Analyzing 3D-Printed Samples on a PerkinElmer DMA 8000

San Jose State University, Charles W Davidson College of Engineering

BME 207 - Experimental Methods in Biomedical Engineering

Christopher Heying

December 13, 2024

Table of Contents

Executive Summary

The objective of our experiment is to determine how Material Type, Print Orientation, and Infill Density of a 3D sample will influence its Storage Modulus, Loss Modulus, and Tan Delta using a Dynamic Mechanical Analysis (DMA) machine.

Material Type was determined to have a statistically significant effect on the Storage Modulus and Tan Delta, with p-values of 0.008 and 0.001, respectively. No other input factors exhibited statistically significant effects on response variables. These results are unexpected and require further testing with improved sample size and true randomization.

Objective and Significance

The objective of our experiment is to determine how Material Type, Print Orientation, and Infill Density influence Storage Modulus, Loss Modulus, and Tan Delta for 3D printed specimens using the PerkinElmer 8000, a dynamic mechanical analysis machine. For our experiment, we will evaluate two materials: PETG and PLA, which will be printed in different orientations (x, y, z, and 45°) and with varied infill densities (20%, 40%, and 60%). As such, we will want to measure Storage Modulus, Loss Modulus, and the Tan Delta for all combinations of input factors. Each specimen will be a 20mm x 10mm x 4mm rectangular prism with a hexagonal infill geometry.

All three response variables are ratio data, as they all have a meaningful zero point. Storage Modulus (G') represents the stored energy in a material, indicating its stiffness [1]. It is crucial for understanding how the material deforms under stress. Loss Modulus (G'') reflects the energy dissipated as heat, indicating the material's viscosity [1]. This measurement helps assess how much of the material's response is elastic versus viscous. Tan Delta is the ratio of the loss modulus to the storage modulus $tan\delta = G''/G'$, representing the damping characteristics of the material [1].

Because the Storage Modulus, Loss Modulus, and Tan Delta of a material are all dependent on the temperature that the material is subjected to, we will be recording the maximums for each response variable since these maximums occur at particular temperature thresholds unique to the material [2]. The max Storage Modulus will occur when no external heat is applied to the material, as a lack of energy will increase the materials stiffness. Therefore, the max Storage Modulus is obtained at around 25ºC. The max Loss Modulus will occur near the glass transition temperature (T_g), which is when an amorphous material experiences mechanical loss by transitioning from a hard and brittle state to a soft and flexible state [2]. The glass transition temperature for PLA is between 55-65ºC and the glass transition temperature for PETG is between 75-85ºC [2]. The max Tan Delta will also occur near the glass transition temperature, and indicates the highest viscous to elastic response under deformation [3].

The DMA8000 allows for precise evaluation of the mechanical properties of polymers, which are used in the fabrication of medical devices due to their ease of processing, flexibility, and biocompatibility. Polymers intended for medical use must demonstrate sufficient tensile strength and durability to ensure the longevity and functionality of the device. Thus, understanding their mechanical behavior is critical, as polymeric materials play a pivotal role in the overall performance and success of medical implants and devices[4].

PETG and PLA plastics are widely used in medicine, as they have high impact resistance, durability, chemical stability and non-toxicity. They are also biocompatible, that is, they do not react adversely with biological tissues. This property makes them suitable for the manufacture of enclosures and components of medical equipment, such as breathing apparatus and automatic injection systems. Its compatibility with antimicrobial agents such as silver nanoparticles increases resistance to bacterial growth, which is crucial for medical applications where sterility is important [5].

Results and Discussion

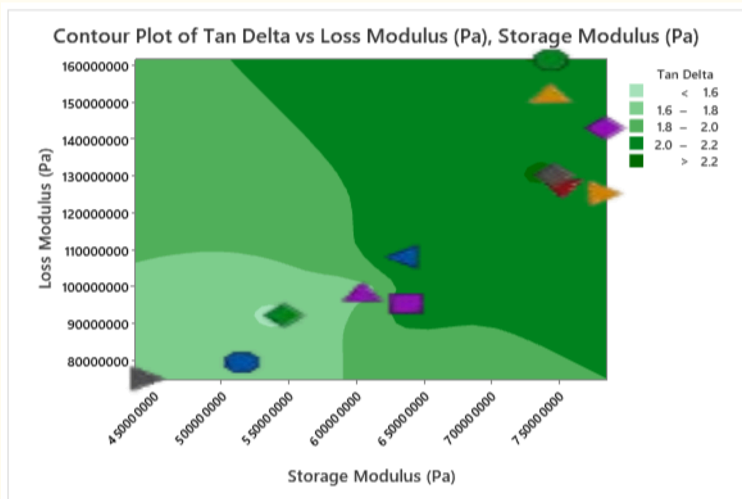
Modifications to DoE

In the original design for our experiment, we were to test 3 material types: PLA, ABS, and TPU. However, because we sourced our 3D printed samples from the SJSU Makerspace, we were reliant on the materials they had available. ABS was not available, so we substituted PETG for ABS. Also, due to time constraints and complexity, we removed TPU from Material Types to test. TPU has a very low glass transition temperature (T_g) of -20 to -40ºC, which would require the use of liquid nitrogen [6]. This would further complicate the procedure given our time constraints, and was therefore removed from the experimental design. Also, we originally planned to perform 3 replicates per unique factor level combination. Due to time constraints, testing of replicates was not feasible and our experimental design was updated to perform only 1 test per unique factor level combination.

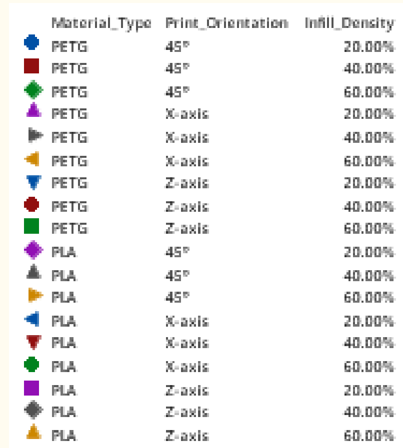
Results

Data were collected from 15 of the available 24 samples. It was determined that the samples printed in a y-orientation had 100% Infill Density due to limitations with 3D printing. As such, only 1 sample printed in the y-orientation for each Material Type was tested since the Infill Density between samples was constant. Of the PLA samples, 10 of the 12 samples were tested. Of the PETG samples, only 5 of the 12 samples were tested. Thus, only 15 of the 24 samples were tested and recorded. However, specimens printed in the y-orientation had 100% Infill Density due to 3D-printing limitations. Therefore, y-oriented specimens were omitted from analysis. Also, there was one outlier confirmed via a Grubbs outlier test, which was identified as PLA-45^{o}-40%. Incomplete testing and omission of data yields a small sample size (n = 12) of data that is unbalanced.

Figure 1.

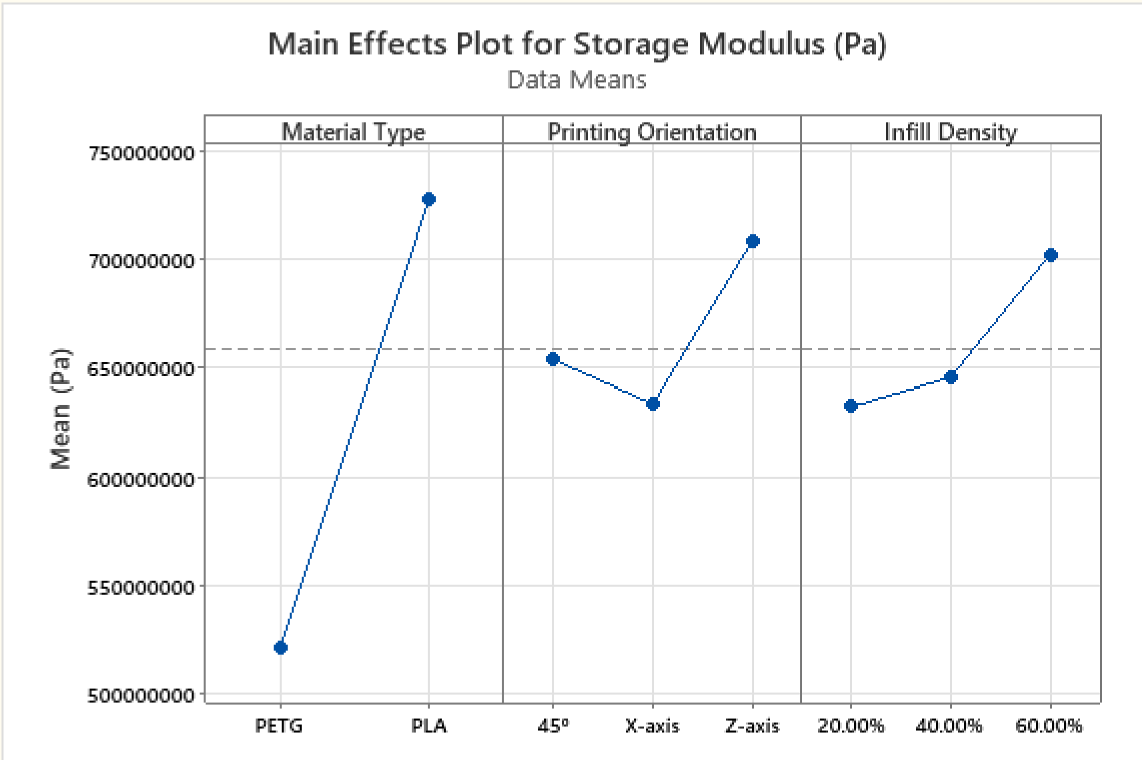


Legend



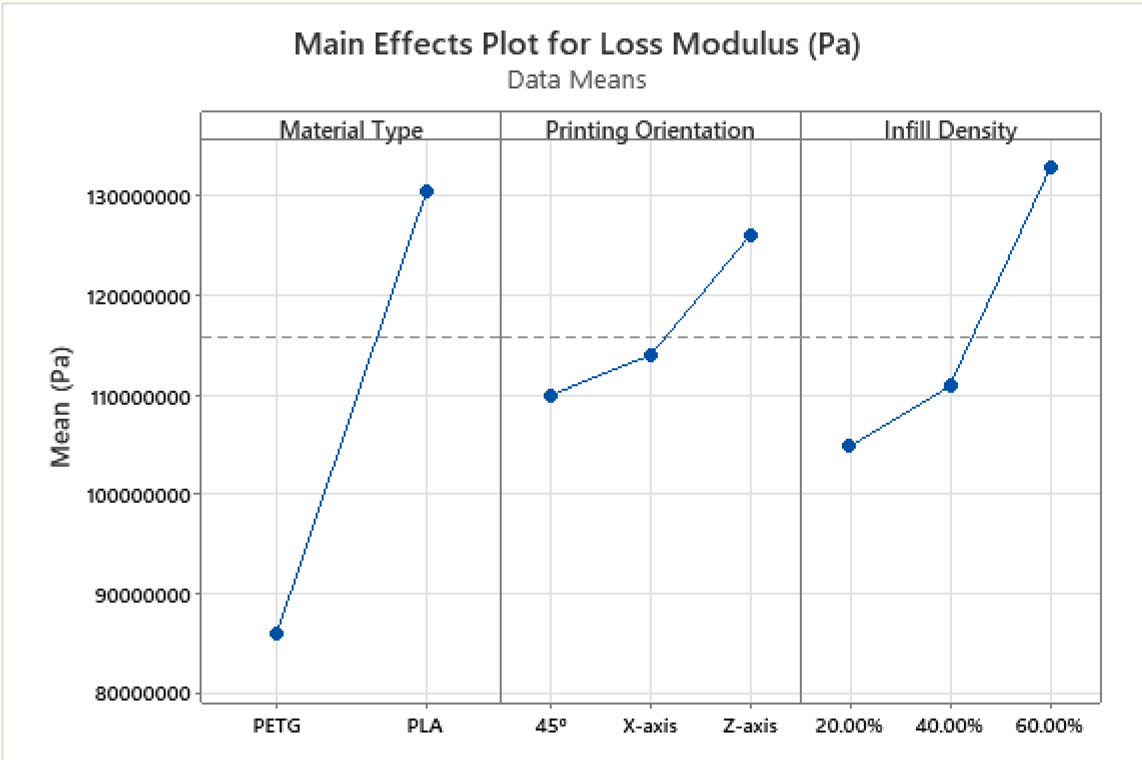
A contour plot of Storage Modulus, Loss Modulus, and Tan Delta demonstrates the effects for each combination of Material Type, Print Orientation, and Tan Delta. It can be observed that factor combinations that contribute to the largest combined magnitudes of response variables are located in the upper right corner of the contour plot. Some notable combinations being PLA-X-40% and PLA-Z-40%. The factor combinations that contribute to the smallest combined magnitudes of response variables are located in the bottom left corner of the contour plot, most notably of which is PETG-45º-60% and PETG-X-40%.

Figure 2.



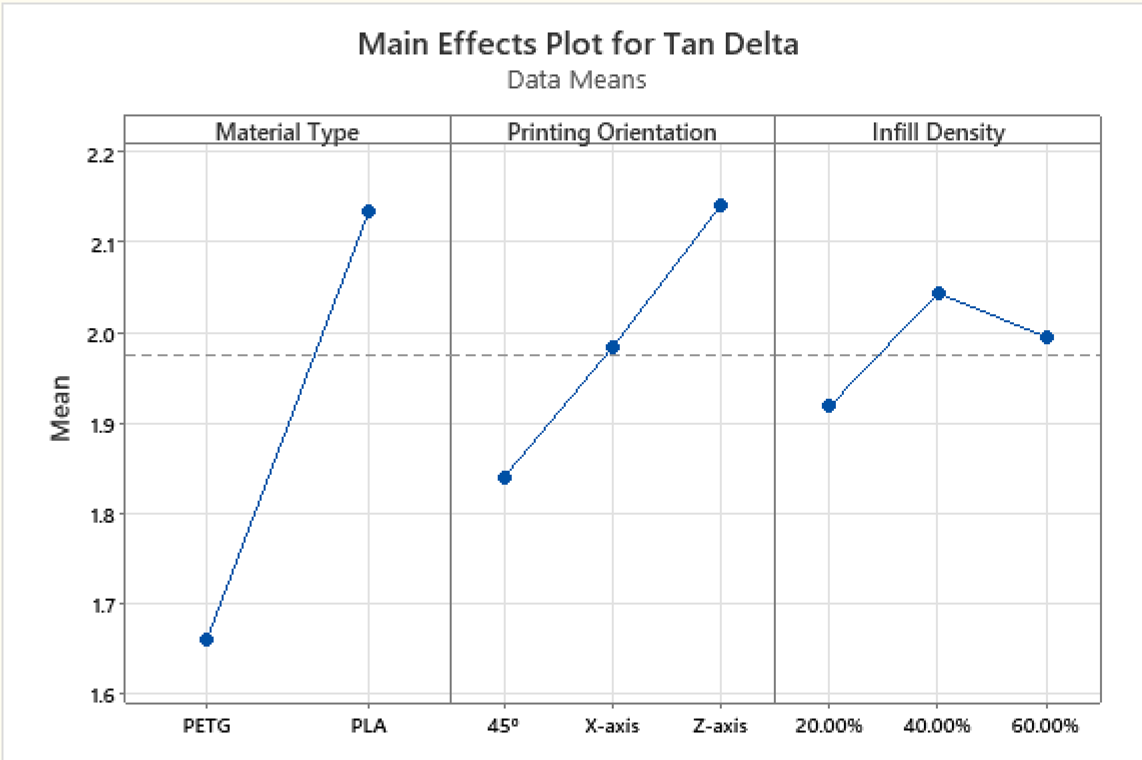
Storage Modulus is influenced by Material Type and Infill Density. Switching from PETG to PLA increases the Storage Modulus. A sample with Print Orientation on the X-axis decreases the Storage Modulus. A sample with Print Orientation on the Z-axis increases the Storage Modulus. As we increase the Infill Density of the 3D-printed sample, we increase the Storage Modulus.

Figure 3.



Loss Modulus is influenced by Material Type and Infill Density. Switching from PETG to PLA increases the Loss Modulus. Switching the Print Orientation of the 3D-printed sample from 45º to X-axis to Z-axis increases the Loss Modulus. As we increase the Infill Density of the 3D-printed sample, we increase the Loss Modulus.

Figure 4.



Tan Delta is influenced by Material Type. Switching from PETG to PLA increases the Tan Delta. Switching the Print Orientation from 45º to X-axis to Z-axis increases the Tan Delta. However, no clear effect on Tan Delta is observed from changes to Infill Density

Discussion

A Mixed Effects Model was performed using Minitab with a significance level of 0.05. This was the most effective method to analyze the data since our data is approximately normal, unbalanced, and contains a low sample size (n = 12). The Mixed Effects Model is able to account for these drawbacks, as well as handle unequal variances in the data. A Mixed Effects Model will also analyze the effect that different operators may have on experimental results. The results are unexpected and likely contain errors.

We observed that Material Type has a statistically significant effect on the Storage Modulus and Tan Delta, with p-values of 0.008 and 0.001, respectively. This is expected as PLA is known to have higher Storage Modulus values compared to PETG [7]. Since there are only two Material Types, no Post Hoc Analysis is required. Unexpectedly, by convention, Material Type has no statistically significant effect on Loss Modulus since the p-value is 0.054, which does not surpass the significance level threshold. This is unexpected since PLA is known to have higher Loss Modulus values compared to PETG [7]. These values are reflected in Figure 2, where we can see a clear difference in Loss Modulus means between PLA and PETG.

We observed that Printing Orientation has no statistically significant effect on Storage Modulus, Loss Modulus or Tan Delta, as the p-values are 0.450, 0.928, and 0.756, respectively. This is unexpected as Printing Orientation influences stiffness from the 3D-printing deposition direction [8]. We would have expected similar results of Storage Modulus, Loss Modulus, and Tan Delta for samples printed in the X-, Z-orientations (or horizontal orientations) since they are in the deposition direction [8]. It was also expected that a sample printed at 45^{o} would yield a lower Storage Modulus than the horizontal oriented samples, but have a higher Loss Modulus and Tan Delta. Instead, the 45^{o} sample had an average Storage Modulus and a lower Loss Modulus and Tan Delta.

We observed that Infill Density has no statistically significant effect on Storage Modulus, Loss Modulus or Tan Delta, as the p-values are 0.913, 0.547, 0.559, respectively. This is unexpected as the density of the material would influence stiffness [9]. By increasing infill density, we would be increasing the stiffness of the material while inversely decreasing the viscoelastic and damping characteristics of the material, resulting in an increase to Storage Modulus and a decrease to both Loss Modulus and Tan Delta [9, 10]. Even though there is no statistically significant effect of Infill Density on any response variable, the main effects plots for each response variable shows that as we increase Infill Density, we increase Storage Modulus, but unexpectedly increase the Loss Modulus as well. There is no clear pattern between Infill Density and Tan Delta.

The high degree of unexpected results is attributed to error from a low sample size (n = 12) and a lack of full randomization. The low sample size is attributed to an elimination of replicates, change in experimental design, incomplete data, and omission of y-orientation samples from the data. Eliminating replicates and removing the TPU Material Type from our experimental design was necessary given our groups time constraints from extended training duration on the DMA8000. Training time was extended out of scheduling conflicts with the trainer. Incomplete data is attributed to unexpected closure of the SJSU Lab, preventing our group from performing any more experimental runs. The amount of data points were further reduced since samples printed in the y-orientation technically have 100% Infill Density. Because 100% Infill Density is not a part of our experimental design, this data was omitted from statistical analysis, therefore further reducing the sample size. Our design also suffered from a lack of true randomization since our group decided to only randomize Print Orientation and Infill Density while assigning Material Type more so as a blocking factor. This was done to improve the efficiency for performing experiments since PETG and PLA have different glass transition temperatures, requiring different settings and timings to be set for each material on the DMA machine. However, this introduces potential confounding effects regarding timing of test runs and test run order. Also, given the incomplete data, the data missing is for the PETG Material Type, which may contribute to an unrealized effect of PETG on response variables.

Conclusion

It is apparent that Material Type has a significant influence on Storage Modulus and Tan Delta. This may be attributed to materials having unique mechanical properties. However, we found Material Type to not have a statistically significant effect on Storage Modulus. Infill Density and Printing Orientation were determined to have no effect on Storage Modulus, Loss Modulus, or Tan Delta. These results are unexpected and attributed to error from a low sample size and a lack of full randomization. For future experiments, sample size should be increased with the addition of replicates, full randomization should be employed, fabrication of y-oriented samples should be reevaluated, and the infill geometry should be varied (hexagonal, triangular, rectilinear, etc), as storage modulus, loss modulus, and tan delta are influenced by a sample's microstructure [11].

Appendix A: References

[1] D. Dunson, “Characterization of polymers using dynamic mechanical analysis (DMA),” *EAG Appl. Note*, pp. 1–8, 2017.

[2] What are the glass transition temperatures for PLA and PETG? ChatGTP. 2024 at ChatGPT

[3] J. A. Jansen, “Determining Glass Transition Temperature Using DMA,” The Madison Group, Jan. 03, 2023. https://madisongroup.com/determining-glass-transition-temperature-using-dma/

[4] D. S. Jones, “Dynamic mechanical analysis of polymeric systems of pharmaceutical and biomedical significance,” *Int. J. Pharm.*, vol. 179, no. 2, pp. 167–178, Mar. 1999.

[5] Prajapati, S., Sharma, J. K., Kumar, S., Pandey, S., & Pandey, M. K. (2024). A review on comparison of physical and mechanical properties of PLA, ABS, TPU, and PETG manufactured engineering components by using fused deposition modeling. *Emerging Materials Research*. https://doi.org/10.1016/j.matpr.2024.05.018

[5] Struz J, Trochta M, Hruzik L, Pistacek D, Stawarz S, Kucharczyk W, Rucki M. Wear and Dynamic Mechanical Analysis (DMA) of Samples Produced via Fused Deposition Modelling (FDM) 3D Printing Method. Polymers (Basel). 2024 Oct 28;16(21):3018. doi: 10.3390/polym16213018. PMID: 39518228; PMCID: PMC11548368.

[6] What is the glass transition temperature for TPU? ChatGTP. 2024 at ChatGPT

[7] Aryabhat Darnal, Zaryab Shahid, Himani Deshpande, Jeeeun Kim, Anastasia Muliana,Tuning mechanical properties of 3D printed composites with PLA:TPU programmable filaments,”Composite Structures”,Volume 318,2023,117075,ISSN 0263-8223,https://doi.org/10.1016/j.compstruct.2023.117075.(https://www.sciencedirect.com/science/article/pii/S0263822323004191)

[8] Anant Prakash Agrawal, Virendra Kumar, Jitendra Kumar, Prabhu Paramasivam, Seshathiri Dhanasekaran, Lalta Prasad,An investigation of combined effect of infill pattern, density, and layer thickness on mechanical properties of 3D printed ABS by fused filament fabrication,Heliyon,Volume 9, Issue 6,2023,e16531,ISSN 2405-8440,https://doi.org/10.1016/j.heliyon.2023.e16531.(https://www.sciencedirect.com/science/article/pii/S2405844023037386)

[9] Thumsorn S, Prasong W, Kurose T, Ishigami A, Kobayashi Y, Ito H. Rheological Behavior and Dynamic Mechanical Properties for Interpretation of Layer Adhesion in FDM 3D Printing. Polymers (Basel). 2022 Jul 3;14(13):2721. doi: 10.3390/polym14132721. PMID: 35808766; PMCID: PMC9268954.

[10] Aw YY, Yeoh CK, Idris MA, Teh PL, Hamzah KA, Sazali SA. Effect of Printing Parameters on Tensile, Dynamic Mechanical, and Thermoelectric Properties of FDM 3D Printed CABS/ZnO Composites. Materials (Basel). 2018 Mar 22;11(4):466. doi: 10.3390/ma11040466. PMID: 29565286; PMCID: PMC5951312.

[11] Fan Liu, Yawen Zhao, Licheng Chen, Zili Yuan, Tao Shi, Shushan Cui, Dongli Zou, Lifeng He, Dawu Xiao, Zhaoping Luo, Bin Su, Tao Fa, Kezhao Liu, A phenomenological study on the microstructure dependence of the internal friction in U–Nb shape memory alloy, Materials Science and Engineering: A, Volume 883, 2023, 145470, ISSN0921-5093, https://doi.org/10.1016/j.msea.2023.145470.(https://www.sciencedirect.com/science/article/pii/S0921509323008948)

Appendix B: Data Table

Standard Order	Run Order	Material Type	Print Orientation	Infill_Density	Max Storage Modulus (Pa)	Max Loss Modulus (Pa)	Max Tan Delta (Pa)	Operator
8	1	PLA	Z-axis	40%	747,767,709	130,490,789	2.3196630	Brian
11	2	PLA	45º	40%	339,284,429	72,251,384	2.1129550	Pedro
2	3	PLA	X-axis	40%	752,651,730	127,504,140	2.0198600	Chris
6	4	PLA	Y-axis	60%	762,304,384	148,791,399	2.1011980	Pedro
5	5	PLA	Y-axis	40%				
1	6	PLA	X-axis	20%	635,288,554	108,382,440	2.1788110	Pedro
7	7	PLA	Z-axis	20%	633,678,104	95,448,440	2.1011980	Pedro
10	8	PLA	45º	20%	785,162,712	143,362,556	2.0329060	Chris
9	9	PLA	Z-axis	60%	743,837,513	152,169,163	2.0020740	Chris
12	10	PLA	45º	60%	782,102,267	125,401,327	2.1618150	Albina
4	11	PLA	Y-axis	20%				
3	12	PLA	X-axis	60%	743,051,151	161,613,302	2.2444280	Albina
17	13	PETG	Y-axis	40%	678,321,384	111,591,029	1.5836290	Albina
13	14	PETG	X-axis	20%	599,772,967	97,722,832	1.6832330	Albina
24	15	PETG	45º	60%	540,104,961	92,028,881	1.5695990	Brian
22	16	PETG	45º	20%	507,349,988	79,360,295	1.5948080	Pedro
20	17	PETG	Z-axis	40%				
18	18	PETG	Y-axis	60%				
21	19	PETG	Z-axis	60%				
15	20	PETG	X-axis	60%				
23	21	PETG	45º	40%				
14	22	PETG	X-axis	40%	436,595,181	74,762,963	1.7889000	Brian
19	23	PETG	Z-axis	20%				
16	24	PETG	Y-axis	20%				