Modeling of extraction of silica rendemen husk rice (*Oryza sativa* L.) by microwave extraction assisted (MAE) using response surface methodology (RSM)

D W Indriani, T R Wardhani
Department of Agricultural Engineering, Faculty of Agricultural Technology
Universitas Brawijaya, Jl Veteran Malang, Indonesia

*Corresponding author: dinawahyu@ub.ac.id

Abstract. Nutrient immobilization technology in plants has been developed to increase the ability of the soil to grow plants. Immobilization is a technique to trap compounds in tablets so that they can be used to retain nutrients in the soil, especially in paddy fields. The purpose of this study was to determine the effect of the use of synthetic fertilizer concentration from rice husk in the manufacture of tablets, as well as to optimize the yield response with the independent variables of KOH concentration and time using Response Surface Methodology (RSM). The result of this research is that the highest organic matter content contained in the material after extraction is 52.54%. Optimization was carried out using Response Surface Methodology (RSM) with the Central Composite Design (CCD) method on Design-Expert software with the independent variables being KOH concentration (2.5%, 5%, 7.5%) and time (5 minutes, 7.5 minutes, 10 minutes while) the response variable is the result. The summary statistics data model suggested by the software is linear where the lowest standard deviation value is 0.6130, the highest R² value is 0.8469, the highest adjusted R² value is 0.8341, the highest R² predicted value is 0.8063, and the PRESS value (the lowest prediction error sum of square) was 11.41. Then the result of the response from the software is 5.033%.

1. Introduction

Rice husk is a hard layer (kariopsis) consisting of lemma and palea [1]. Rice husk is one of the skins that wraps around rice seeds. Rice husks, often referred to as husks, will separate and become waste that is rarely used. Rice husk is a processed product that has many benefits, one of which is used as a nutrient in plants. In general, rice husk is used as a planting medium such as organic fertilizer. One of the chemical compositions in rice husk is silica. Silica is the result of polymerization of silicic acid.

Silica can be produced from rice husk extraction. One of the extraction methods that can be used is the Microwave-Assisted Extraction (MAE) method. The advantages of Microwave-Assisted Extraction (MAE) are that it can control the desired temperature, the time used is fast, and the solvent used is small. However, extraction using Microwave-Assisted Extraction (MAE) has not obtained optimal results. Therefore, the data generated from the extraction of rice husk silica was statistically and mathematically optimized for analysis using Response Surface Methodology (RSM) with Central Composite Design (CCD) in Design Expert software. This was done to determine the most optimal conditions. The advantage of using this optimization method is that it can minimize time and costs.

There are several modelling techniques, one of which is Response Surface Methodology (RSM), which is a statistical technique and a mathematical technique that functions to analyze a problem, especially in quantitative research. The independent variable that will affect the response variable [2]. In this study using the Central Composite Design (CCD) design on Design Expert 11 software. In the
Central Composite Design (CCD) design, several statistical models were produced that function to determine the optimum results, including fit summary, sequential model sum of square, lack of fit summary statistics, ANOVA, and combination of optimum points. Therefore, an optimization method with Response Surface Methodology (RSM) is used to minimize time and cost.

Therefore, the research entitled modelling the yield of rice husk silica extraction (Oryza sativa L.) with Microwave Assisted Extraction (MAE) Using Response Surface Methodology (RSM) aims to determine the modelling of the yield of variations in KOH concentration and time using Response Surface Methodology (RSM) and Central Composite Design (CCD). The use of a modelling method with Response Surface Methodology (RSM) to be able to determine the optimum point of the comparison of the concentration of KOH with time to the yield of silica extract of rice husk produced. In the previous study, the largest yield was 6.51% which was tested using X-Ray Fluorescence (XRF) analysis. Thus, the yield produced using the Microwave Assisted Extraction (MAE) method has not produced optimal silica, so modelling is carried out using the Response Surface Methodology (RSM).

2. Materials and method
This research was conducted in March–July 2021, and took place at the Bioprocess Engineering Laboratory, Department of Agricultural Engineering, Faculty of Agricultural Technology, Universitas Brawijaya Malang.

2.1. Materials
The materials used for the manufacture of rice husk silica, among others, rice husks, KOH, HCl, and distilled water. Variations in the concentration of KOH used include 2.5%, 5%, 7.5%. The material used to optimize the data is the yield of rice husk silica extraction.

2.2. Methods
Rice husks were washed and mashed using a blender, then soaked in a KOH solution with a concentration of 2.5%, 5%, 7.5%. Then it was left for 1 hour and heated in the microwave for 5 minutes, 7.5 minutes, 10 minutes then allowed to stand for 24 hours and the filtrate and residue were separated. Added HCl, then baked in a silica gel oven at 110 for 3 hours. Furthermore, silica powder is formed, which is the yield. And continued with yield optimization using Response Surface Methodology (RSM) with the Central Composite Design (CCD) method on Design-Expert software with independent variables, namely the concentration of KOH (2.5%, 5%, 7.5%) and time (5 minutes), 7.5 minutes, 10 minutes) while the response variable is the result. This research design will optimize the yield of rice husk extract. By using the independent variables, namely the concentration of KOH and time with the desired response variable is the amount of yield (%). There are 2 independent variables, namely the concentration of KOH (%) and time (minutes).

3. Result and discussion
3.1. Response Surface Methodology (RSM) of Rice Husk Silica
In this research, optimization of Response Surface Methodology (RSM) was used to determine the optimum yield point resulting from the extraction of rice husk silica. This optimization uses two factors, namely variations in KOH concentration and time variations. The design used in this study is the Central Composite Design (CCD). Optimization using Design Expert 11, by using rice husk silica extraction yield from table 1 and continued by filling in the response variable in the form of yield with units of percent.

Based on table 1, the resulting graph trends tend to increase in each treatment. The highest silica yield value of 5.3%, namely the treatment of 7.5% KOH concentration with a heating time of 10 minutes, while the lowest silica yield value of 1.48%, namely the treatment of 2.5% KOH concentration with a heating time of 5 minutes.

Based on the analysis of variance ANOVA showed that the KOH concentration factor had a significant effect on the results (P > 0.05), while the heating time factor had no significant effect on the extraction value of silica (P > 0.05). According to Prasetyo, [14] the higher the concentration of KOH added, the more yields are produced. The extraction time also gives an effect on the yield produced. The shorter the extraction time, will give a low yield because not all components are extracted properly.
The longer the heating time, the contact time between husk and KOH solution will be greater, so that the yield will be even greater.

Table 1. Rice husk silica extraction yield.

| Treatment | Yield (%) |
|-----------|-----------|
| KOH 2.5% time 5 minute | Test 1 1.71% Test 2 1.50% Test 3 1.24% Average 1.48% |
| KOH 5% time 5 minute | 4.10% |
| KOH 7.5% time 5 minute | 5.73% |
| KOH 2.5% time 7.5 minute | 2.04% |
| KOH 5% time 7.5 minute | 4.27% |
| KOH 7.5% time 7.5 minute | 5.83% |
| KOH 2.5% time 10 minute | 2.07% |
| KOH 5% time 10 minute | 4.41% |
| KOH 7.5% time 10 minute | 5.90% |

Table 2. Variable Central Composite Design (CCD) with Response Surface Methodology (RSM) on design expert.

| Variable | Level |
|----------|-------|
| KOH Concentration (%) | -α (-1.41) -1 0 +1 +α (1.41) |
| Time (minute) | 1.46447 2.5 5 7.5 8.53553 |
| 3.96447 5 7.5 10 11.0355 |

Based on table 2, there are independent variables with several levels. The independent variables in this study were the concentration of KOH and time. There are -α and which aim to reduce bias. Where -α is -1.41 and is 1.41 obtained from 2k/4 = 1.414.

Table 3 shows that the lowest value produced by the yield was in the 22nd run with the addition of a 2.5% KOH concentration and a time of 5 minutes with a yield of 1.24%. While the highest yield was on the 7th run with the addition of a KOH concentration of 7.5% and a time of 10 minutes, with a yield of 5.9%.

The results in Table 3 show that the most significant values are found in the model and the concentration of KOH where the p-value <0.05. While at the time, the p-value is 0.0803, so that time variations can affect the yield. According to Windiarsih, [16] the longer the material and solvent are in contact, the higher the yield is obtained. The concentration of solvent and the length of time the extraction process can have a significant effect on the yield produced, so the longer the time used, the higher the yield. However, if the extraction time is too long, the solvent will reach a saturation point, causing the results to degrade [17].

In table 4, the sequential model sum of squares can be interpreted as a summary of several models used. So, the model suggested by the software, namely Linear vs. Mean, resulted in a sum of square value of 49.88; df by 2; mean square of 24.94; F-value of 66.38; and p-value of <0.0001. The linear vs mean model was chosen because it resulted in a p-value <0.05 (5%), which means that the model is significant. The smaller the p-value generated, the more significant the model.

Table 3. Actual and coded research design on Central Composite Design (CCD).

| Std Run | X1 | X2 | KOH Concentration (%) | Time (minute) |
|---------|----|----|-----------------------|---------------|
| 9 1     | 1.000 | 2.5 | 10 |
| 24 2    | 0.000 | 1.414 | 5 | 11.0355 |
| 13 3    | -1.414 | 0.000 | 1.46447 | 7.5 |
| 18 4    | 1.414 | 0.000 | 8.53553 | 7.5 |
| Std | Run | Coded Variable | Actual Variable | KOH Concentration (%) | Time (minute) |
|-----|-----|----------------|-----------------|-----------------------|---------------|
| 19  | 5   | 0.000          | -1.414          | 5                     | 3.96447       |
| 22  | 6   | 0.000          | 1.414           | 5                     | 11.0355       |
| 11  | 7   | 1.000          | 1.000           | 7.5                   | 10            |
| 25  | 8   | 0.000          | 0.000           | 5                     | 7.5           |
| 6   | 9   | 1.000          | -1.000          | 7.5                   |               |
| 15  | 10  | -1.414         | 0.000           | 1.46447               | 10            |
| 2   | 11  | -1.000         | -1.000          | 2.5                   | 10            |
| 10  | 12  | 1.000          | 1.000           | 7.5                   | 10            |
| 7   | 13  | -1.000         | 1.000           | 2.5                   | 10            |
| 8   | 14  | -1.000         | 1.000           | 2.5                   | 10            |
| 17  | 15  | 1.414          | 0.000           | 8.53553               | 7.5           |
| 1   | 16  | -1.000         | -1.000          | 2.5                   | 5             |
| 27  | 17  | 0.000          | 0.000           | 5                     | 7.5           |
| 23  | 18  | 0.000          | 1.414           | 5                     | 11.0355       |
| 16  | 19  | 1.414          | 0.000           | 8.53553               | 7.5           |
| 4   | 20  | 1.000          | -1.000          | 7.5                   | 5             |
| 5   | 21  | 1.000          | -1.000          | 7.5                   | 5             |
| 3   | 22  | -1.000         | -1.000          | 2.5                   | 5             |
| 12  | 23  | 1.000          | 1.000           | 7.5                   | 10            |
| 20  | 24  | 0.000          | -1.414          | 5                     | 3.96447       |
| 14  | 25  | -1.414         | 0.000           | 1.46447               | 7.5           |
| 26  | 26  | 0.000          | 0.000           | 5                     | 7.5           |
| 21  | 27  | 0.000          | -1.414          | 5                     | 3.96447       |

**Table 4.** Model selection based on sequential model sum of square.

| Source          | Sum of Square | Df | Mean Square | F-value | p-value |
|-----------------|---------------|----|-------------|---------|---------|
| Mean vs Total   | 308.73        | 1  | 308.73      |         |         |
| Linear vs Mean  | 49.88         | 2  | 24.94       | 66.38   | <0.0001 |
| Linear vs Mean  | Suggested     |    |             |         |         |
| Quadratic vs 2FI| 0.0211        | 2  | 0.0105      | 0.0247  | 0.9756  |
| Cubic vs        | 1.06          | 2  | 0.5315      | 1.28    | 0.3011  |
| Cubic vs        | Aliased       |    |             |         |         |
| Residual        | 7.89          | 19 | 0.4154      |         |         |
| Total           | 367.63        | 27 | 13.62       |         |         |

**Table 5.** Model selection based on lack of fit tests.

| Source | Sum of Squares | df | Mean Square | F-value | p-value |
|--------|----------------|----|-------------|---------|---------|
| Linear | 1.18           | 6  | 0.1964      | 0.4509  | 0.8350  |
| 2FI    | Suggested      |    |             |         |         |
| Quadratic | 1.12 | 5  | 0.2275      | 0.5223  | 0.7563  |
| Cubic  | 0.0532         | 1  | 0.0532      | 0.1222  | 0.7308  |
| Pure Error | 7.84 | 18 | 0.4355      |         |         |
Based on table 5, the lack of fit test is defined as an inaccuracy test with the influence of the results of the p-value and F-value. Sources suggested by the software are linear with a sum of squares value of 1.18; df of 6; mean square of 0.1964; F-value is 0.4509; p-value 0.8350. A Linear source was chosen because it has the highest p-value or > 0.05 (5%).

Table 6. Model selection based on summary statistics.

| Source | Std. Dev. | R² | Adjusted R² | Predicted R² | PRESS | Suggested |
|--------|-----------|----|-------------|--------------|-------|-----------|
| Linear | 0.6130    | 0.8469 | 0.8341 | 0.8063 | 11.41 |          |
| 2FI    | 0.6247    | 0.8476 | 0.8277 | 0.7898 | 12.38 |          |
| Quadratic | 0.6530 | 0.8480 | 0.8117 | 0.7448 | 15.03 |          |
| Cubic  | 0.6445    | 0.8660 | 0.8166 | 0.7280 | 16.02 | Aliased  |

Table 6 Summary Statistics, which is a summary of several models based on the R² value. In the data above, the significance is linear with a standard deviation of 0.6130; R² of 0.8469; adjusted R² of 0.8341; predicted R² of 0.8063; and PRESS of 11.41. The data suggested by the model has requirements based on the standard deviation, and PRESS has a low value. While in R², adjusted R², predicted R² has a high value. According to Faridah [12], a significant summary statistics model has an R² value close to 1.

Table 7. ANOVA analysis results yield response.

| Source    | Sum of Squares | df | Mean Square | F-value | p-value | 
|-----------|----------------|----|-------------|---------|---------|
| Model     | 49.88          | 2  | 24.94       | 66.38   | <0.0001 |
| A-Konsentra | 48.63          | 1  | 48.63       | 129.42  | <0.0001 |
| B-Time    | 1.25           | 1  | 1.25        | 3.33    | 0.0803  |
| Residual  | 9.02           | 24 | 0.3757      |         |         |
| Lack of Fit | 1.18           | 6  | 0.1964      | 0.4509  | 0.8350  |
| Pure Error | 7.84           | 18 | 0.4355      |         |         |
| Cor Total | 58.90          | 26 |             |         |         |

Based on table 7, ANOVA (Analysis of Variance) is to determine the relationship between variables. In the table above, the data that is significant or suggested by the software is the data that has the smallest p-value. Then the value that has the smallest p-value is the source model with a sum of squares value of 49.88; df of 2; mean square of 24.94; F-value of 66.38; p-value of <0.0001. Determination of significant data based on the value of the smallest p-value, then look for the difference in the value of the F-value and the smallest p-value, so that the data is said to be significant.

Figure 1. Contour plot and 3D graph of yield response.
Based on Figure 1 contour plot and 3D graph of yield response with variable KOH concentration and time. The picture above shows a linear graph. The blue color in the graph above shows the response with the lowest value. The resulting linear model is due to variations in concentration and time used.

The color in the graph is the yield value. The red color on the graph shows the optimum peak point and the blue color on the graph represents the lowest response. However, in Figure 1 there is no peak or red graph. This is due to variations in time and solvent [15].

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{figure1.png}
\caption{Graph of actual distribution and predicted yield response.}
\label{fig:figure1}
\end{figure}

Table 8. Optimal point solution.

| Number | Konsentrasi | Time | Rendemen | Desirability |
|--------|-------------|------|----------|--------------|
| 1      | 7,500       | 10,000 | 5,033    | 0.814        | Selected    |

Based on on table 8, the combination of optimum points was obtained from data processing using Design Expert 11. The most optimum point was obtained at a KOH concentration of 7.5% and a time of 10 minutes with a yield of 5.033 and a desirability value of 0.814. Desirability is a value desired by the response. If the desirability value obtained is close to 1, then it is appropriate [13].

3.2. Polynomial Equations of Husk Silica with MAE

Based on the analysis obtained through the software obtained second-order polynomial equations, including the equation of the code variable and the actual variable. The polynomial equation model based on the coded factors is as follows:

\[ Yield = 3.38 + 1.42X1 + 0.2285X2 \]  \hspace{1cm} (1)

From the data above, the equation is used to calculate the solubility results. Equations that use coding factors are used to make predictions about the response given at each level for each factor. So, the factor that has the highest level is symbolized by the symbol +1, while the lowest level is symbolized by the symbol -1. The benefit of the equation symbolized by the code is that it can identify the relative influence of the existing factors by comparing the coefficient factors.

While the second-order polynomial equations in the form of actual variables are as follows:

\[ Yield = -0.150901 + 0.569383X1 + 0.091396X2 \]  \hspace{1cm} (2)

From the above equation data, it can be used to make predictions about the response given at a certain level of each factor. Levels are specified in the original units for each factor. Equations are not allowed to explain the relative influence on each factor, because the scale of the coefficients has been changed and allows the units for each factor in it and the intersection is not at the center of the design area.
4. Conclusion
The results of the modelling of rice husk silica extraction using Response Surface Methodology (RSM) obtained predictive data from Design-Expert software, namely at a KOH concentration of 7.5% and a time of 10 minutes. The recommended sequential model of sum of square data is linear vs. mean with a p-value of <0.0001. Lack of fit test data suggested by the software is linear with a p-value of 0.8350. The summary statistics data model suggested by the software is linear where the lowest standard deviation value is 0.6130, the highest R2 value is 0.8469, the highest adjusted R2 value is 0.8341, the highest R2 predicted value is 0.8063, and the PRESS value (the lowest prediction error sum of square) was 11.41. Then the result of the response from the software is 5.033%.

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