

Optimization of palm fruit sterilization by microwave irradiation using response surface methodology

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Abstract. This study reported optimization of palm fruit sterilization process by microwave irradiation. The results of fractional factorial experiments showed no significant external factors affecting temperature of microwave sterilization (MS). Response surface methodology (RSM) was employed and model equation of MS of palm fruit was built. Response surface plots and their corresponding contour plots were analyzed as well as solving model equation. The optimum process parameters for lipase reduction were obtained from MS of 1 kg palm fruit at microwave power of 486 Watt and heating time of 14 minutes. The experimental results showed reduction of lipase activity in the present work under MS treatment. The adequacy of the model equation for predicting the optimum response value was verified by validation data ($P > 0.15$).

1. Introduction

Processing of oil palm fruits involves operations of fruit sterilization, threshing, digestion, oil expression, and oil clarification. Palm oil mill sterilizes fresh palm fruit bunches (FFB) within 24-48 hours prior extraction to avoid palm oil deterioration. The aim of sterilization is to protect palm oil quality especially content of free fatty acid (FFA) less than 5-6% [1]. According to Malaysian Palm Oil Board (MPOB), the maximum FFA grade in CPO is 5% [2]. Sterilization of palm fruit carries out in batches with high pressurized steam (15 – 45 psi) for 90 minutes. Digestion and pressing extracts oil from mesocarp of sterilized palm fruits. Overall, processing of oil palm fruits generates wastewater with high organic concentration [3]. Current sterilization technology has weakness because it requires long operation time, needs high energy to produce pressurized steam and discharge palm oil mill effluent (POME). To improve the sterilization process, this study proposes microwave sterilization to reduce sterilization time and avoids POME generation. Several authors reported heating or sterilization of oil palm fruit by microwave irradiation [4, 5, 6, 7, 8, 9, 10].

Sterilization objective in this study is to inactivate lipase. Lipase may hydrolyze oil into free fatty acid in the presence of water. Parameters importance to sterilization process are time and temperature. Sterilization time should considers *D*-value of lipase. The *D*-value of lipase defines as time to inactivate 90% of lipase activity at certain temperature. Sarah and Taib (2013) reported *D*-value of lipase from microwave sterilization of oil palm fruit ranges between 8.3 minutes ($T = 68^{\circ}\text{C}$) to 16.9 min ($T = 82^{\circ}\text{C}$) [8]. Sarah et al. also reported performance of steam batch process and microwave irradiation during sterilization of palm fruits. Microwave sterilization carried out for 14 to 17 minutes at temperature of 70 to 76°C, while laboratory scale steam batch sterilization proceeded for 2 hours at



temperature of 105°C. The previous published experimental results for microwave sterilization of oil palm fruit showed that the optimum process parameters are time, mass and power [9]. Based on the results, this study investigates effect of main constituent like sterilization time, mass and power on heat generation during sterilization and quality of palm oil. For this study, we therefore evaluated the effects of sterilization time, mass and power on microwave sterilization process, and develop a response surface model using a Central Composite Design (CCD) for lipase inactivation. The development of response surface prediction to describe lipase inactivation in this study should be very beneficial to construct application of microwave sterilization.

2. Materials and Methods

2.1. Materials

This study used oil palm fruit bunch *Dura* variety taken around campus of University Sumatera Utara-Medan as raw material, and chemicals such as ethanol, sodium hydroxide etc for analysis of FFA content. Experiments carried out in microwave oven (R-249 IN (S)/(W)) with frequency of 2450 MHz. We measured temperature using thermocouple type K (Krupp and Closs size diameter of 3 x 300 mm (Mineral Insulated) C/w Cable 2 m) connected with thermo controller (Shimaden).

2.2. Sterilization of oil palm fruit by microwave irradiation

Prior sterilization process, we placed oil palm fruitlets inside microwave cavity and adjusted microwave power and heating time. Temperature measurement conducted during sterilization period. Temperature of oil palm fruitlet increased due to polar water molecules of oil palm fruitlet were rotating and vibrating to align with the changing polarity around them when expose to microwaves. The movement of the molecule creates heat [11].

2.3 Design of experiment

This study applied response surface methodology (RSM) to optimize microwave sterilization process using software Minitab 2017 trial version. The most common experimental design in RSM is CCD. CCD is optimized design for fitting quadratic models because it has equal predictability in all direction from the centre [12]. In order to find out the best microwave sterilization condition, a series of experiments conducted by three independent variables to obtain predictive model allowed for the design of a minimal number of experimental runs. We choose three different parameters like sterilization time, mass and power as main variables and CCD arrangement for this study is shown in Table 1. In this study, significant level was adjust to 5% error ($\alpha = 0.05$) with confidence level of 95%.

3. Results and Discussions

3.1 Predictive models to evaluate effect of sterilization time, mass and microwave power to temperature

The values of temperature 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:

$$Y = 62.17 - 4.01X_1 + 6.27X_2 + 4.40X_3 - 0.06X_1^2 - 3.11X_2^2 - 0.14X_3^2 - 1.29X_1X_2 + 1.14X_1X_3 - 0.19X_2X_3 \quad (1)$$

A summary of the analysis of variance (ANOVA) for model (1) is shown in Table 2. The value of adjusted determination coefficient (R_{Adj}^2) (0.16) and determination coefficient (R^2) (0.55) indicated low 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 -value = 1.39 and probability > 0.306).

Figure 1(a) show relationship of residual from ANOVA test plotted against fitted value. Residual data points for temperature are not uniformly or disperse randomly and do not form a specific pattern. Figure 1(a) indicates residuals model are equally dispersed and show clear correlation with the fitted

value. Figure 1(b) show distribution of residuals data versus sequences (order) tends to be random which indicates independent variables are related or correlated.

Table 1. Experimental design

Mass (g)	Power Level	Time (min)
750	Medium	12
1000	Medium Low	8
750	Medium	12
330	Medium	12
750	Medium	12
1000	Medium High	16
500	Medium High	8
500	Medium High	16
750	Low	12
750	Medium	18.728
750	High	12
1000	Medium Low	16
500	Medium Low	8
750	Medium	12
750	Medium	12
500	Medium Low	16
1171	Medium	12
750	Medium	12
1000	Medium High	8
750	Medium	5.272

Figure 1(c) shows normalized curve of microwave power and temperature relationship. The linear regression line is a predictor of temperature data set during observation. The residual point is corresponding to straight line which indicates the residual is normally distributed. Thus supported by the results of Kolmogorov-Smirnov (KS). Figure 1(c) shows value of KS is 0.097 while KS table is 0.294. This concludes $KS_{\text{calculation}} < KS_{\text{table}}$ which indicates no deviation with the model.

Table 2. Results of ANOVA test

Source	Sum of squares	Degree of freedom	Mean squares	F-value	Prob > F
Model	1186.26	9	131.806	1.39	0.306
Lack of fit	519.24	5	103.849	1.22	0.418
Pure error	427.07	5	85.414		
Corrected total	2132.57	19			

3.2 Effect of sterilization time, mass and microwave power on temperature

The graphical representations of regression Eq.(1) as response surface and contour plots were obtained using Minitab software. As maximum initial and final sterilization time, mass and microwave power list in Table 1, optimum parameters for microwave sterilization of oil palm fruit were optimized. Three dimensional response surface plots generated by Minitab were analyzed to evaluate the impact of two dependent variable while holding the third variable constant as shown in Figure 2.

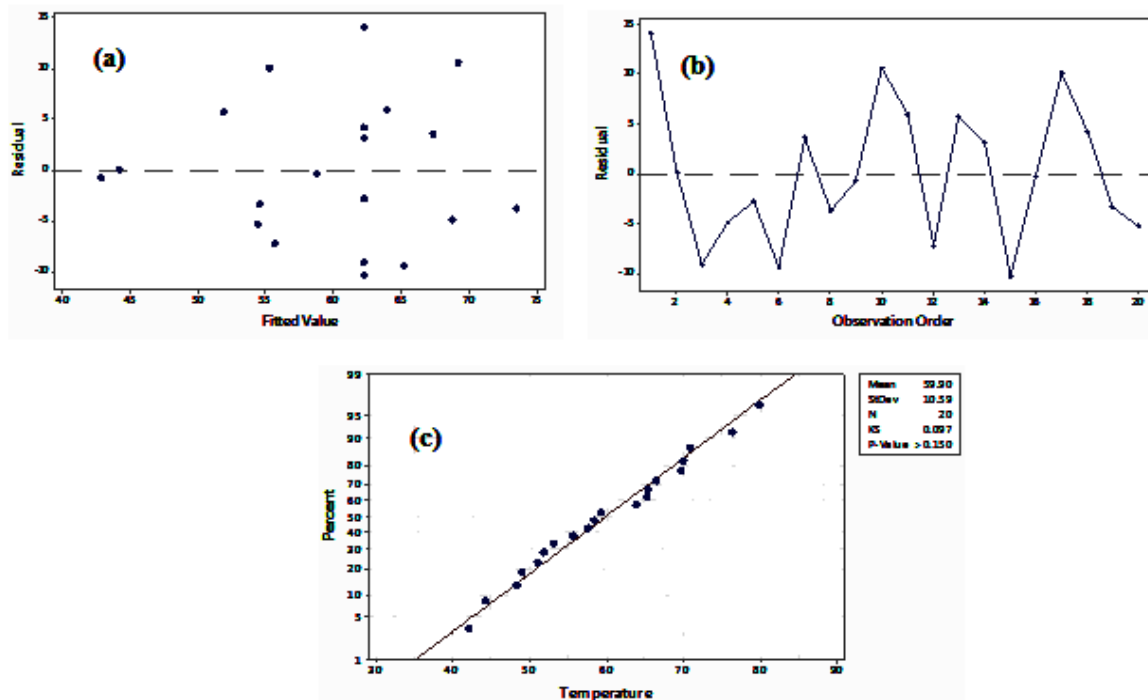


Figure 1. Linear regression modelling of temperature data

Figure 2 shows relationship sterilization time, mass and microwave power on temperature of sterilization process. Figure 2(a) shows microwave power and sterilization time influenced temperature of sterilization at constant sample mass. Microwave power and sterilization time influenced temperature in both linear and quadratic positions. The increase of temperature corresponded to the rise of microwave power level and sterilization time. Increment of microwave power resulted high intensity of microwave energy applied into the oil palm fruit material. The microwave energy penetrates into palm fruits core and water molecules of palm fruits re-oriented themselves as response to microwave energy, creating internal kinetic energy of inter molecular friction. The kinetic energy instantly converted into thermal energy which indicates by the rise of fruit interior temperature, and then exterior temperature. Thus created instant heating and sterilization occurred very fast. Sarah, M et al.(2014) reported similar phenomena while investigated effectivity of steam batch process and microwave irradiation to sterilize oil palm fruit. Microwave irradiation required only 14 to 17 minutes to inactivate lipase activity as compared to conventional steam batch process that carried out for 1.5 to 2 hours. They also reported temperature for irradiation process (70 – 76.5°C) lower than conventional process (105°C). Steam batch sterilization process disposed waste water, while microwave irradiation none [12].

Figure 2(c) shows mass and microwave power had less correlation with temperature at constant sterilization time. This study observed increment of sample mass did not influenced the rise of temperature at linear position (please see Figure 2(c)), or during sterilization at constant time and microwave power. Figure 2(c) also show sample mass and microwave power influenced the rise of temperature at quadratic position while sterilization time was constant. Increment mass of sample resulted lower intensity of microwave energy applied into the oil palm fruit material and power density was low, thus lower temperature of sterilization process obtained. On the contrary, increment of microwave power increase microwave intensity resulted greater power density and high temperature obtained for sterilization process. Power density express the number of microwave energy utilized by each palm fruit samples.

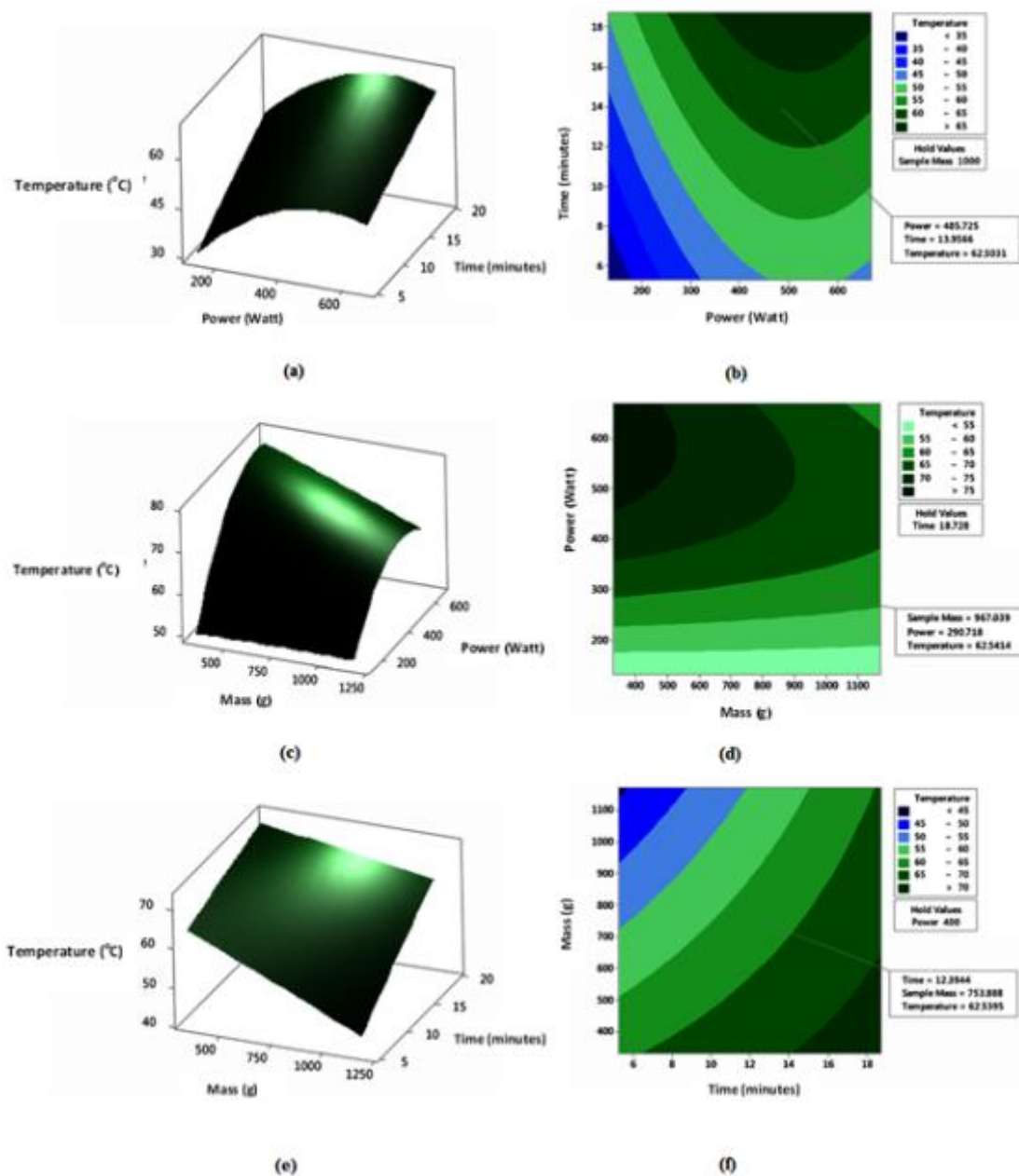


Figure 2. Response surface plots and its contour plots of effect: sterilization time and microwave power (a and b), mass and microwave power (c and d) and mass and sterilization time (e and f) on temperature

Figure 2(e) shows mass and sterilization time influenced temperature of sterilization at constant microwave power. Mass and sterilization time influenced temperature in both linear and quadratic positions. The increase of temperature corresponded to the reduction of mass and increment of sterilization time. Time plays an important role in the sterilization process. Prolonged heating of low power density treatment might obtain high temperature, on the contrary it might cause overheating process for small sample during treatment with high power density that destroyed the sample. Other study reported overheating as thermal runaway which defines as uncontrolled rise of temperature during microwave irradiation [13]

3.3. Optimum conditions

Figure 2(b, d and f) show contour plots of correlations and relationships between sterilization time and microwave power (Figure 2(b)), mass and microwave power (Figure 2(d)) and mass and sterilization time (Figure 2(f)) to temperature of sterilization. This study observed temperature for sterilization of oil palm fruits or lipase inactivation process should proceed between temperature of 44.2°C to 79.8°C with average temperature of 60.8°C.

The temperature of sterilization resulted from this study was lower as compared to commercial sterilization of FFB which is generally carried out at temperature of 131°C and pressure of 40 psi. The temperature of 60.8°C is used as a benchmark to determine the range of contour plots in which the optimal temperature is located as shown in Figure 2(b, d and f). The optimal conditions of palm fruits sterilization are tabulated in Table 3.

Table 3. The optimum conditions of palm fruit sterilization

Independent variables	Temperature (°C)	Power (Watt)	Time (minutes)	Mass (g)
Time and Power	62.50	485.72	13.96	1000
Mass and Power	62.54	290.72	18.73	967.04
Mass and Time	62.54	400	12.39	753.89

Based of RSM study, the best operating conditions to obtain optimum temperature of 62.5°C for oil palm sterilization in this study were sterilization using 1000 g oil palm fruitlets heated by 485.7 Watt microwave power for 13.96 minutes. Meanwhile, during data verification in laboratory, this study observed the best combination of mass, power and time to obtain optimum temperature from experiments oil palm sterilization were 1000 g, 240 Watt and 8 minutes respectively. Temperature obtained from this treatment was 44.2°C. By using those process combination, the FFA content in oil palm fruit in this study were observed below standard.

4. Conclusion

Overall, this study concluded microwave power and sterilization time strongly influenced temperature required to sterilize oil palm fruitlets by microwave irradiation. While mass less influenced temperature sterilization. The optimum temperature for microwave sterilization of palm fruit obtained from RSM were 1000 g, 485.7 Watt, and 13.96 minutes with temperature of 62.5°C. From energy saving point of view, the optimum temperature obtained was 44.2°C from experiment with combination of mass, power and time were 967.04 g, 290.72 Watt, was 18.73 minutes respectively.

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