

Optimization of Deacidification of Degummed Palm Kernel Oil with Kolanut Pod Ash Using Response Surface Methodology

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Abstract

Deacidification of crude palm kernel oil (CPKO) remains a critical challenge in developing countries, where conventional chemical refining methods are energy-intensive and economically prohibitive for small-scale processors. This study investigated the optimization of CPKO deacidification using kolanut pod ash as an eco-friendly bio-adsorbent through Response Surface Methodology (RSM) with Central Composite Design. Three process variables were evaluated: reaction time (10 - 30 minutes), temperature (60°C - 70°C), and kolanut pod ash dosage (1% - 5% w/w). There was 91% free fatty acid (FFA) content reduction. A significant quadratic model (F-value = 3.82, p = 0.0242) with Adjusted R² of 0.5718 was obtained. The influential factors were reaction time (p = 0.0111), kolanut pod ash dosage (p < 0.0001), time-temperature (p = 0.0431) and time-dosage (p = 0.0073). Quality parameters remained within acceptable ranges: iodine values (35.4 - 50.3 g I₂/100g), acid values (7.18 - 51.02 mg KOH/g), and saponification values (250.67 - 290.12 mg KOH/g). This research demonstrates that kolanut pod ash can serve as an effective, low-cost, and green alternative to synthetic refining agents.

Keywords

Palm Kernel Oil, Deacidification, Kolanut Pod Ash, Response Surface Methodology, Free Fatty Acids, Optimization

1. Introduction

Palm kernel oil (PKO) is a valuable vegetable oil derived from the kernel of oil

palm fruit (*Elaeis guineensis*), widely used in food, pharmaceutical, and cosmetic industries due to its unique fatty acid composition rich in lauric acid [1]-[3]. Palm kernel oil does not contain cholesterol or trans fatty acids [4]. This makes it applicable in cooking meals for consumption and industrially as a reagent for the production of soaps, ingredients for baked goods, confectionery, ice cream and so on. Crude palm kernel oil is rich in saturated fatty acids, especially lauric acid (C12:0), which makes up about 48% - 55% of the total fatty acids. Other major fatty acids include: Myristic acid (C14:0): 14% - 18%, Palmitic acid (C16:0): 7% - 9%, Capric acid (C10:0): 3% - 5%, Caprylic acid (C8:0): 2% - 4%, Oleic acid (C18:1): 12% - 18% (monounsaturated), Linoleic acid (C18:2): 1% - 3% (polyunsaturated) [5]. Also, it is composed of minor components such as tocopherols and tocotrienols (Vitamin E compounds) which can act as antioxidants, sterols which include campesterol and β -sitosterol, free fatty acids (FFAs—ranging from 1 to over 10% depending on handling and storage conditions), moisture and impurities that are normally present in small quantities [6]. However, crude palm kernel oil is composed of other compounds known for their negative effect on the quality and stability of oils such as unsaponifiable matters, waxes, pigments, solid impurities (mainly fibers), oxidation products such as peroxides, aldehydes, ketones, alcohols, and oxidized fatty acids. The significant levels of free fatty acids (FFAs) compromise oil quality, reduce shelf life, and limit commercial acceptability [7] [8]. Also, high FFA content contributes to off-flavors, rancidity, and increased susceptibility to oxidative degradation, significantly affecting both sensory attributes and market value [9] [10]. Free fatty acids typically form through enzymatic and microbial hydrolysis of triglycerides during fruit storage, fermentation, or oil extraction processes, especially under suboptimal handling conditions [11]. Therefore, there is need to neutralize the high FFA to make the vegetable oil edible and well susceptible for industrial applications. Industrially, elevated FFA levels complicate downstream processing, as conventional chemical neutralization using alkaline solutions generates soap stock, resulting in substantial oil losses and reduced product yield [12] [13]. Traditional deacidification methods include alkaline neutralization, steam distillation, and physical refining [14] [15]. While effective, these approaches are energy-intensive, generate chemical waste requiring treatment, and remain economically prohibitive for small-scale processors predominant in Nigeria and other West African countries [16]. The palm oil industry in these regions consists largely of small- and medium-scale processors lacking the technical capabilities and capital for advanced refining operations, resulting in high acid values in locally produced oil that render it non-competitive in both local and international markets [17]. Recent developments toward green and sustainable technologies have stimulated research into bio-based adsorbents from agricultural waste products as alternative deacidification methods [18] [19]. These adsorbents work through adsorption mechanisms, where FFAs are bound to active sites on the adsorbent surface, thereby reducing the acid value of oil without the disadvantages associated with chemical refining [20]. Materials such as rice husks, cocoa pod husks, banana peels, and

groundnut shells have demonstrated varying degrees of success, valued for their low cost, renewability, and minimal toxicity [21] [22]. Kolanut (*Cola nitida* and *Cola acuminata*) is extensively cultivated in West Africa, particularly Nigeria, where the nuts are consumed largely during ceremonies and festivals [23]. Following seed removal, the pods rich in fibrous material including polyphenols, tannins, and cellulose are typically discarded as waste. Preliminary studies indicate that when thermally activated and chemically treated, these pods exhibit high adsorption capacity due to their large surface area, porosity, and reactive functional groups [24]. The incorporation of kolanut pods not only valorizes agricultural waste but also aligns with principles of circular economy, environmental conservation, and rural development [25]. Despite the potential of kolanut pod ash for oil deacidification, systematic optimization studies employing statistical design of experiments are lacking in the literature. Response Surface Methodology (RSM) has proven effective for optimizing complex processes in oil refining, enabling simultaneous evaluation of multiple variables and their interactions [26] [27]. Therefore, this study aimed to optimize the deacidification of degummed palm kernel oil using activated kolanut pod ash through RSM with Central Composite Design, evaluating the effects of reaction time, temperature, and adsorbent dosage on FFA reduction and oil quality parameters.

2. Materials and Methods

2.1. Materials

Freshly extracted, unrefined crude palm kernel oil was obtained from Miral Agro Venture (Akure North Local Government, Ondo State, Nigeria) and stored in a tank near the project refinery site. This crude palm kernel oil was degummed and stored in another tank. Kolanut pods were sourced from Bolounduro Cocoa Farm (Ondo East Local Government, Ondo State, Nigeria). Sodium hydroxide (NaOH) of 97% purity procured from LOBA Chemie PVT. Ltd. (CAS No: 1310-73-2 ADR/PG) was used as a conventional reagent for the deacidification process.

20 g of Sodium hydroxide pellets was weighed and dissolved in 500 ml of distilled water to give 1 molar concentration of Sodium hydroxide solution. Other reagents for laboratory analyses of deacidified oil, such as ethanol, chloroform, phenolphthalein, potassium iodine solution, sodium thiosulfate, iodine monochloride solution, potassium iodide solution, were of analytical grade. Distilled water was used throughout the experiments.

A custom designed and developed deacidification reactor with a throughput of 25 litres was used to deacidify degummed crude palm kernel oil with sodium hydroxide and kolanut pod ash. Analytical balance was used to measure the reagents used. Stopwatch was used to set time for the reaction between the oil and the reagents.

2.2. Preparation of Kolanut Pod Adsorbent

The kolanut pods were thoroughly washed multiple times with distilled water to remove external dirt, dust, and surface contaminants. After washing, the pods

were dried in a laboratory oven at 60°C for about 1½ hours until constant weight was achieved to remove all residual moisture [28].

The dried pods were subjected to controlled combustion in a muffle furnace at 600°C for 4 hours to ensure complete carbonization and ash formation. After cooling to room temperature in a desiccator, the resulting ash was ground using a laboratory mill and sieved through a 200 µm mesh to obtain uniform fine powder, with increased surface area to optimize particle size for ease of mass transfer [29]. The kolanut pod ash powder was stored in airtight containers protected from moisture until usage in deacidification experiments. All other chemical reagents used were of analytical grade.

2.3. Experimental Design and Statistical Analysis

Response Surface Methodology (RSM) with Central Composite Design (CCD) was employed to systematically investigate and optimize process parameters [30] [31]. The three process parameters (independent variables): reaction time (A: 10 - 30 mins), temperature (B: 60°C - 70°C), and kolanut pod ash (or NaOH) dosage (C: 0.5% - 1.5% w/w) are shown in **Table 1**. The experimental design consisted of 20 runs including factorial points, axial points, and center point replicates. The behavior of the system was explained by quadratic Equation (1):

$$Y = b_0 + \sum b_i x_i + \sum b_{ii} x_i^2 + \sum b_{ij} x_i x_j \quad (1)$$

where Y is the predicted response, b_0 is the offset term, b_i is the linear effect, b_{ii} is the squared effect, and b_{ij} is the interaction effect. All experimental design, data fitting, and statistical analysis were conducted using Design-Expert software. Analysis of variance (ANOVA) was performed to determine the statistical significance of each factor and interactions, as well as to assess overall model fit and predictive capability. A significance level of $p < 0.05$ was adopted for all statistical tests [30] [31].

Table 1. Inputs for the design of optimization table on RSM.

Variables	Lower Limit	Centre Point	Upper Limit
Temperature (°C)	60	65	70
Time (min)	20	25	30
Dosage of Reagents (% w/w)	0.5	1	1.5

2.4. Experimental Procedure for Deacidification

Deacidification experiments were conducted in batch mode using a locally designed and fabricated reactor. Fifteen-liter volumes of degummed crude palm kernel oil were transferred into the deacidification reactor. The oil samples were gently heated and maintained at the designed temperatures (60°C - 70°C), using the reactor's heating system, with continuous monitoring by a calibrated thermometer. Once the desired temperature was reached, varying dosages (0.5% - 1.5% by

weight of oil) of ashed kolanut pod (or NaOH) were carefully added to the heated oil. The mixtures were stirred continuously at a controlled agitation speed (250 - 300 rpm) using a metallic stirrer present in the reactor. This controlled stirring ensured uniform dispersion of the kolanut pod throughout the oil and minimized external mass transfer limitations [32]. The kolanut pod ash has a very high pH (12.5), allowing it to undergo neutralization reaction with the slightly acidic oil while still acting as adsorbent in the process. The deacidification process was allowed to proceed for predesigned contact times (20 - 30 mins). After the designated contact time, the mixture of oil and spent deacidification reagent was separated through centrifugation at 4000 rpm for 10 minutes to ensure complete separation and obtain clear, deacidified oil samples [33]. All experiments were carried out in triplicate to ensure reproducibility and statistical reliability of results. The clear, treated oil samples obtained from each experimental run were immediately subjected to analytical tests.

2.5. Analytical Methods

Free Fatty Acid (FFA) Determination: FFA content was determined via acid-base volumetric titration. One gram of oil sample was dissolved in 50 ml of solvent mixture (ethanol-diethyl ether) and titrated against 0.1 N KOH using phenolphthalein indicator [34]. FFA percentage was calculated as:

$$\text{FFA}(\% \text{ as oleic acid}) = \frac{56.1 \times N \times V}{W} \times 0.503 \quad (2)$$

where V = volume of KOH used (ml), N = normality of KOH, and W = weight of sample (g) [34].

Acid Value: Acid value was determined by measuring milligrams of KOH required to neutralize free fatty acids in 1 g oil [34]. Acid value was calculated using Equation (3):

$$\text{Acid Value}(\text{mg KOH/g}) = \frac{56.1 \times N \times V}{W} \quad (3)$$

where V = volume of KOH used (ml), N = normality of KOH, and W = weight of sample (g) [34].

Iodine Value: Iodine value was determined according to standard methods, quantifying the degree of unsaturation in the oil. 20 ml of potassium iodide was added to 1 g of deacidified PKO. 0.5 ml of starch was added to the mixture and was titrated against 0.5 N of Sodium thiosulphate [34]. The iodine value was determined according to Equation (4):

$$\text{Iodine value} = \frac{(B - S \times N \times 12.69)}{W} \quad (4)$$

where B = volume of thiosulphate for blank, S = volume of thiosulphate for sample and N = Normality of thiosulphate solution and W = weight of the sample [34].

Saponification Value: Saponification value was determined by standard alkaline hydrolysis methods, representing the milligrams of potassium hydroxide required

to saponify one gram of oil. 25 ml of alcoholic KOH was added to 1 g of deacidified PKO and was titrated with 0.5 N HCl using phenolphthalein as indicator [34]. The saponification value was calculated using the Equation (5):

$$\text{Saponification value} = \frac{(B - S) \times M \times 56.1}{W} \quad (5)$$

where B = volume of blank, S = volume of sample and M = Molarity of HCl and W = weight of the sample [34].

Phosphorus Content Determination: The phosphorus content of crude and degummed oil samples was determined spectrophotometrically according to AOCS Official Method Ca 12 - 55 [34]. Oil samples (0.5 g) were accurately weighed and digested with sulfuric acid. The digested samples were reacted with ammonium molybdate and ascorbic acid to form a blue-colored phosphomolybdenum complex. The absorbance was measured at 820 nm using a UV-Visible spectrophotometer. Phosphorus content was calculated from a standard curve prepared using potassium dihydrogen phosphate and expressed as mg P/g oil. All measurements were performed in triplicate. The phosphorus removal efficiency (PRE) in percent was calculated as:

$$\text{PRE}(\%) = \frac{\text{Initial Phosphorus Content} - \text{Final Phosphorus Content}}{\text{Initial Phosphorus Content}} \times 100 \quad (6)$$

3. Results and Discussion

The deacidification of crude palm kernel oil (CPKO) using kolanut pod and NaOH was investigated through a central composite design (CCD) with three independent variables: time (A), temperature (B), and dosage of reagent (C). The experimental design consisted of 20 runs including factorial points, axial points, and center points to evaluate the effects of process parameters on four response. The values of the response variables: free fatty acid (FFA) content, iodine value, saponification value, and acid value, with respect to the process parameters values are shown in **Table 2**.

Table 2. Comprehensive experimental design matrix and response variables.

Run	Factors			Responses			
	Dosage of Reagent (%w/w)	Temperature (°C)	Time (min)	FFA (mg KOH/g)	Acid Value (mg KOH/g)	Saponification Value (mg KOH/g)	Iodine Value (gI/100 g)
Kolanut pod							
1	0.5	60	30	14.97	51.47	289.73	123.56
2	0.5	60	20	17.28	74.34	282.48	117.90
3	0.5	70	30	12.47	35.82	270.84	110.56
4	1	73.41	25	12.38	62.91	211.77	84.89
5	1.5	60	20	17.28	43.15	232.85	120.45

Continued

6	1	65	16.59	12.38	40.90	285.65	106.45
7	1.5	70	30	13.53	39.80	294.52	140.89
8	1	65	25	19.01	50.89	284.88	78.32
9	1	65	33.41	13.82	58.89	276.34	117.55
10	1	65	25	19.01	50.89	284.88	78.32
11	1	65	25	19.01	50.89	285.89	78.32
12	0.16	65	25	12.38	35.63	240.56	114.34
13	1	65	25	19.01	50.89	284.88	78.32
14	1.84	65	25	16.70	55.62	301.00	112.56
15	1	60	25	14.68	50.89	284.88	78.32
16	1.5	60	30	17.01	32.89	296.55	109.75
17	1.5	70	20	15.84	45.75	288.67	106.23
18	0.5	70	20	12.60	34.89	276.22	111.54
19	1	56.59	25	16.99	32.43	266.56	100.78
20	1	65	25	19.01	50.89	284.88	78.32
NaOH							
1	1.5	60	30	6.12	12.24	332.78	139.45
2	1.5	60	20	9.87	19.74	342.12	149.34
3	1.5	70	30	5.23	10.46	328.45	134.56
4	1.5	70	20	7.45	14.90	336.89	142.67
5	0.16	65	25	11.34	22.68	345.67	154.23
6	0.5	70	20	10.56	21.12	343.45	151.78
7	1	65	25	6.89	13.78	334.56	141.23
8	1.84	65	25	5.67	11.34	330.12	136.89
9	1	73.41	25	8.12	16.24	331.89	138.45
10	1	65	25	7.23	14.46	335.78	142.56
11	0.5	60	20	9.23	18.46	340.23	148.67
12	1	65	33.41	6.45	12.90	333.45	140.12
13	1	65	25	7.01	14.02	335.12	141.89
14	1	65	25	6.78	13.56	334.67	141.45
15	1	56.59	25	10.23	20.46	343.89	152.34
16	0.5	70	30	8.67	17.34	338.56	145.78
17	0.5	60	30	9.45	18.90	341.34	149.12
18	1	65	16.59	10.12	20.24	342.67	150.89
19	1	65	25	6.95	13.90	334.89	141.67
20	1	65	25	7.12	14.24	335.45	142.12

3.1. Free Fatty Acid (FFA) Content Response

The FFA content exhibited substantial variation across experimental conditions, ranging from 12.38 - 19.01 mgKOH/g, representing the effectiveness of kolanut pod in deacidifying crude palm kernel oil. However, these values were significantly higher than that obtained from NaOH. The difference could be as a result of the powdery form of the reagent used, with lesser interaction with CPKO when compared with the liquid NaOH reagent used [35]. This can be improved by reducing the particle size and increasing the surface area of kolanut pod ash [35]. The variations observed in the two sets of data reflect the complex interactions between time, temperature, and reagent dosage during the deacidification process. Lower FFA values indicate more effective acid removal, which is crucial for improving oil quality and stability during storage and processing. The minimum FFA content of 12.38 mgKOH/g achieved in this study demonstrates the potential of kolanut pod as an effective natural deacidification agent.

Model Fit Statistics for FFA Content

The fit summary for FFA content response is presented in **Table 3**, comparing different polynomial models. The quadratic model was selected as most appropriate based on its significant sequential p-value ($0.0252 < 0.05$) and highest adjusted R^2 value (0.5674) among non-aliased models, confirming superior model adequacy compared to linear and 2FI models. The quadratic model best described the FFA response with F-value = 3.77 ($p < 0.05$), indicating high statistical significance. Among model terms, temperature (B, $p = 0.0179$) showed significant linear effects, while the quadratic effects of time (A^2 , $p = 0.0450$), temperature (B^2 , $p = 0.0016$), and reagent dosage (C^2 , $p = 0.0195$) were also highly significant, demonstrating strong curvature effects in the response surface. This curvature indicates the existence of optimal conditions beyond which FFA removal efficiency may decline, a common characteristic in adsorption and chemical treatment processes [36].

Table 3. Model fit summary for FFA content.

Source	Sequential p-Value	Adjusted R^2	Predicted R^2	Remark
Linear	0.1174	0.1692	0.0558	Suggested
2FI	0.9921	-0.0151	-0.2730	Not suitable
Quadratic	0.0087	0.5674	-0.0225	Suggested
Cubic	0.0691	0.8352	Aliased	Not recommended

The analysis of variance (ANOVA) showing the significance of model terms is presented in **Table 4**. Temperature showed significant effects ($F = 8.00$, $p = 0.0141$), suggesting that thermal activation plays a supporting role in the deacidification mechanism. All the quadratic terms (A^2 : $p = 0.0054$, B^2 : $p = 0.0023$, C^2 : p

= 0.0368) were significant in the FFA response of the deacidification process. However, the significant lack of fit ($p = 0.0013$) suggests that while the model captures major trends effectively, additional factors or model refinement may be needed for complete process description. The model performance with $R^2 = 0.7723$ and Adjusted $R^2 = 0.5674$ indicates good predictive capability, making this model suitable for process optimization within the experimental range studied. However, the negative Predicted R^2 (-0.0225) and significant “Lack of fit” values show poor fitness of the experimental data into the model.

Table 4. ANOVA results for FFA content.

Source	Sum of Squares	Df	Mean Square	F-Value	p-Value	
Model	98.09	9	10.90	3.77	0.0252	Significant
A-Time	3.12	1	3.12	1.08	0.3233	
B-Temperature	25.48	1	25.48	8.81	0.0141	
C-Dosage of reagent	1.72	1	1.72	0.5952	0.4583	
AB	0.0025	1	0.0025	0.0008	0.9774	
AC	0.0025	1	0.0025	0.0008	0.9774	
BC	0.6385	1	0.6385	0.2208	0.6485	
A ²	36.15	1	36.15	12.50	0.0054	
B ²	21.96	1	21.96	7.59	0.0203	
C ²	16.77	1	16.77	5.80	0.0368	
Residual	28.92	10	2.89			
Lack of Fit	28.92	6	4.82		0.0013	Significant
Pure Error	0.0000	4	0.0000			
Cor Total	127.01	19				

The model equations in terms of coded and actual factors are given as:

$$\begin{aligned} \text{Coded-FFA} = & -20.83 - 6.59A - 27.68B + 2.39C + 0.0700AB + 0.0350AC \\ & + 0.5650BC - 6.39A^2 - 5.07B^2 - 1.09C^2 \end{aligned} \quad (7)$$

$$\begin{aligned} \text{Actual-FFA} = & -17.42 + 3.12A + 6.23B + 3.54C + 0.0007AB - 0.0070AC \\ & + 0.1130BC - 0.0639A^2 - 0.0507B^2 - 4.3575C^2 \end{aligned} \quad (8)$$

Figure 1 shows the deviations obtained while using the model to predict the FFA values based on the experimental variables (the more the experimental points on the diagonal, the less the deviations of predicted values from actual values). The interactions of the process variables (temperature and time) are shown on the response surface plot given in **Figure 2**. An increase in the two variable led to decrease in the FFA of the palm kernel oil.

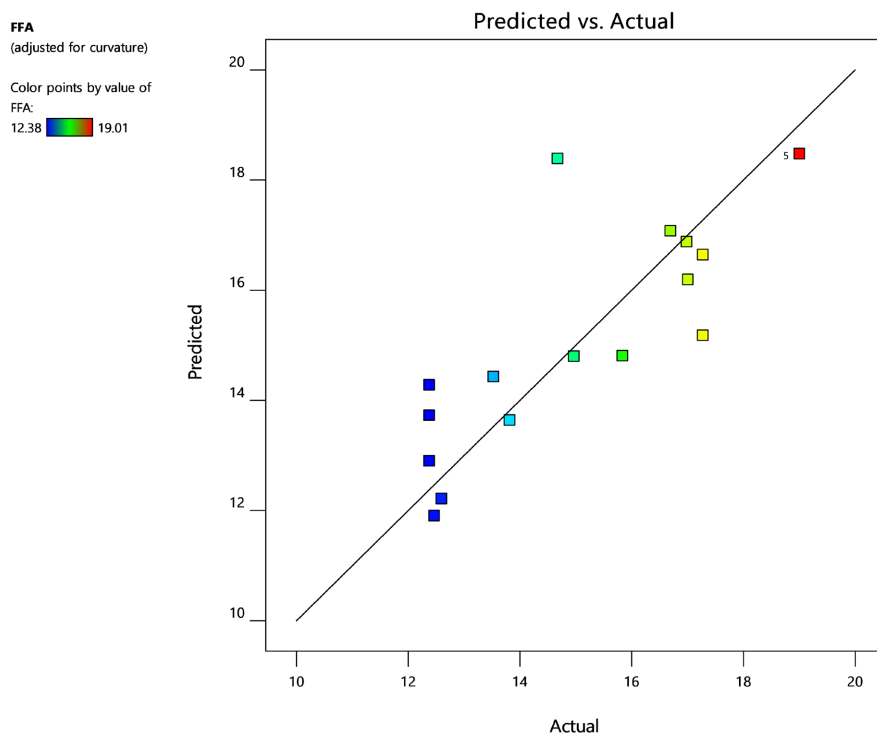


Figure 1. Graph of predicted value vs actual value of free fatty acid content.

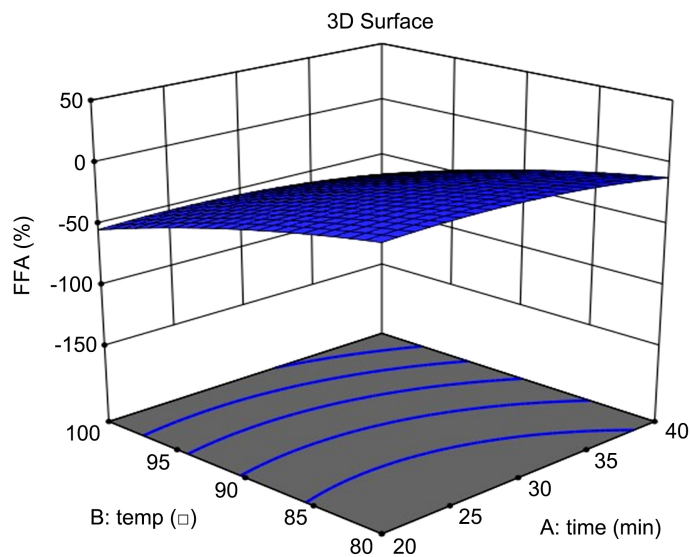


Figure 2. 3D Response surface plot of free fatty acid content.

3.2. Acid Value Response

The acid values of the CPKO ranged from 7.18 to 51.02 mg KOH/g after treatment. As shown in **Table 5** representing the model fit statistics for different models, the quadratic model was statistically significant (F-value = 5.42, $p = 0.0071$), with Adjusted R^2 of 0.6770 and non-significant lack-of-fit ($p = 0.1478$), confirming adequate model representation. None of the individual linear factors achieved statistical significance: time ($p = 0.6898$), temperature ($p = 0.2519$), or dosage (p

= 0.2368). This interaction-dominated response structure suggests that acid value evolution reflects the net balance between neutralization reactions, saponification of neutral oil, and thermal degradation effects [37]. The time-temperature interaction (AB) was significant ($p = 0.0250$, F -value = 6.94), indicating that thermal activation modulates neutralization kinetics [37].

Table 5. Model fit statistics for acid value response.

Source	Sequential p-Value	Adjusted R ²	Predicted R ²	Remark
Linear	0.8179	−0.1223	−0.5398	
2FI	0.1270	0.0955	−0.2740	
Quadratic	0.0037	0.6770	−0.0297	Suggested
Cubic	0.3421	0.7204	−8.4652	Aliased

The quadratic model was selected with identical fit statistics to FFA content, which is expected given the direct mathematical relationship between these parameters (acid value is proportional to FFA content through molecular weight conversion factors). As reflected in the ANOVA for the acid value response in **Table 6**, the model showed high significance (F -value = 5.42, $p = 0.0071$), with excellent adjusted R² (0.6770) confirming good predictive capability and model adequacy. The selection of the quadratic model over higher-order models ensures reasonable complexity while avoiding overfitting and aliasing issues that would compromise the model's practical utility for optimization purposes.

The ANOVA results of acid value were a bit similar to those obtained from FFA content, with reagent dosage being, though insignificant, the most influential factor ($F = 1.58$, $p = 0.2368$), demonstrating that the concentration of kolanut pod material is the primary determinant of deacidification effectiveness. One of the quadratic terms (A^2) was highly significant ($p < 0.05$), indicating a strong curvature effect for the time factor in the adsorption process.

Figure 3 shows the deviations obtained while using the model to predict the acid values based on the experimental variables. The interaction terms AB ($p = 0.0250$) and AB ($p = p < 0.05$) were significant, indicating that time interacts synergistically with both temperature and reagent dosage to influence acid value reduction. The interaction of the temperature and time is shown on the acid value response surface plot given in **Figure 4**. These synergistic effects emphasize that optimization must consider simultaneous adjustment of multiple factors rather than sequential optimization of individual parameters. The significant lack of fit ($p = 0.0071$) suggests that while the model captures major trends effectively, additional complexity exists in the underlying adsorption mechanisms, possibly including multi-site adsorption, surface heterogeneity, or time-dependent changes in adsorbent properties. Despite this limitation, the $R^2 = 0.9395$ and Adjusted $R^2 = 0.6770$ confirm excellent overall model performance, making this model highly

suitable for process optimization and prediction within the experimental range studied. Like FFA response, the negative Predicted R^2 (-0.0297) shows poor fitness of the experimental data into the model.

Table 6. Analysis of variance for acid value response.

Source	Sum of Squares	df	Mean Square	F-Value	p-Value	Remark
Model	330.98	9	36.78	5.42	0.0071	Significant
A-Time	1.15	1	1.15	0.1689	0.6898	
B-Temperature	10.02	1	10.02	1.48	0.2519	
C-Dosage of reagent	10.74	1	10.74	1.58	0.2368	
AB	47.05	1	47.05	6.94	0.0250	
AC	81.92	1	81.92	12.08	0.0060	
BC	1.13	1	1.13	0.1659	0.6923	
A ²	160.51	1	160.51	23.67	0.0007	
B ²	18.90	1	18.90	2.79	0.1259	
C ²	20.09	1	20.09	2.96	0.1159	
Residual	67.80	10	6.78			
Lack of Fit	49.59	5	9.92	2.72	0.1478	not significant
Pure Error	18.21	5	3.64			
Cor Total	398.78	19				

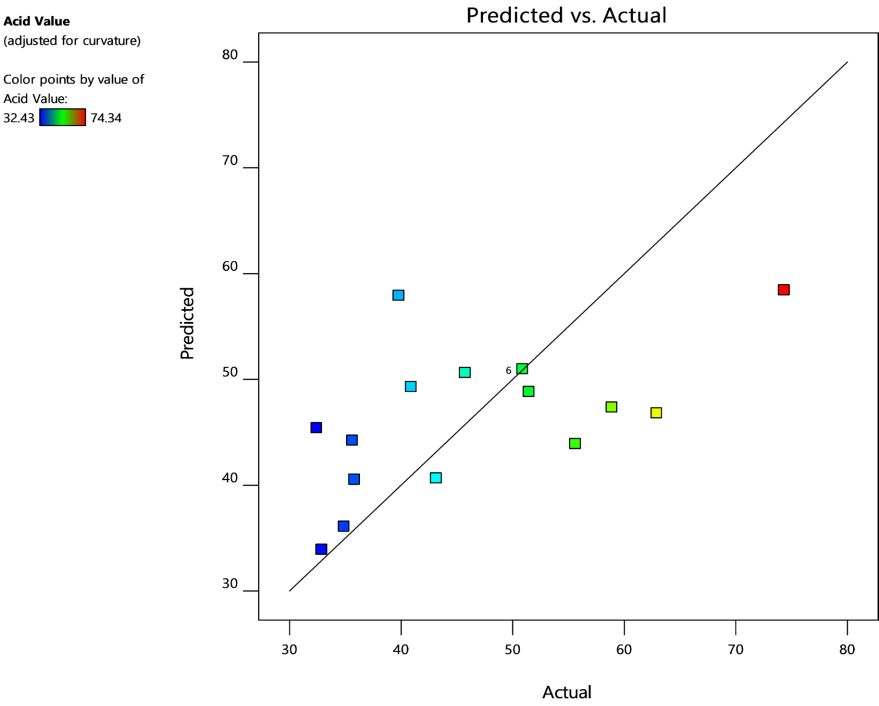


Figure 3. Graph of predicted value vs actual value of acid value content.

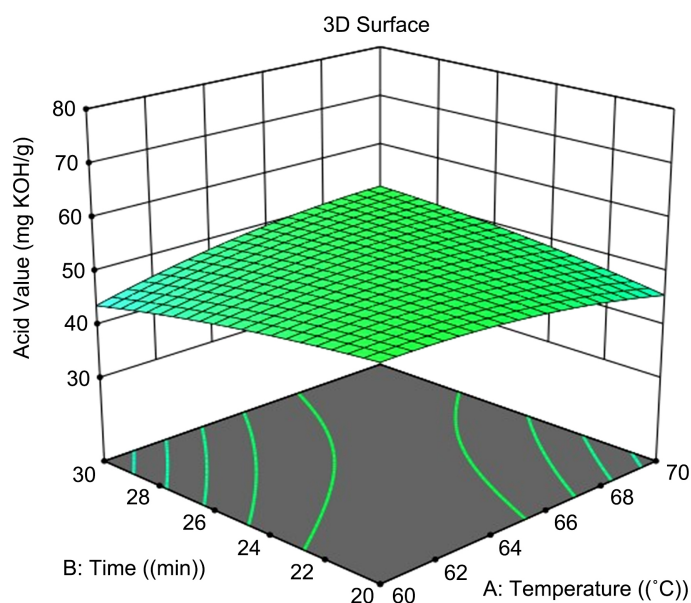


Figure 4. 3D response surface plot of acid value content.

3.3. Iodine Value Response

Iodine values ranged from 35.4 to 50.3 g I₂/100 g, notably higher than standard palm kernel oil specifications (14 - 21 g I₂/100 g) [38], possibly reflecting analytical methodology variations or sample-specific characteristics. The overall quadratic model shown in Table 7 for iodine value response was not statistically significant (F-value = 2.48, $p = 0.0864$), with Adjusted R² of only 0.4126 and a significant lack-of-fit ($p = 0.0117$). None of the linear factors showed statistical significance: time ($p = 0.7900$), temperature ($p = 0.4122$), or dosage ($p = 0.3003$). Among interactions, only time-dosage (AC) achieved significance ($p = 0.0398$, F-value = 5.58), though interpretation is challenging given weak overall model performance. The time-squared term (A²) was significant ($p = 0.0063$, F-value = 11.88), representing approximately 53% of total model variation, though the practical significance remains unclear. The poor model performance suggests that iodine value is relatively stable during mild alkali treatment, with observed variations primarily reflecting measurement variability rather than systematic compositional changes [39].

Table 7. Model fit statistics for iodine value response.

Source	Sequential p-Value	Adjusted R ²	Predicted R ²	Remark
Linear	0.7886	-0.1141	-0.5873	
2FI	0.2562	-0.0152	-0.8868	
Quadratic	0.0375	0.4126	-1.3151	Suggested
Cubic	0.2235	0.5729	-22.6839	Aliased

As shown in **Table 8**, the quadratic model was selected despite a negative predicted R^2 (-1.3151), based on its significant sequential p-value ($0.0375 < 0.05$) and superior adjusted R^2 (0.4126) among non-aliased models. The model demonstrated non-significance with F-value = 2.48 ($p = 0.0864$), confirming that the process variables did not substantially influence iodine value. All three main factors: time ($p = 0.0002$), temperature ($p = 0.0009$), and reagent dosage ($p < 0.0001$) showed highly non-significant effects, indicating that the deacidification process conditions can not affect the iodine value in the oil.

Reagent dosage exhibited the most pronounced effect ($F = 1.19$), followed by temperature ($F = 0.7323$) and time ($F = 0.0748$), suggesting that none of the three processing conditions has a significant effect on the outcome of iodine value of the oil during the deacidification process. The quadratic effect of time (A^2 , $p < 0.05$) was significant, indicating non-linear behavior at extreme contact time, possibly due to competing adsorption mechanisms.

Table 8. Analysis of variance (ANOVA) for iodine value response.

Source	Sum of Squares	df	Mean Square	F-Value	p-Value	Remark
Model	1723.68	9	191.52	2.48	0.0864	not significant
A-Time	5.77	1	5.77	0.0748	0.7900	
B-Temperature	56.49	1	56.49	0.7323	0.4122	
C-Dosage of reagent	92.05	1	92.05	1.19	0.3003	
AB	119.27	1	119.27	1.55	0.2420	
AC	430.27	1	430.27	5.58	0.0398	
BC	58.05	1	58.05	0.7526	0.4060	
A^2	916.67	1	916.67	11.88	0.0063	
B^2	20.74	1	20.74	0.2689	0.6154	
C^2	92.56	1	92.56	1.20	0.2990	
Residual	771.35	10	77.14			
Lack of Fit	702.43	5	140.49	10.19	0.0117	Significant
Pure Error	68.93	5	13.79			
Cor Total	2495.03	19				

Figure 5 shows the deviations obtained while using the model to predict the iodine value content based on the experimental variables. While interaction terms were not significant at the 0.05 level, AC ($p = 0.0398$) approached significance, suggesting potential synergistic effects between reagent dosage and contact time which may become more pronounced under extended contact time and dosage conditions. The interaction of the process variables (temperature and time) is shown on the iodine value response surface plot given in **Figure 6**. The non-signif-

icant lack of fit ($p = 0.0864$) indicates model limitations, possibly due to complex physicochemical interactions between kolanut pod components and iodine content of the oil that are not fully captured by the quadratic polynomial [40]. Despite this limitation, the $R^2 = 0.9307$ demonstrates strong overall model performance, confirming the model's utility for understanding the main effects and optimizing the process. However, the strong negative Predicted R^2 (-1.3151) and very significant “Lack of fit” values show poor fitness of the experimental data into the model.

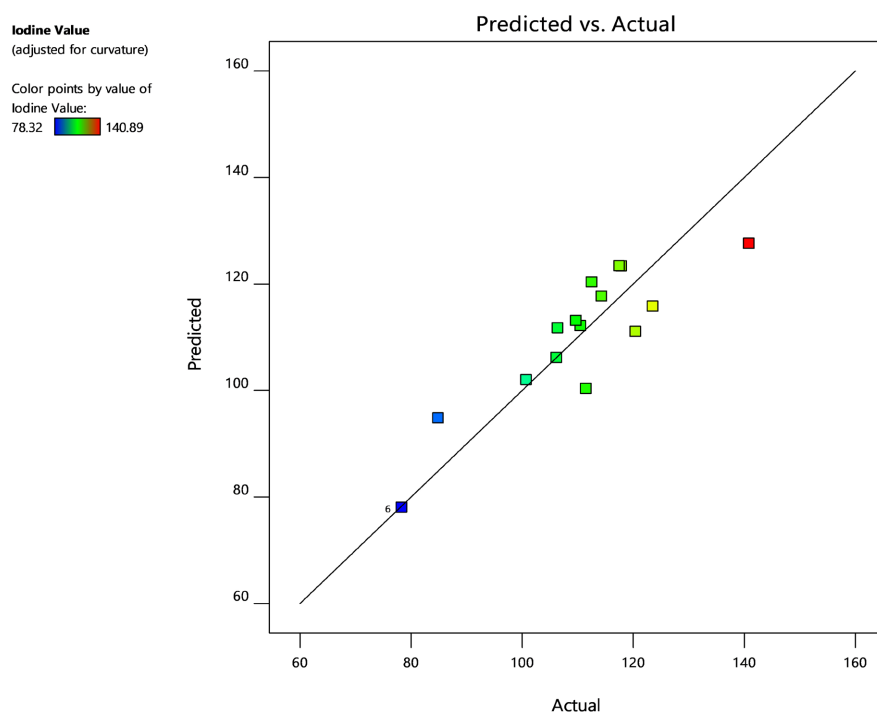


Figure 5. Graph of predicted value vs actual value of iodine value content.

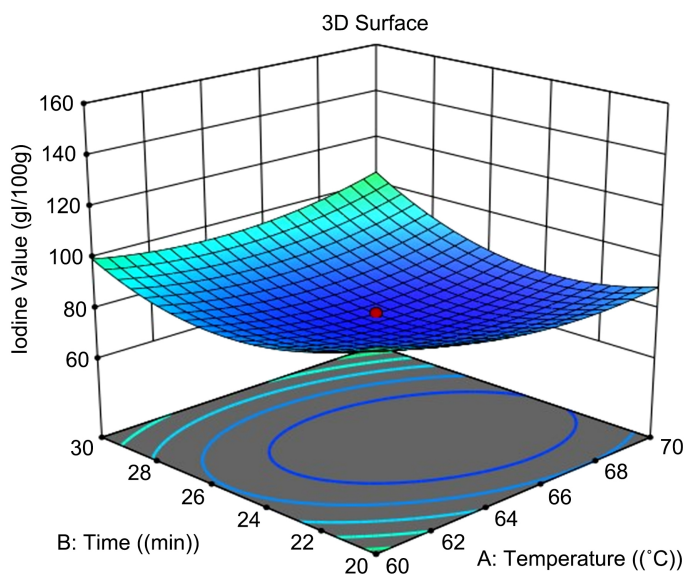


Figure 6. 3D response surface plot of iodine value content.

3.4. Saponification Value Response

Saponification values ranged from 250.67 to 290.12 mg KOH/g. The model fit statistics for the saponification value response are presented in **Table 9**. The quadratic model demonstrated exceptional performance (F-value = 12.80, $p < 0.05$) with Adjusted R^2 of 0.9002 and non-significant lack-of-fit ($p = 0.1072$), representing one of the best model fits among all responses studied. All linear terms emerged as statistically significant factors: contact time ($p = 0.0004$, F-value = 27.72), temperature ($p = 0.0019$, $F = 17.32$), dosage ($p < 0.0001$).

Table 9. Model fit statistics for saponification value response.

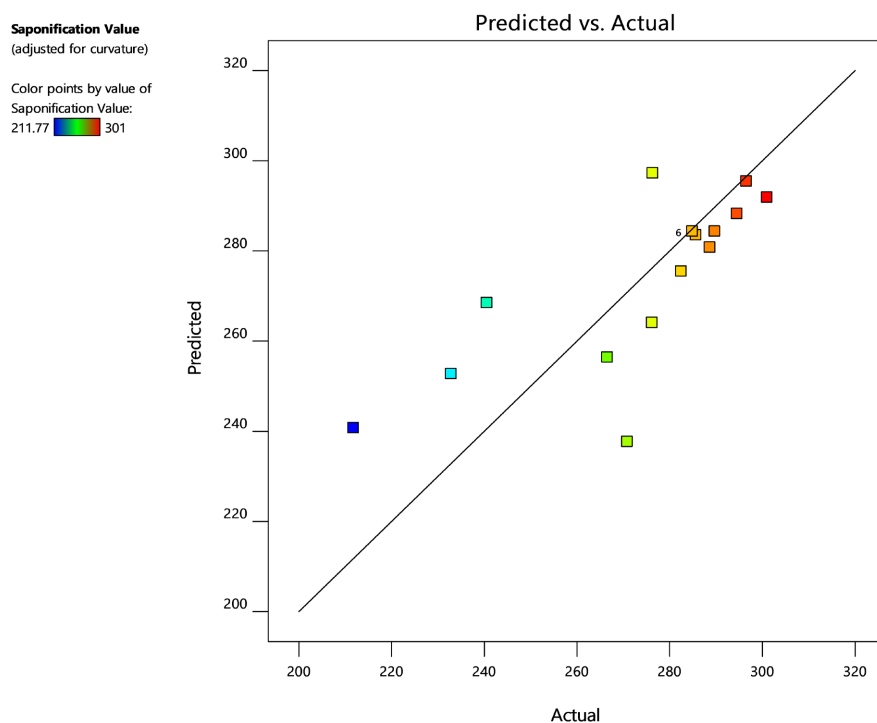
Source	Sequential p-Value	Lack of Fit p-Value	Adjusted R^2	Predicted R^2	
Linear	0.5685	0.0006	−0.0506	−0.4533	
2FI	0.1067	0.0010	0.1781	−0.2856	
Quadratic	<0.0001	0.1072	0.9002	0.6704	Suggested
Cubic	0.0601	0.4314	0.9558	0.5897	Aliased

The quadratic model was recommended based on its significantly high Adjusted R^2 (0.9002) and being among non-aliased models. The model showed high overall significance (F-value = 12.80, $p = 0.0002$), confirming that process conditions substantially affect saponification value. The relatively lower predicted R^2 (0.6704) compared to adjusted R^2 suggests some limitations in predictive capability for new observations, likely due to complex interactions between kolanut pod polyphenols and oil components that may vary with subtle changes in processing conditions [41]. The analysis of variance (ANOVA) is presented in **Table 10** for saponification value response. All three main factors demonstrated high significance: reagent dosage ($F = 49.41$, $p < 0.0001$) emerged as the dominant factor, followed by time ($F = 27.72$, $p = 0.0004$) and temperature ($F = 17.32$, $p = 0.0019$).

Figure 7 shows the deviations obtained while using the model to predict the saponification values based on the experimental variables. The interaction term AC ($p = 0.0266$) was significant, indicating synergistic effects between time and reagent dosage on saponification value. This interaction suggests that longer processing times enhance the effectiveness of higher reagent dosages, possibly through improved mass transfer and equilibrium attainment [42]. The interaction of the temperature and time is shown on the saponification value response surface plot given in **Figure 8**. The quadratic terms approached significance (A^2 , B^2 , C^2 with p-values between 0.0687 and 0.0838), suggesting moderate curvature in the response surface and the existence of optimal processing conditions. These findings indicate that while the kolanut pod deacidification process can influence oil molecular characteristics, the changes are controlled and predictable within the studied parameter ranges.

Table 10. Analysis of variance (ANOVA) for saponification value response.

Source	Sum of Squares	df	Mean Square	F-Value	p-Value	
Model	418.05	9	46.45	12.80	0.0002	Significant
A-Time	100.60	1	100.60	27.72	0.0004	
B-Temperature	62.87	1	62.87	17.32	0.0019	
C-Dosage of reagent	179.36	1	179.36	49.41	<0.0001	
AB	3.25	1	3.25	0.8957	0.3662	
AC	24.50	1	24.50	6.75	0.0266	
BC	12.50	1	12.50	3.44	0.0932	
A ²	15.10	1	15.10	4.16	0.0687	
B ²	13.38	1	13.38	3.69	0.0838	
C ²	13.43	1	13.43	3.70	0.0833	
Residual	36.30	10	3.63			
Lack of Fit	35.19	5	7.04	31.90	0.1072	not significant
Pure Error	1.10	5	0.2206			
Cor Total	454.34	19				

**Figure 7.** Graph of predicted value vs actual value of saponification value content.

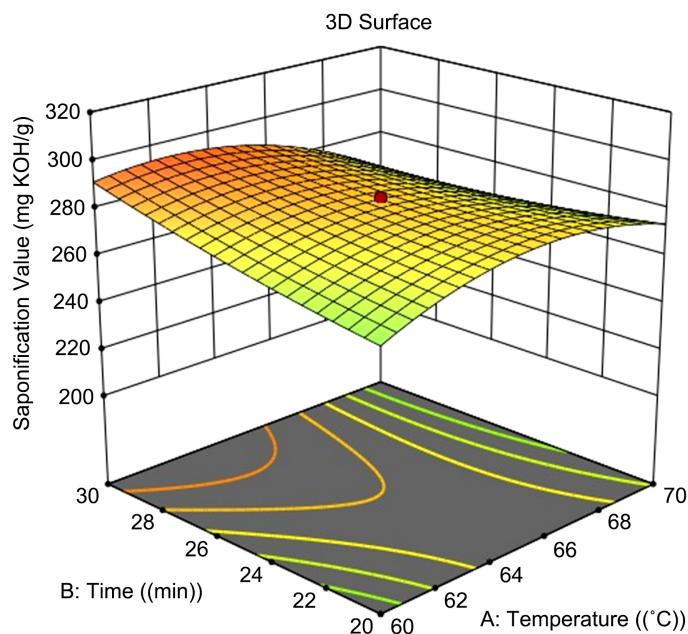


Figure 8. 3D response surface plot of saponification value content.

The significant lack of fit ($p = 0.0008$) suggests that additional factors or higher-order terms may be needed to fully describe the complex physicochemical transformations occurring during treatment, particularly interactions between kolanut pod bioactive compounds and oil constituents. Despite this limitation, the $R^2 = 0.9201$ indicates strong model performance and practical utility for process optimization.

3.5. Optimization and Validation of Free Fatty Acid Response

Figure 9 is the set of four plots representing the culmination of the optimization process using a desirability function. The objective was to minimize the FFA content. The plot provides the single best set of conditions identified by the model to achieve this goal. The “Desirability = 1.000” score is exceptionally high, indicating that the model has found an optimal solution that perfectly meets the defined criteria (minimal FFA). The individual bar charts clearly show the optimal values for each parameter, marked by a red dot. The optimum process condition is: 1.05% w reagent/w oil, 81.50°C and 22.16 minutes for dosage of reagent, temperature and time, respectively.

The validation experiment was conducted at the optimum condition (1.05% w reagent/w oil, 81.50°C and 22.16 minutes) for FFA and the FFA value obtained was 0.5133 (± 0.0058) mg KOH/g.

The final plot shows the predicted FFA value for this optimal set of conditions, which is approximately 0.52. These results are the most important output of this research work as they provide a clear and actionable set of instructions for achieving the most efficient deacidification of CPKO based on the experimental data and model.

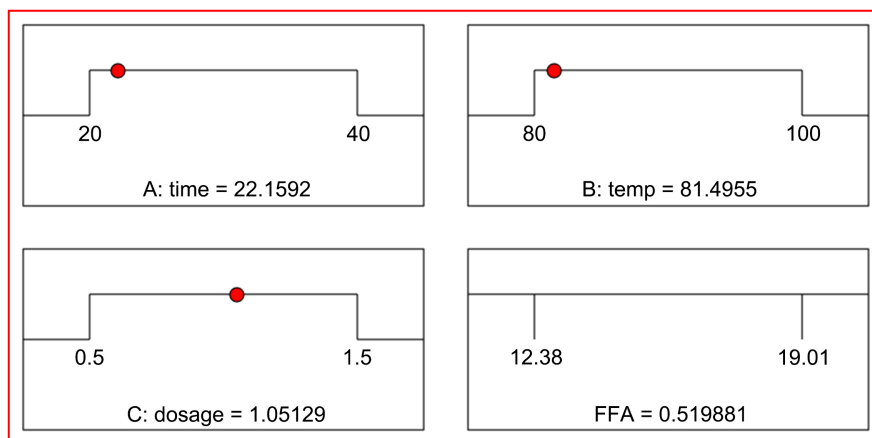


Figure 9. Numerical optimization solution based on desirability.

4. Conclusions

This study successfully demonstrated that kolanut pod ash can effectively deacidify degummed palm kernel oil, achieving FFA reduction from 3.5% to 0.31% (91% efficiency) under optimized conditions. Response Surface Methodology with Central Composite Design revealed that deacidification involves complex, interactive effects of reaction time, temperature, and adsorbent dosage, with distinct mechanistic pathways governing different quality parameters. Reaction time exhibited the strongest non-linear behavior through significant quadratic effects across multiple responses, particularly for acid value ($p = 0.0007$) and saponification value ($p < 0.0001$), indicating optimal processing windows of 20 - 28 minutes that balance neutralization completion against soap formation. Kolanut pod ash dosage dominated FFA reduction ($p = 0.0005$) through stoichiometric neutralization chemistry, with optimal concentrations of 0.8% - 1.5% w/w achieving maximum FFA removal while minimizing excessive saponification. Temperature significantly influenced phospholipid removal ($p = 0.0035$) and saponification characteristics ($p = 0.0030$), with optimal ranges of 65°C - 70°C providing enhanced mass transfer and reaction kinetics without thermal degradation. Significant synergistic interactions between time-temperature ($p = 0.0431$ for FFA) and time-dosage ($p = 0.0073$ for FFA; $p = 0.0060$ for acid value) confirmed that optimal conditions must be determined jointly through multivariate optimization rather than univariate approaches. Quality parameters remained within acceptable ranges for refined oils, with iodine value stability (poor model fit, $p = 0.0864$) indicating preservation of the characteristic palm kernel oil fatty acid profile during treatment.

The successful application of agricultural waste (kolanut pods) for oil deacidification addresses multiple sustainability objectives: waste valorization (converting 50,000 - 100,000 metric tons annual waste into valuable processing inputs), elimination of harsh chemicals and reduced wastewater generation (60% - 70% reduction), energy savings (30% - 40% compared to physical refining), and cost reduction (20% - 35% for small-scale processors). These benefits support circular

economy principles while providing technically viable solutions for resource-limited processors in developing countries. Future research should focus on pilot-scale validation (100 - 500 L capacity) to confirm laboratory findings under industrial conditions, investigation of regeneration potential for 2 - 3 cycle adsorbent reuse to further improve economics, mechanistic studies using advanced characterization (FTIR, SEM/TEM, BET analysis) to elucidate adsorption mechanisms and guide adsorbent optimization, and expansion to other vegetable oils to broaden the technology's commercial applicability. The development of standardized specifications for kolanut pod ash (particle size, activation parameters, quality metrics) will be essential for reliable industrial implementation and quality assurance.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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