Modeling Microwave Oven Sterilization of Palm Fruit using Response Surface Methodology (RSM)

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Received:
28 June 2025

Revised:
11 September 2025

Accepted:
11 September 2025

Published:
29 September 2025

DOI:
10.29303/jrpb.v13i2.1190

ISSN 2301-8119, e-ISSN 2443-1354

Available at
http://jrpb.unram.ac.id/

Abstract: The conventional steam-based sterilization of crude palm oil (CPO) in Indonesia is known to be energy- and water-intensive, as well as time-consuming. This necessitates more efficient and sustainable alternatives, such as microwave oven heating. While microwave applications in CPO sterilization have been explored as a more efficient and sustainable alternative, existing studies often lack comprehensive optimization and a systematic investigation of key process parameters. This study aimed to optimize fresh fruit bunch (FFB) sterilization using a microwave oven to identify optimal operating parameters for maximizing oil extraction yield and minimizing Free Fatty Acid (FFA) content. The Central Composite Design (CCD) within Response Surface Methodology (RSM) was employed as a statistical technique to model the process and ensure the validity of the results. Three independent variables were investigated: sterilization time (6–20 min), fruit mass (396–1104 g), and fruit maturity level (ripe and over-ripe), resulting in twenty-six experimental combinations. Optimal conditions for maximum yield and lowest FFA were determined at 6 min sterilization time and 550 g fruit mass for both maturity levels. Conversely, suboptimal conditions at 11.5 min and 150 g fruit mass for both maturity levels resulted in the lowest yield and highest FFA. This research provides crucial insights for developing an optimized microwave sterilization technique, enhancing palm oil processing efficiency and product quality. The findings underscore the potential for a scalable, low-energy sterilization method that could significantly reduce the environmental footprint of CPO production, instilling optimism about the positive impact of this research.

Keywords: CCD; CPO; Dry heat sterilization; Electromagnetic magnetic; FFA

INTRODUCTION

Background

The Indonesian economy derives significant benefits from its domestic palm oil industry. In 2021, palm oil exports reached US$35 billion, representing an impressive 52% increase compared to the previous year (Gabungan Pengusaha Kelapa Sawit Indonesia, 2022). Beyond its strong export performance, the industry also plays a critical socio-economic role by directly employing over five million smallholder farmers and indirectly supporting the livelihoods of an additional 16 million people engaged in processing and distribution activities (Ma'rufah et al., 2023).

Palm oil processing involves several stages, beginning with fresh fruit bunch (FFB) reception and continuing through oil extraction and clarification. Each stage ensures high-quality production (Jusoh et al., 2013; Pakdeechot et al., 2021). Among these stages, sterilization constitutes the most critical pre-treatment step, as it substantially influences both downstream processing efficiency and the quality of the final oil (Thang et al., 2021). This process typically entails subjecting FFBs or loose fruits to pressurized steam at elevated temperatures, most commonly 140 °C and 40 psi for 75–90 min (Mohammad et al., 2021). Sterilization serves multiple purposes: facilitating kernel release from the shell, softening the mesocarp, and, most importantly, inactivating lipase enzymes that otherwise promote free fatty acid (FFA) formation (Liew et al., 2015).

Although steam sterilization remains the dominant method in the industry, it is resource-intensive, requiring large amounts of water, energy, and time. These drawbacks underscore the need for more sustainable and efficient alternatives. Microwave technology has emerged as a promising solution, offering advantages such as reduced FFA content, elimination of steam usage, improved environmental sustainability, and a significant reduction in processing time to only 14–17 min (Chang et al., 2015; Law et al., 2018; Liew et al., 2015; Nokkaew, 2014; Sarah et al., 2018; Sarah & Taib, 2013).

In addition to sterilization technology, fruit maturity is another crucial factor influencing palm oil quality and processing efficiency. Bruised or overripe fruits exhibit higher FFA levels, reducing oil quality. Furthermore, variations in fruit ripeness contribute to the observed standard deviation in release efficiency, as ripe fruits generally detach more easily from the bunch than unripe fruits (Ali et al., 2014).

Recent studies have explored the feasibility of microwave sterilization at both laboratory and industrial scales. Sovia et al., (2024) reported that microwave sterilization for seven minutes achieved optimal results, producing a sterilization temperature of 94.06 °C, a moisture content of 0.55%, a yield of 17.81%, an FFA content of 0.51%, a deterioration of the bleachability index (DOBI) of 3.88 nm, and a carotene concentration of 675.52 ppm. These findings highlight the potential of microwave sterilization but also emphasize the necessity of systematic optimization to maximize efficiency and product quality.

Process optimization is essential for translating laboratory findings into industrial applications. In this context, Response Surface Methodology (RSM) provides a robust statistical framework for optimizing process variables and assessing their interactions. RSM allows researchers to obtain comprehensive data with fewer experiments than traditional factorial designs (Kumaradevan et al., 2015; Montgomery, 2017). Specifically, the Central Composite Design (CCD) within RSM offers an efficient approach to modeling complex response surfaces, incorporating additional data points beyond the initial experimental scope (Kusuma et al., 2015).

Aims

This study aims to precisely determine the optimal parameters for oil palm fruit sterilization using RSM, thereby developing an optimized and practical method.

METHODS

Material and Chemicals

Fresh fruit bunches (FFB) of 20-year-old Tenera oil palm. The cleaned fresh palm fruits (Figure 1). The chemicals used in the quality analysis include sodium hydroxide (NaOH) solution, phenolphthalein indicator, propanol, and hexane.

(a) (b)

Figure 1. (a) Fresh palm fruit (a) Ripe, and (b) over-ripe

Microwave Oven Sterilization Method

The microwave oven sterilization was performed using a domestic Sharp R-728 model, which has a maximum power output of 900 W and operates at a wave frequency of 2.45 GHz. The dimensions of its PTFE-coated cavity are 51.4 cm (w) x 30.8 cm (h) x 42.9 cm (d).

The fresh palm fruits (fruit) were separated from the bunch, which has two categories of maturity (ripe and over-ripe) with various masses (150 to 550 g). The fruits were placed in the microwave-safe plate evenly distributed (one layer) and heated by microwave irradiation at high mode and various times (6.00 to 11.50 min). After sterilization, the palm fruit was extracted using a manual hydraulic pressing tool. This tool can separate CPO from the mesocarp without breaking the palm shell. The CPO was filtered and weighted to evaluate its yield and FFA content. The yield of CPO was calculated according to equation 1 (Pakdeechot et al., 2021).

$$\text{Yield } (\%, w/w) = \frac{m_1}{m_2} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where m_1 is mass of crude palm oil (g), m_2 is mass of fresh palm fruits (g)

FFA content of palm oil was determined by the AOCS Official Method Ca 5a- 40, 3 g of each sample were diluted with 40 mL and heated using a hot plate at a temperature of 135 °C for 1 min. After that, three drops of phenolphthalein solution were added and titrated using KOH that had been diluted and its normality was known (N) until the solution turned red (V), to evaluate FFA by three replicantions. The FFA of CPO was calculated acoording to the equation 2 (American Oil Chemists' Society, 2009).

$$FFA\ (\%) = \frac{V \times N \times (25.6)}{m} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Where FFA is free fatty acid, V is volume of KOH (mL), N is normality of KOH (N), and m is mass of crude palm oil (g)

Design of Experiment

A CCD will be employed, resulting in a total of 26 experimental runs using the Design-Expert version 9.0.4.1 (Stat-Ease Inc., Minneapolis, MN, USA) to optimize the chosen key factors namely sterilization time (x_1), a numeric factor varied from 6 to 11.50 min; fruit mass (x_2), a numeric factor varied from 150 to 550 g; and fruit maturities (x_3), a categorical factor with two levels: "ripe" and "over-ripe". The CCD is capable of accommodating this categorical factor by multiplying the number of runs required for the numeric factors by the number of categorical combinations. Responses measured in this study will include yield of CPO, targeted for maximization, and FFA content, targeted for minimization. The experimental designs is shown in Table 1.

Table 1. Presents the Central Composite Design (CCD) matrix alongside the corresponding CPO

Run	Time (min)	Mass (g)	Maturity	Yield (%)	FFA (%)
1	8.75	350.00	Over-ripe	17.42	1.75
2	8.75	350.00	Ripe	18.55	0.77
3	8.75	67.16	Ripe	12.05	2.15
4	12.64	350.00	Over-ripe	17.09	2.59
5	8.75	350.00	Ripe	19.40	0.79
6	8.75	67.16	Over-ripe	11.92	2.28
7	6.00	550.00	Over-ripe	18.91	0.67
8	11.50	150.00	Over-ripe	12.36	2.48
9	4.86	350.00	Over-ripe	18.34	0.52
10	8.75	350.00	Ripe	18.61	0.79
11	8.75	632.84	Ripe	17.70	2.40
12	6.00	150.00	Ripe	13.22	0.69
13	8.75	350.00	Ripe	18.44	0.82
14	8.75	350.00	Over-ripe	18.31	1.70
15	6.00	150.00	Over-ripe	11.91	0.96
16	11.50	550.00	Over-ripe	16.38	2.43
17	8.75	350.00	Over-ripe	16.19	2.57
18	4.86	350.00	Ripe	19.14	0.47
19	11.50	150.00	Ripe	14.53	2.22
20	6.00	550.00	Ripe	19.98	0.79
21	11.50	550.00	Ripe	16.78	2.55
22	8.75	350.00	Over-ripe	15.74	2.57
23	8.75	350.00	Ripe	18.38	2.51
24	8.75	632.84	Over-ripe	17.98	1.51
25	12.64	350.00	Ripe	18.80	2.55
26	8.75	350.00	Over-ripe	16.22	2.66

The regression analysis was performed to estimate the response function as a second order polynomial (Montgomery, 2017).

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i x_i + \sum_{i=1}^{n} \beta_{ii} {x_i}^2 + \sum_i \sum_j \beta_{ij} x_i x_j + \varepsilon_{ij)} \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (3)$$

Where Y is the pedicted response, β0 is model coefficient, βi are Linear influence on response, βij are Effect of interaction on response, βii are Quadratic Effect of variables on the response and x_i and x_j are the the levels of the independent variables. In the present study, two variables were involved, so values i and j were taking from 1 to 2.

The collected data will be analyzed using RSM to fit a model for each response. Analysis of Variance (ANOVA) will validate these models and their regression coefficients; a p-value below 0.05 will indicate statistical significance. Furthermore, response surface plots and contour maps will be generated to visualize the intricate relationships between the factors and responses. Ultimately, numerical optimization techniques within the Design-Expert software will pinpoint the ideal combination of sterilization time, mass, and fruit maturity to achieve the desired outcomes of maximized yield and minimized FFA content of CPO.

RESULTS AND DISCUSSION

Effect of Independent Variables on Yield of Crude Palm Oil

Table 1 presents the CCD matrix alongside the corresponding CPO yield and FFA content obtained for each experimental run. The analysis of variance (ANOVA) for the response surface model, detailed in Table 2, reveals a highly significant model F-value of 21.32, indicating a mere 0.01% probability that such an F-value would occur due to random noise. Furthermore, the model exhibits satisfactory adequacy as evidenced by its R^2 value. The significant model terms ($p < 0.05$) in this study include B, C, the interaction term A*B, and the quadratic term B^2. Moreover, the lack of fit F-value of 1.58 is not statistically significant compared to the pure error, with a 26.60% chance of such a value occurring by chance. This is a desirable outcome as it confirms the model's adequate fit to the experimental data.

Table 2. Analysis of variance (ANOVA) for response surface quadratic model to identify significant factors affecting the crude palm oil yield.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	144.92	8	18.11	21.32	< 0.0001*
A-Time	2.42	1	2.42	2.85	0.1098
B-Mass	83.63	1	83.63	98.44	< 0.0001*
C-Maturity	10.86	1	10.86	12.79	0.0023*
A*B	6.99	1	6.99	8.23	0.0107*
A*C	0.1406	1	0.1406	0.1655	0.6892
B*C	0.4201	1	0.4201	0.4945	0.4914
A^2	0.0125	1	0.0125	0.0147	0.9050
B^2	39.57	1	39.57	46.58	< 0.0001*
Residual	14.44	17	0.8496		
Lack of Fit	9.24	9	1.03	1.58	0.2660
Pure Error	5.21	8	0.6507		
Cor Total	159.36	25			

Standard deviation: 0.9217; R^2: 0.9094; Adj R^2: 0.8667; Pred R^2: 0.7581; Adeq precision: 18.1184
*Significant variable

For the fitted model, the coefficient of determination (R^2) was 0.9094. This implies that 90.94% of the variation in the sample related to microwave oven sterilization of fresh palm fruit can be attributed to the independent variables, while only 9.06% of the total variation remains unexplained by the model. Overall, it can be concluded that the developed model accurately represents the true relationship among the selected parameters (Table 2). The predicted R^2 of 0.7581 aligns well with the adjusted R^2 of 0.8667, evidenced by a minimal difference of less than 0.2, indicating a sound overall mean. The adequate precision ratio 18.118 (exceeding the desired value of 4) confirms a strong signal. Therefore, this model provides a reliable tool for navigating and optimizing the design space. Response surface curves have been generated to analyze variable interactions and identify optimal settings for maximizing the desired response.

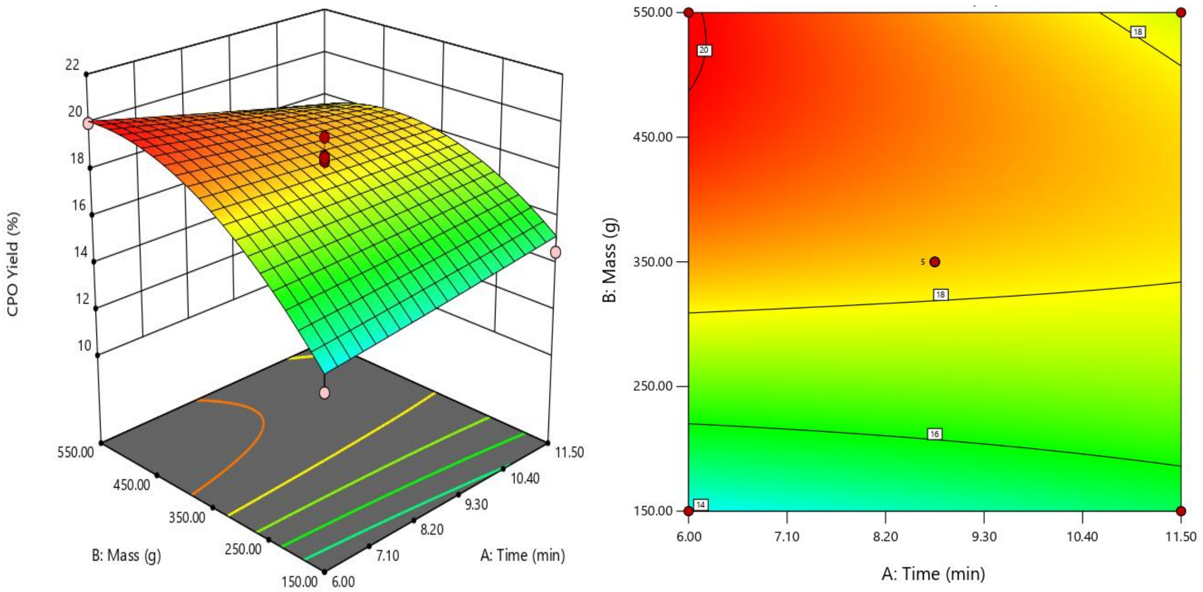
Table 2 shows that microwave oven sterilization time did not significantly affect CPO yield but significantly affected CPO FFA content (Table 4), due to different biological processes. CPO yield is primarily a function of the initial oil content in the fruit's mesocarp, which remains stable at harvest depending on the variety, mass, and maturity. In contrast, microwave oven sterilization time is related to the temperature that denatures the lipase enzyme, thereby reducing or stopping FFA formation.

The CPO yield (Y) data collected under various experimental conditions, including sterilization time and mass at different fruit maturities (ripe and over-ripe), can be expressed by the following second-order polynomial equation, as detailed in Table 3. The equation indicated that yield was mainly influenced by sterilization time.

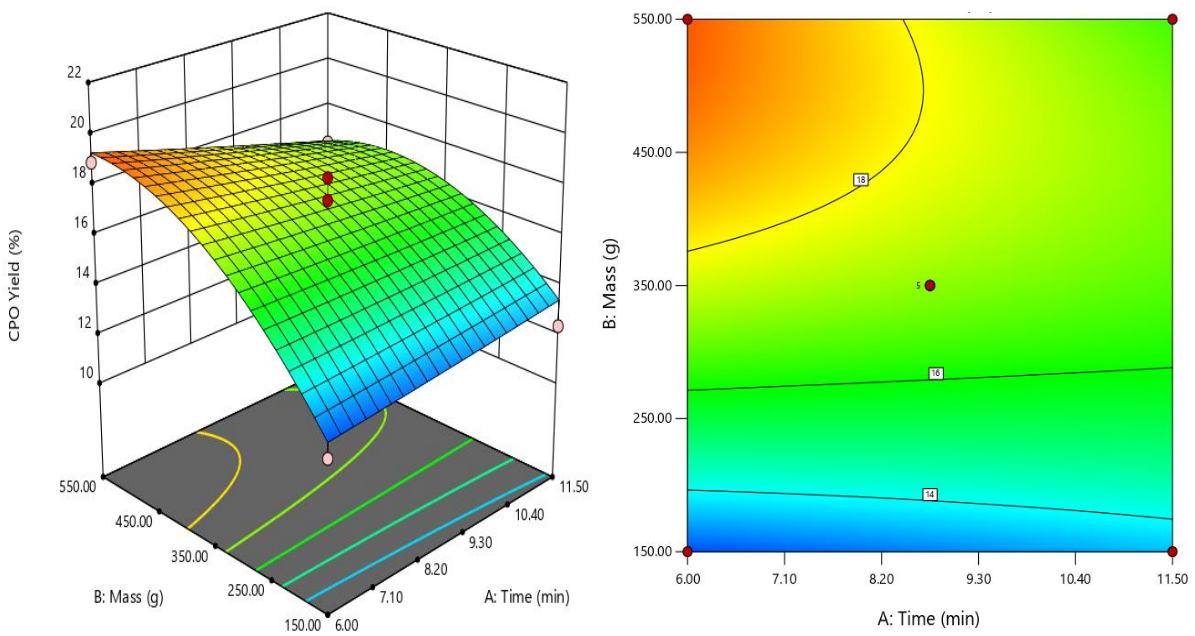
Table 3. Final equation in terms of actual factors of CPO yield

Source	Ripe	Over-Ripe
Model coefficient	5.527255	4.263960
A-Time	0.418247	0.350069
B-Mass	0.055006	0.056627
A*B-Time*Mass	− 0.001700	− 0.001700
A^2-$Time^2$	0.003960	0.003960
B^2 -$Mass^2$	− 0.000042	− 0.000042

The response surface curves and contour plots for the microwave oven sterilization of CPO are shown in Figure 1. These figures will help explain how sterilization time and the mass of fruit affect the yield of CPO from different stages of fruit maturity. Figure 1a illustrates the interaction regarding CPO yield for ripe fruit, while Figure 1b focuses on the effect on CPO yield for over-ripe fruit. For ripe fruit (Figure 1a), the highest CPO yield, which was 19.40%, was achieved with 8.75 min of sterilization and 350 g of fruit. Additionally, increasing either the sterilization time or the mass of the fruit, within the studied ranges, resulted in higher yields. Similarly, for over-ripe fruit (Figure 1b), the maximum CPO yield was 18.31%, also achieved with 8.75 min of sterilization and 350 g of fruit.



(a)



(b)

Figure 1. Three dimensional response surfaces and their respective two dimensional contour plots for the effects of sterilization time and fruit mass on the yield of crude palm oil at different fruit maturities, i.e. (a) Ripe, and (b) Over-ripe

Longer exposure duration of microwave radiation will result in higher oil yield because more cell walls of oil palm fruit had been ruptured due to the heat generated (Cheng et al., 2011). Chow & Ma (2007) and Pakdeechot et al. (2021) also reported the same behaviour upon prolonged heating.

Effect of Independent Variables on Free Fatty Acid of Crude Palm Oil

The analysis of variance for the response surface model is summarized in Table 4. The results indicate that the model F-value is 11.73, suggesting that the model is significant. There is only a 0.01% chance that an F-value of this magnitude could occur due to random noise. Additionally, the model demonstrates a satisfactory level of adequacy, as indicated by the R^2 value. In this study, term A is identified as a significant model term ($p < 0.05$). The Lack of Fit F-value is 0.61, which suggests that the lack of fit is not significant when compared to the pure error. There is an 80.18% chance that a lack of fit F-value of this size could arise from noise. A non-significant lack of fit is advantageous, as it indicates that the model adequately represents the data.

Table 4. Analysis of variance (ANOVA) for response surface linear model to identify significant factors affecting the FFA of crude palm oil.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	10.75	3	3.58	11.73	< 0.0001*
A-Time	9.70	1	9.70	31.75	< 0.0001*
B-Mass	0.0256	1	0.0256	0.0838	0.7750
C-Maturity	1.03	1	1.03	3.37	0.0801
Residual	6.72	22	0.3055		
Lack of Fit	3.46	14	0.2474	0.6076	0.8018
Pure Error	3.26	8	0.4072		
Cor Total	17.47	25			

Standard deviation: 0.5527; R^2: 0.6153; Adj R^2: 0.5629; Pred R^2: 0.4992; Adeq precision: 11.9918
*Significant variable

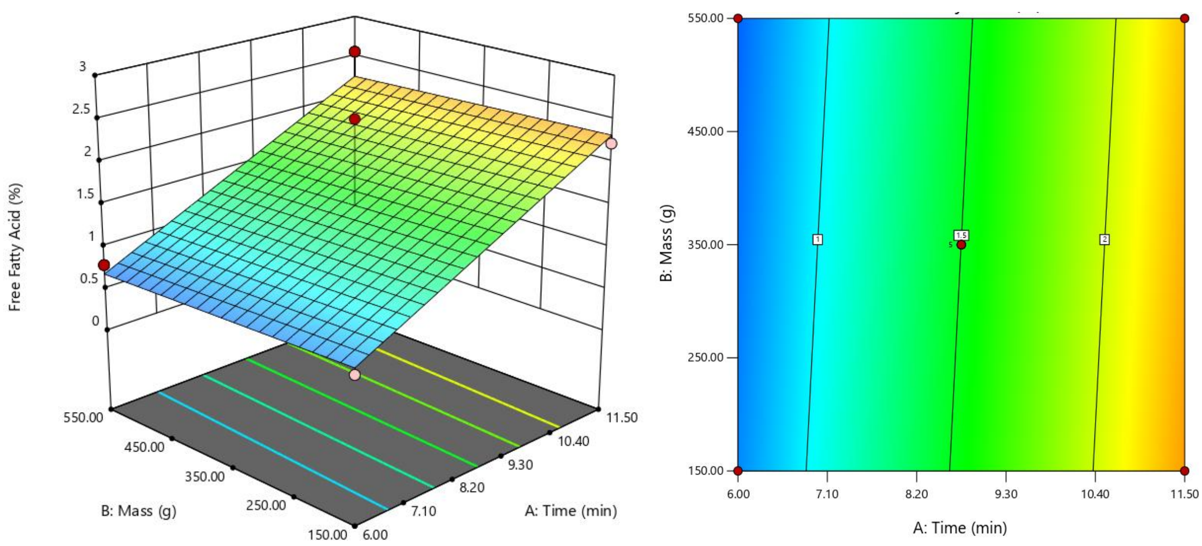
The coefficient of determination (R^2) for the fitted model was 0.6153. This indicates that 61.53% of the variation in the sample related to microwave oven sterilization of fresh palm fruit can be explained by the independent variables, while 38.47% of the total variation remains unexplained by the model. Overall, we can conclude that the developed model accurately represents the true relationship among the selected parameters (as shown in Table 2). The predicted R^2 value is 0.7581, which suggests that the overall mean aligns reasonably well with the adjusted R^2 value of 0.5629, as the difference between the two is less than 0.2. Furthermore, a ratio of 11.992 indicates an adequate signal, as a ratio greater than 4 is considered desirable. This model can be effectively used to navigate the design space. Response surface curves are plotted to analyze the interaction of variables and identify the optimal level of each variable for achieving the minimum response.

The CPO FFA content (Y) data collected under various experimental conditions, including sterilization time and mass at different fruit maturities (ripe and over-ripe), can be expressed by the following linear equation, as detailed in Table 5. The equation indicated that FFA was mainly influenced by sterilization time.

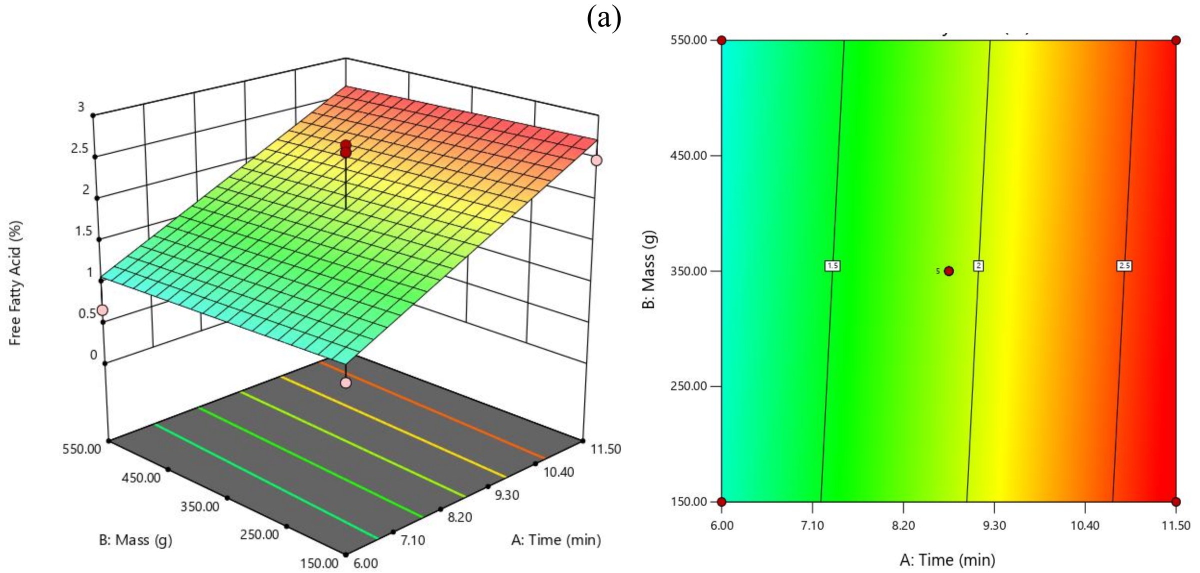
Table 5. Final equation in terms of actual factors of CPO Free Fatty Acid

Source	Ripe	Over-ripe
Model coefficient	− 0.906381	− 0.508695
A-Time	0.283100	0.283100
B-Mass	− 0.000200	− 0.000200

The response surface curves and contour plots for the microwave oven sterilization of CPO are shown in Figure 2. These figures will help explain how sterilization time and the mass of fruit affect the FFA of CPO from different stages of fruit maturity. Figure 1a illustrates the interaction regarding CPO FFA for ripe fruit, while Figure 1b focuses on the effect on CPO FFA for over-ripe fruit. For ripe fruit (Figure 2a), the highest CPO FFA, which was 2.55%, was achieved with 11.50 minutes of sterilization and 550 grams of fruit. Additionally, increasing either the sterilization time or the mass of the fruit, within the studied ranges, resulted in higher FFA. Similarly, for over-ripe fruit (Figure 2b), the maximum CPO FFA was 2.66%, also achieved with 8.75 minutes of sterilization and 350 grams of fruit.



(a)



(b)

Figure 2. Three dimensional response surfaces and their respective two dimensional contour plots for the effects of sterilization time and fruit mass on the free fatty acid of crude palm oil at different fruit maturities, i.e. (a) Ripe, and (b) Over-ripe

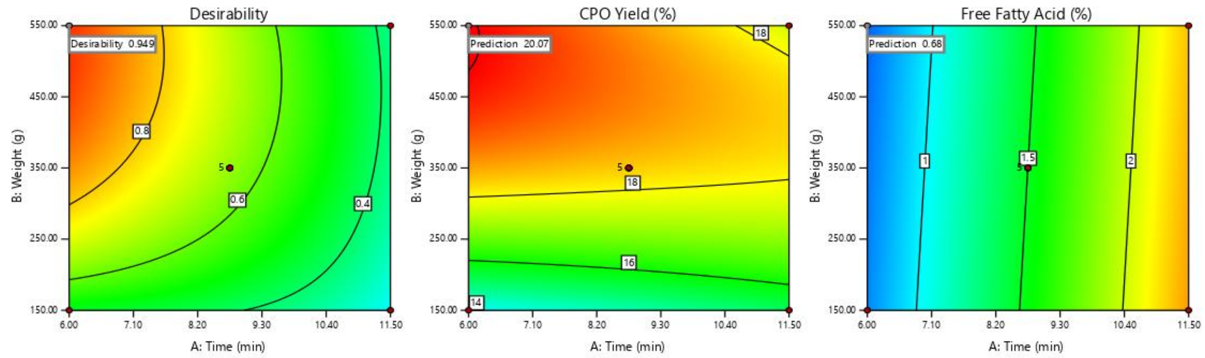
FFA content are crucial for assessing CPO quality. The quality standard for FFA in CPO is set at a maximum of 5%. In this study, the average FFA content obtained through microwave oven sterilization were below this standard. However, the FFA content observed in our research were higher than those reported by Makky et al., (2020). Makky et al., (2020) used a 720 W microwave oven for 4 min, resulting in a FFA content of 0.29%. This is due to the longer sterilization time (6 min) and higher power (900 W).

Optimization and Model Verification

The aim of the optimization process for microwave oven sterilization of fresh palm fruits was to achieve the highest yield of CPO while minimizing the amount of FFA. The optimal conditions resulted in a maximum CPO yield of 20.070% and a minimum FFA content of 0.682% at ripe maturity (Table 6).

Table 6. Predicted Optimum Conditions of Palm Fruit Microwave Oven Sterilization

Variables	Minimum	Maximum	Optimum
Time	6.00	11.50	6.00
Mass	150	550	550
Maturity	Ripe	Over-ripe	Ripe



(a) (b) (c)

Figure 3. Predicted values of the responses obtained at optimum conditions (a) Desirability; (b) CPO yield, and (c) Free Fatty Acid

The image presents contour plots highlighting the response variables—Desirability, CPO Yield, and FFA content—predicted at optimal operating conditions represented by specific values of Time (around 6 min) and Weight (around 550 g).Desirability: The highest predicted desirability value is approximately 0.949. This indicates that the chosen conditions maximize the overall desirability, balancing multiple response objectives. A desirability of 1 implies an almost ideal setting for the process, considering all factors simultaneously. CPO Yield: The predicted CPO yield is about 20.07% under optimal conditions, reflecting a comparatively high oil extraction efficiency. This suggests that increasing the sample weight significantly enhances oil yield while ensuring process efficiency quickly without compromising yield. FFA: The predicted FFA content is 0.68%, which is considerably low, signifying good oil quality. Low FFA content are critical to maintaining the quality and stability of palm oil since elevated FFA often leads to rancidity and spoilage.

CONCLUSION

In this research, microwave oven sterilization of palm fruit was conducted. The effects of sterilization time, mass, and maturity evaluated using a CCD. The CPO yield model demonstrated an adjusted determination coefficient ($RAdj^2$) of 86.67% and a coefficient of determination (R^2) of 90.94%. For FFA, the $RAdj^2$ was 56.29%, and the R^2 was 61.53%. Analysis indicated that while time did not significantly affect CPO yield, it significantly influenced FFA. Conversely, mass and maturity significantly impacted CPO yield but did not significantly influence FFA. Consequently, microwave irradiation is essential for oil palm fruit sterilization. The optimized microwave oven sterilization conditions for a ripe maturity level were determined as 6 min for sterilization time and 550 g for fruit mass. Under these optimal conditions, a maximum CPO yield of 20.070% and a minimum FFA content of 0.682% were achieved at the ripe maturity level. Future studies may explore scale-up using industrial microwave systems and assess cost-benefit performance.

ACKOWLEDGEMENT

We would like to thanks to PT. Agro Mitra Madani for supporting facilities during this research.

FUNDING

This research was funded by the Faculty of Agriculture's Basic Research scheme which is sourced from the University of Jambi's "Program Penerimaan Negara Bukan Pajak (PNBP)" No 295/UN21.11/PT.01.05/SPK/2023.

CONFLICT OF INTEREST

The author declares no conflict of interest.

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