Response Surface Methodology to Optimize Microwave Sterilization of Palm Fruit

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Abstract: This study employed response surface methodology (RSM) to optimize microwave sterilization (MS) of oil palm fruit (OPF). RSM used experimental design CCD (central composite design) arrangement with three experimental factors: mass (330–1171 g), microwave power (80–800 Watt) and sterilization time (5.3–18.7 minutes) as main variables. This study adjusted significant level to 5% error (α = 0.05) with confidence level of 95%. MS affected moisture loss during MS significantly indicated by p-value experimental factors less than 0.05. The p-value of mass, power and time were 0.0001, 0.001, and 0.002 respectively. RSM provided predictive model to evaluate effect of sterilization time, mass and microwave power to moisture loss. Evaluation on normalized curve of MS treatment and lack of fit test of predictive model resulted p-value of 0.011 or less than 0.05 indicated no deviation of experimental data with the model. Determination of optimum condition for MS was conducted by evaluated the surface and contour plots obtained from response surface study. The optimum condition of MS was obtained from combination of heating 350.7 g palm fruit using microwave power of 639.29 W for 18.728 minutes. The moisture loss during optimum MS treatment was observed approximately 32.07%.

1. Introduction
The most important process in palm oil mill is sterilization. Conventional palm oil mill carries out sterilization using saturated steam at 40 psi (140°C) for 75-90 minutes. Sterilization aims to deactivate lipase enzymes, softens the fruit, and facilitates fruit detachment [1]. Steam sterilization has disadvantages such as requires long operation time and generates liquid waste water. Currently, there are few issues in accordance with sterilization process such as high oxidation risks and over-sterilization that may lead to poor bleach ability of the resultant oil [2]. Microwave sterilization offers high efficiency in time and power during sterilization process.

As compare to conventional water or steam heating, microwave volumetric heating has significant advantages in reducing sterilization time and protect oil quality [3]. In microwave heating, material absorbed microwave energy during their interaction with ionic constituent movements and polar molecules inside the material. The process eliminates POME production as no steam required for sterilization. Therefore, microwave-heating technology is regarded as a green and fast pre-treatment process which could be employed in the palm oil sterilization process [4]. Advantages of oil palm fruit sterilization with microwave energy in palm oil quality are higher vitamin E and carotene content [5].
Some authors reported microwave sterilization of oil palm fruit. Sarah and Taib (2013) concluded lipase inactivation by microwave irradiation should consider \( D \)-value of lipase obtained from similar process. \( D \)-value of lipase ranges between 8.3 min (\( T = 68^\circ \text{C} \)) to 16.9 min (\( T = 82^\circ \text{C} \))\[6\]. Sarah et al. reported performance of steam batch and microwave irradiation process during sterilization of palm fruits. Microwave sterilization observed proceed faster than steam batch sterilization. It carried out for 14 to 17 minutes at temperature of 70 to 76\( ^\circ \text{C} \), while steam batch proceeded for 2 hours at temperature of 105\( ^\circ \text{C} \).

In this study, effect of main constituent such as sterilization time, mass and power on heat generation during sterilization was investigated. Response surface model using a Central Composite Design (CCD) for lipase inactivation was employed to optimize microwave sterilization process. Optimization used Response Surface Methodology (RSM) that enables evaluation of the effects of many factors and their interactions on response variables.

2. Material and Methods

2.1. Materials

Material in this study is oil palm fruit bunch \( Dura \) variety. The material supplied by plantation around campus of University Sumatera Utara-Medan. Sterilization carried out in domestic microwave oven (Model: R-249 IN (S)/(W)) with frequency of 2450 MHz. We observed temperature using thermocouple type K (Krupp and Closs size diameter of 3 x 300 mm (Mineral Insulated) C/w Cable 2 m). This thermocouple connected with thermo controller (Shimaden).

2.2. Levels of Research Study

This study conducted in four steps: design of experiment, data verification, statistical analysis and response surface methodology as shown in Figure 1.

![Figure 1. Scheme of research study](image)

Design of experiment. In this study we adjusted level of parameters of sterilization process such as mass, power level and time. Maximum and minimum level of experimental design is shown in Table 1. CCD is optimized design for fitting quadratic models with equal predictability in all direction from the centre \[7\]. The experimental design is shown in Table 2.

| Parameters | Minimum Level | Maximum Level |
|------------|---------------|---------------|
|            | Value         | Code          |
| Mass       | 500 g         | 1000 g        |
| Power Level| Medium        | Low           |
|            | -1            | +1            |
| Time       | 8 min         | 16 in         |

Data verification. This study conducted sterilization process of oil palm fruit to verify data and determined moisture loss. Prior sterilization process, we detached the fruit from the bunch, cleaned and put it in the dry place. The fruits weighed into various mass as shown in Table 2, put it inside microwave oven cavity, and adjusted microwave power and heating time according to design of experimental (Table 2). During sterilization, this study observed temperature to monitor maximum sterilization temperature to avoid prolonged heating. After sterilization completed, we determined
moisture loss using Eq. (1). Moisture loss defines as ratio between balance of initial and final mass to their initial.

\[
\% \text{ Moisture Loss} = \frac{M_{\text{final}} - M_{\text{initial}}}{M_{\text{initial}}} \times 100\% \tag{1}
\]

Statistical analysis. Prior statistical analysis, moisture loss resulted from experiment used to determine mathematic model required to explain correlation between significant factor in microwave sterilization and moisture loss. This study used ANOVA to evaluate the most significant factor contributed to moisture loss during sterilization. Significant level was adjust to 5% error (\( \alpha = 0.05 \)) with confidence level of 95%.

Response surface methodology. Response surface methodology (RSM) was used to optimize microwave sterilization process using software Minitab 17 trial version. This method resulted surface and contour plots to observe the optimum condition from sterilization process.

| Table 2. Level of parameters of sterilization process |
|---|---|---|---|
| Runs | Mass (g) | Power level | Time (min) |
| 1 | 500 | Medium Low | 8 |
| 2 | 1000 | Medium Low | 8 |
| 3 | 500 | Medium High | 8 |
| 4 | 1000 | Medium High | 8 |
| 5 | 500 | Medium Low | 16 |
| 6 | 1000 | Medium Low | 16 |
| 7 | 500 | Medium High | 16 |
| 8 | 1000 | Medium High | 16 |
| 9 | 330 | Medium | 12 |
| 10 | 1171 | Medium | 12 |
| 11 | 750 | Low | 12 |
| 12 | 750 | High | 12 |
| 13 | 750 | Medium | 5 |
| 14 | 750 | Medium | 18 |
| 15 | 750 | Medium | 12 |
| 16 | 750 | Medium | 12 |
| 17 | 750 | Medium | 12 |
| 18 | 750 | Medium | 12 |
| 19 | 750 | Medium | 12 |
| 20 | 750 | Medium | 12 |

3. RESULTS AND DISCUSSIONS

3.1. Predictive Model to Evaluate Effect of Sterilization Time, Mass and Microwave Power to Moisture Loss

The moisture loss (Y) data obtained under the different experimental conditions (sterilization time \((X_1)\), mass \((X_2)\) and microwave power\((X_3)\)) is related by the following second order polynomial equation.

\[
Y=4.49 -3.53X_1 +3.15X_2 +2.68X_1 +1.81X_1^2 -0.1X_2^2 + 0.55X_3^2 -1.16X_1X_2 -0.99X_1X_3 + 0.54X_2X_3 \tag{2}
\]

A summary of the analysis of variance (ANOVA) for model mathematic represented by Eq. (2) is shown in Table 3. The value of adjusted determination coefficient \((R_{Adj}^2 = 0.79)\) and determination coefficient \((R^2 = 0.89)\) indicated high degree of correlation between the observed and predicted value. The ANOVA test of regression model demonstrated insignificant correlation between sterilization time, mass and to temperature \((F\text{-value} = 1.39\) and probability \(> 0.306)\).
Figure 2(a) shows relationship of residual from ANOVA test plotted against fitted value. Residual data points for moisture loss are not uniformly or disperse randomly and do not form a specific pattern. Figure 2(a) indicates residual model are equally dispersed and shows clear correlation with the fitted value.

Table 3. Results of anova test from eq. (2)

| Source          | Sum of squares | Degree of freedom | Mean squares | F-value | Probability > F |
|-----------------|----------------|-------------------|--------------|---------|----------------|
| Model           | 475.37         | 9                 | 52.819       | 8.91    | 0.001          |
| Lack of fit     | 58.25          | 5                 | 11.651       | 57.66   | 0.000          |
| Pure error      | 1.01           | 5                 | 0.202        |         |                |
| Corrected total | 534.64         | 19                |              |         |                |

Figure 2(b) shows distribution of residuals data versus sequences (order) tends to be random which indicates independent variables are related or correlated. Figure 2(c) shows normalized curve of residual percentage and moisture loss relationship. The linear regression line is a predictor of moisture loss data set during observation. The residual point approaches to straight line which indicates the residual is normally distributed. The results verified by Kolmogorov-Smirnov (KS). Figure 1(c) shows value of KS is 0.222 while KS table is 0.294. This concludes KS<sub>calculation</sub>< KS<sub>table</sub> which indicates no deviation with the model.

3.2. Predictive Model to Evaluate Relationship Between Temperature and Moisture Loss

The effect of temperature (X) on moisture loss (Y) is shown in predictive model mathematic represented by Eq. (3). The R² obtained from Eq. (3) is 34.55% which indicates low correlation between moisture loss and temperature as shown in Figure 3.

\[ Y(\%) = -11.598 + 0.294X \]  \hspace{1cm} (3)

Figure 3 shows low relationship between temperature and moisture loss because of the deviation between the observed data and predict results obtained from the model. On the contrary, ANOVA results of the Eq. (3) concluded that temperature has significant effect on moisture loss indicated by F value approach zero (significant \( F = 0.006420038 \)). The result of the ANOVA test for predictive model (3) is shown in Table 4.

3.3. Effect of Sterilization Time, Mass and Microwave Power on Moisture Loss

The graphical representations of regression Eq.(1) as response surface and contour plots were obtained using Minitab software trial version. Optimization of oil palm fruit sterilization by microwave irradiation conducted with respect to maximum and minimum sterilization time, mass and microwave power tabulated in Table 1. Three dimensional response surface plots generated by the software were analyzed to evaluate the impact of two dependent variables on moisture loss at constant independent variable as shown in Figure 4. Figure 4 shows relationship between sterilization time, mass and microwave power on moisture loss. Figure 4(a) shows microwave power and sample mass influenced moisture loss at constant time. Moisture loss increases if mass was reduced at increment of microwave power. Moisture loss represents the heat generated after polar molecules rotation and vibration inside the fruit as microwaves oscillate \[8\]. This polar molecule movement produces friction and then energy is dissipated as heat \[9\].

Figure 4(c) shows time and microwave power influenced moisture loss of sterilization at constant sample mass. The increase of moisture loss corresponded to the rise of microwave power level and sterilization time. The water content of oil palm fruit at the same drying time will decrease when the microwave power is used higher. Rapid mass transfer occurs at higher microwave levels because more heat is generated on the material. Figure 4(e) shows mass and sterilization time influenced moisture loss of sterilization at constant microwave power. Moisture loss increases as the mass becomes smaller and the sterilization time increases.
Figure 2. Linear regression modelling of moisture loss data

Table 4. Result of anova test from eq. (3)

|          | df | SS      | MS      | F       | Significance F |
|----------|----|---------|---------|---------|----------------|
| Regression | 1  | 184.7180709 | 184.7180709 | 9.501934 | 0.006420038    |
| Residual  | 18 | 349.9209041 | 19.44005023 |         |                |
| Total     | 19 | 534.638975  |         |         |                |

Figure 3. Relationship between temperature and moisture loss
Figure 4. Response surface plots and its contour plots of effect: sample mass and microwave power (a and b), time and microwave power (c dan d) and mass and sterilization time (e and f) on moisture loss

Table 5 The optimum conditions of palm fruit sterilization

| Independent variables | Moisture Loss (%) | Power (Watt) | Time (minutes) | Mass (g) |
|-----------------------|-------------------|-------------|----------------|---------|
| Mass and Power        | 32.09             | 644.97      | 18.73          | 354.28  |
| Time and Power        | 31.86             | 641.19      | 17.95          | 330.00  |
| Mass and Time         | 36.57             | 800         | 18.22          | 352.45  |

4. Conclusion
Overall, this study concluded microwave power, sterilization time, and mass strongly influenced moisture loss in oil palm sterilization by microwave irradiation. The optimum moisture loss for microwave sterilization of palm fruit obtained from RSM were 354.28 g, 644.97 Watt, and 18.73 minutes with moisture loss of 32.09%.
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