

# Processing of Olein Fraction Red Palm Oil with Minimal Refining Method and Optimization of Deodorization Process

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## ABSTRACT

Red Palm Olein (RPOn) is the result of refining and fraction of Crude Palm Oil (CPO). In the conventional refining of CPO, carotene which is a pro-Vitamin A is intentionally removed to get a clear colored oil.. This research is comparing between two methods are Method I and Method II. Method I is started from degumming process (acid degumming), Neutralization, deodorization, and then fractionation. Meanwhile, Method II is started from degumming process (water degumming), fractionation and then deodorization. Optimization using RSM method with CCD program, carried out in the deodorization process of the selected by method II. Method II is producing RPOn with 0.1949% moisture content, 0.8743 mE/kg peroxide value, 0.148% FFA, 57.5380 ppm of beta carotene and 100.5526 ppm of tocopherol alpha. Improvement of the deodorization process using RSM by setting the temperature to 90°C, vacuum pressure to 50 cmHg and 2.98 hours of deodorization process will be produce RPOn with 0.03% FFA.

**Keywords:** Red Palm Olein, Response Surface Method, Degumming, Beta Caroten, FFA.

## 1. INTRODUCTION

Crude palm oil (CPO) comes from the fruit of the palm oil (*Elaeis guineensis*), a tropical plant species native to West Africa. CPO is extracted from the palm mesocarp with a mechanical screw-press extraction process [1], [2]. In refining palm oil process, part of carotene is removed through bleaching process that followed with deodorization in high temperature that can spoil the carotene for getting clear palm oil [3]. CPO with rich beta carotene is important in the deficiency context of Vitamin A. Red Palm Oil (RPO) is the result of refining from CPO that produce high carotene content, is signed with red oil product. In the final product, RPO have a lot of varian product based on fractionation process. There are 5 variant of RPO: Red Palm Oil, Red Palm Olein, Red Palm Stearin, Red Palm Super Olein, and Red Palm mid-fraction [4].

RPO application research has been done for food product, healt, and skin care lotion [4]–[10]. The effort for maintaining nutrition value in CPO process ever did it using supercritical carbondioxide method, molecular

distillation, and membrane technology [11]–[14]. In this time, the refining of CPO especially in cooking oil manufacturing process (RBDPO) has damaged carotenoid pigments in bleaching process [15]. Several stages of the CPO purification process, namely degumming, neutralization, and deodorization can reduce the carotene content

Correction in refining palm oil steps, especially removing bleaching's step will produce palm oil with high carotenoids, known as Red Palm Oil (RPO). Degumming process is the first process (pretreatment) in refining process for removing Gum. Gum is general term for minor content in CPO that complex compounding of phospholipid, glycolipid, sugar, protein, and other water soluble compound in CPO [16], [17]. Phospholipid reduction's oil in bleaching process will be absorbed on the bleaching clay which will reduce its effectiveness in the ability to remove chlorophyll and its derivatives [18]–[21]. The phospholipid can cause decolorization oil, has function as the main precursor off flavor and causes loss of neutral lipids during neutralization in the refining general process [22]. Moreover, Phospholipid or gum is

considered as undesirable substance because can make dirty equipment pipe in high temperature process [23].

The degumming process that is usually done in palm oil processing is by using acid. It is degumming process whose process uses phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) dan warm water. Giving phosphoric acid and water (60 °C) is intended for removing non-hydratable phosphatides. Phosphoric acid can change it to hydratable shape. Hydratable phosphatides like phosphatidycholine (lesitin), can easily remove from oil using water [24], [16], [20]. Besides of degumming process, neutralization using NaOH and high temperature deodorization can damage the carotene [25], [26].

By modifying the method, processing or controlling the temperature factor, the length process of conventional refining process is expected to maintain minor components (carotene and tocopherol) and still maintain oil quality based on FFA parameters, moisture content and PV. With consideration potential nutrition value, then refining research and fractionation in CPO being Red Palm Olein (RPOn) with minimal processing method which is a modification of the conventional refining process. It started from degumming process, neutralization, deodorization, and fractionation. Comparison of the two selected refining process are followed by optimization of the deodorization using controlling the temperature, vacuum pressure and process time.

**2. METHODS**

This research uses CPO from PT Wilmar Nabati Indonesia. The process of refining palm oil into RPOn uses two processing methods. In method I is started from degumming process (acid degumming), neutralization, deodorization, and then fractionation. Meanwhile, method II is started from degumming process (water degumming), fractionation and then deodorization. Each method using different arrangement process except degumming process. In method I degumming process using acid degumming and method II using water degumming. Method I and method II using different arrangement refining process. Another difference is only method I using neutralization process with NaOH.

Water degumming is a treatment on CPO using water (60 °C). Acid degumming as like as water degumming process but there are addition of phosphoric acid (H<sub>3</sub>PO<sub>4</sub>). Neutralization using NaOH with excess 0.10% of oil weight. Fractionation using dry fractionation method is saving oil in room temperature within 24 hours, Deodorization process carried out in 80 °C temperature with 80 mbar vacuum pressure within an hour[27].

From RPOn that produce from the best selected method, the next step is to optimize the deodorization process. This optimize is using Response Surface Methodology (RSM) with Central Composite Design (CCD). Alpha value is calculated based on the formula  $\alpha$

$= 2k/4$ , k is the number of variable tested. This experiment is used three independent variables: temperature (A), pressure (B), time (C). Observe the responses is FFA percentage. . In addition, then 20 runs included 8 factorial points, 6 axial points, and 6 replicates at the center point to determine the experimental error for this study.

Experimental data is obtained will be analyzed using a second-order polynomial to find the connection between independent variable with FFA.

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i < j} \beta_{ij} X_i X_j + \varepsilon \tag{1}$$

Y is response (FFA),  $\beta_0$  is intercept  $\beta_i$ ,  $\beta_{ii}$ ,  $\beta_{ij}$  are linear, quadratic, and interactive coefficient, respectively, k is the number of factor, and  $X_i$  and  $X_j$  are independent variables under study.

**Table 1.** The center composite program of deodorization process with three independent variable.

Code	Name	Low code	High code	Low	High
A	Deodorization temperatur (0C)	-1	1	90	150
B	Vacuum pressure (cmHg)	-1	1	50	60
C	Time (hour)	-1	1	1	3

**2.1. Determination of FFA, Peroxide value and Moisture content**

The FFA of oil sample were determined using AOCS Official recommended method Ca 5a-40. Moisture content (AOCS Official Method Ca 2c-25 2012). Peroxide value (SNI 3741:2013)

**2.2. Determination of alpha tocopherol**

Oil sample were dissolved in etanol; the absorbance was measured at 446 nm. The carotenoid content of oil samples was expressed as mg alpha-tokoferol/kg oil (ppm)

**2.3. Determination of beta-carotenoids**

Carotenoids were determined according to PORIM test method (PORIM, 1995). Oil Sample were dissolved in hexane; the absorbance was measured at 291 nm. The carotenoid content of oil samples was expressed as mg beta-carotene/kg oil (ppm)

**2.4. Statistical Analysis**

The statistical analysis was performed using Microsoft Excel. Analysis of variance (ANOVA) was used to the oil quality parameters at significance level ( $\alpha$ ) of 0.05. Further significance difference was tested by Tukey’s test.. Process optimization using Response Surface Method (RSM) with Central Composite Design (CCD) and using Design Expert 6.0.8 software.

**3. RESULTS AND DISCUSSION**

**3.1. Method I vs Method II**

with warm water and sufficient heating treatment to remove excess water in water degumming, and then deodorization process in 80°C temperature with 80 mbar vacuum pressure within an hour, FFA is distilled in the vapor phase that causes FFA’s decrease in RPOn process [28].

The concentration of Beta Carotene in method I and method II in the end of process are not indicating the significant differences, but during the neutralization process it shows the decrease and tends to increase after fractionation process. The concentration of alpha tocopherol is showing decrease in method I (5.19%) although in method II (7.08%).

**Table 2.** Influence of CPO Processing Toward Moisture Content, Peroxide Value, FFA, Beta-Carotene, Alpha Tecoferol in RPOn Processing

	Moisture content (%)	Peroxide value (mE/kg)	FFA (%)	Beta carotene (ppm)	AlfaTokoferol (ppm)
Crude Palm Oil (CPO);	0.2385 ±0.0504	2.3086 <sup>a</sup> ±0.1105	1.1376 <sup>a</sup> ±0.0881	53.5940 <sup>abc</sup> ±1.1928	108.5526 <sup>a</sup> ±0.9977
Method I					
Acid Degumming (GPO);	0.2266 ±0.0216	1.9473 <sup>b</sup> ±0.0910	0.6126 <sup>b</sup> ±0.0214	52.1020 <sup>bc</sup> ±1.2739	99.5264 <sup>d</sup> ±0.8746
Neutralized Palm Oil (NPO);	0.2314 ±0.0344	1.6313 <sup>c</sup> ±0.0015	0.0562 <sup>de</sup> ±0.0289	50.1840 <sup>c</sup> ±3.2602	91.4738 <sup>e</sup> ±1.5528
Neutralized Deodorized Palm Oil (NDPO);	0.1955 ±0.0644	1.1919 <sup>d</sup> ±0.1068	0.0715 <sup>de</sup> ±0.0388	51.3900 <sup>bc</sup> ±2.9906	93.3946 <sup>e</sup> ±1.0713
Neutralized Deodorized Palm Olein (RPOn);	0.1998 ±0.0142	0.8742 <sup>e</sup> ±0.0882	0.0459 <sup>e</sup> ±0.0491	53.4820 <sup>abc</sup> ±4.6142	102.9210 <sup>bc</sup> ±1.2600
Method II					
Water Degumming (GPO);	0.2202 ±0.0305	1.9066 <sup>b</sup> ±0.0899	0.6984 <sup>b</sup> ±0.0362	53.2120 <sup>abc</sup> ±2.8787	100.5526 <sup>cd</sup> ±1.3398
Degummed Palm Olein (GPOn);	0.2131 ±0.0199	0.8354 <sup>e</sup> ±0.1078	0.2396 <sup>c</sup> ±0.0379	55.9240 <sup>ab</sup> ±1.2595	104.9474 <sup>b</sup> ±1.3693
Deodorized Palm Olein (RPOn);	0.1949 ±0.0191	0.8743 <sup>e</sup> ±0.1119	0.1480 <sup>cd</sup> ±0.0214	57.5380 <sup>a</sup> ±0.0371	100.8686 <sup>cd</sup> ±0.6059

RPOn process is using method I and method II. In every process is indicating the differences (Table 2) except moisture content parameter. There is a decrease in the number of peroxides (62.13%) and FFA (95.97), after acid degumming, neutralization, deodorization, fractionation process (method I), meanwhile in method II after water degumming process, fractionation, and deodorization process, peroxides value (62.12%) and FFA (86.99%). This significant decrease of FFA because in method I, neutralization process is using NaOH to remove FFA through the saponification process, whereas in method II, the decrease because of washing process

**3.2. Deguming**

In degumming method (acid degumming and water degumming) as a pretreatment, this research used sedimentation in water-separation process; the oil would be carried over (losing). Acid degumming that used H<sub>3</sub>PO<sub>4</sub> will dissolve in water; it was marked by the acid pH. Washing should be done continuously until water’s pH back to normal (pH before adding acid/neutral). Doing many washing will increase chance of losing oil. In degumming process, moisture content didn’t show any different; it was between 0.220% until 0.226%. The result

of this research showed that acid degumming method, which phosphate acid in 60 °C decreased beta-carotene from 53.59 ppm to 52.10 ppm (3%), while in water degumming method decreased from 53.59 ppm to 53.21 ppm (1%), the decreases didn't seem any different. The decreased also happened in alpha tocopherol from 108.55 ppm to 99.53 ppm (8%) in acid degumming method; while in water degumming method from 108.55 ppm to 100.55 ppm (7%), the differences was clear. The decreases of beta-carotene and tocopherol was accordance with research by You et al [18] , it showed that using phosphate acid 0.49 into 200 gram CPO in 60 °C would decrease beta-carotene from 680.67 ppm into 625.00 ppm, in other word there are 55.67 ppm decreases (8%), and also in a research by Nur Sulihatimarsyilia et al., [25] there were decreasing in carotenoid and tocopherol after acid degumming and water degumming process. The decreasing of beta-carotene and alpha tocopherol is not only due to phosphate acid but also the water that was used in degumming process. Degumming process threw away not only water but also oil that contained beta-carotene [25], [26], [29].

Content of FFA in CPO that has been degummed with water decreased along with gum's elevation. Phospholipid removal in CPO (using degumming) caused decreasing FFA in oil, from 1.13% to 0.61% (0.52%) in acid degumming and from 1.13% to 0.70% (0.43%) in water degumming. FFA decreasing was detected in drying process which losing over water that used in degumming before another step. A good handle of degumming process that restrained/decreased hydrophilic process was conditioned time zone the water contacted with oil and also the separation. The similar result showed in Nur Sulihatimarsyilia et al [25], where FFA in PPFO decreased from 5.30% to 5.11% in water degumming process.

Degumming method as a pretreatment from this research-result showed that using water degumming and acid degumming seemed no differences according the observation parameter (FFA, moisture content, beta-carotene, alpha-tocopherol, peroxide value). However the process was different, water degumming didn't use chemical (phosphate acid), so it was more natural. The use of water in water degumming process was lesser than acid degumming process. In acid degumming, washing process would continuously do until pH back to normal. Usage of water and water separation with sedimentation method would take more time because washing process was done repeatedly. Beside that losing neutral oil in acid degumming process (2.75%) was more than water degumming (1.87%).

### **3.3. Neutralization**

According to the result of research, average of FFA palm oil after neutralization was decreased to 0.06% from FFA degree 1.14%, along with the average of rudiment oil decreased 5.7% from rudiment before (degummed palm oil). This was showed that added NaOH in degummed palm oil gave a significant result in

decreasing FFA in oil through soap process. It can be happened in several neutral oil, so that neutral oil can be thrown [30]. Compared with research conducted by Mayamol et al. [27], influence of neutralization on reduction of FFA showed that a tenfold reduction in the percentage of FFA had occurred for NPO and increasing the neutral oil loss. Widarta, Nuri and Haryati [31] reported that neutralization stage of NRPO (Neutralized Red Palm Oil) for pilot plant scale could reduce FFA to 0.13%.

The average of moisture content palm oil after neutralization didn't show any differences, in 0.20%. the highest of moisture content was depend on separation process and NaOH concentration using low soluble alkali NaOH caused emulsion between oil and soap. That emulsion trapped the water from soluble alkali NaOH and the sample, so it spread into emulsion and can't be separated. In concentration of higher soluble alkali NaOH, the emulsion didn't happen, the soluble alkali NaOH along with free fat acid shaped soap degree from neutralization was lower. Neutralization process used so many water, especially washing remains NaOH. The oil was washed in water so that remains NaOH and soap would be thrown perfectly, it was marked by pH that nearly 7 (neutral). The amount of water and time in water-oil separation was the weakness of neutralization process, where the volume of water and oil were 1:1 [27], [30].

### **3.4. Last Product Red Palm Olein (RPOn)**

The result of research showed that using method I and II, the quality of RPOn was not seem any differences in physic and chemical observation (moisture content, FFA, PV), but there are clear differences in some component observation on first source, CPO. Oil inclined to react with oxygen auto-oxidation, it was not only incline with fat oil composition but also another component in it, such as natural pro-oxidant and anti-oxidant like carotenoid and tocopherol, or some certain heavy metal like copper and iron that can be worked as pro-oxidant. Thus, low number of pre-oxidant in RPOn can be connected with some carotenoid in high concentration and also tocopherol that function as anti-oxidant, so that fat oxidation process can be prevented. Beta-carotene could stabilize radical component from oil with hydrogen donor to lipid radical and radical peroxy [32]. In carotenoid observation ( $\beta$ -carotene) and alpha tocopherol, RPO that had no enhanced in concentration, it was similar with CPO (Table 2). Through deacidification process with NaOH or through deodorization process in 80 °C and vacuum 80 mbar, beta-carotene still survived.

**Table 3.** Quality of Crude Characteristic and Red Palm Oil Compare with Another Research

Sampel	FFA (%)	PV (mE/kg)	$\beta$ -karoten (ppm)	Tokoferol alpha (ppm)
CPO <sup>a</sup>	3.53	2.32	56.00	187.00
CPO	1.14	2.31	53.59	108.55
Red Palm Olein <sup>a</sup>	0.04	0.10	33.30	166.00
Red Palm Oil <sup>b</sup>	1.27			
Red Palm Oil <sup>c</sup>	0.49	0.12		
Refined Palm Oil <sup>d</sup>	0.14	1.08		
Red Palm Olein (Metode I)	0.05	0.87	53.48	102.92
Red Palm Olein (Metode II)	0.15	0.87	57.54	100.87

<sup>a</sup> [4]; <sup>b</sup> [33]; <sup>c</sup> [34]; <sup>d</sup> [30];

From Table 3, showed that quality of RPOn in this research was similar with other research, FFA's score war near <0.1%, meanwhile PV was still between 0.1. to 0.08 mE/kg. While  $\beta$ -carotene concentration in method I and II could maintain its concentration (not significant) and a little decline in alpha tocopherol concentration (5.19% and 7.08%). Comparing parameter quality with

other research showed that method I and II could be used to produce high quality RPOn. Between method I and II, method I had advantage in FFA's low percentage. This was because in its process NaOH used as chemical material and it worked effectively to lower FFA. In method II, FFA can be lower by repairing deodorization

**Table 4.** Experimental Design with Observed and Predicted Values

No	Coded			Observed FFA	Predicted FFA
	A- Temperature	B- Pressure	C- Time		
1	-1	1	-1	0.153	0.241
2	-1	-1	-1	0.153	0.222
3	1	1	1	0.179	0.199
4	0	0	1.682	0.025	0.051
5	0	-1.682	0	0.230	0.189
6	0	0	0	0.460	0.484
7	1.682	0	0	0.281	0.028
8	1	-1	-1	0.051	0.128
9	-1	1	1	0.025	0.038
10	0	0	0	0.486	0.484
11	0	0	0	0.508	0.484
12	1	1	-1	0.051	0.147
13	0	1.682	0	0.306	0.221
14	-1.682	0	0	0.204	0.151
15	1	-1	1	0.178	0.180
16	0	0	-1.682	0.330	0.178
17	0	0	0	0.535	0.484
18	-1	-1	1	0.026	0.019
19	0	0	0	0.383	0.484
20	0	0	0	0.510	0.484

process in controlling temperature, pressure and time a process take to get optimum solution in using RSM.

**3.5. Optimize Deodorization in RPOn Process**

Optimize process in this research was done in deodorization process from method II with 3 variables independent such as temperature, pressure and time using responses of FFA percentage. The target of deodorization was volatile compound [35], so that it was done in high temperature of processing RBDPO [21]. Because this research was making RPO so the temperature had to be as low as possible, it was for decreasing FFA[29], [34].

RSM was employed to evaluate the relation between the responses (FFA) and the 3 reaction variables. The result in every point based on CCD experimental plans is shown in Table 4. The responses from regression analysis are correlated with 4 variables independent using polinomial equation orde 2. The scores that are observed are variation such as 0.025% in temperature 90°C pressure - 60 cmHg, and time 3 hours, or 0.5353% in temperature 120oC, pressure -55 cmHg and time 2 hours.

**Table 5.** Summary of regression coefficients, P-values, and fitting parameter model responses

Response model	FFA (%)	
Sugested model	Quadratic	
Model P-value	0.0029*	
R2	0.8612	
Adj. R2	0.7362	
Pred. R2	0.0973	
Adeq.Prec	7.172	
Lack of Fit	0.0562	
Parameters	RC	p-value
Intercept	0.4806	
A	0.0169	0.5102
B	0.0094	0.7115
C	-0.03765	0.1601
A2	-0.1078	0.0012
B2	-0.1078	0.0022
C2	-0.1307	0.0003
AB	9.55E-05	0.9977
AC	0.0638	0.0775
BC	2.72E-05	0.9993

A: Temperature (°C); B: Vacuum Presure (cmHg); C: Time (hour); RC: regression coefficient; R<sup>2</sup>: determination coefficient; Adj. R<sup>2</sup>: adjusted R<sup>2</sup>; R<sup>2</sup>: predicted R<sup>2</sup>; Adeq.Prec : Adequate precicision; \*P < 0.05

Statistic test result using Design Expert, anova are used to know the effect from variable to Y responses, showed that the model had a real impact or significant to responses where p-value = 0.003 or smaller than 0.05

[36]. The p-value of the lack of fit was 0.0562, indicating that it was not significant. Therefore, the number of experiments was sufficient to determine the effect of the variables on FFA. Model suitability is tested too using equation of regression and coefficient determination (R<sup>2</sup>). The high value of R<sup>2</sup> (0.8612) indicates that the fitted model can be used to predict reasonably precise outcomes.

From Table 5, factor of pressure (A<sup>2</sup>), temperature (B<sup>2</sup>) and time (C<sup>2</sup>) were found to be significant at the 95% confidence level according to computed F-value (high) and p-value at 5% level. The statistical tests indicate that the selected model is satisfactory for predicting the FFA within the scope of the studied variables, and also shows that the quadratic model is valid in the present study. The smaller the p-value for parameter, more significant the parameter is.

**3.6. Optimization Responses FFA**

Criteria or objective function that is used to set the optimum conditions which are suitable with the limitations (constraints) as in Table 6. Goal constraints that are given with minimum temperature, pressure and time would lower the damage risk at minor content that had nutrition and also decreased resources. Given constraints are expected to give minimum FFA percentage.

**Table 6.** Constraints Responses for Optimum

Name	Goal	Lower Limit	Upper Limit
A:Temperature (0C)	minimize	90	150
B: Vacuum Pressure (cmHg)	minimize	50	60
C: Time(hour)	is in range	1	3
FFA (%)	minimize	0.0253	0.5353

From the constraints criteria in Table 6, software Design Expert offered 8 optimum solutions, in first row highest desirability score was 0.9999 that temperature treatment, pressure and temperature would give optimum FFA (Table 7).

According to result in Table 7, there were 8 optimum solutions chosen by FFA lowest and highest desirability score. FFA responses that are offered by RSM model was in temperature 90 °C, pressure -50 cmHg and time 2.98 hours. That solution result would show FFA quality in 0.03%. Desirability result on the exact optimal solution showed 0.99. Desirability score 1 indicated response the perfect case, but desirability 0 indicated response the thrown case [37].

From result of optimum solution chosen, was done in validation test (Table 8) 3 times test with hour that compromised into 3 hours, is gained FFA score with differences 0.02 to 0.05 higher than predicted score, but

**Table 7.** Optimal Solution

No.	Temperature (°C)	Pressure (cmHg)	Time (Hour)	FFA (%)	Desirability	
1	90.00	50.00	2.98	0.03	0.999999	Selected
2	90.00	50.51	3.00	0.04	0.973472	
3	90.00	50.00	2.86	0.07	0.971939	
4	93.51	50.00	3.00	0.05	0.962533	
5	90.00	50.00	1.00	0.22	0.849369	
6	90.00	50.00	1.09	0.24	0.837624	
7	90.00	50.00	1.88	0.26	0.812908	
8	116.68	50.00	1.00	0.29	0.646898	

observed score that can be expected. From the results of the chosen optimum solution, a validation test (Table 8) was carried out 3 times with a compromised time of 3 hours, the FFA score was obtained with a difference of 0.02 to 0.05 higher than the predicted score. This shows that using a temperature of 90 °C, a vacuum pressure of 50 cmHg for 3 hours gives results that are not different from the RSM model

#### 4. CONCLUSION

Purifying method, minimum CPO to RPO is started with water degumming, fractionation then deodorization process (method II) has given parameter quality (moisture content and PV) that were not significant with purifying process that involved chemical (method I) in its process, beta-carotene and alpha tocopherol too. Decreased FFA content in method II can be optimized by controlling parameter in temperature, vacuum pressure and time of deodorization. Using RSM process in temperature 90 °C, pressure -50 cmHg and time 2.98 hours would get RPO with FFA score in 0.03%.

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