Experimental study on parameters of 3D printing process for PEEK materials

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Abstract. In order to satisfy the demand of the efficient and precise manufacturing of artificial bone, the research on the process of 3D printing is carried out for artificial bone material PEEK. Aiming at the high efficiency, low consumables and high storage modulus, response regression mathematical models of parameters are established and the optimized process parameters are obtained based on the Response Surface Methodology. The obtained optimal parameters of 3D printing are that the layer thickness is 0.43mm, the density of internal filling is 55.05% and the number of outer contour is 1.39. Verification experiment is carried out and its results are verified the correctness of optimization results and the regression model. This study provides the basis for the selection of process parameters for 3D printing of PEEK materials.

1. Introduction
There are a variety of materials used to make artificial bone. Among them, special engineering plastic Polyetheretherketone (PEEK) has become the most promising artificial bone matrix composite material with its excellent wear resistance, biocompatibility and chemical stability [1]. It can be used independently as an artificial bone replacement material [2]. In addition, the bones of different people are different in structure, morphology, etc. which requires the customization of artificial bones. The advantages of 3D printing technology in the rapid manufacture of personalized complex structures just meet these needs [3]. Therefore, it is necessary to further study the application of 3D printing in artificial bone manufacturing.

In order to achieve the high efficiency and high quality manufacturing for 3D printing of PEEK materials, the optimal combination of process parameters for printing needs to be determined firstly. For the current FDM technology, printing time, the consumption of printing material have been the subject of general attention, but there are little research on the mechanical properties of printed specimens [4]. In this study, the 3D printing artificial bone material PEEK has been experimentally studied considering the printing time, the consumption of printing material and the storage modulus of the specimen. A mathematical model has been constructed by Response Surface Methodology and the 3D printing process parameters has been optimized and verified.

2. Experimental method
Cubes with a length of 34 mm, a width of 12 mm and a height of 3 mm were printed. The printing
time and the length of the printed material are recorded by the control system, and the amount of printed material is calculated based on the length of the material. The storage modulus is measured by the dynamic thermo mechanical tester DMA-Q800. Experimental design of the process for optimum 3D printing parameter was conducted according to Response Surface Methodology technique using a Box-Behnken design matrix. With reference to the previous study [5], the layer thickness called factor A, the density of internal filling called factor B, and the contour called factor C were chosen as the objects of study in terms of printing time, the consumption of printing material, and storage modulus. The levels of each factor are shown in Table 1.

| Table 1. Parameters and levels of 3D printing experiment |
|-----------------------------------------------|
| Level | A [mm] | B [%] | C [outer turns] |
|-------|--------|-------|----------------|
| -1    | 0.2    | 30    | 2              |
| 0     | 0.3    | 50    | 4              |
| +1    | 0.4    | 70    | 6              |

3. Results and discussions

3.1. Results of experiment
A total of 17 runs of the Box-Behnken experimental design for both specimens are shown in Table 2. The output response $R_1$ is the printing time, the output response $R_2$ is the consumption of printing material, and the output response $R_3$ is the storage modulus.

| Table 2. Results of printing experiment |
|-----------------------------------------|
| No. | Influencing factors | Responses |
|-----|---------------------|------------|
|     | A [mm] | B [%] | C [No.] | $R_1$ [s] | $R_2$ [cm$^3$] | $R_3$ [MPa] |
| 1   | 0.4    | 50    | 6       | 516      | 1123.04      | 1847.6      |
| 2   | 0.3    | 30    | 2       | 481      | 1097.53      | 1510.69     |
| 3   | 0.3    | 50    | 4       | 566      | 1189.2       | 1325.58     |
| 4   | 0.2    | 50    | 6       | 822      | 1227.78      | 1743.84     |
| 5   | 0.3    | 70    | 6       | 633      | 1233.96      | 1473.31     |
| 6   | 0.3    | 50    | 4       | 569      | 1197.35      | 1315.58     |
| 7   | 0.2    | 50    | 2       | 630      | 1265.8       | 1545.09     |
| 8   | 0.4    | 30    | 4       | 467      | 1034.08      | 1602.46     |
| 9   | 0.3    | 70    | 2       | 530      | 1237.51      | 1341.7      |
| 10  | 0.4    | 50    | 2       | 432      | 1010.71      | 1621.93     |
| 11  | 0.2    | 30    | 4       | 705      | 1208.45      | 1663.64     |
| 12  | 0.3    | 30    | 6       | 612      | 1193.33      | 1800.51     |
| 13  | 0.2    | 70    | 4       | 737      | 1296.17      | 1276.57     |
| 14  | 0.3    | 50    | 4       | 570      | 1190.7       | 1319.58     |
| 15  | 0.3    | 50    | 4       | 567      | 1197.3       | 1333.58     |
| 16  | 0.3    | 50    | 4       | 570      | 1190         | 1322.58     |
| 17  | 0.4    | 70    | 4       | 491      | 1112.72      | 1473.35     |

3.2. Establishment of regression model
For the second-order polynomial response surface, the mathematical expression between the design variable and the optimization objective function is shown in equation (1).

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i X_i + \sum_{i=1}^{n} \beta_i X_i^2 + \sum_{i<j}^{n} \beta_{ij} X_i X_j + \varepsilon$$  \hspace{1cm} (1)
Where $Y$ is the output response, $\beta$ is the regression coefficient of the response surface, $X$ is the design variable, $i$ and $j$ are the variable subscripts, $n$ is the number of design variables, $\varepsilon$ is the statistical error. After regression fitting, the effects of layer thickness, the density of internal filling, and peripheral contour test factors on responses including printing time, the consumption of printing material, and storage modulus can be expressed by equations (2), (3) and (4):

$$R_1 = 841.7 - 2673.0A + 2.3B + 85.5C - 1.0AB - 135.0AC - 0.2BC - 3380.0A^2 - 5.5 \times 10^{-3} B^2 - 0.5 C^2 \tag{2}$$

$$R_2 = 1103.9 + 322.1A + 4.3B - 6.6C - 1.1AB + 187.9AC - 0.6BC + 3190.3A^2 + 0.1 B^2 + 49.2 C^2 \tag{3}$$

$$R_3 = 4018.2 - 11512.2A - 14.9B - 301.6C + 32.2AB + 33.6AC - 1.0BC + 16934.4A^2 + 0.1 B^2 + 49.2 C^2 \tag{4}$$

3.3. Analysis of interaction among various factors
For each response value, the effect of the interaction among the different factors is shown in Figure 1. The three factors in this experiment are as shown in each of the three sides of the cube. The vertices of the cube represent the magnitude of each response value $[6-8]$. It can be seen from the figure that under the influence of the interaction among the factors, the magnitude of the response value changes significantly. This shows that the interaction among the factors has a great influence on the change of the response value. The interaction among the factors should be fully considered.

(a) Normal probability distribution of printing time residuals  
(b) Normal probability distribution of residuals of printed material consumption  
(c) Normal distribution of storage modulus residuals

**Figure 1.** Normal probability distribution of residuals.
According to the regression models of equations (2) to (4), the three-dimensional response surface and the contour map of interaction among the experimental factors are respectively constructed, and the other two factors are considered when a certain factor is fixed at the center value. The effect of the interaction of partial factors on the output response is shown in Figure 2.

![Figure 2. Interaction between factors](image)

The significance analysis of the printing time model by three-factor interaction can be concluded that as the layer thickness increases, the printing time decreases, the consumption of printing material decreases, and the storage modulus decreases firstly and then increases. The reason is that the increase of layer thickness leads to a decrease in the number of layers, which reduces the path through which the nozzle passes, and thus the printing time is reduced [9].

As the density of internal filling increases, the printing time increases and the consumption of printing material tends to increase. The greater the density of internal filling, the more the print path is required for the nozzle, and the printing time increases. The gap of outer peripheral contour in the same layer is zero, therefore that the outer contour region of the same layer can be understood as a region having a filling density of one hundred percent, so the increase in the number of outer contours corresponds to an increase in the density of the same layer of entire test piece, and thus the consumption of printing material is increased.

As the number of outer contours increases, the printing time increases, the consumption of printing material increases, and the storage modulus decreases firstly and then increases. The reason is that the area occupied by the outer contour conflicts with the internal filling area. Due to the interaction, the effect of the internal filling area is reduced when the number of outer contours begins to increase, and the storage modulus of the test piece is reduced. However, with the number of outer contours increases, the effect of contour region becomes larger, and the storage modulus increases again. Therefore, the storage modulus decreases firstly and then increases.
3.4. Analysis of relationship between predicted value and actual value

The relationship between the predicted value of the output response selected in this experiment and the actual value is shown in Figure 3. It can be seen from the figure that the predicted value is almost on the same line as the actual value, which indicates that the model established in this paper has high accuracy.

![Figure 3. Relationship between predicted value and actual value of output response in 3D print experiment](image)
4. Optimization and verification

4.1. Optimization of process parameters

The optimization condition targets of 3D printing time and the consumption of printing material are set to minimum, and the optimization target of storage modulus is set to the maximum. The factors selected in the three regression models of equations (2), (3) and (4) are optimized. Three models are considered at the same time, the optimal combination of 3D printing process parameters is layer thickness 0.43mm, density of internal filling is 55.05% and the number of outer contour is 1.39. The result is shown in Figure 4.

![Overlay Plot](image)

**Figure 4.** Optimization results of 3D printing process

4.2. Verification of 3D printing experiment

The verification experiment was carried out using the optimal parameters. The 3D printing time, consumption of printing material and storage modulus of the test piece are 453s, 1003.33cm³ and 1766.65MPa. The predicted values of printing time is 429s, it differs from the true value by 5.59%. The predicted values of consumption of printing material is 938.305cm³, it differs from the true value by 6.93%. The predicted values of storage modulus is 1859.84MPa, It differs from the real value by 5.01%. The 3D printing results successfully validated the model established in this study.

5. Conclusion

In this paper, the Response Surface Methodology was used to optimize the process parameters of 3D printing. Appropriate factors were selected for the study. By analyzing the numerical relationship
between the output response and the factors, regression fitting is performed to obtain the regression equation of each output response to the corresponding factors. By the significant analysis of each output response, the influence of each output response corresponding to the factors in the paper is extremely significant. The interaction among the factors has a great influence on the output response, which indicates that it is necessary to consider the interaction when optimizing the factors in this experiment. The optimal selection of the regression models is constructed, and the optimum combination of 3D printing parameters is as follows: the thickness is 0.43 mm, the density of internal filling is 55.05%, and the number of contour is 1.39. Verification experiment was carried out and it verified the correctness of the model and optimization results.

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