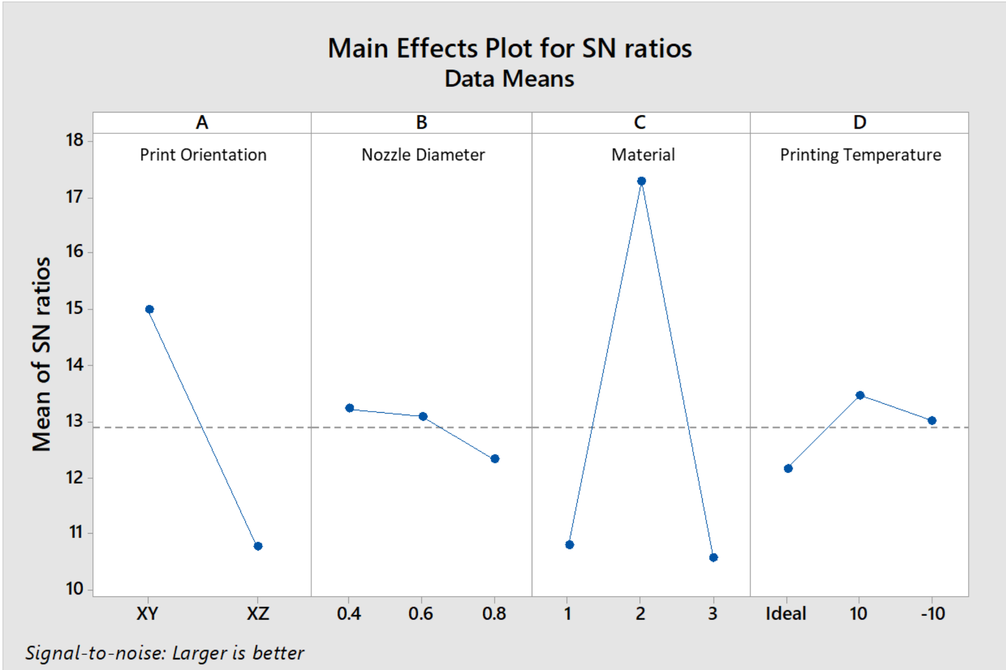

Figure 8: Main effects plot for SN ratios for tensile testing

Table 3: Mean ultimate tensile strength for different factors and corresponding levels, indicating ranks of influential factors.

Level	Print Orientation	Nozzle Diameter	Material	Temperature
1	15.00	13.23	10.80	12.16
2	10.78	13.10	17.30	13.48
3		12.33	10.56	13.02
Delta	4.22	0.90	6.75	1.31
Rank	2	4	1	3

CONCLUSION

This research investigates three commercially available LW-PLA filaments by testing their tensile strength, degradation temperature, glass transition temperature, and VE capability. Maximum VE is achieved by varying extrusion temperature while measuring the wall thickness of a specimen. Followed by flow calibration to achieve the lowest density possible with the discovered VE. These settings are used to fabricate tensile specimens with varying parameters including ND, PO, extrusion temperature, and material type. In addition, TGA and DSC are employed to extract thermal properties of the materials.

Thermal properties of all three materials are all within proximity of one another. The degradation temperature ranges from 296 C to 314 C, while the glass transition temperature ranges from 45 C to 55 C. Additives within CF LW-PLA are only at 7% compared to 20% for SS and eSun. The Results indicate that high VE leads to a lower density as expected, but also yields to a significant decrease in tensile strength. This explains why the material type was the most influential factor since it is highly correlated with part density. Specimens fabricated in XY orientation exhibited superior properties compared to the XZ counterparts.

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