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# Application of RSM Method in Bio-composite Materials (Polymethyl-Methacrylate/Hydroxyapatite) Tension Strength Optimization by 3D Printing Machine Process

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***Abstract***

*It is necessary to develop optimization methods to improve synthetic bone structure for application in human bone implants. Synthetic bone made of polymethyl-methacrylate (PMMA) composites are frequently employed in the medical field (PMMA, on the other hand, has restricted mechanical qualities, as well as being less compatible, rigid, and non-bioactive. This research mixed PMMA material with hydroxyapatite (HA) material. The material's composition is PMMA: MMA = 1: 1, with a hydroxyapatite (HA) to PMMA powder ratio of 0.50: 1 (w/w). The material will be printed through a 3D Printing machine which has a 1.5 mm nozzle. This 3D Printing machine undergoes periodic development, but the results obtained are not in accordance with the needs, especially the tensile strength of the specimen. Therefore, it is necessary to conduct research to optimizing the printing parameters of the 3D Printing machine. Experimental results and analysis using the RSM method show that printing parameters of the 3D printing machine on PMMA/HA material to get the highest tensile strength was at the point of 13.670 mm/s for the perimeter speed parameter, 76.330 mm/s for the infill speed parameter and 33.670% point for the fill density*

***Keywords**: RSM, Tensile Strength, PMMA*

## 1. INTRODUCTION

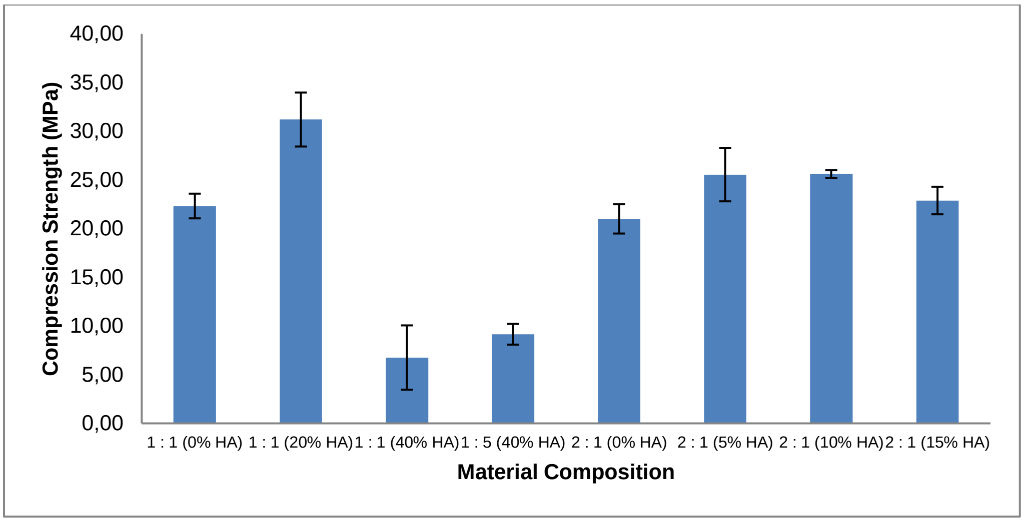
Autograft and allograft are alternative solutions for repairing damaged human bone structures. Autograft is a bone replacement from human bone structure, while allograft is a bone replacement from materials other than human bones. The purpose of replacing human bone structure is to repair, maintain and replace bone structures damaged by disease, accidents and trauma [1]. The material of hydroxyapatite (HA) includes osteoblast linkages that can build new bone tissue and is biocompatible, bioresorbable, bioinert, bioactive, non-toxic, and osteoconductive, making it an alternative bone implant material [1]. The substance of polymethyl-methacrylate (PMMA) is extensively used in the orthopedic sector as an implant to replace damaged bone, but it can also be developed as an alternative material for prosthetics [2].

Polymethyl-methacrylate (PMMA) and hydroxyapatite (HA) materials are printed using two methods: manually and by 3D printing machines. 3D Printing technology is driving big changes, particularly in the material development industry. Since the 1980s, this technology has been known as Additive Layer Manufacturing. This technology is well-known among academics and the manufacturing industry since it has a significant economic impact [3], [4]. Fused Deposition Modeling (FDM) technology is a well-known, low-cost 3D printing technique with additive features [5]. FDM was first introduced in the early 1990s by the American company Stratays Inc. FDM technique works by extruding thermoplastic material through a nozzle at a specific heat temperature, then building the product layer by layer.

Rapid prototyping refers to materials that are printed using a CAD application on a 3D printing equipment. Rapid prototyping printing is used to create complicated product or part models that can be processed quickly [6], [7]. Rapid prototyping can also help to save time during the manufacturing process [8]. The 3D Printing machine process parameters must be optimized for printing composite materials made of polymethyl-methacrylate (PMMA) and hydroxyapatite (HA). Air gap, raster angle, raster width, interior style part, layer thickness, part fill style, part x, y, z shrinkage factor, and contour width are all factors that affect the quality and strength of printed specimens [5].

The parameters of the 3D printing machine process that have been noticed to analyze the performance of the 3D printing machine, such as layer thickness, temperature, and raster angle, have also been carried out in earlier research [9]. The parameters employed in this study are perimeter speed, infill speed, and fill density. These parameters were chosen because they are thought to have an impact on mechanical strength, and they have been used in prior studies [7]. Because these parameters have not been set, further studies are needed to determine the optimum printing parameters to produce printed materials with the highest tensile strength. Some optimization approaches methods, including Taguchi technique, genetic algorithms (GA), artificial neural networks (ANN), factorial design, and the response surface method (RSM) are commonly used [5].

The response surface method (RSM) was chosen in this study because it gives accurate predictions and can explain the influence of variable interactions. Figure 1 depicts previous research on the composition ratio of polymethyl-methacrylate (PMMA) with hydroxyapatite (HA) concentrations.




*Graphic 1. Compressive strength of hydroxyapatite materials with varied composition (Sekarjati & Tontowi, 2018)*

According to research result by Sekarjati & Tontowi (2018), the composition with maximum compressive strength was found in the PMMA:MMA ratio of 1: 1 (w/v), and with addition of 20% hydroxyapatite (HA) from overall mixture, as shown in Figure 1. When the hydroxyapatite (HA) concentration is replaced by PMMA powder, the ratio becomes 0.50:1 (w/w) [7]. This study aims to obtain optimal parameters on the 3D Printing machine based on these compositions, using the response surface method (RSM) in order to obtain the highest tensile strength.

## 2. METHODS

PMMA powder and MMA liquid (ISO 1567 type 1 class 1, acrylic denture materials, heat curing type) and HA powder material are used to make ASTM D638 Type 1 specimens (Bio-nano carbonate, BATAN). The specimens in this study were created with the Inventor 2017 software and saved in *stl format so that they could be translated to G-code for use on a 3D printer. The 3D printing machine will produce a specimen with a length of 165 mm, a width of 19 mm, and a thickness of 3 mm. Three parameters were chosen to produce the best results: perimeter speed (the speed of the outer printing process), infill speed (the speed of the inner printing process), and fill density (the density between the perimeter speed and infill speed patterns). These settings were chosen because they have an impact on the printing process [7]. The extrusion speed of the 3D printing machine is 60mm/min and 80 mm/min with an extrusion length of 20 mm, flowing homogeneously and continuously. The extruded specimens were heat treated for 2 hours at a temperature of 70°-80°C in an electric oven (type SO-181). The electric oven (type SO-181) is preheated for 4 hours to reach a temperature of 70°-80°C. The extruded products are heat treated in order to meet the ASTM D638 Type 1 specimen size. Following the heat treatment, the specimens were put through a mechanical test (tensile strength).

*Figure 1. Specimen according to ASTM D638 Type 1.*

To create the specimen shown in Figure 2, the powder material was measured with an Ohaus brand digital balance with a 0.0001-gram precision, while the liquid material was measured with an injection needle. After that, the material is manually combined in a porcelain bowl with a spatula. The mixture of these components is used as the 3D printing machine's input material. Before being heat treated in an electric oven, the printed specimens were measured with a caliper (type SO-181).

*Figure 2. ASTM D638 Type 1 3D Printing Tool. Specimen Printing*

A Design of Experiment (DoE) using Minitab 19 software was used to collect data on each parameter (perimeter speed, infill speed, and fill density). This data is used as a reference for 3D printing machine parameter settings for specimen printing in order to achieve the best tensile strength. Furthermore, as in earlier studies, the data is evaluated to optimize the printing process parameters and can properly forecast. [5], [9]. First-order regression modeling, which is expressed in a first-order polynomial linear equation, is one of two stages of analysis for the response surface approach. The first-order model was created using Minitab 19 software and regression analysis. The first-order model's output was calculated using the following equation:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 \quad (1)$$

A polynomial's degree is increased by using second order. If the regression analysis fails, the analysis is repeated in the second order, with data from the axial point. The second-order model's outcomes are calculated using the following equation:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 \quad (2)$$

## 3. Result and Discussion

### 3.1 Order One Data Analysis

Using the RSM approach, tensile strength testing was performed on PMMA and HA specimens to determine their tensile strength. Table 1 summarizes the results of the first-order experiment. This data explains that the coded variable is the value of the actual variable.

*Table 1. Responses to the Results of the First Order Experiment*

| ***Coded Variable*** | | | ***Actual Variable*** | | | ***Tensile Strength*** |
|---|---|---|---|---|---|---|
| **X1** | **X2** | **X3** | **Perimeter Speed (mm/s)** | **Infill Speed (mm/s)** | **Fill Density (mm/s)** | ***(N/mm²)*** |
| 1 | -1 | 1 | 40 | 50 | 60 | 6.53 |
| -1 | 1 | -1 | 20 | 70 | 40 | 8.78 |
| 0 | 0 | 0 | 30 | 60 | 50 | 1.66 |
| 0 | 0 | 0 | 30 | 60 | 50 | 3.6 |
| 0 | 0 | 0 | 30 | 60 | 50 | 2.58 |
| -1 | -1 | -1 | 20 | 50 | 40 | 8.18 |
| -1 | 1 | 1 | 20 | 70 | 60 | 8.59 |
| 0 | 0 | 0 | 30 | 60 | 50 | 11.45 |
| 1 | -1 | -1 | 40 | 50 | 40 | 6.15 |
| 1 | 1 | -1 | 40 | 70 | 40 | 10.37 |
| -1 | -1 | 1 | 20 | 50 | 60 | 7.94 |
| 1 | 1 | 1 | 40 | 70 | 60 | 9.2 |

The tensile strength response regression model was created using Minitab 19 software using the data in Table 1. Table 2 shows the results of the regression model calculations.

*Table 2. Tensile Strength Response Regression Model*

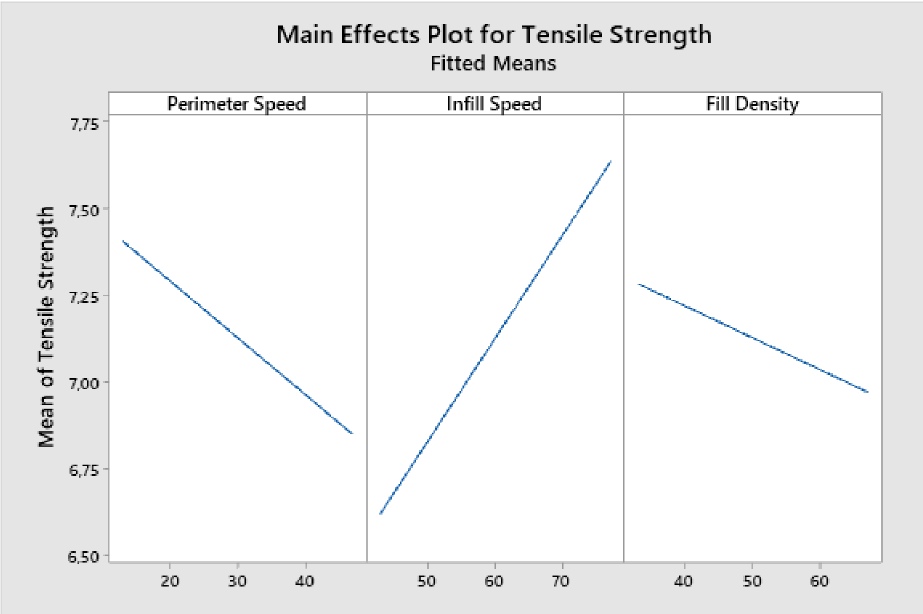
| **S** | **R-sq** | **R-sq(adj)** | **R-sq(pred)** |
|---|---|---|---|
| 2.886 | 1.33% | 0.00% | 0.00% |

The coefficient of determination is 0.0133 (Table 2). This value indicates that the independent variables (perimeter speed, infill speed, and fill density) have a very low influence on the response variable (tensile strength), as evidenced by the fact that the higher the R2 value, the greater the independent variable's influence on the response variable [10]. The F-value and P-value were subjected to an analysis of variance of the lack of fit test using Minitab 19 software to reinforce the validity of the study's findings.

*Table 3. Response Tensile Strength on Order One: Analysis of Variance*

| *Source* | *DF* | *Adj SS* | *Adj MS* | *F-value* | *P-value* |
|---|---|---|---|---|---|
| *Model* | 4 | 1.681 | 0.4202 | 0.05 | 0.995 |
| *Blocks* | 1 | 0.030 | 0.0301 | 0.00 | 0.953 |
| *Linear* | 3 | 1.651 | 0.5502 | 0.07 | 0.977 |
| *Perimeter Speed* | 1 | 0.353 | 0.3534 | 0.04 | 0.840 |
| *Infill Speed* | 1 | 1.186 | 1.1855 | 0.14 | 0.711 |
| *Fill Density* | 1 | 0.112 | 0.1116 | 0.01 | 0.909 |
| *Error* | 15 | 124.899 | 8.3266 | | |
| *Lack-of-Fit* | 11 | 61.942 | 5.6311 | 0.36 | 0.920 |
| *Pure Error* | 4 | 62.957 | 15.7393 | | |
| Total | 19 | 126.580 | | | |

The lack of fit F-value of 0.36 is below the F-table value of 9.01, and the lack of fit P-value of 0.920 is above the value of = 0.05, indicating that there is no variation between the model produced and the real model, allowing it to be characterized with a linear line. Figure 4 shows the results for the maximum tensile strength response values at perimeter speed 10 mm/s, infill speed 80 mm/s, and fill density 30%. These data are used to calculate the ensuing response when given at various levels, however it cannot be stated to be the best result because it is confined to only one response [11].




*Graphic 2. Response Tensile Strength Main Effect Plot*

The model is well described by the experiment given in the first order, because it meets the constraints such as the F value being below the F table and the P value being above the value. However, the coefficient of determination (R2) is still quite low, resulting in a weak relationship between the independent variable and the response variable in the regression model that is created. As a result, a second-order analysis is required to enhance the value of the coefficient of determination (R2).

### 3.2 Analysis of Second-Order Data

By adding six axial points and two central points to a central composite design, this second-order experiment uses a central composite design. Minitab 19 software was used to evaluate the outcomes of each second-order response. Table 4 shows the experimental data for the second order.

*Table 4. The responses to the results of the second order experiment.*

| ***Coded Variable*** | | | ***Actual Variable*** | | | ***Tensile Strength (N/mm²)*** |
|---|---|---|---|---|---|---|
| **X1** | **X2** | **X3** | **Perimeter Speed (mm/s)** | **Infill Speed (mm/s)** | **Fill Density (mm/s)** | |
| 0 | -1.633 | 0 | 30 | 43.67 | 50 | 8.29 |
| 0 | 0 | 0 | 30 | 60 | 50 | 6.67 |
| 0 | 0 | 1.633 | 30 | 60 | 66.33 | 6.65 |
| 1.633 | 0 | 0 | 46.33 | 60 | 50 | 9.16 |
| -1.633 | 0 | 0 | 13.67 | 60 | 50 | 9.73 |
| 0 | 0 | -1.633 | 30 | 60 | 33.67 | 6.65 |
| 0 | 0 | 0 | 30 | 60 | 50 | 4.43 |
| 0 | 1.633 | 0 | 30 | 76.33 | 50 | 5.74 |

Using the Minitab 19 program, the data in Table 4 was processed to create a tensile strength regression model. Table 5 shows the results of the second-order regression model calculation.

*Table 5. Tensile Strength Response Regression Model*

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 2.877 | 41.16% | 0.00% | 0.00% |

The coefficient of determination (R2) is 0.4116 (Table 5). This number indicates that the response variable (tensile strength) is influenced by the independent factors (perimeter speed, infill speed, and fill density). This is demonstrated by the fact that the higher the R2 value, the stronger the independent variable's influence on the response variable [10]. The F-value and P-value were subjected to an analysis of variance of the lack of fit test using Minitab 19 software to reinforce the validity of the study's findings.

*Table 6. Second-order analysis of variance for response tensile strength*

| ***Source*** | ***DF*** | ***Adj SS*** | ***Adj MS*** | ***F-value*** | ***P-value*** |
|---|---|---|---|---|---|
| *Model* | 10 | 52.103 | 5.2103 | 0.63 | 0.760 |
| *Blocks* | 1 | 0.030 | 0.0301 | 0.00 | 0.953 |
| *Linear* | 3 | 1.651 | 0.5502 | 0.07 | 0.976 |
| *Perimeter Speed* | 1 | 0.353 | 0.3534 | 0.04 | 0.841 |
| *Infill Speed* | 1 | 1.186 | 1.1855 | 0.14 | 0.714 |
| *Fill Density* | 1 | 0.112 | 0.1116 | 0.01 | 0.910 |
| *Square* | 3 | 46.149 | 15.3829 | 1.86 | 0.207 |
| *Perimeter Speed*Perimeter Speed* | 1 | 37.498 | 37.4980 | 4.53 | 0.062 |
| *Infill Speed*Infill Speed* | 1 | 7.908 | 7.9076 | 0.96 | 0.354 |
| *Fill Density*Fill Density* | 1 | 5.358 | 5.3575 | 0.65 | 0.442 |
| *2-way Interaction* | 3 | 4.274 | 1.4245 | 0.17 | 0.913 |
| *Perimeter Speed*Infill Speed* | 1 | 3.976 | 3.9762 | 0.48 | 0.506 |
| *Perimeter Speed*Fill Density* | 1 | 0.016 | 0.0162 | 0.00 | 0.966 |
| *Infill Speed*Fill Density* | 1 | 0.281 | 0.2812 | 0.03 | 0.858 |
| *Error* | 9 | 74.477 | 8.2752 | | |
| *Lack-of-Fit* | 5 | 11.520 | 2.3040 | 0.15 | 0.971 |
| *Pure Error* | 4 | 62.957 | 15.7393 | | |
| Total | 19 | 126.580 | | | |

The lack of fit F-value of 0.15 is below the F-table value of 5.05, and the lack of fit P-value of 0.971 is above the value of = 0.05, according to the results of the tensile strength response variance analysis (Table 6). (H0 or null hypothesis was not rejected.) This result indicates that the produced model and the actual model are identical. The response surface approach can provide a graph model with a 3D curve to show the ideal locations of each response for each parameter that impacts the response. Figure 5 shows the surface plot data for the tensile strength response.

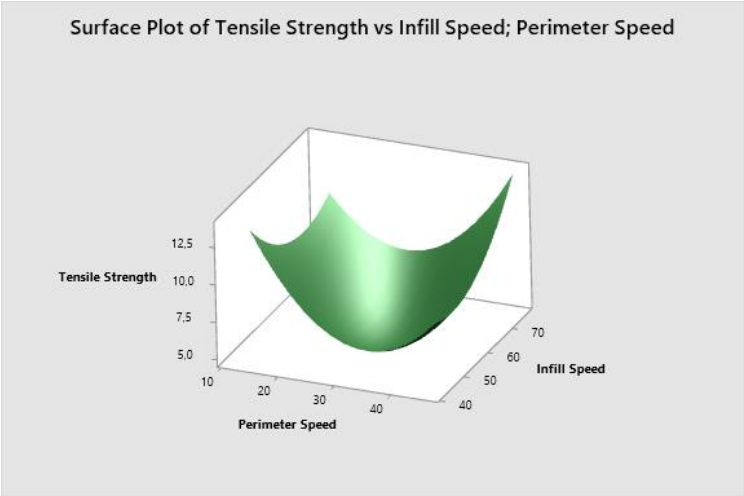
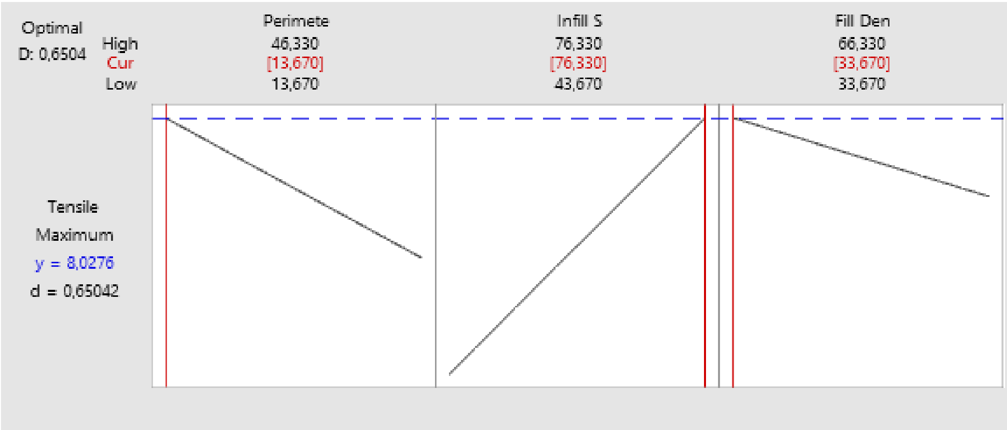



*Figure 3. Tensile Strength of the Surface Plot*

### 3.3 Parameter Optimization of Tensile Strength

At this point, the Minitab 19 program was used to perform an optimization analysis of the 3D printing machine parameters. The greatest tensile strength value was determined through optimization. Figure 6 depicts the optimization outcomes received.




*Graphic 3. Tensile Strength Optimization Response*

The figure illustrates that the perimeter speed parameter should be 13,670 mm/s, the infill speed parameter should be 76,330 mm/s, and the fill density parameter should be 33,670 mm/s for the best tensile strength. The composite desirability rating on the optimization plot indicates how optimal the combination of factors is for the overall response. The composite desirability value is a number between 0 and 1 that indicates how desirable something is. The composite desirability value in this experiment is 0.6504. This number is near to one, indicating that the resultant combination is excellent [12].

## 4. Conclusion

The optimal parameters for the PMMA/HA material 3D printing machine to obtain the highest tensile strength are at the point of 13,670 mm/s for perimeter speed parameters, 76,330 mm/s for infill speed parameters, and 33,670 mm/s for fill density parameter, according to the results of the Minitab 19 software analysis using the Response Surface Method (RSM).

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