Process Optimization to Preserve β-Carotene During Palm Fruit Irradiation

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Abstract. Excluded free fatty acid content, palm oil quality was indicated by the presence of minor constituents in palm oil such as β-carotene. Heating utilizing microwave energy degrades β-carotene. This study carried out process optimization to evaluate significant parameters affected palm oil quality during sterilization by microwave irradiation and to obtain optimum process parameters to preserve β-carotene concentration in palm oil. Central composite design (CCD) and response surface methodology (RSM) were used. The optimum β-carotene concentration in this study was 404 ppm obtained from microwave sterilization with combination of mass, power and time approximately 117 grams, 669.087 watts and 5 min respectively.

1. Introduction
Palm fruit is native tropical fruit utilized to produce edible oil from its mesocarp (palm oil) and kernel (palm kernel oil) [1]. The primary stage in processing oil palm fruit is sterilization where the palm fruit will be placed into a sterilizer with pressurized steam of 40 psi and a temperature of 140 °C. The sterilization process provides several benefits, such as deactivating the lipase enzyme, facilitating the threshing of fruit from the bunches and it also causes breakage in the cell wall of palm fruit mesocarp, so that less oil will be left in the palm fruit mesocarp after the digestion process [2]. A lot of the contents contained in palm oil among others are minor constituents which is divided into two categories. The first category consists of fatty acid derivatives, such as partial glycerides (mono and diacyl glycerol), phosphatides, esters and sterols. The second category includes classes of compounds not related chemically to fatty acids called non-glyceride constituents. The non-glyceride fraction of palm oil consists of sterols, triterpene alcohols, tocopherols, phospholipids, chlorophylls, carotenoids, volatile flavour components such as aldehydes and ketones [3]. Among those minor constituents, the most important are carotenoids and vitamin E. Both carotenoids and tocopherols are responsible for palm oil stability and nutritional value. Carotenoid content in palm oil is found primarily between 700-800 ppm as β-carotene [4, 5] while vitamin E is present in crude palm oil (CPO) and residual oil from palm pressed fibre (PFO) at concentration ranged between 600–1000 and 2000–4000 ppm, respectively [6].

Nowadays, sterilization of oil palm fruit can be performed by using microwave energy. Several studies reported heating and sterilization of oil palm fruit by microwave irradiation. This sterilization method can save time and energy. Save time means sterilization carried out very fast, while saving energy indicates by lower temperature requirement to conduct sterilization [5, 7, 8, 9, 10, 11, 12].
Sarah and Taib (2013) reported sterilization of palm fruit utilizing microwave energy carried out very fast (less than 17 min) with respect to decimal reduction time ($D$-value) of lipase at temperature between 70 to 85°C. They observed CPO extracted from sterilized fruits contained significant amount of vitamin E concentration after heated by microwave energy at various $D$-value such as 1300 ppm ($D$-value = 8.33 min) and 1333.33 ppm ($D$-value = 16.95 min) respectively. In the same report, they also observed that microwave sterilization of palm fruit for more than 9.71 min destroyed some carotenoids. This typical crude palm oil contained β-carotene ranging only between 300.16-360.40 ppm or 50% lower as compare to carotenoids content in palm oil obtained from extraction of fruits from conventional sterilization [4, 5]. Structure of carotenoids with conjugated double bonds makes its vulnerable to heat. Heating of oil palm fruit at temperature of 60°C above accelerates degradation of carotenoids which occurred slowly at ambient temperature [4]. Concerning advantages of microwave sterilization and with respect to previous studies, it is important to carry out microwave sterilization process that quarantines minor β-carotene loss in palm oil during sterilization. In fact, β-carotene acts as pro vitamin A that prevents human body from diseases.

Sarah (2018) reported method to preserve carotenoids during Sterilization of Palm Fruit Using Microwave Irradiation. This method was developed from $D$-value of lipase and $D$-value of carotenoids that offered several combinations of time and temperature required by microwave sterilization to preserve carotenoids in palm oil product [13]. All those process combinations were not represented optimum condition required for this kind sterilization. Optimum parameters for microwave sterilization should be determined by process optimization.

In this study, we conducted process optimization for β-carotene preservation to obtain better CPO quality through microwave sterilization. Optimization aims to improve process parameters so as to study key parameters influences microwave sterilization performance and their relationship with CPO quality especially β-carotene content in palm oil. This study used Minitab 17 Statistical Software trial version to optimize microwave sterilization process of palm fruit. This study used central composite design (CCD) to adjust level of parameters during microwave sterilization process, such as sample mass, microwave power and time of sterilization. The levels of parameters in the experimental design 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 aims to estimate RSM model of second order equation to determine the point that gives the optimum response condition. Experimental design of this study is shown in Table 1. Statistical analysis is also carried out on independent variables that aims to study their effect on the response parameters so that the optimum condition can be obtained.

2. Materials and Methods
2.1 Materials and rigs of experiment
The main raw material used in this study is mature palm fresh fruit bunches Dura variety taken around the area of Universitas Sumatera Utara. This experiment used a commercial microwave oven (Sharp R-249 IN (S)/(W)) which has dimensions of 319 x 211 x 336 mm at frequency of 2450 MHz and maximum power of 800 watt. Palm fruit extraction process was conducted utilizing fabricated manual hydraulic presser with a capacity of 1-2 kg.

2.2 Sterilization process
The fruitlets were weighed and arranged in the centre of the microwave oven cavity. Adjustment of power and time for sterilization conducted with respect to experimental design as shown in Table 1. After the sterilization process was completed, the oil palm fruits were inserted into the hydraulic presser, and the oil was collected for quality determination test.

2.3 Determination of β-carotene
Determination of β-carotene content carried out using Shimadzu 1700 UV/VIS spectrophotometer. The palm oil sample was weighed as much as 0.04 grams and put into a 10 ml flask. The sample is then dissolved with n-hexane until it reaches the boundary mark and shaken until homogeneous. Measurements were taken at a wavelength of 446 nm of homogeneous diluted sample absorption.
2.4 Statistical analysis and optimization using response surface methodology

ANOVA was used in this study to evaluate the most significant factor contributed to crude palm oil quality especially carotenoids content in crude palm oil. Significant level was adjusted to 5% error ($\alpha = 0.05$) with confidence level of 95%. Optimization microwave sterilization performed by using Minitab trial version software and Response surface methodology (RSM) method was used to obtain second order polynomial model. This method also resulted surface and contour plots to observe the optimum condition from microwave sterilization process. A second order polynomial model was predicted and resulted several empirical model \[8\] as follow:

$$Y = \beta_0 + \sum_{i=1}^{2} \beta_i X_i + \sum_{i=1}^{2} \beta_{ii} X_i^2 + \sum_{i<j} \beta_{ij} X_i X_j$$  \hspace{1cm} (1)

The response variable ($Y$) represent carotenoids as $\beta$-carotene. Where $X_i$ and $X_j$ are independent variables while $\beta_0$, $\beta_{ii}$, and $\beta_{ij}$ are regression coefficients for intercept, linear, quadratic and interaction term, respectively.

### Table 1. Experimental design

| Run | Mass (g) | Power Level   | Time (min) | Run | Mass (g) | Power Level | Time (min) |
|-----|----------|---------------|------------|-----|----------|-------------|------------|
| 1   | 500      | Medium Low    | 8          | 11  | 750      | Low         | 12         |
| 2   | 1000     | Medium Low    | 8          | 12  | 750      | High        | 12         |
| 3   | 500      | Medium High   | 8          | 13  | 750      | Medium      | 5.27       |
| 4   | 1000     | Medium High   | 8          | 14  | 750      | Medium      | 18.72      |
| 5   | 500      | Medium Low    | 16         | 15  | 750      | Medium      | 12         |
| 6   | 1000     | Medium Low    | 16         | 16  | 750      | Medium      | 12         |
| 7   | 500      | Medium High   | 16         | 17  | 750      | Medium      | 12         |
| 8   | 1000     | Medium High   | 16         | 18  | 750      | Medium      | 12         |
| 9   | 330      | Medium        | 12         | 19  | 750      | Medium      | 12         |
| 10  | 1171     | Medium        | 12         | 20  | 750      | Medium      | 12         |

3. Results and Discussions

3.1 Predictive model to evaluate effect of process parameters to $\beta$-carotene concentration

Concentration of $\beta$-carotene ($Y$) data obtained under different combination of experimental factors such as mass ($X_1$), microwave power ($X_2$) and sterilization time ($X_3$) related by second degree polynomial model to explain relationship between process parameters and carotenoids concentration is shown in Eq. (2).

$$Y_{\beta\text{-carotene}} = 312 + 10.4X_1 + 44.2X_2 + 2X_3 + 8.4X_1^2 - 6.1X_2^2 + 26.1X_3^2 + 5.4X_1X_2 + 11.6X_1X_3 - 20.9X_2X_3$$  \hspace{1cm} (2)

Statistical analysis of experimental results indicated power was the only response with significant effect ($p = 0.037$) on the model with respect to $p$ value less than $\alpha$ (significant at $p$ value $< 0.05$). The equation describing the relationship between $\beta$-carotene concentration with sample mass factor, power and time with determination coefficient $R^2$ (correlation coefficient) = 49.28%. This indicates that only 49.28% of the total variation in the results obtained is represented in the equation. Residual assumption test such as independence test, identity test, and normal distribution test were conducted to evaluate the model.

3.2 Equation model analysis of $\beta$-carotene

Figure 1(a) show relationship between residual from ANOVA test plotted against fitted value. Residual data points for $\beta$-carotene concentration are scattered randomly and do not form a specific pattern. It indicates residuals of the model obtained equal distribution. Figure 1(b) show that the distribution of residual data versus sequence (order) tends to be random and not patterned, it shows
that among the independent variables are affected each other. Figure 1(c) show residual normality plot of β-carotene concentration. The linear regression line is a predictor of temperature data set during observation. Based on the residual normality plot, the resulting residual point has corresponded or approached the specified straight line. This is proved by The Kolmogorov-Smirnov (KS) statistical score. The KS calculate statistical score obtained was 0.088, while the KS table for $\alpha = 0.05$ and the observation number 20 was 0.294 [14]. This study obtained KS calculate less than KS table indicated residual of the obtained model has been normally distributed, it means that the observed experimental responses approached to the predicted results.

![Residual normality plot](image)

![Residual normality plot](image)

![Residual normality plot](image)

**Figure 1.** Equation model analysis of β-carotene concentration

### 3.3 Response surface and contour plot for β-carotene

The response surface model was obtained by plotting the response contour (β-carotene concentration) against three factors influenced the response such oil palm mass sample, microwave power and sterilization time. Figure 2 shows a point of 3D saddle plot response of surface and contour plot of β-carotene concentration for mass and power variables at constant sterilization time. In this situation sterilization time was adjusted for 5 min. Palm oil product with minimum concentration of β-carotene as depicted in Figure 2(a) was observed with concentration less than 350 ppm after irradiated palm fruit of 1083.21 gram by microwave energy using microwave power of 139.23 watts. On the contrary, palm oil with maximum concentration of β-carotene more than 425 ppm obtained at combination of 338.95 grams palm fruit and microwave power of 436.75 watts. The optimum condition to obtain palm oil with high β-carotene content at constant sterilization time as depicted in Figure 2 is discussed below.
caused by overheating, the presence of oxygen and contact with metals that initiate oxidation so that relatively unstable carotenoid characteristic is highly possible for β-carotene concentration in palm oil. The previous figures show significant quadratic effect of various mass and time to β-carotene concentration at constant microwave power of 400 watt. Minimum β-carotene concentration in palm oil (less than 330 ppm) as depicted in Figure 3(a) and 3(b) obtained if sterilization of 474.14 grams palm fruit occurred for 12.02 min. To obtain β-carotene concentration in palm fruit more than 450 ppm (maximum), approximately 1153.87 grams of palm fruit should be irradiated for 18.37 min.

Figure 3(a) and 3(b) display a point of minimum response from mass and time to β-carotene concentration with respect to process variables mass and sterilization time.

Figure 4(a) and 4(b) shows a 3D surface and contour plot of β-carotene concentration for power and time variables at constant mass (750 gram). To obtain β-carotene concentration at minimum level (250 ppm), sterilization should be conducted for 17.14 min using power of 165.15 watts while maximum β-carotene concentration (more than 500 ppm) obtained after irradiation for 18.63 min using power of 668.94 watts. Power increment was observed provide a large thermal effect that indicated by rapid temperature rise that related to heat penetration which is a key factor in the sterilization of oil palm fruits, meanwhile sterilization of small palm fruit sample with a small mass runs faster than large mass samples. Increasing time and temperatures for sterilization can promote β-carotene loss. The relatively unstable carotenoid characteristic is highly possible for β-carotene degradation, this is caused by overheating, the presence of oxygen and contact with metals that initiate oxidation so that the composition and activity of carotenoid antioxidants decreases [2, 15].
4. Conclusion

Microwave sterilization can be used to obtain optimum β-carotene concentration in palm oil using CCD dan RSM methods using Minitab software. The optimum β-carotene concentration in this study was 404 ppm obtained from microwave sterilization with combination of mass, power and time approximately 1170 grams, 669.087 watts and 5 min respectively. The concentration of β-carotene was not influenced directly by mass, power and time in the sterilization process, but influenced by other factors such as the presence of oxygen and contact with the metal at the extraction stage, which initiates the oxidation so that the composition and activity of the substance antioxidants decreased.

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