Investigation of Additive Manufacturing of Biocompatible Objects: Optimization of Degradation, Mechanical and Physical Properties

Y. Kartal * (https://orcid.org/0000-0002-5102-7655)

Kırıkkale University, Faculty of Engineering and Natural Sciences, Department of Mechanical Engineering, Kırıkkale Turkey.

email: y.kartal@kku.edu.tr

Abstract-The effect of production inputs on product properties in the production of polyethylene terephthalate (PET) material by additive manufacturing method was investigated and optimum production parameters were tried to be determined. The degradation of PET material in different infill patterns, mechanical and physical properties of the samples produced at different print speeds, layer thicknesses and infill patterns were investigated. In the study, it was determined that the most effective input on the mechanical properties of the samples was the infill pattern, followed by the print speed and the least effective input was the layer thickness. However, it was determined that the most effective inputs on the surface roughness and dimensional accuracy of the produced samples were layer thickness and infill pattern, respectively. Since the degree of influence of the infill pattern on mechanical and physical properties is higher than other production inputs, hydrolytic degradation properties of 9 different infill patterns were investigated.

Keywords: Degradation, additive manufacturing, optimization, biopolymer, green production

Graphical abstract

Graphical summary is given in Figure 1.

1. Introduction

Polyethylene terephthalate (PET) is one of the material types with a very high production amount due to its consumption in recent years. 3965 tonnes of PET fibre was used in 2024 [1]. This material, which is especially used in the packaging sector, is preferred due to its superior chemical resistance and biocompatibility. Depending on the high production amount of PETs, production technologies are also widespread. However, the use of PETs in additive manufacturing has been limited so far. In particular, the effect of production parameters on degradation properties has been little studied. In addition, the study of the effect of production parameters on mechanical and physical properties has also been limited. In this study, optimum production parameters for PET material production by additive manufacturing method were investigated.

Especially in the last decade, additive manufacturing using recycled materials has become one of the most interesting application areas. The application of additive manufacturing (AM) methods to polymer materials is particularly noteworthy. Biocompatible [2], wear-resistant [3], electrically conductive [4], chip-reinforced objects [5] have been produced using polymers and their properties have been studied. However, the investigation of the degradation property, which is an important data in the recycling of products, has remained limited. In the limited number of studies conducted in this context, properties such as sensor production [6] or mechanical properties of the produced materials [7] have been investigated. Determination of the degradation properties of PET materials in the additive manufacturing method, which has become an interesting application area in the last decade, is undoubtedly of vital importance in terms of preventing environmental problems caused by PET materials.

Additive manufacturing, which is based on the principle of producing objects layer by layer, is an indispensable application area due to its advantages such as almost no waste material, dimensional accuracy and relatively fast production [8]. One of the most common application principles of this method is the extrusion method. In this method, production is defined by laying and solidifying the filament, which is called filament and which is brought into a certain form of the material to be produced, in a hot metal area called nozzle after being heated, and laying and solidifying the next layer on the layer formed [9, 10]. Today, the use of PETs produced with high technologies in additive manufacturing method is promising in terms of using the products for many years. There are studies using recycled PET materials in the literature [11, 12, 13]. When the studies are analysed, it can be concluded that PETs will be used in the Eİ method especially from the end of the 2020s [14, 15, 16].

The aim of this study was to investigate the effect of print speed, layer thickness and infill pattern on the degradation, mechanical and physical properties of the produced samples. For this purpose, 3 different print speed, layer thickness and infill pattern were used. The most appropriate and most effective production parameters were tried to be determined by ANOVA analysis performed in Minitab application in accordance with Taguchi L9 design. In the analyses, it was determined that the production parameters were effective on tensile strength, toughness, surface hardness, surface roughness and dimensional accuracy values. In the study, the effect of infill pattern, which is one of the inputs affecting mechanical and physical properties, on the hydrolytic degradation property was also analysed.

2. Materials and methods

This study was carried out using polyethylene terephthalate. The PET granules were extruded into filaments with a diameter of 1.75 mm by extrusion method. The products were produced with three different print speeds (30, 45, 60 mm/s), layer thickness (0.12, 0.16, 0.2) and infill pattern (line, zigzag, co-centered) using extrusion method, which is one of the additive manufacturing production methods. Table and nozzle temperatures were kept constant at 65 °C and 220 °C, respectively. In the slicing programme used in the study, the highest degree and multiples of 0.6 and 0.8 of this degree were used for both print speed and layer thickness. ANOVA analyses were performed in Taguchi L9 model to find the optimum combination of production parameters and the most effective input. ANOVA (Analysis of Variance) is a widely used statistical formula for comparing the variances between the means of results obtained from different groups [17]. ANOVA is not only the most widely used method for analysing parameters, but also gives the most reliable results [18]. ANOVA was used in the analyses of the parameters in the study due to these advantages. Experimental parameters and degrees are given in Table 1.

2.1. Mechanical analyses

Tensile strength, toughness and hardness analyses were performed. Tensile strength test specimens were determined by taking the arithmetic mean of the tests performed on 5 specimens produced in the same parameters in INSTRON brand 8516 model device in accordance with ASTM D638 standard. The Instron 8516 mechanical testing machine is a servo-hydraulically controlled machine. In addition to tensile and compression tests, it also provides bending tests. Notch impact test specimens were produced in accordance with ISO 179-1:2010(E) and 5 specimens produced in the same parameters were tested on TERCO brand 3016 model device with 16 joule energy value and the arithmetic average of the values were taken. The MT3016 impact tester is a robust, reliable, efficient and easy-to-use impact tester manufactured to the appropriate standards. The surface hardness of the samples was determined by the average of 5 Shore D measurements taken from different parts of the sample. Figure 2 shows the design and production steps of the specimens prepared for the tests. Figure 3 shows the data obtained during and after the tests.

2.2. Physical analyses

Dimensional stability and surface roughness values were carried out on hardness specimens by non-destructive methods. The investigations were carried out on Mitutoyo brand Heightgage HS-60 model and TMR 120 testers for dimensional stability and surface roughness values, respectively, and the average of the 5 data obtained was taken.

2.3. Hydrolytic degradation analysis

The samples were ultrasonically cleaned in pure water for 8 hours to remove any dirt or dust residue. The products were kept in a 50 ml solution containing 3 wt% phosphate buffered saline at 38°C. The samples were kept in this atmosphere for 180 days. Weight loss was measured with an electronic weighing scale with an accuracy of 0.0001 g. The samples were produced in 9 different infill patterns with Creality slicing programme. The reason why the study was carried out with Creality slicing programme is that it has 12 different infill patterns. The reason why the study was carried out in 9 of the 12 infill patterns available is that the 3 unused patterns were produced from other patterns. Table 2 shows the infill patterns used.

3. Results and discussion

The optical microscope image produced depending on the layer thickness is given in Figure 4 (at 100x magnification).

3.1. Mechanical properties

The study was designed in accordance with Taguchi L9 method. The inputs are print speed (PS), layer thickness (T) and infill pattern (LP). Three different degrees of each input were considered. The tensile strength (US), toughness (T) and hardness (H) values of each sample were determined and given in Table 3. Table 3 consists of six columns; the second, third and fourth columns show the production inputs and parameters, while the fifth, sixth and seventh columns show the tensile strength, toughness and hardness values obtained in the research, respectively. The arithmetic mean values of tensile strength (US), toughness (T) and hardness (H) values are 48.85, 5.55 and 74.89, respectively. Figure 5 shows the main effect plots obtained from Minitab analysis performed with Taguchi analysis data.

3.1.1. Stress strain analyses

Since high specimen tensile strengths are generally desired, the analysis was carried out using the "larger is better" principle in Taguchi analysis. The signal to noise values at the end of the analysis and the tensile strength values measured at each level are given in Figure 6.

When the signal to noise ratio graph in Figure 6 is investigated, it can be determined that the most suitable production parameters are 60 mm/s, 0.2 mm and zigzag for print speed, layer thickness and infill pattern, respectively. The production parameters for the sample with the lowest tensile strength value are for print speed: 30 mm/s, for layer thickness: 0.12 and for infill pattern: line combination.

After determining the most suitable parameters for production, ANOVA analysis was performed to investigate the degree of influence of the inputs. The ANOVA analysis showed that the most effective production input on tensile strength value was almost equal for two data; print speed and infill pattern, but the least effective production input was layer thickness. In the literature, layer thickness was found to be the least effective input [19]. In addition, ANOVA analysis determined that the reliability rate of the study was 85.94%.

With the increase of the print speed, higher strength is obtained between the layers since the new layer will be layered on the previous layer in a shorter time, and therefore the most effective parameter in tensile strength is the print speed. Similar results were also found in the literature. Megri A.E. et al. reported an increase in mechanical properties with increasing print speed and stated that the reason for this was the decrease in the production time of each layer with increasing print speed [20].

3.1.2. Surface hardness analyses

Surface hardness value is generally desired to be high depending on the area of use. Therefore, in the Taguchi analysis of the study, the analysis was carried out with the principle of "larger is better". The signal to noise values as a result of the analysis and the surface hardness values measured at each level are given in Figure 7.

Among the inputs, the effect of infill pattern and print speed on the surface hardness value (from the signal to noise ratio) is almost similar and the least effective input is layer thickness. From the obtained signal to noise ratio graph, the most suitable production parameters were determined as 45 mm/s, 0.2 mm and zigzag for print speed, layer thickness and infill pattern, respectively. ANOVA analysis showed that the reliability rate of the study was 97.45%.

One of the most effective parameters on surface hardness is print speed. Depending on the increasing print speed, the surface hardness increased due to the better adhesion of the layers completed in a shorter time. In addition, different infill patterns on the sample surface cause different hardness on the sample surface, which is another effective parameter.

3.1.3. Toughness analyses

The effect of the production parameters on the toughness properties of the produced samples was analysed in Minitab application using the " larger is better" principle in Taguchi analysis. The signal to noise values at the end of the analysis and the toughness values measured at each level are given in Figure 8.

From the obtained signal to noise ratio graph, the most suitable production parameters were determined as 30 mm/s, 0.12 mm and co-centered for print speed, layer thickness and infill pattern, respectively. These are the recommended production data for the studies to be carried out in order to obtain the highest toughness values. The production parameters with the lowest toughness values are for print speed: 45 mm/s, for layer thickness: 0.16 and for infill pattern: line combination.

The ANOVA analysis shows that the most effective production input on the toughness value is the infill pattern with an effect rate of 82.90%, while the least effective production input is the print speed. In addition, the ANOVA analysis showed that the reliability rate of the study was 91.83%. The effect of layer thickness and print speed on mechanical properties is known from previous studies [21, 22].

The most effective parameter on toughness is the infill pattern. The infill pattern is also one of the most effective parameters on tensile strength and surface hardness. Depending on different infill patterns, the reaction to the force applied to the sample will be different. The infill pattern that can accumulate the applied force in a region within the sample will have higher mechanical properties. Therefore, one of the most effective parameters on mechanical properties is the infill pattern.

3.2. Physical property analyses

The measured surface roughness (SR) and dimensional accuracy (DA) values are given in Table 4. Table 4 consists of six columns. The fifth and sixth columns show the surface roughness and dimensional accuracy values obtained in the research, respectively. The main effect graphs obtained from the Minitab analysis performed to make the surface roughness and dimensional accuracy values more meaningful are given in Figure 9.

3.2.1. Surface roughness

Taguchi analyses used to investigate the effect of production parameters on the surface roughness values of the samples were performed using the "smaller is better" principle. The signal to noise values at the end of the analysis and the surface roughness values measured at each level are given in Figure 10.

As can be seen in Figure 10 obtained from Taguchi analysis to determine the production parameters for the lowest surface roughness, the most suitable production parameters are: line for infill pattern, 60 mm/s for print speed and for layer thickness: 0.12 mm. ANOVA analysis was performed to determine the most effective production input on surface roughness and the reliability of the model, and it was determined that layer thickness had an effect degree of 49.94%, respectively. The other effective inputs are infill pattern and print speed, respectively, according to the degree of influence. The reliability rate of the analysis using Minitab application is 92.33%. In the literature, surface roughness first increases and then decreases with the increase in print speed, which is consistent with the results in this study [23].

With increasing layer thickness, the surface roughness will increase as the layer traces on the surface will become more noticeable. Similarly, Jayakumar N., et al. reported that layer thickness is the most effective parameter on surface roughness [24].

3.2.2. Dimensional accuracy

Since the dimensional accuracy of the parts produced by additive manufacturing is desired to be high, the analysis was carried out with the "smaller is better" principle in Taguchi analysis. The signal to noise values and dimensional stability values measured at each level are given in Figure 11.

High dimensional accuracy of the produced samples is one of the main advantages of additive manufacturing. When determining the production parameters, it is aimed to determine the parameters that produce the samples with the most precise dimensional accuracy. Therefore, in Taguchi analyses, the optimum production parameters were determined as 45 mm/s, 0.12 mm and zigzag for print speed, layer thickness and infill pattern, respectively. Similarly, print speed and layer thickness affected the dimensional accuracy value in the study conducted in the literature [25].

After determining the most suitable parameters for production, ANOVA analysis was performed to investigate the degree of influence of the inputs. The ANOVA analysis showed that the most effective manufacturing input on the dimensional accuracy value was infill pattern with 90.07% effect. Surprisingly, the influence of the other two inputs remained equal and very limited. Furthermore, the ANOVA analysis showed that the reliability of the study was 93.13%.

The effective parameter in the dimensional accuracy determined by the measurements taken from the surface of the sample is the infill pattern. The dimensional accuracy will change with the change of the infill pattern. This effect is more effective than layer thickness and print speed.

3.3. Hydrolytic degradation analysis

In order to determine the effect of the production pattern on the degradation of the samples, the samples were weighed after 180 days in an atmosphere containing 3% phosphate buffered saline. The degradation of objects produced by additive manufacturing has also been one of the subjects of investigation in the literature. Chen F., et al. investigated the effect of structural voids on degradation in additive manufacturing [26]. When the studies in the literature are investigated, it can be indicated that there are very few studies investigating the effect of infill pattern on degradation. Therefore, in this study, the effect of infill pattern on degradation in additive manufacturing was investigated. The results obtained are given in Table 5. The graphs derived from the data in Table 5 are given in Figure 12.

As can be seen from Table 5, the effect of the infill pattern on the degradation property is proportional and this effect varies depending on the type of pattern. Degradation is actually an indicator of the solubility of the polymer and is a critical property for the recycling of polymers commonly found in nature such as PET. Among the samples produced in different infill patterns by additive manufacturing process, the most and least degraded sample patterns are cross and zigzag structures, respectively.

4. Conclusions

The use of PET material, which was transformed from granule to filament with a diameter of 1.75 mm by plastic injection moulding method, in additive manufacturing method was investigated. The effect of additive manufacturing production inputs was analysed by Taguchi analysis in Minitab application. The most effective production input and the reliability of the analysed model were determined by ANOVA analysis. The data obtained in the study are as follows;

- As a result of the ANOVA analysis on the surface roughness value among the production inputs, layer thickness was determined as the most effective input.

- The most effective input in the tensile strength, toughness and dimensional accuracy properties of the produced parts is the infill pattern.

- The toughness value of the produced materials was affected by the production inputs. In order to obtain samples with the highest toughness value, the production parameters should be as follows; print speed: 30 mm/s, layer thickness: 0.12 mm and infill pattern: co-centered.

- The most effective input in determining the toughness value is the infill pattern and the other two inputs are layer thickness and print speed, respectively.

- The degree of influence of the available inputs on surface roughness is layer thickness, infill pattern and print speed, respectively. Since the layer thickness is also the diameter of the material exiting the hot nozzle, it is actually expected that the layer thickness is the most influential input on the surface roughness.

- The specimens with the lowest surface roughness are those with print speed: 45 mm/s, layer thickness: 0.16 mm and infill pattern: zigzag. These parameters are recommended for low surface roughness production.

-The degree of influence of the inputs on the dimensional accuracy, which is one of the data that determines the physical properties, with their contribution rates, respectively; infill pattern with 89.89% influence rate, layer thickness and print speed with equal 1.6% influence rate.

- The best dimensional accuracy values are 45 mm/s for print speed, 0.12 mm for layer thickness and zigzag for infill pattern.

- Since the most degraded layer geometry is zigzag, this geometry can be selected if the recycling of the products produced in the additive manufacturing process is desired to be in the shortest time.

- In this study, mechanical, physical and degradation properties were investigated by using biocompatible PET material. In future researches, biocompatible polymeric materials such as PLA, ABS, TPU can be used instead of PET material.

- The environmental impact of the degradation rate of PET can be investigated in future studies. However, the degradation mechanism and degradation rate of polymeric materials such as PLA, ABS, TPU can be investigated.

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CONFLICT OF INTEREST

The author of this work declares that he has no conflicts of interest.

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Biography

Yunus Kartal received his master's degree from Kırıkkale University in 2017 in Turkey. He received his PhD from Kirikkale University in 2023. From 2014-2024, Yunus worked as a Research Assistant at Kırıkkale University. He is currently working as an Assistant Professor at the Department of Mechanical Engineering, Faculty of Engineering and Natural Sciences, Kırıkkale University. His research interests include additive manufacturing, optimization in manufacturing and machining.

Captions of Figures

Figure 1. Graphical abstract

Figure 2. a) STL format of the tensile specimen, b) STL format of the toughness specimen, c) STL format of the specimen where degradation and surface hardness were measured e) Manufactured tensile specimen f) Produced toughness sample g) Produced degradation and surface hardness measured sample

Figure 3. Images during and after the tests

Figure 4. Optical microscope image depending on layer thickness, a) sample with a layer thickness of 0.12 mm, b) sample with a layer thickness of 0.16 mm, c) is the sample with a layer thickness of 0.2 mm.

Figure 5. Main effect plots a) Tensile strength data b) Toughness values c) Hardness values

Figure 6. a) Tensile strength signal to noise ratios b) Tensile strength values

Figure 7. a) Surface hardness signal to noise ratios b) Surface hardness values

Figure 8. a) Signal to noise ratios for toughness b) Toughness measurements

Figure 9. Main effect plots a) for surface roughness data b) obtained for dimensional accuracy values

Figure 10. a) Surface roughness signal to noise ratios b) Surface roughness measurements

Figure 11. a) Dimensional accuracy signal to noise ratios b) Dimensional accuracy measurements

Figure 12. Degradation rates

Captions of Tables

Table 1. Study parameters and grades

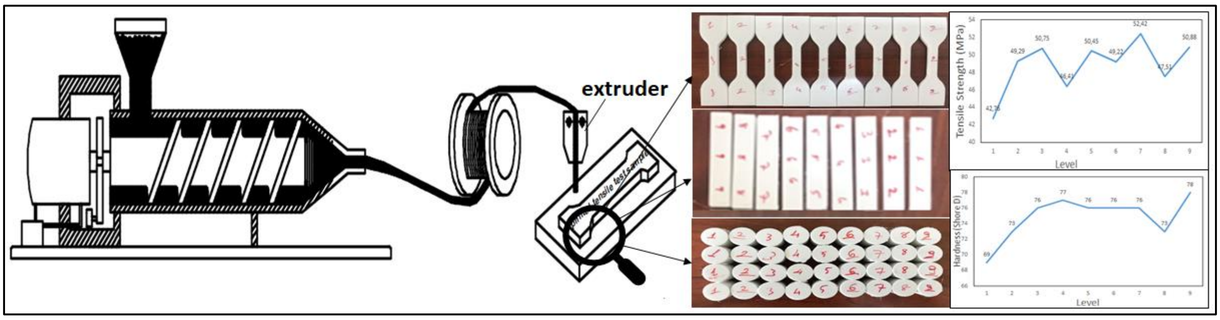
Table 2. Production patterns in degradation analyses

Table 3. Production parameters and results

Table 4. Production parameters and results

Table 5. Production parameters and results

FIGURES



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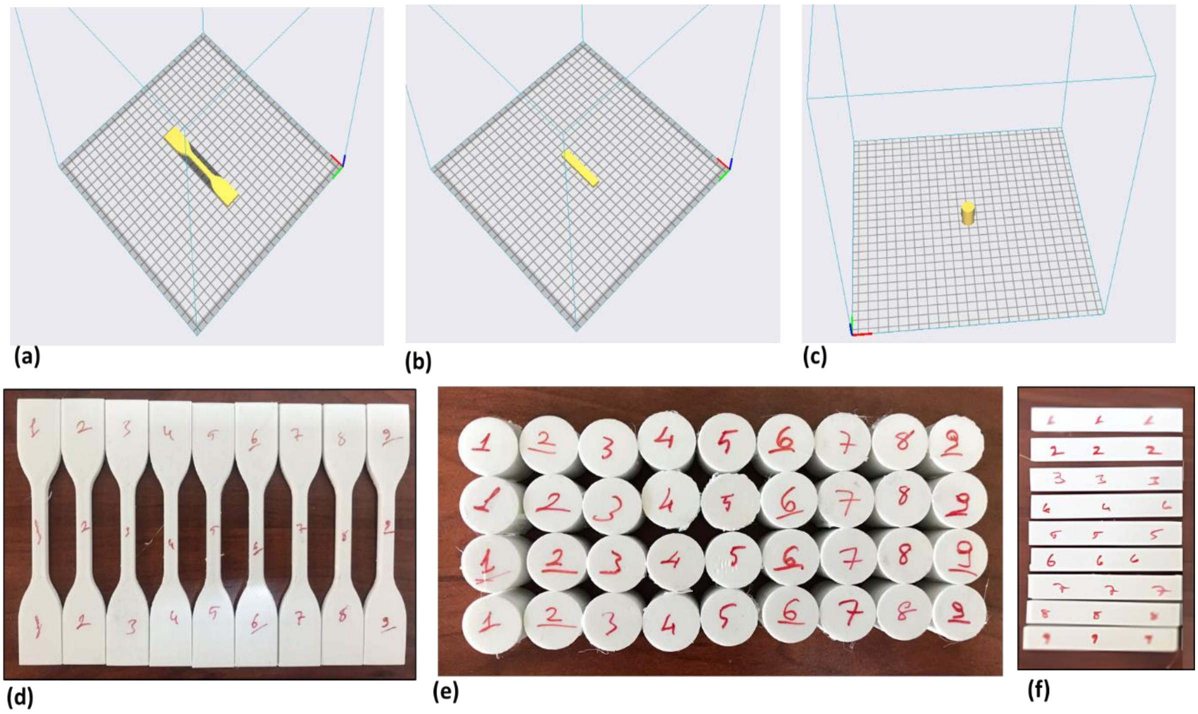

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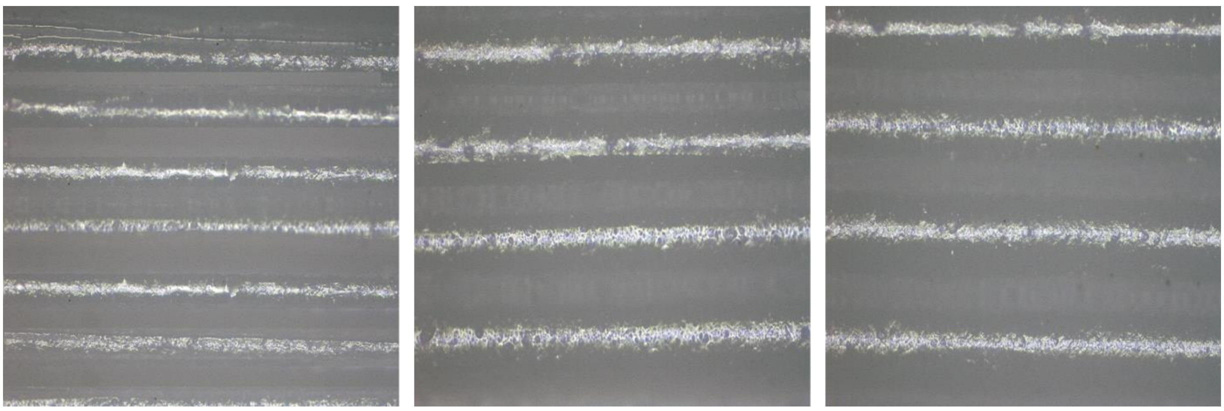

a) b) c)

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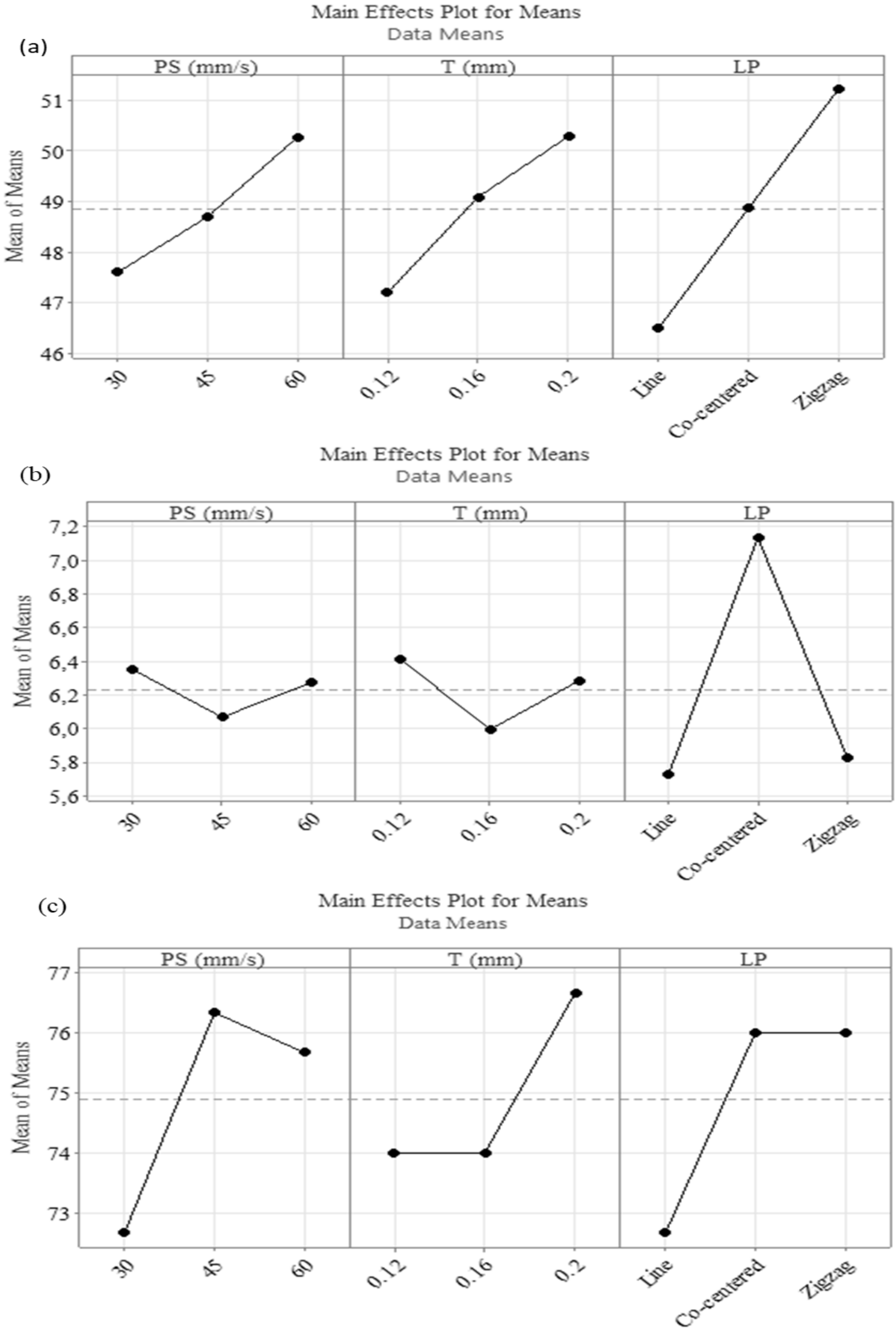

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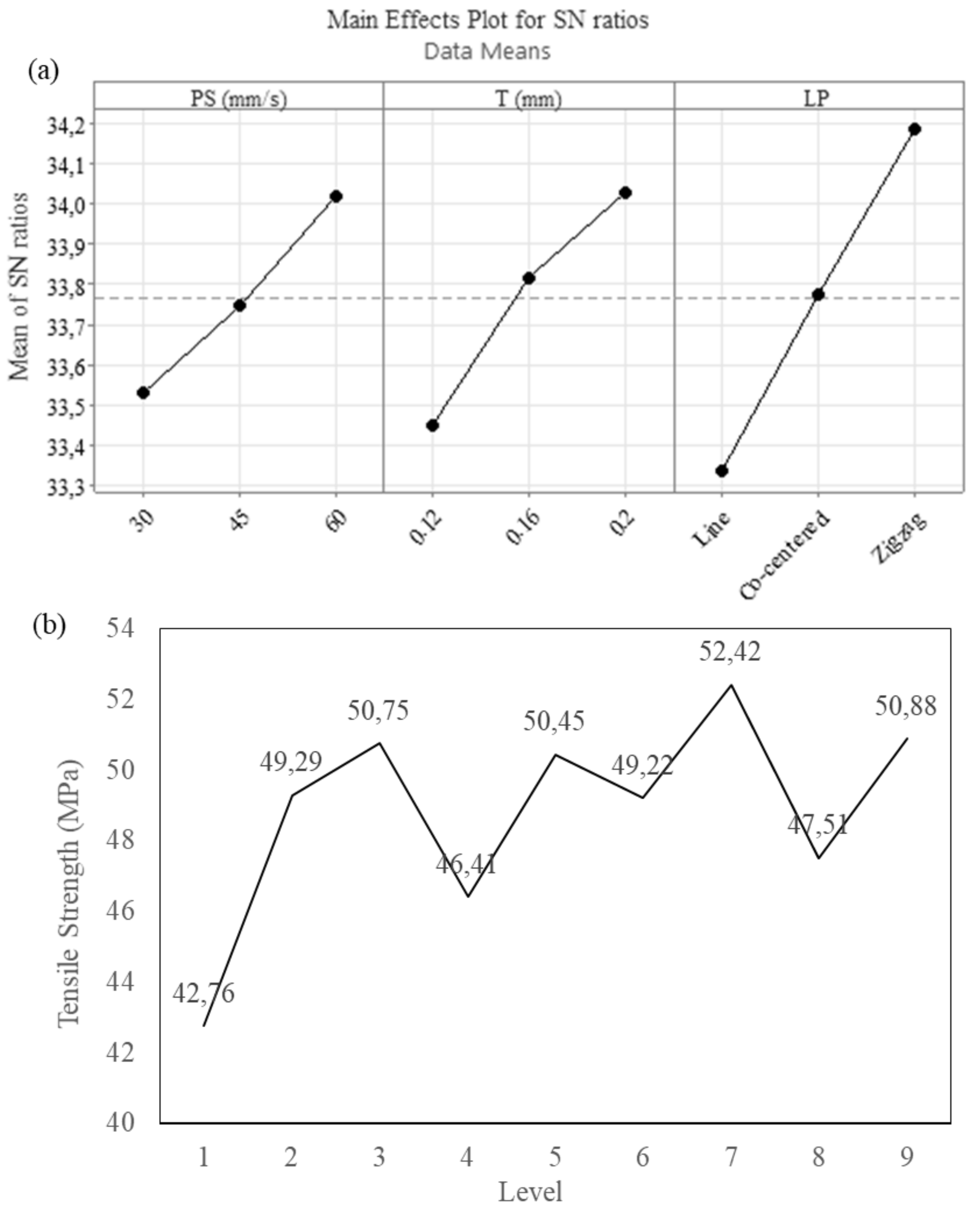


Figure 6. a) Tensile strength signal to noise ratios b) Tensile strength values

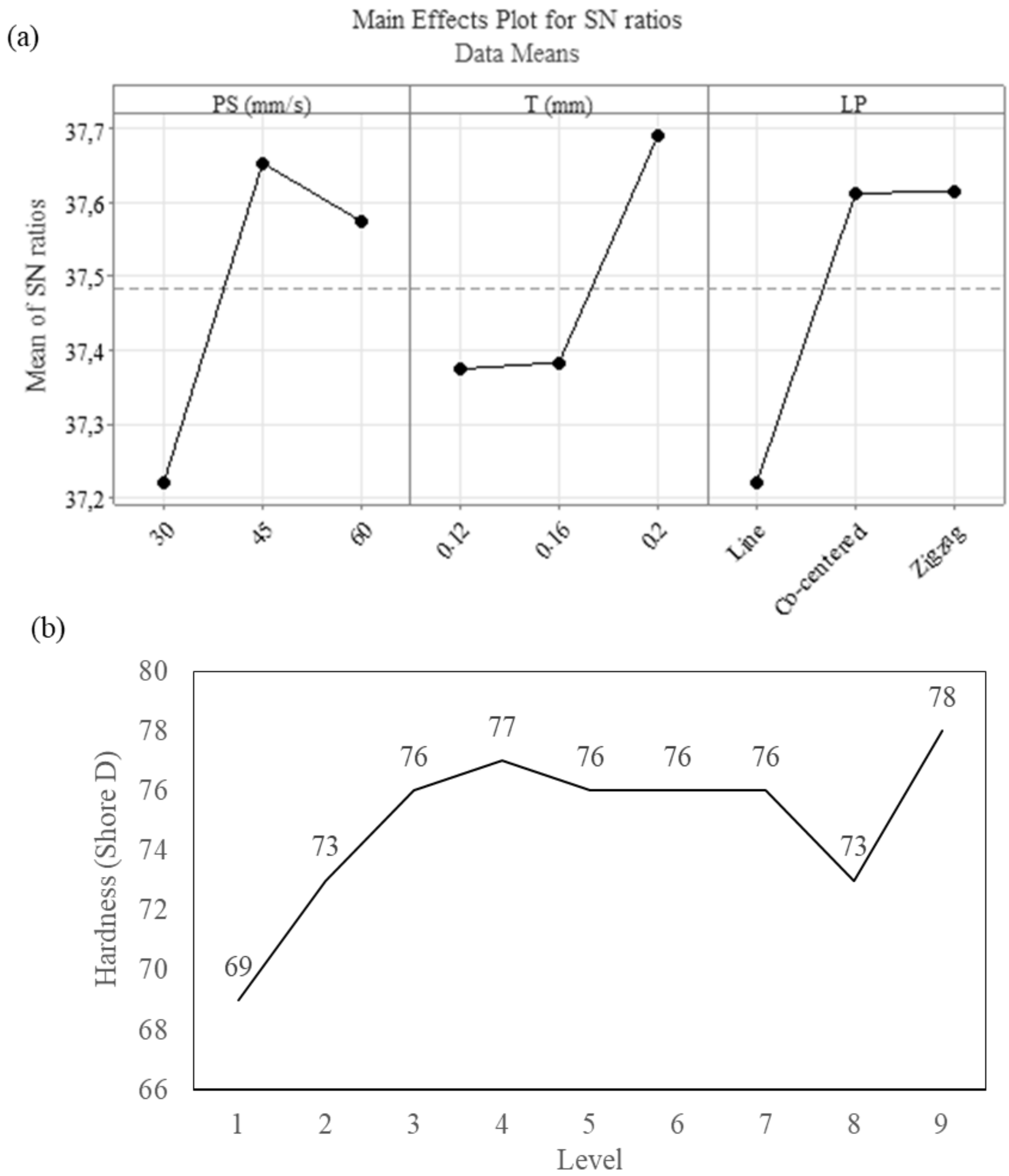


Figure 7. a) Surface hardness signal to noise ratios b) Surface hardness values

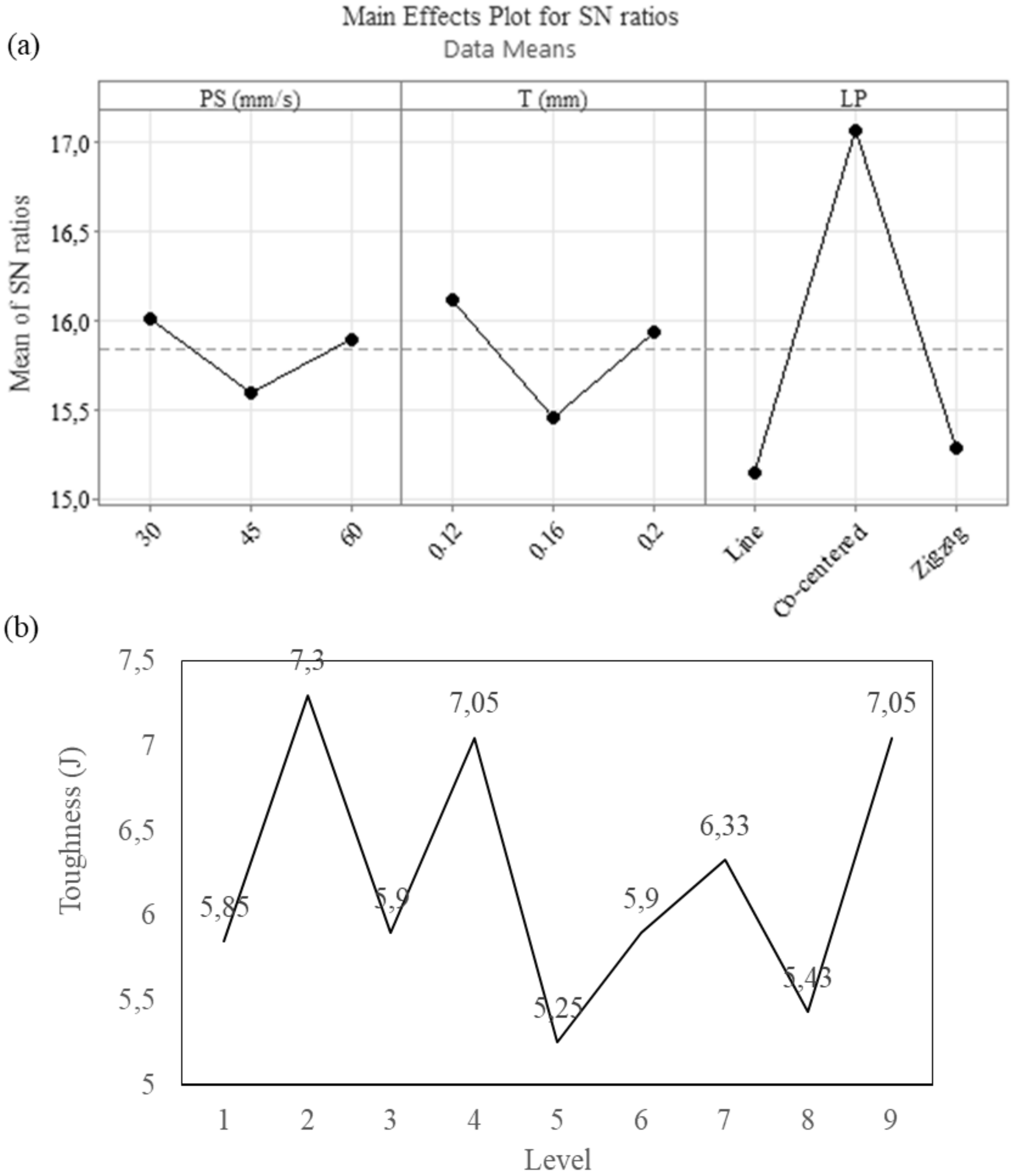


Figure 8. a) Signal to noise ratios for toughness b) Toughness measurements

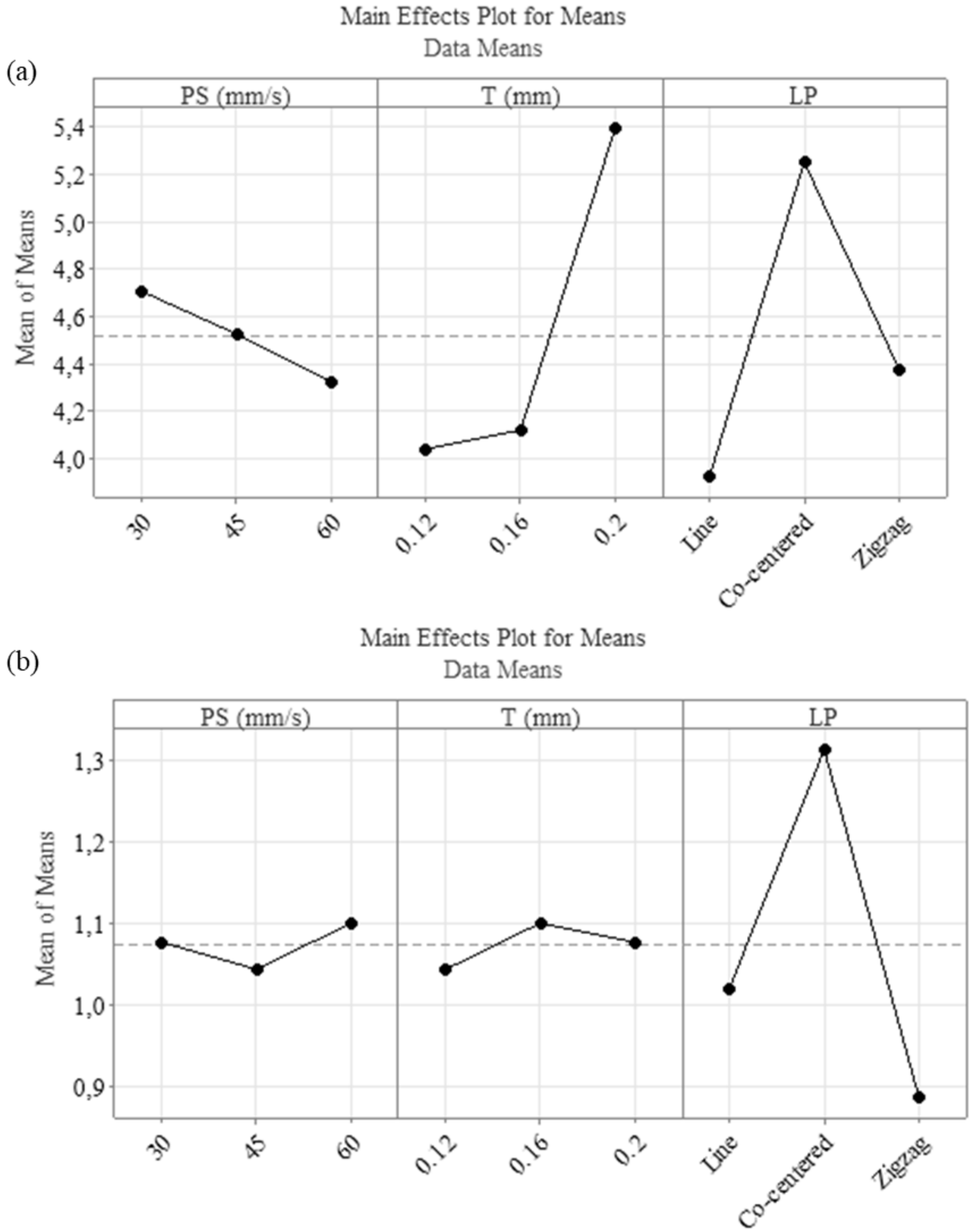
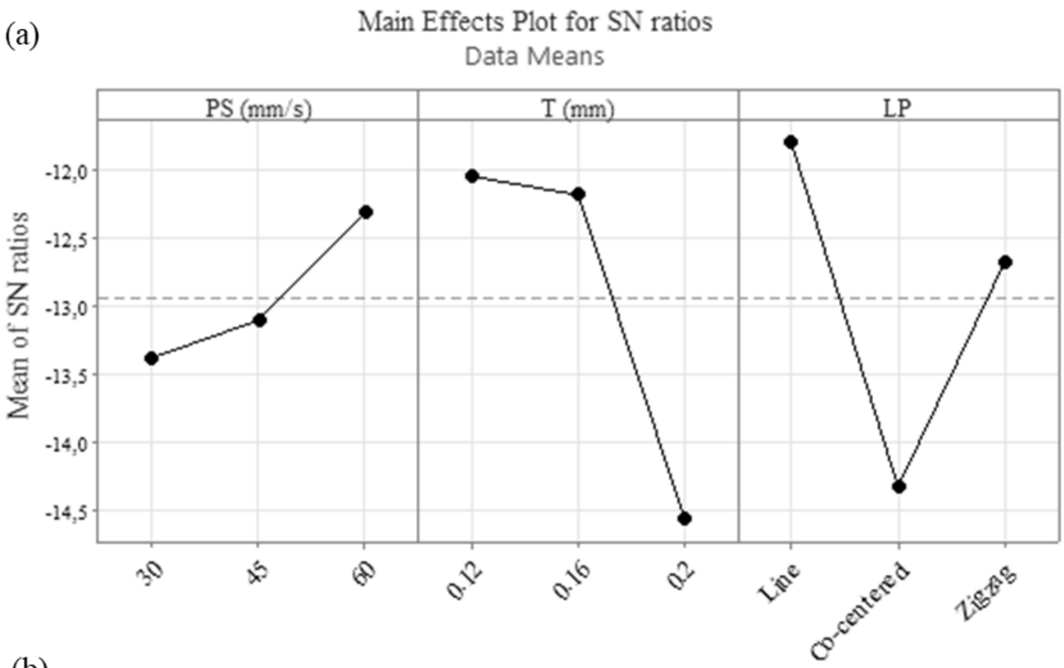


Figure 9. Main effect plots a) for surface roughness data b) obtained for dimensional accuracy values

(a)



(b)

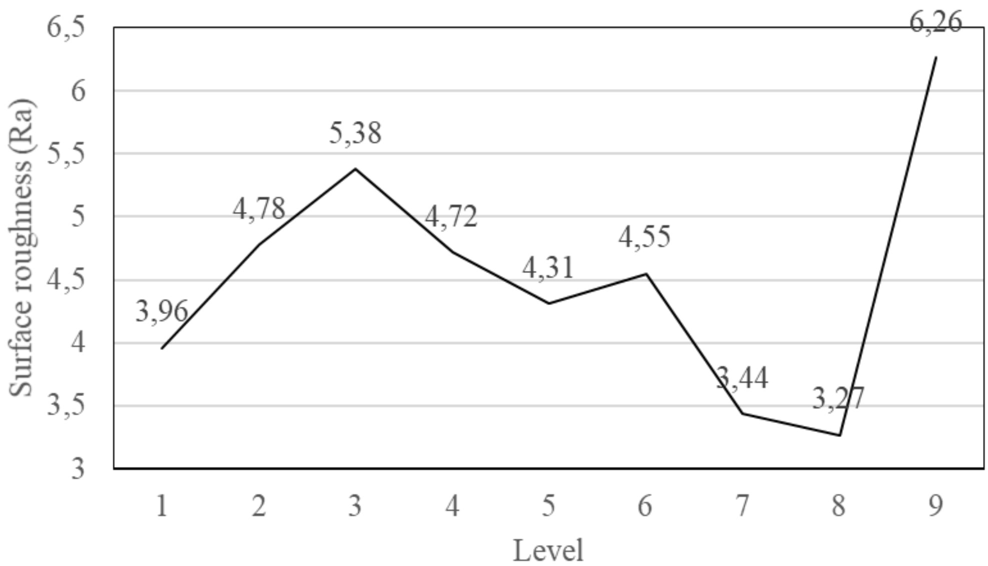


Figure 10. a) Surface roughness signal to noise ratios b) Surface roughness measurements

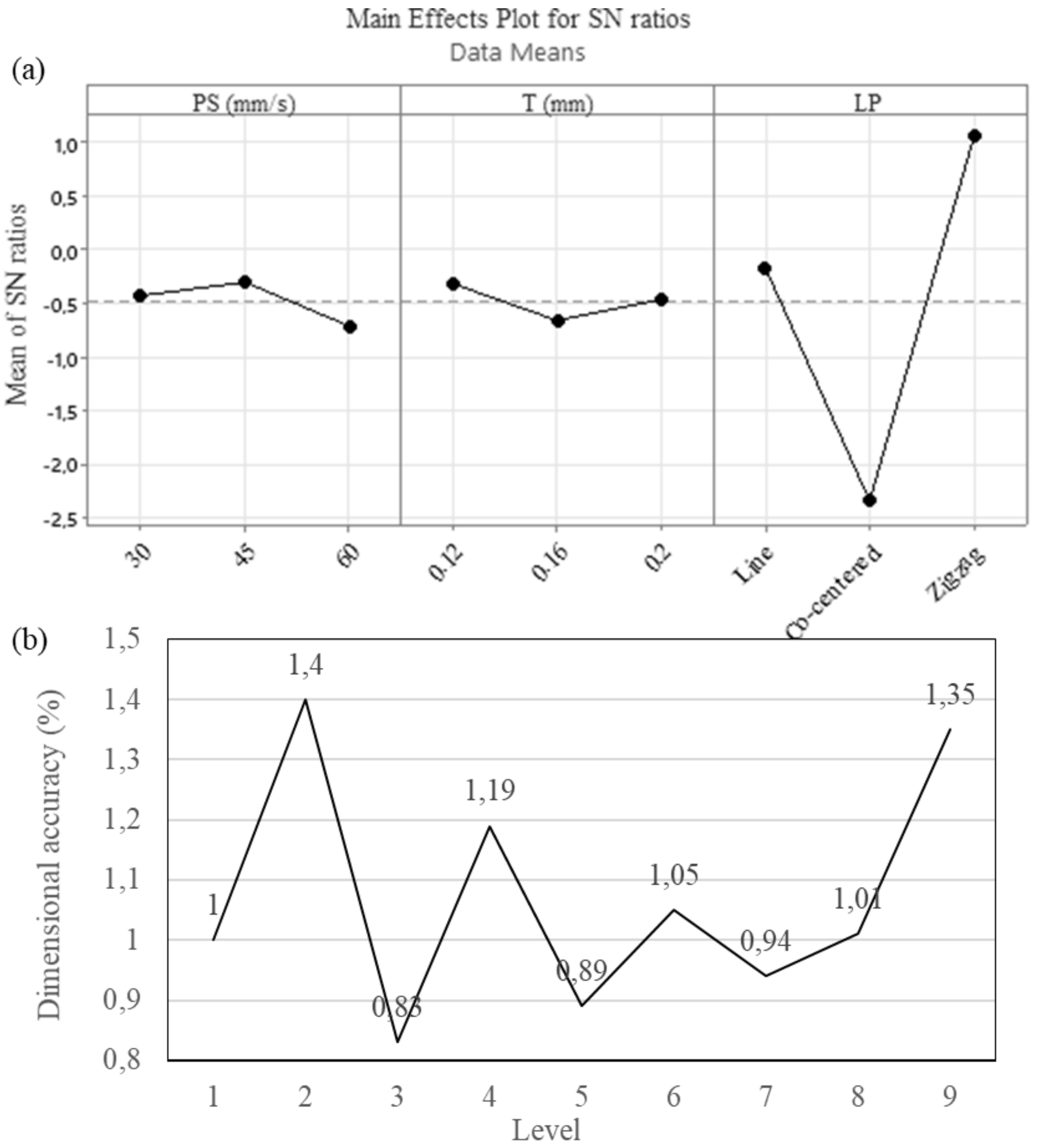


Figure 11. a) Dimensional accuracy signal to noise ratios b) Dimensional accuracy measurements

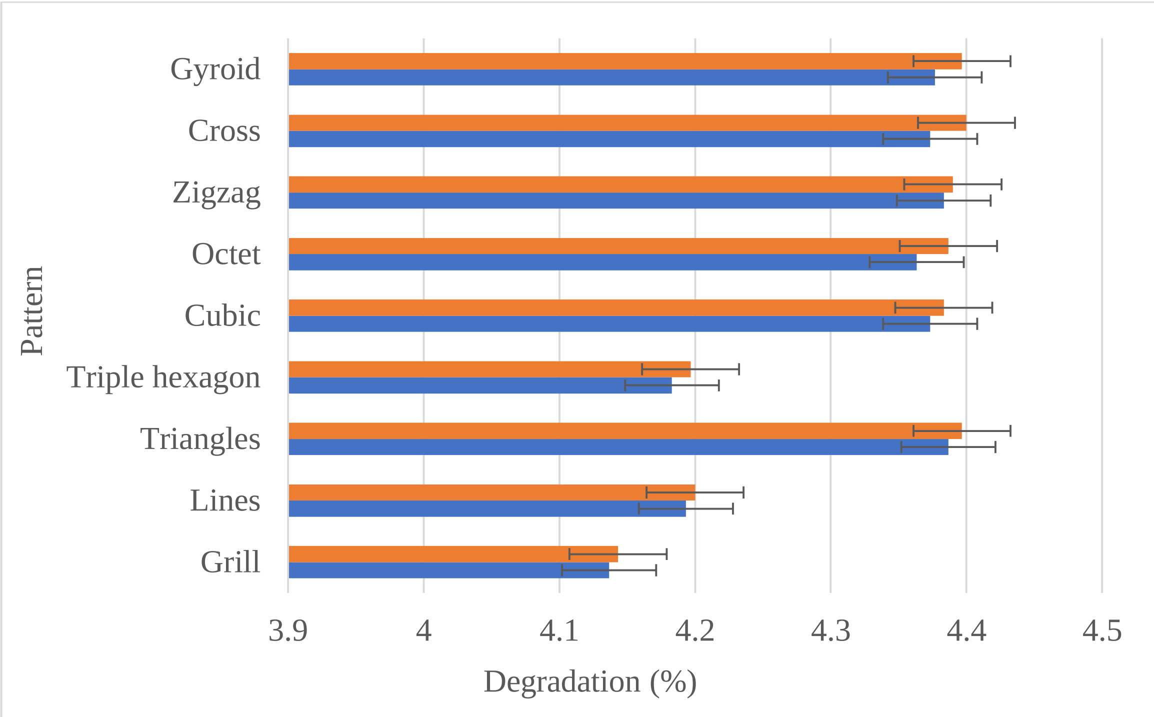


Figure 12. Degradation rates

TABLES

Table 1. Study parameters and grades

Level	Print speed (mm/s)	Layer thickness (mm)	Infill pattern
1	30	0.12	Line
2	30	0.16	Co-centred
3	30	0.20	Zigzag
4	45	0.12	Co-centred
5	45	0.16	Zigzag
6	45	0.20	Line
7	60	0.12	Zigzag
8	60	0.16	Line
9	60	0.20	Co-centred

Table 2. Production patterns in degradation analyses

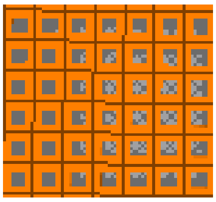
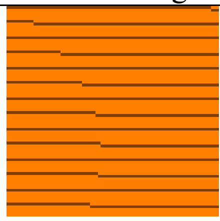
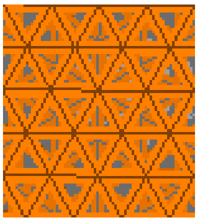
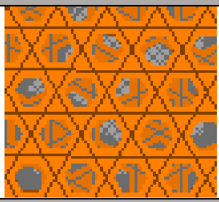
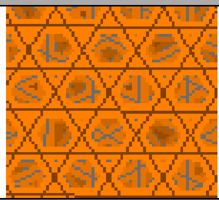
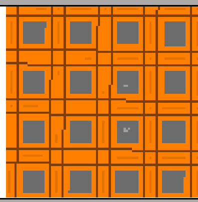
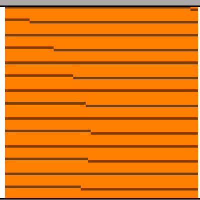
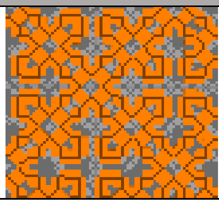
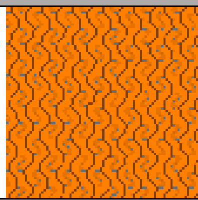
Pattern design	Pattern design	Pattern design
		
Grill	Line	Triangles
		
Triple hexagon	Cubic	Octet
		
Zigzag	Cross	Gyroid

Table 3. Production parameters and results

Level	PS (mm/s)	T (mm)	LP	US	T	H
1	30	0.12	Line	42.76	5.85	69
2	30	0.16	Co-centred	49.29	7.30	73
3	30	0.20	Zigzag	50.75	5.90	76
4	45	0.12	Co-centred	46.41	7.05	77
5	45	0.16	Zigzag	50.45	5.25	76
6	45	0.20	Line	49.22	5.90	76
7	60	0.12	Zigzag	52.42	6.33	76
8	60	0.16	Line	47.51	5.43	73
9	60	0.20	Co-centred	50.88	7.05	78

Table 4. Production parameters and results

Level	PS (mm/s)	L (mm)	LP	SR	DA
1	30	0.12	Line	3.96	1
2	30	0.16	Co-centred	4.78	1.4
3	30	0.20	Zigzag	5.38	0.83
4	45	0.12	Co-centred	4.72	1.19
5	45	0.16	Zigzag	4.31	0.89
6	45	0.20	Line	4.55	1.05
7	60	0.12	Zigzag	3.44	0.94

8	60	0.16	Line	3.27	1.01
9	60	0.20	Co-centred	6.26	1.35

Table 5. Production parameters and results

Level	Infill pattern	First weighing (g)	Weighing after 180 days (g)	Difference (%)
1	Grill	4.1367	4.1433	0.159547
2	Lines	4.1933	4.20	0.159779
3	Triangles	4.3867	4.3967	0.227962
4	Triple hexagon	4.183	4.1967	0.327516
5	Cubic	4.3733	4.3833	0.22866
6	Octet	4.3633	4.3867	0.536291
7	Zigzag	4.3833	4.39	0.152853
8	Cross	4.3733	4.4	0.610523
9	Gyroid	4.3767	4.3967	0.456965