JJMIE

Volume 18, Number 2, June. 2024
ISSN 1995-6665
Pages 421- 429
https://doi.org/10.59038/jjmie/180214
Editor-in-Chief: Prof. Ali M. Jawarneh

Jordan Journal of Mechanical and Industrial Engineering

Exploring Shore D Hardness Variations Under Different Printing Conditions and Post-processing Treatments

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Received 14 Dec 2023 *Accepted 1 Mar 2024*

Abstract

This study investigated the influence of two key printing parameters—namely, layer thickness and infill percentage and also the annealing effect, on the hardness of PLA and ABS components. Employing the design of experiment (DOE) methodology, the most significant factor influencing the Shore D hardness values for each material was established, in addition to determining the optimal printing parameters that yield maximum hardness in the printed parts. Our findings reveal that layer thickness significantly impacts the hardness of 3D printed PLA and ABS specimens, with printing infill percentage exerting a comparatively smaller influence, while in case of PLA annealed samples, the most significant factor is infill percentage.

Keywords: 3D printing, PLA, ABS, annealing, DOE, Shore D hardness.

1. Introduction

Additive manufacturing, commonly known as 3D printing, has emerged as a revolutionary technology in the field of manufacturing, offering unparalleled design freedom and rapid prototyping capabilities. As this technology continues to evolve, understanding the mechanical properties of 3D-printed materials becomes paramount, especially when considering applications in engineering, aerospace, and medical fields. Shore D hardness, a measure of a material's resistance to indentation, stands as a crucial parameter in evaluating the mechanical performance of 3D-printed components.

Poly-lactic acid (PLA) and acrylonitrile butadiene styrene (ABS) are among the most widely used thermoplastic polymers in 3D printing, each offering distinct advantages and challenges. PLA, derived from renewable resources, has gained popularity for its biodegradability and ease of use. ABS, on the other hand, is valued for its durability and thermal resistance. Additionally, the post-print annealing process has been explored as a means to enhance the mechanical properties of PLA, potentially expanding its range of applications.

The comprehensive examination of major material properties, including tensile and flexural strength [1–10], impact resistance [11–15], and fracture toughness [16–18], has been extensively conducted for various common material, such as ABS, PLA, polycarbonate, and nylon.

Among the many fields of applications (automotive industry, aerospace, medical applications) of 3D printing technology is the manufacture of spur gears, since it offers a range of advantages, from customization and rapid prototyping to the ability to create complex geometries.

Material hardness has a significant impact on the properties and performance of 3D printed spur gears. Spur gears with higher hardness can show better wear resistance, making them more durable over time. This is especially important for gears that are subject to heavy loads or constant friction.Polymers with higher hardness can be less elastic, which can reduce their ability to absorb shock. This can lead to a greater susceptibility to cracking or other forms of damage when subjected to impact.Hardness can also influence spur gears grip. The gears with a lower hardness may have better grip on slippery surfaces, while gears with a higher hardness may be better suited for rough surfaces.Lower hardness polymers can allow gears to be more flexible, which can be beneficial in certain applications where flexibility is essential. On the other hand, the hardness of the polymer can also influence the 3D printing process itself. Harder polymers may require different print settings, such as higher temperatures or slower print speeds, to ensure proper adhesion between layers and prevent warping.

Maguluri et al. [19] investigated the impact of three crucial printing variables—namely, infill percentage, extrusion temperature, and printing speed—on the hardness of components made from polylactic acid (PLA). Employing Taguchi's Design of Experiment (DOE) methodology, they efficiently assessed printing configurations that enhance part hardness while minimizing the number of experiments. The analysis utilized signal-to-noise (S/N) ratios to pinpoint optimal parameters, and the individual contributions of these variables were quantified through analysis of variance (ANOVA). The results

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underscored the substantial influence of extrusion temperature on the hardness of 3D printed PLA specimens, whereas printing speeds exhibited a relatively minor effect on this property.

In a parallel study, Vishwas et al. [20] concentrated on appraising the impact of process parameters (pattern orientation, layer thickness, and shell thickness) in Fused Deposition Modeling (FDM) on crucial characteristics such as ultimate tensile strength and dimensional accuracy of ABS and Nylon materials. Utilizing Taguchi analysis, they discovered that the orientation angle and coating thickness significantly affected ultimate tensile strength and moderately influenced dimensional accuracy. The emphasis was on achieving optimal manufacturing outcomes.

Similarly, another investigation [21] employed ANOVA analysis to identify notable factors influencing the hardness of PLA 3D printed parts, pinpointing nozzle diameter as a significant factor. Additionally, for tensile strength, the study determined that the critical factor was the printing direction.

This scientific paper aims to delve into the intricacies of Shore D hardness in 3D-printed PLA, annealed PLA, and ABS, with a meticulous consideration of various printing parameters. By systematically examining the impact of key factors such as infill percentage and layer thickness and also post-processing heat treatments, we seek to unravel the nuanced relationships between printing conditions and material hardness.

The significance of this research lies in its potential to optimize 3D printing processes for specific applications. Understanding how printing parameters influence Shore D hardness not only provides insights into material behavior but also guides the engineering of components with tailored mechanical properties. The outcomes of this study are anticipated to contribute to the broader knowledge base of additive manufacturing, offering practical implications for industries relying on the precision and reliability of 3D-printed materials. The paper's novelty lies in its focused exploration of specific printing parameters, consideration of the annealing effect, use of a structured methodology, identification of key factors, optimization goals, and material-specific findings. These elements collectively contribute to the originality and significance of the research, offering valuable insights for advancing the field of 3D printing and material science.

2. Methodology

The hardness of 135 3D printed samples was evaluated according to the standard ISO 868:2003(En), Plastics and Ebonite — Determination of Indentation Hardness by Means of a Durometer (Shore Hardness) [22], in 3 points on each sample. The shape and dimensions of the samples are presented in Figure 1.

The average hardness for the tested samples were calculated and used for statistical analysis performed using the MiniTab software.

The hardness was measured using the device presented in Figure 2, on the specimens constructed for the tensile test, for each investigated material, namely PLA, ABS and PLA annealed. The hardness values were determined on a number of 135 samples, with filling percentages of 50%, 75% and 100%, and layer height 0.10 mm, 0.15 mm and 0.20 mm.

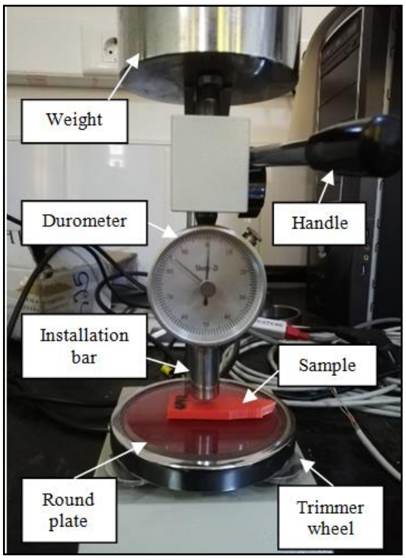


Figure 2. Shore D harness testing device [23].

In Table 1 can be seen the characteristics extracted from providers data sheets, for PLA and ABS filaments utilized in the study.

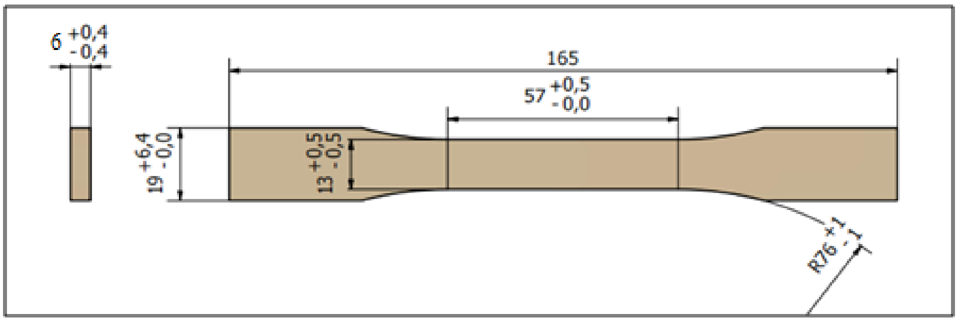


Figure 1. Shape and dimensions of 3D printed specimens used for hardness test.

Table 1. Characteristics of PLA and ABS filaments used in the present study

Materials	Extrusion temperature (°C)	Bed temperature (°C)	Density (g/cm^3)	Ultimate tensile Strength (MPa)	Specific deformation (%)	Charpy impact strength (kJ/m^2)
PLA	210 ± 10	25-60	1.31±0.02	15.5-72	34.5 ± 8.1	5.7 ± 0.4
ABS	210 + 40	110 ± 10	1.10	33.9	4.8	10.5

The printing process utilized the Raise E2 3D printer, which has a volume capacity of 330×240×240mm. Specifically, for the current study (refer to Table 2), the chosen printing parameters were as follows: build orientation in the X-Y model using lines and oriented at 45 degrees.

Table 2. Printing specifications

Printing settings	PLA	ABS
Shell width (mm)	1	1
Infill speed (mm/s)	70	40
Estimated print time (min)	46	60
Estimated filament used (g)	10.6	10.6
Extruder temperature (°C)	210	240
Bed temperature (°C)	60	110
Platform addition	Raft only	Raft only

The software generating the infill is ideaMaker, using alternatives layers made with lines (135,45 degrees orientation) as can be seen in Figure 3.

For the annealing heat treatment, PLA material samples were subjected to a 3-hour period at a temperature of 75°C, with a gradual cooling process in an oven. The experimental design for each mechanical property involved the utilization of the design of experiments (DOE) - full factorial design method through Minitab 19 software [24–27]. Table 3 presents the levels of each investigated parameter.

Table 3. Design of experiments analysis – levels of analyzed parameters.

Parameter	Level		
	1	2	3
Infill percentage, %	50	75	100
Layer thickness, mm	0.10	0.15	0.20

It resulted, therefore, an orthogonal array of 3^2 values. It resulted, therefore, an ortogonal array of 9 values, based on 2 parameters and 3 levels.

3. Results and discussion

3.1. Shore D hardness experimental investigation

The mean values of Shore D hardness are presented in Figure 4, making a comparison between different materials (PLA, PLA annealed and ABS), and highlighting the influence of printing parameters (infill percentage and layer thickness) on the obtained results.

It can be seen that PLA samples exhibited the highest values of Shore D hardness, for all considered printing parameters, while the lower Shore D hardness was obtained in case of ABS 3D printed samples.

Comparable observations regarding the Shore D hardness of ABS material are corroborated in the bibliographic source [28]. According to this reference, the average Shore D hardness value before exposure to heat treatment is 65.82, while the research presented in this work indicates an average pre-treatment Shore D value of 60.13. Similar results for the material ABS, of Shore D hardness are identified in reference [28], where the average values before heat treatment is 65.82 Shore D, compared to the results obtained in this study where it was obtained an average value of 60.13 Shore D.

The hardness values are similar to those obtained by the same Shore D method in other studies in the analyzed bibliography: values above 60 Shore D for PLA [29,30] and around the average of 78 Shore D [31,32].

3.2. Statistical analysis

Pareto charts extracted from MiniTab software (Figure 5) in the context of examining the Shore D hardness of PLA (polylactic acid, a type of biodegradable plastic often used in 3D printing), the Pareto chart shows the impact of two factors on the Shore D hardness of PLA (Figure 5, a).

On the graph are listed the influencing factors A and B, each with a bar indicating the "Standardized Effect". The standardized effect is a measure of the impact of each factor on the response of the studied variable, in this case Shore Dhardness. Factor A has a significantly greater effect than factor B, as seen by the length of its bar. The exact value of the standardized effect for factor A (layer thickness is 2.776), which is above the reference line for significance (indicated by the red dotted line). Factor B (infill percentage) has a shorter bar, indicating that its effect is smaller and below the significance threshold.

In conclusion, in this specific study, layer thickness in 3D printing of PLA has a greater impact on Shore D hardness than infill percentage. If the red dotted line represents the level of statistical significance ($\alpha = 0.05$), then we can infer that only the layer thickness has a statistically significant effect on the Shore D hardness of PLA.

Another Pareto chart (Figure 5 b) aims to evaluate the standardized effects of different factors on Shore D hardness, this time for ABS (acrylonitrile butadiene styrene, another common material used in 3D printing).As in the previous graph, we have two terms, A and B, with the following characteristics:factor A stands for "Layer thickness, mm" and has a standardized effect of 2.776, indicated by the long blue horizontal bar that exceeds the significance line (dotted red line). This suggests that layer thickness is a significant factor in determining the Shore D hardness of ABS.Factor B stands for "Infill percentage, %" and has a significantly shorter bar, suggesting that it has less effect on the Shore D hardness of ABS and is not considered significant in this context as it does not cross the significance line.

The alpha value (α) indicated as 0.05 represents the significance level used for statistical hypothesis testing. If a bar exceeds the red dotted line, then the effect of that factor is considered statistically significant at that significance level.

Therefore, in this study, it can be inferred that the layer thickness has a significant impact on the Shore D hardness of the ABS material under the given conditions, while the filling percentage has no significant effect. This analysis could be used to optimize the 3D printing process to achieve a desired Shore D hardness of ABS printed objects.

Figure 5 c) shows the Pareto graph for heat-treated PLA material. Factor B is now the one with the largest standardized effect, having a value of 2.776. This is indicated by the long blue horizontal bar, which significantly exceeds the significance line (dotted red line), suggesting that it is a significant factor in determining the Shore D hardness of PLA.

Factor A, on the other hand, has a shorter bar, indicating a smaller effect on Shore D hardness of treated PLA. For this graph, the terms are reversed compared to the previous graphs, thus: factor A, which is "Layer thickness, mm", has less impact, and factor B, which is "Infill percentage, %", is now the most influential factor.Based on this graph, it can be concluded that the filling percentage is a significant factor in influencing the Shore D hardness of PLA annealed due to a better adhesion of the layers determined by the heat treatment above the glass transition point which increases the penetration resistance of the surface with a tougher body. Although the average hardness values are not significantly higher for the heat-treated samples, the statistical analysis reveals the change in the influence of the determining factors (filling percentage and layer thickness) which signifies a change in the internal structure of the material.

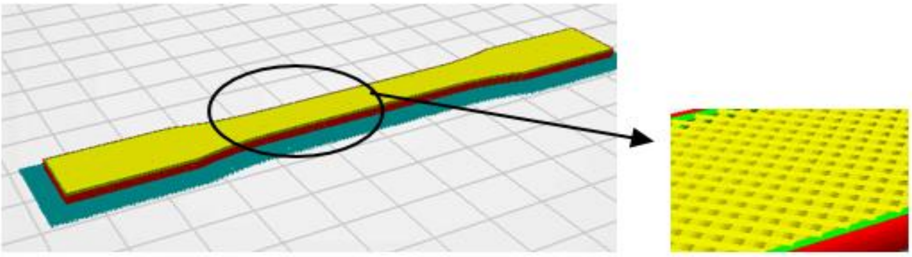


Figure 3. Infill aspect of 3D printed specimens.

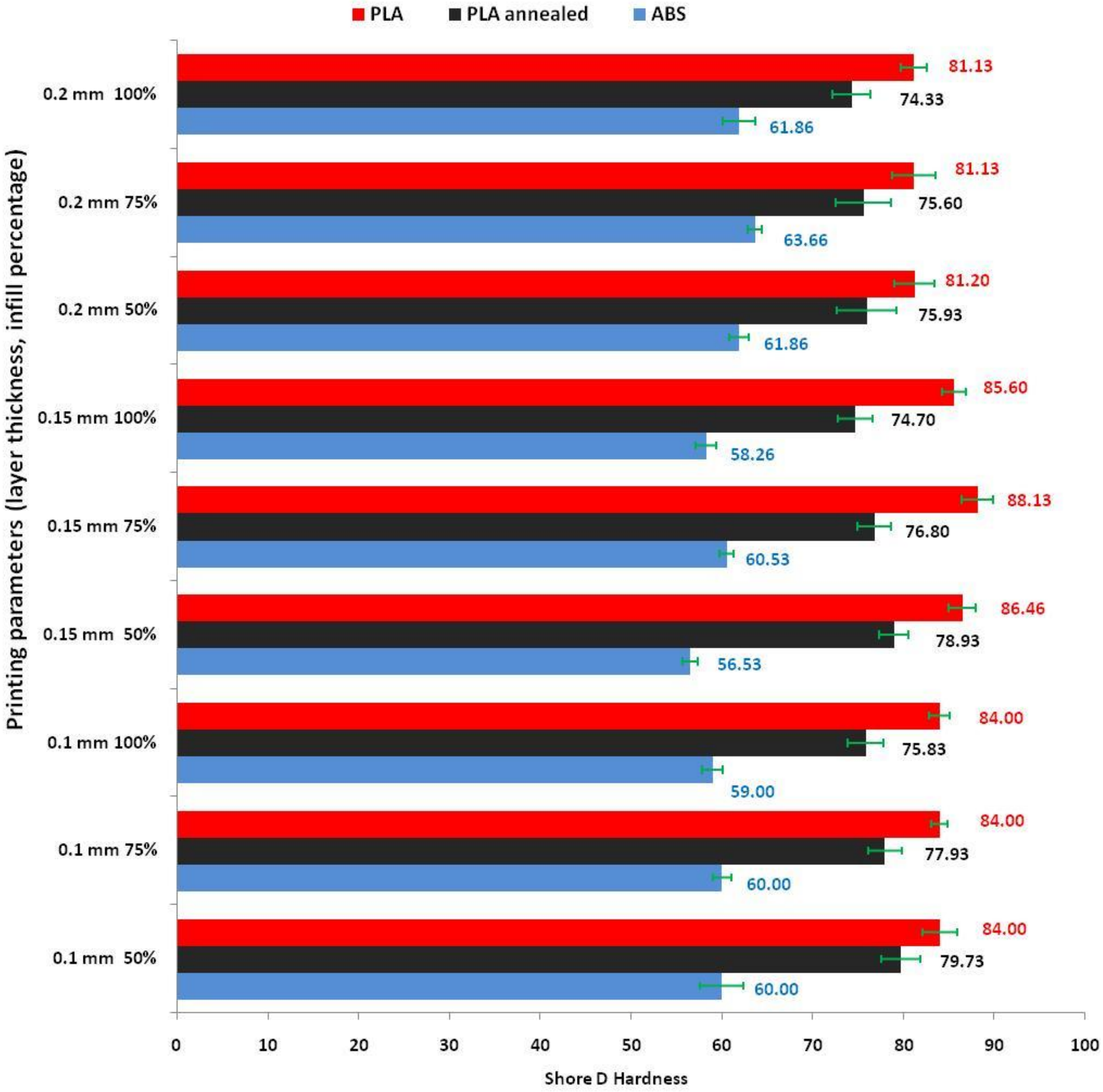
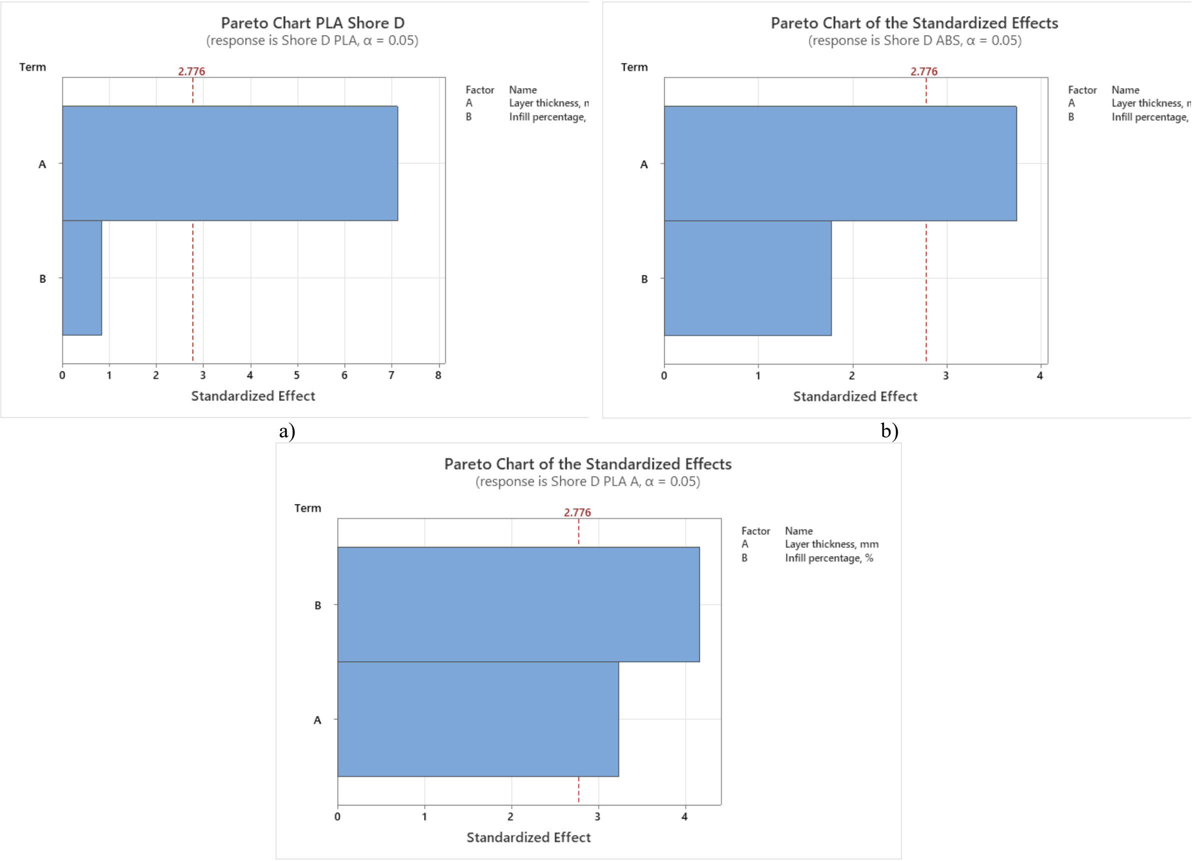


Figure 4. Shore D Hardness results for PLA, PLA annealed and ABS materials considering different printing parameters.



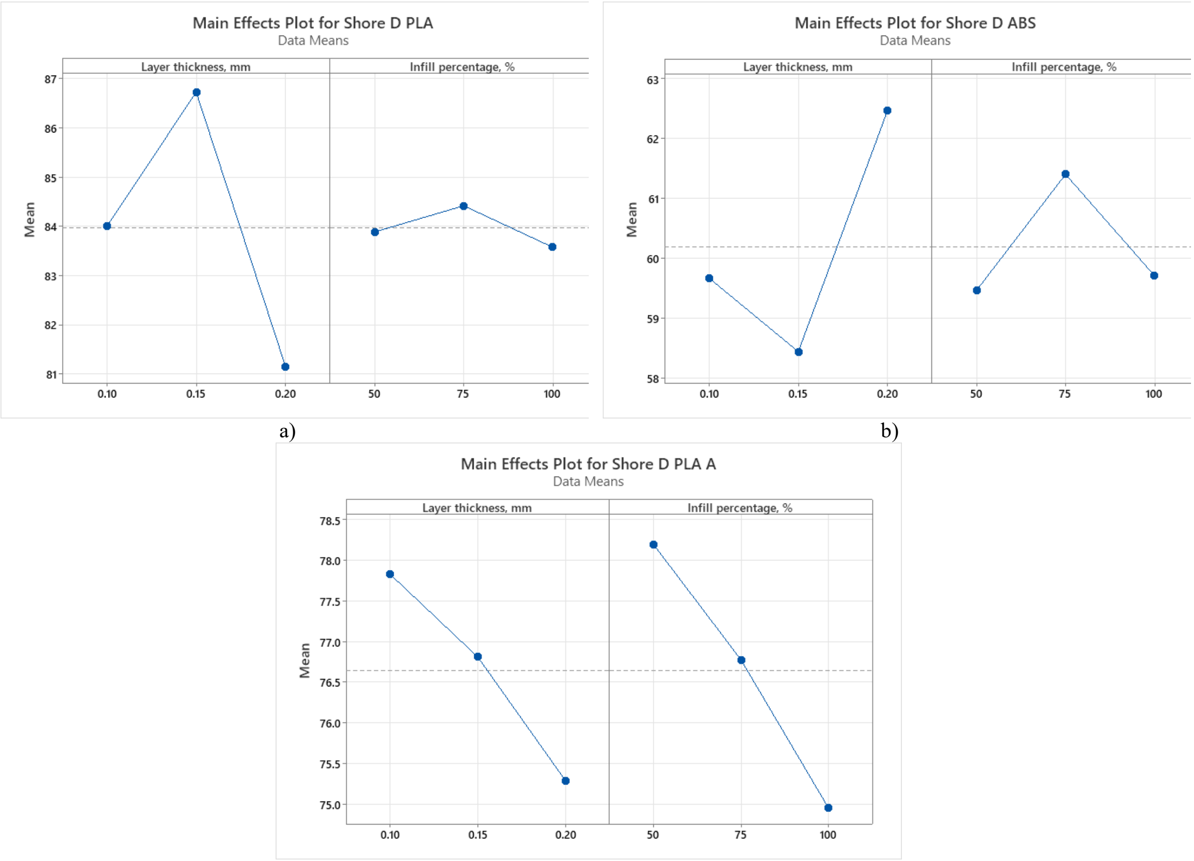
a) b)

c)

Figure 5. Pareto charts regarding the influence of printing parameters on Shore D hardness for: a) PLA, b) ABS, c)PLA annealed.

The three plots in Figure 6 are main effects plots for Shore D hardness for PLA and ABS, showing the mean responses for different levels of layer thickness and infill percentages. In the first plot (Shore D PLA) the mean Shore D hardness reaches a point maximum at a layer thickness of 0.15 mm and decreases at both 0.10 mm and 0.20 mm. This pattern suggests that an intermediate layer thickness is optimal for achieving the highest hardness. The decrease in hardness at thinner and thicker layer thicknesses could be attributed to inadequate bonding between layers, leading to reduced structural integrity and hardness. For the fill percentage, the average hardness decreases slightly as the fill percentage increases from 50% to 100%. The slight decrease in average hardness with increasing infill percentage from 50% to 100% suggests that higher infill densities may lead to a slight reduction in hardness. This could be due to the increased presence of internal voids and reduced material density at higher infill percentages, resulting in lower overall hardness. In the second graph (Shore D ABS): the average Shore D hardness increases significantly at a layer thickness of 0.20 mm and then decreases at a thickness of 0.15 mm, due to the ability of thicker layers to provide better interlayer adhesion, resulting in higher hardness. However, beyond a certain thickness, excessive material deposition could lead to reduced hardness due to issues such as over-extrusion or poor cooling. The average Shore D hardness has a maximum at one percent of filling of 75%, with lower values at 50% and 100%. This trend indicates that an intermediate infill percentage is optimal for achieving maximum hardness, due to a balance between material density and internal structure. In the third plot (Shore D treated PLA): a constant decrease in average Shore D hardness is correlated with increasing layer thickness from 0.10 mm to 0.20 mm, indicating that thinner layers result in higher hardness. The decrease in hardness with thicker layers could be attributed to reduced layer adhesion and increased porosity, which negatively impact the overall hardness of the annealed printed parts. Also, a steady decrease in average hardness is observed as the filling percentage increases from 50% to 100%, similar to as-built PLA samples.

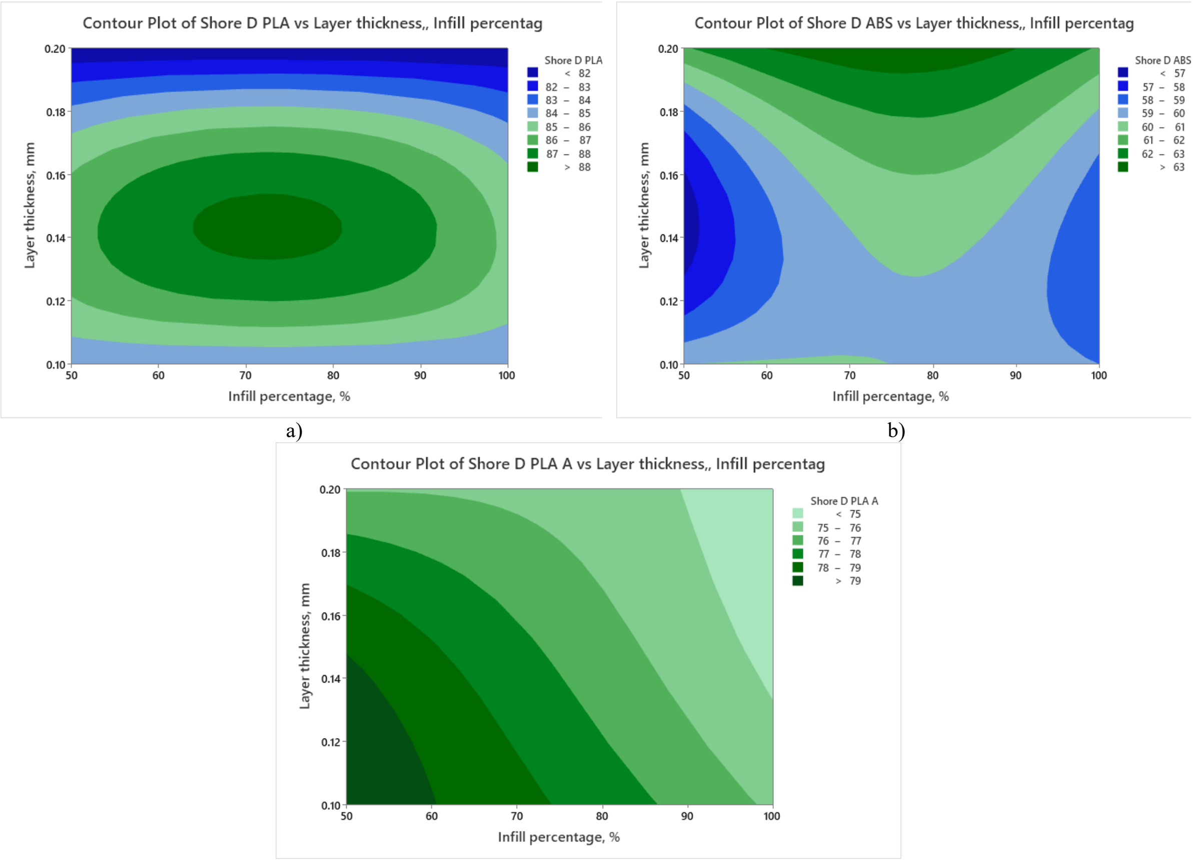
Comparing the three graphs, for ABS, the layer thickness of 0.15 mm seems to be optimal for obtaining the highest Shore D hardness, while for PLA, this effect varies depending on the type of PLA used (with or without heat treatment).In all cases, a filling percentage of 75% is the value that produces the highest Shore D hardness, indicating that an intermediate filling percentage is more effective than the extreme values for both materials. We note that, for PLA, increasing the layer thickness consistently leads to a decrease in Shore D hardness, suggesting that a thinner layer could contribute to a higher hardness of the finished part. These observations are preferably used to optimize 3D printing settings to achieve desired levels of hardness, taking into account the differences between materials and their specific properties.



a) b)

c)

Figure 6. Main effect plots for: a) PLA, b) ABS, c)PLA annealed.



a) b)

c)

Figure 7. Contour plots for:a) PLA, b) ABS, c) PLA annealed.

Contour plots showing the relationship between Shore D hardness, layer thickness and infill percentage for PLA and ABS are shown in Figure 7.

In the first graph (Shore D PLA) the green areas represent the higher Shore D hardness values, which are concentrated around a layer thickness of about 0.15 mm and a fill percentage of around 70-80%.

Lower values of Shore D hardness are represented in blue and occur at layer thicknesses of less than or greater than 0.15 mm and at varying infill percentages.

In the second graph (Shore D ABS) the blue areas indicate the lower Shore D hardness values and are more prominent at layer thicknesses of 0.10 mm and 0.20 mm, regardless of infill percentage.

The higher hardness values for ABS are represented by the green areas and are located at a layer thickness of approximately 0.15 mm, with the filling percentage varying between 65% and 90%.

The third graph (Shore D PLA treated) higher harnesses are indicated by the dark green area and appear to be concentrated at lower layer thicknesses and fill percentages below 60%.

Lower Shore D hardness values are represented by lighter green and are associated with greater layer thicknesses and higher fill percentages.

When comparing the three contour plots, for PLA, there is a general trend where a medium layer thickness and a higher fill percentage lead to a higher Shore D hardness. However, this trend is different for treated PLA, where higher hardness is associated with lower layer thicknesses.

For ABS, the maximum Shore D hardness is higher at a layer thickness of 0.2 mm, with less sensitivity to fill percentage variations compared to PLA.

4. Conclusions

The analysis of Shore D hardness values for PLA, PLA annealed, and ABS materials, taking into account various printing parameters like infill percentage and layer thickness, has yielded several significant observations. PLA consistently exhibited the highest Shore D hardness values across all printing parameters, establishing it as a material with notable rigidity. In contrast, ABS 3D printed samples consistently displayed lower Shore D hardness, aligning with findings from relevant literature.

In terms of statistical analysis, the study revealed distinct influences of printing parameters on Shore D hardness for PLA and ABS. For PLA, the layer thickness was identified as having a significantly greater impact than the infill percentage, underscoring the importance of optimizing layer thickness in the 3D printing process. Conversely, for ABS, layer thickness emerged as a significant factor influencing Shore D hardness, while the effect of infill percentage was less pronounced.

The insignificant influence of infill percentage on the Shore D hardness of 3D printed PLA and ABS parts, compared to layer thickness, can be understood through the mechanics of the printing process and the properties of the materials involved. In 3D printing, layer thickness directly impacts the resolution and structural integrity of the printed object. Thicker layers may result in fewer layers overall, potentially reducing the overall strength and hardness of the part. Conversely, thinner layers allow for finer detail and more uniform distribution of material, potentially leading to stronger and harder prints.

On the other hand, infill percentage primarily affects the internal structure and density of the printed part. While it may influence factors such as weight and material usage, its impact on surface properties such as hardness, as measured by the Shore D scale, is often minimal. This is because the Shore D hardness test primarily evaluates the resistance of the material's surface to indentation, which is determined more by the material composition and outer layers rather than the internal structure. While infill percentage may affect the overall density of the part, its influence on the material's fundamental properties is relatively limited. Additionally, 3D printing involves melting and extruding filament layer by layer to build the object, with the outer layers typically solid regardless of infill density. As the Shore D hardness test measures surface hardness rather than internal structure, changes in infill percentage may not significantly affect the measured propertiesThe examination of annealed PLA samples indicated a shift in influencing factors. Layer thickness exhibited a diminished effect, while infill percentage became a significant factor. This suggests that heat treatment influenced the internal structure and adhesion of layers in PLA, altering the factors that primarily determine Shore D hardness.

Main effects plots provided additional insights, revealing varying optimal conditions for PLA hardness depending on the type of PLA used (with or without heat treatment). For ABS, the plots indicated a significant increase in hardness at a layer thickness of 0.15 mm and a 75% infill percentage, highlighting specific conditions that contribute to enhanced material hardness.

Contour plots further illustrated the intricate relationships between Shore D hardness, layer thickness, and infill percentage for PLA and ABS. The general trend for PLA suggested that moderate layer thickness and higher infill percentages led to increased hardness. Treated PLA, however, exhibited higher hardness associated with lower layer thicknesses. ABS demonstrated higher Shore D hardness at a layer thickness of 0.2 mm, with less sensitivity to infill percentage variations compared to PLA.

Regarding the increase in hardness at 50% infill compared to 100% infill, one potential explanation for the observed phenomenon is the influence of interlayer adhesion on overall hardness. At higher infill percentages, there may be increased interlayer bonding, resulting in a more cohesive structure and higher hardness. However, beyond a certain limit, excessive infill density may lead to reduced interlayer bonding or increased internal stresses, compromising the overall hardness of the part.

In conclusion, this comprehensive study sheds light on the nuanced interactions between printing parameters and Shore D hardness for different 3D printing materials. The findings underscore the material-specific considerations and emphasize the need for precise optimization of printing conditions to achieve desired hardness levels. This research contributes valuable insights to the ongoing efforts aimed at enhancing the accuracy and reliability of 3D printing processes across diverse applications.

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