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Enhancing Palm Kernel Oil Refining: Synergistic Effects of Hydrogen Peroxide and Bentonite Clay on Bleaching Efficiency

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ABSTRACT: Optimizing the refining process of palm kernel oil, particularly its bleaching phase, is crucial for removing free fatty acids and impurities. This study investigates the synergistic effects of hydrogen peroxide (H_2O_2) and bentonite clay on palm kernel oil bleaching using a two-stage refining process. Key findings indicate that pre-treating with H_2O_2 before clay application yields optimal results at 90 ℃ with a 2: 1.5 H_2O_2-to-bentonite ratio. Notably, H_2O_2 pre-treatment effectively removes free fatty acids, albeit with a slight increase in moisture content. Further optimization using Response Surface Methodology (RSM) with Box-Behnken Design revealed optimal reaction conditions of temperature: 98.7°C, H_2O_2 volume: 19.763 mL, bentonite clay amount: 9.868 g, and contact time: 22.681 mins. These predicted optimal values demonstrate significant potential for industrial application and future research, enhancing the efficiency and quality of palm kernel oil refining.

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The oil palm tree (*Elaeis guineensis*) produces seeds that are used to make palm kernel oil, which is a staple in the edible oil market and a flexible component of many consumer goods. The second most popular oil in the lauric acid category is palm kernel oil. It is made from the dried kernels of *Elaeis Guinensis*, the oil palm (Timms, 1986). Palm kernel oil makes up less than 5 % of all-natural oils and fats, but it is an essential feedstock for the oleochemical industry. The palm kernel oil industry is defined by the extraction of oil from palm kernel oil, and it plays an important role in supplying vegetable oil for a variety of applications ranging from cooking to industrial processing (Nde and Anuanwen, 2020). PKO is obtained from the flesh of oil palm kernel's fruits and has a high lauric acid concentration and a sharp melting point, making it suited for usage in confectionary fats (Dian *et al., 2017*). Palm kernel oil is a by-product of palm oil manufacturing, and its production is increasing with palm oil output (Atasie and Akinhanmi, 2009). Palm kernel oil is proven to be more unsaturated than other oils, therefore it can be hydrogenated to make a wider range of goods. Palm kernel oil can be used alone or in combination with other oils to create cake icing, biscuit dough, ice cream, filling creams, and imitation whipped cream.

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They can also be used to make sharp melting margarines, amongst other things. (Musa, 2010). Due to its many uses, which include food processing and cooking, the palm kernel oil (PKO) sector holds a dominant position in the worldwide edible oil market (Chew and Nyam, 2019). The refining procedures that are essential to improving PKO's marketability are coming under more and more scrutiny as consumer preferences for premium edible oils change over time.

The treatment of crude PKO involves several stages to get rid of impurities and pigments and to improve the general quality of the oil. The stages include Degumming, Neutralization, Bleaching and Deodorization. Even after the degumming and neutralization treatment, the oil still holds undesirable odours, impurities, and colour pigments that need to be eliminated before the finished product can be accepted by consumers. A bleaching procedure, can eliminate some of the lingering pollutants in a way that is qualitatively significant. Bleaching results in an improvement of the product's oxidation stability, as well as the first flavour, final aroma, and overall aroma. Additionally, it helps the processing that comes after it by absorbing trace levels of soaps, metal ions that promote oxidation, peroxides that are in the process of breaking down, and other trace contaminants. The oil that has been neutralized and bleached is referred to as NB-Oil. Hydrogen peroxide is used in the bleaching process to remove various oxidized compounds like peroxides, aldehydes, ketones, epoxies, hydro peroxides etc, from oils, including palm kernel oil (Choe and Min, 2006; Finnegan *et al., 2010*; Li *et al., 2018*; Wu *et al., 2019*). Activated clay and hydrogen peroxide (H_2O_2) combined are employed to maximize the benefits of both strategies. Pigments are treated with H_2O_2 via oxidation reaction, and contaminants are physically adsorbed by the clay. This two-step procedure improves the bleaching efficacy overall and potentially result in an improved purification of the PKO. When clay and H_2O_2 are combined, their synergistic effect is optimised since the activated clay required is less, hence minimising cost. Hence, the objective of this paper is to evaluate the synergistic effects of hydrogen peroxide (H_2O_2) and bentonite clay on palm kernel oil bleaching, aiming to improve refining efficiency.

MATERIALS AND METHODS

Material preparation: Palm kernel oil (PKO), bleaching clay (bentonite), and hydrogen peroxide were sourced locally in Benin City, Edo State. The crude PKO underwent water degumming following (Salawudeen *et al., 2014*), and neutralization using the acid-base method (Anakhu and Ibrahim, 2020). The neutralized oil was separated via centrifugation, with soapy water in the heavy phase and oil in the light phase. Deaeration reduced moisture to 0.1% prior to bleaching (Gibon *et al., 2007*). Bleaching experiments varied temperature, peroxide concentration, bentonite mass, and contact time. Bleaching agents were mixed with the oil under low pressure and stirred with a magnetic stirrer before filtration. The filtered oil was collected for characterization.

The oil's moisture content was determined and calculated for using the oven dry method (Olaoye and Adekanye, 2018). The oil's Free fatty acid (FFA) content was determined by the conventional titration method (Satyarthi *et al., 2009*) by titrating a sample of crude PKO against 0.1 M NaOH using 1% phenolphthalein solution as the indicator. The acid value and saponification (sap) value of the oil was determined following the existing method (Amin Paulin and Ahou Irène, 2019; Anakhu and Ibrahim, 2020; Sahar *et al., 2018*). The peroxide value was calculated by measuring the amount of iodine produced by the reaction of peroxides (formed in fat or oil) with iodide ions as previously established *(Cai et al., 2017*).

Determination of moisture content for PKO: Moisture content was determined for crude and refined PKO using the oven-dry method (Olaoye and Adekanye, 2018). A 10–15 g oil sample was dried at 120°C for two hours, and moisture content was calculated using Eq. **Error! Reference source not found.**:

$$\frac{(W_1 + W_2) - W_3}{W_2} \times 100 \quad (1)$$

Where, W_1, W_2, and W_3 represent dish weight, sample weight, and post-drying weight, respectively.
Note: Standard moisture content for crude and refined PKO is 0.5% and 0.1% respectively (Olaoye and Adekanye, 2018).
Determination of %FFA of crude and refined PKO: FFA content was determined via titration (Satyarthi *et al., 2009*). A 1–3 g sample was mixed with ethanol and phenolphthalein, then titrated with 0.1 M NaOH. FFA content was calculated using Eq. **Error! Reference source not found.**:

$$FFA\ (\%) = (CbVb \times 20) \div Msample \quad (2)$$

Where: C_b is base concentration, V_b ($V_2 - V_1$) is base volume, and M_{sample} is the mass of the sample.

The recommended % FFA for crude and refined PKO is 5% max and 0.1% max respectively (Anakhu and Ibrahim, 2020).

Determination of Acid Value of Oil: Acid value was determined using 1 g of oil dissolved in benzene and ethanol, with phenolphthalein as an indicator, titrated against 0.1 M NaOH (Anakhu and Ibrahim, 2020). Acid value was calculated with Eq. **Error! Reference source not found.**.

$$\text{Acid value} = \frac{40 \times \text{N} \times \text{V}}{\text{W}_{\text{oil}}} \quad (3)$$

Where N represents the normality of the NaOH solution, V represents the volume of NaOH solution (in mL) used in the titration, and W_{oil} represents the weight of the oil sample (in grams).

Determination of Saponification Value: The saponification value was determined using alcoholic NaOH and titration against 0.5 M HCl (Olaniyi *et al., 2014*). It was calculated using Eq. **Error! Reference source not found.**.

$$\text{Saponification value} = \frac{28.05 \times (\text{V}_\text{B} - \text{V}_\text{s})}{\text{W}_\text{s}} \quad (4)$$

Where V_B, V_S and W_S are blank volume, sample volume, and sample weight, respectively.

Determination of the Peroxide Value: To determine the peroxide value, 10–12 g of oil is weighed into a conical flask. Then, 30 mL of Acetic Acid-Chloroform mixture is added and shaken. After adding 1 mL of saturated potassium iodide solution and shaking for 1 minute, 30 mL of distilled water is added and shaken again. The initial burette reading of 0.01 N sodium thiosulfate is recorded, and 0.5 mL of 1% starch solution is added as an indicator. The mixture is titrated until the color disappears, and the peroxide value is calculated using Eq. **Error! Reference source not found.**.

$$\text{Peroxide value} = \frac{\text{V} \times \text{N} \times 1000}{\text{W}_\text{S}} \quad (5)$$

Where, W_S = Sample weight, N = Normality of $Na_2S_2O_3$, V = Volume of $Na_2S_2O_3$ (Final burette reading – Initial burette reading)

Process variables (temperature, contact time and clay dosage): Bleaching temperatures ranged from 90–120°C, affecting adsorption kinetics and oil viscosity (Bot and Flöter, 2013). Contact times varied between 15–45 minutes, with 30 minutes being optimal. Bleaching performance and oil color were influenced by these variables, as noted in prior studies (Gibon, 2012). The amount of bentonite used ranged from 5–10 g. In some cases, the bentonite was used alone while in other cases it was dosed first into the oil before peroxide was added and then in other cases, it was added to the mixture of peroxide and oil. The volume of peroxide concentration varied from 10–20 mL with 15 mL being more prominent.

Statistical Tools Used: In this study, several statistical tools were employed to optimize the bleaching conditions for palm kernel oil (PKO). Response Surface Methodology (RSM) was applied, utilizing Box-Behnken Design (BBD) to assess the effects of temperature, H_2O_2 amount, clay amount, and contact time on response variables such as FFA, moisture content, acid value, saponification value, and peroxide value. Multiple regression analysis was used to establish the relationships between process parameters and responses. Analysis of Variance (ANOVA) was performed to evaluate the statistical significance of the regression models, with a lack of fit test to verify model adequacy. Design Expert 13 software was utilized for experimental design, data analysis, and optimization.

RESULTS AND DISCUSSION

To ascertain the ideal bleaching conditions for PKO, certain variables for variation based on existing literature were selected. Through a thorough review of prior research, I carefully selected parameters poised to exert significant influence on the bleaching process.

This approach ensured a comprehensive exploration of potential factors affecting the optimization of PKO bleaching, setting the stage for experimentation and analysis. An experiment was designed using Box-Behnken Design (BBD) varying the clay dosage, hydrogen peroxide volume, contact time and temperature. The values for the experimental data are shown in The table above, which includes 29 distinct runs, established the pace for our lab experiment. Each variable and its corresponding values were utilized in conducting the test to obtain the results reported in

Acid *Value:* The term "acid value" in fats and oils refers to the measurement of the amount of free fatty acids present in a substance, usually fat or oil. It is stated as the volume of sodium hydroxide (NaOH) needed to offset one gram of the material's free fatty acid content (Aladetuyi *et al., 2014*). A reduction of the acid value content of the other bleached oils when compared to that of the crude PKO clearly shows that the bleaching action was successful in reducing the

unsaturated free fatty acid content. The free fatty acids and organic molecules in the crude oil interacted with the hydrogen peroxide that was introduced in the bleaching process causing the oxidation reaction that broke down the contaminants into less acidic chemicals, lowering the oil's overall acidity. Also, a mix of hydrogen peroxide with bentonite clay in the bleaching process aided the adsorbing of colour pigments, metals, and other impurities from the oil which explains the reduction in the oil's acidity as acidic components bound to these impurities were also removed. Runs 3, 19, and 20 have an acid value that is lower than crude's, but it is still rather high since there is more hydrogen peroxide present than there is clay, and the higher temperatures involved may have had an impact on the creation of peroxides.

Table 2.

Free fatty acid (FFA) content: Given that crude palm kernel oil has the highest free fatty acid content, as shown in the % FFA column, the data displayed in the table above indicates that the refining process assisted in lowering the amount of free fatty acids in the oil.

This is evident by the obvious difference in the FFA content. Oil refinement procedures such as degumming assisted in removing the hydratable gums that contributed to some of the contaminants in the oil, and neutralization assisted in neutralizing the acids that were responsible for the oil's high free fatty acid content using a base which is NaOH thereby increasing the edibility of the oil. Run 29 had the lowest FFA level, demonstrating that if you plan to dose the oil with clay beforehand to remove free fatty acids, the ratio of oil to clay should be roughly 3:1 and with a temperature above 120 ℃.

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Table 1: Runs from Response Surface Optimization Techniques

Run	Hydrogen peroxide (mL)	Clay amount (g)	Temperature (℃)	Contact Time (mins)
1	15	5.0	107.5	45
2	15	10.0	125.0	30
3	15	10.0	107.5	45
4	15	10.0	90.0	30
5	15	7.5	107.5	30
6	15	7.5	90.0	45
7	15	7.5	107.5	30
8	15	7.5	107.5	30
9	10	7.5	90.0	30
10	15	7.5	125.0	15
11	10	7.5	107.5	45
12	10	10.0	107.5	30
13	20	10.0	107.5	30
14	20	7.5	90	30
15	15	10.0	107.5	15
16	15	7.5	107.5	30
17	20	5.0	107.5	30
18	20	7.5	107.5	45

19	10	7.5	107.5	15
20	15	5.0	125.0	30
21	20	7.5	107.5	15
22	15	5.0	90.0	30
23	15	7.5	107.5	30
24	15	5.0	107.5	15
25	15	7.5	125.0	45
26	10	5.0	107.5	30
27	10	7.5	125.0	30
28	15	7.5	90.0	15
29	20	7.5	125.0	30

Acid Value: The term "acid value" in fats and oils refers to the measurement of the amount of free fatty acids present in a substance, usually fat or oil. It is stated as the volume of sodium hydroxide (NaOH) needed to offset one gram of the material's free fatty acid content (Aladetuyi *et al., 2014*). A reduction of the acid value content of the other bleached oils when compared to that of the crude PKO clearly shows that the bleaching action was successful in reducing the unsaturated free fatty acid content. The free fatty acids and organic molecules in the crude oil interacted with the hydrogen peroxide that was introduced in the bleaching process causing the oxidation reaction that broke down the contaminants into less acidic chemicals, lowering the oil's overall acidity. Also, a mix of hydrogen peroxide with bentonite clay in the bleaching process aided the adsorbing of colour pigments, metals, and other impurities from the oil which explains the reduction in the oil's acidity as acidic components bound to these impurities were also removed. Runs 3, 19, and 20 have an acid value that is lower than crude's, but it is still rather high since there is more hydrogen peroxide present than there is clay, and the higher temperatures involved may have had an impact on the creation of peroxides.

Table 2: Results of physicochemical properties of crude and refined PKO when clay is dosed with bentonite first using hydrogen peroxide

	% FFA	Moisture Content	Acid Value	Sap Value	Peroxide Value
Crude PKO	5.60	1.70	13.60	243.84	10.25
Only Bentonite Clay	4.30	1.20	8.85	215.22	8.12
1	4.08	2.40	9.40	195.6	15.17
2	3.90	2.80	8.40	205.26	15.68
3	5.40	2.00	13.20	173.82	14.13
4	4.86	3.40	11.60	126.3	16.24
5	4.66	2.40	9.00	166.62	15.15
6	4.20	2.07	9.25	182.46	15.13
7	4.00	2.64	9.06	162.00	15.17
8	4.03	2.35	9.07	156.00	15.14
9	4.22	1.00	5.20	175.68	12.86
10	4.16	0.80	9.20	191.82	15.24
11	4.26	3.18	9.10	194.76	13.52
12	3.40	3.50	8.40	196.14	13.06
13	3.24	3.00	8.35	200.40	18.92
14	3.00	2.04	8.32	129.24	17.65
15	4.18	2.72	10.20	194.46	16.24
16	4.05	2.64	9.03	156.00	15.73
17	3.35	2.25	8.38	176.46	19.34
18	3.28	2.91	8.34	183.18	18.24
19	4.25	2.33	10.74	188.40	14.21
20	4.17	0.60	10.70	180.72	15.23
21	3.46	2.51	8.60	169.26	16.14
22	3.96	1.24	8.94	164.40	14.96
23	4.03	2.48	9.05	156.00	15.10
24	3.93	2.36	8.87	174.96	15.35
25	4.14	0.44	10.35	200.46	14.28
26	4.26	2.40	9.28	151.44	13.73
27	4.10	0.64	9.09	144.72	12.67
28	4.00	1.26	9.10	156.72	15.10
29	3.20	0.65	8.20	150.84	16.54

Moisture content: The addition of hydrogen peroxide to the bleaching process increased the moisture content of the oil because hydrogen peroxide readily breaks down into water and oxygen when it encounters traces of metals present in oils. This decomposition reaction releases water molecules into the system, contributing to an increase in moisture content, which explains the increase in moisture content of the runs in relation to the crude and when bleaching was done with just bentonite.

Saponification (sap) value: A higher saponification value means a higher level of unsaturation of the oil (Amin Paulin and Ahou Irène, 2019; Olaniyi *et al., 2014*). A low saponification value very likely indicates a higher degree of purity and stability, which is advantageous for edible oils. Hydrogen peroxide is an oxidizing agent that reacts with the unsaturated fatty acids that are present in the oil, affecting the composition of fatty acids in the oil and altering the saponification value. The concentration of hydrogen peroxide, reaction time, temperature, and other process parameters during the bleaching process influenced the extent of oxidative changes and hydrolysis reactions which explains the reduction in saponification value of the oil after bleaching. The saponification values obtained tallies with the claims above as well. Since saponification value measures the amount of base (in this case NaOH) that would be required to turn the oil containing fatty acids into soap; a higher saponification value would mean a higher level of unsaturation of the palm kernel oil and that means more amount of unsaturated fatty acids in the oil. It can thus be observed from the reported values that the saponification value of the oil is lesser when the peroxide and bentonite clay were used to bleach it compared to the saponification of the clay or to the case where just bentonite was used.

Peroxide value: The standard peroxide value for crude PKO lies between 1 – 10 % and it indicates the extent of primary oxidation in the oil, reflecting its freshness and initial stage of rancidity (Olaniyi *et al., 2014*). High peroxide values suggest that the oil has

undergone some oxidative changes due to exposure to oxygen, light and heat, indicating potential quality deterioration. However, controlled oxidation which was duly monitored was involved in this process which was beneficial for the removal of certain impurities and in avoiding excessive oxidative degradation which explains the values gotten after the peroxide value test.

Optimization *using response surface methodology:* It has been established that response surface methodology (RSM) led to the examination of the impact of numerous parameters on response variables in lipid or enzyme processes (Wang and Lu, 2005). **Table 5.**

Table 3 consist of significant runs from The table above, which includes 29 distinct runs, established the pace for our lab experiment. Each variable and its corresponding values were utilized in conducting the test to obtain the results reported in

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Optimization using response surface methodology (Box Behnken Design) BBD was used with four independent variables (process factors) based on coded levels in

Table **4** to find out optimum bleaching conditions and the results is shown

Independent Variable	**Symbol**	**Coded Levels**		
		-1	0	+1
Temperature (℃)	A	90	107.5	125
H_2O_2 amount (mL)	B	10	15	20
Clay amount (g)	C	5	7.5	10
Contact time (min)	D	15	30	45

This is evident by the obvious difference in the FFA content. Oil refinement procedures such as degumming assisted in removing the hydratable gums that contributed to some of the contaminants in the oil, and neutralization assisted in neutralizing the acids that were responsible for the oil's high free fatty acid content using a base which is NaOH thereby increasing the edibility of the oil. Run 29 had the lowest FFA level, demonstrating that if you plan to dose the oil with clay beforehand to remove free fatty acids, the ratio of oil to clay should be roughly 3:1 and with a temperature above 120 ℃.

Table 1 which were selected and used by dosing the oil with the hydrogen peroxide before adding the clay. In this setup, the %FFA is observed to be way less than that observed in The table above, which includes 29 distinct runs, established the pace for our lab experiment. Each variable and its corresponding values were utilized in conducting the test to obtain the results reported in

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20 have an acid value that is lower than crude's, but it is still rather high since there is more hydrogen peroxide present than there is clay, and the higher temperatures involved may have had an impact on the creation of peroxides.

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Table 1. This indicates that lesser amount of free fatty acid is present with this procedure and this can be explained in the sense that the oxidizing effect of the hydrogen peroxide which is in more direct contact with the oil, oxidizes a good amount of the free fatty acids, enhancing the bleaching procedure and the effect of the bentonite clay to be later added.

On the other hand, the moisture and peroxide contents of
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Table 1 is seen to be much more. Since less peroxide is absorbed by the clay this time, there is a higher moisture content and a higher peroxide concentration. To prolong the oil's shelf life and prevent it from going bad too soon, more time and effort would need to be put into heating the oil to evaporate any extra moisture and peroxide. The optimization study's conclusions suggest that treating the oil with peroxide first before adding bentonite increases the effectiveness of hydrogen peroxide supplementation throughout the bleaching process. In addition, to maintain adherence to edibleness requirements, the refining process should include a drying time longer than two hours to enable any residual moisture in the oil to evaporate after the bleaching stage. It has been established that dosing the oil with peroxide first and then adding bentonite would give better results as opposed to blending the peroxide with bentonite and then adding to the oil, therefore our optimal conditions will be obtained from

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Table 2. Taking the tests carried out into consideration, it can be observed that run 9 has the lowest % FFA and acid value which indicates that the bleaching process, which involves the adsorption of impurities by bentonite and oxidative degradation by hydrogen peroxide, successfully eliminated free fatty acids, leading to a cleaner and more stable oil. This is desirable as it enhances the oil's shelf life, stability, as well as its suitability for diverse applications, including food production and industrial uses. The moisture content, saponification value and peroxide value of Run 9 were also within allowable range.

Also, run 5 had the lowest moisture content which is understandable because it was bleached at a higher temperature and even though it had more volume of peroxide introduced, its temperature being higher than that of Run 9 gave it an added advantage. Run 29, although having values within the desirable range, still had its moisture content high due to the volume of peroxide that was introduced during the bleaching stage. From observation, run 5,9 and 29 had the best qualities after refining with run 5 taking the lead. It was observed that the 3 runs operated with the same amount of time and grams of bentonite. Also, comparing the 3 runs, the temperature at which the oil was bleached increased with an increase in volume of peroxide. The optimal temperature and time were observed from The table above, which includes 29 distinct runs, established the pace for our lab experiment. Each variable and its corresponding values were utilized in conducting the test to obtain the results reported in

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Table 5.

Table 3: Results of physicochemical properties of crude and refined PKO when oil is dosed with hydrogen peroxide first before addition of clay

	% FFA	Moisture Content	Acid Value	Saponification Value	Peroxide Value
Crude PKO	1.85	4.64	6.80	124.60	20.25
5	1.08	3.04	5.56	108.56	17.14
9	1.01	3.80	4.80	106.85	20.17
10	1.19	3.16	5.60	110.80	18.96
14	1.10	5.20	6.20	115.22	21.14
29	1.13	4.50	5.40	106.94	18.17

The Temperature, the amount of hydrogen peroxide used, clay amount, and contact time were checked and investigated in the ranges of 90 – 125 ℃, 10 – 20 mL, 5 – 10 (g), and contact time (min) respectively. Responses including the % FFA, acid value, saponification (sap) value, moisture content and peroxide value are obtained. Bleaching experimental factors and their coded levels blend is given in

interacted with the hydrogen peroxide that was introduced in the bleaching process causing the oxidation reaction that broke down the contaminants into less acidic chemicals, lowering the oil's overall acidity. Also, a mix of hydrogen peroxide with bentonite clay in the bleaching process aided the adsorbing of colour pigments, metals, and other impurities from the oil which explains the reduction in the oil's acidity as acidic components bound to these impurities were also removed. Runs 3, 19, and 20 have an acid value that is lower than crude's, but it is still rather high since there is more hydrogen peroxide present than there is clay, and the higher temperatures involved may have had an impact on the creation of peroxides.

Table 2 to be 90 and 30 mins with ratio of hydrogen peroxide to bentonite being 2:1.5.

Optimization using response surface methodology: It has been established that response surface methodology (RSM) led to the examination of the impact of numerous parameters on response variables in lipid or enzyme processes (Wang and Lu, 2005). Optimization using response surface methodology (Box Behnken Design) BBD was used with four independent variables (process factors) based on coded levels in

Table **4** to find out optimum bleaching conditions and the results is shown

Independent Variable	Symbol	Coded Levels		
		-1	0	+1
Temperature (℃)	A	90	107.5	125
H_2O_2 amount (mL)	B	10	15	20
Clay amount (g)	C	5	7.5	10
Contact time (min)	D	15	30	45

Table **4** with the average being 107.5 ℃, 15 mL, 7.5 g and 30 mins for temperature, H_2O_2, clay amount and contact time, respectively. The quadratic model equations that correlates the response to the independent variables in terms of coded factors are given in **Error! Reference source not found.-Error! Reference source not found.** (where A is the temperature, B represents amount of H_2O_2, C is the amount of clay and D is the contact time). Design expert 13 software was used to design bleaching performance and experiment to optimize conditions.

Table 4: Independent variables and their corresponding levels for bleaching of PKO

Independent Variable	Symbol	Coded Levels		
		-1	0	+1
Temperature (℃)	A	90	107.5	125
H_2O_2 amount (mL)	B	10	15	20
Clay amount (g)	C	5	7.5	10

Contact time (min)	D	15	30	45

Table 5: Independent variables versus Response values

Independent Variables					Response Values				
Run	Hydrogen peroxide amount (ml)	Clay amount (g)	Temp. (°C)	Contact Time (min)	% FFA	Moisture Content (%)	Acid Value (mg NaOH/g)	Sap Value (meq NaOH/mg)	Peroxide Value (meq)
1	15	5.0	107.5	45	4.08	2.40	9.40	195.6	15.17
2	15	10.0	125.0	30	3.90	2.80	8.40	205.26	15.68
3	15	10.0	107.5	45	5.40	2.00	13.20	173.82	14.13
4	15	10.0	90.0	30	4.86	3.40	11.60	126.3	16.24
5	15	7.5	107.5	30	4.66	2.40	9.00	166.62	15.15
6	15	7.5	90.0	45	4.20	2.07	9.25	182.46	15.13
7	15	7.5	107.5	30	4.00	2.64	9.06	162.00	15.17
8	15	7.5	107.5	30	4.03	2.35	9.07	156.00	15.14
9	10	7.5	90.0	30	4.22	1.00	5.20	175.68	12.86
10	15	7.5	125.0	15	4.16	0.80	9.20	191.82	15.24
11	10	7.5	107.5	45	4.26	3.18	9.10	194.76	13.52
12	10	10.0	107.5	30	3.40	3.50	8.40	196.14	13.06
13	20	10.0	107.5	30	3.24	3.00	8.35	200.40	18.92
14	20	7.5	90	30	3.00	2.04	8.32	129.24	17.65
15	15	10.0	107.5	15	4.18	2.72	10.20	194.46	16.24
16	15	7.5	107.5	30	4.05	2.64	9.03	156.00	15.73
17	20	5.0	107.5	30	3.35	2.25	8.38	176.46	19.34
18	20	7.5	107.5	45	3.28	2.91	8.34	183.18	18.24
19	10	7.5	107.5	15	4.25	2.33	10.74	188.40	14.21
20	15	5.0	125.0	30	4.17	0.60	10.70	180.72	15.23
21	20	7.5	107.5	15	3.46	2.51	8.60	169.26	16.14
22	15	5.0	90.0	30	3.96	1.24	8.94	164.40	14.96
23	15	7.5	107.5	30	4.03	2.48	9.05	156.00	15.10
24	15	5.0	107.5	15	3.93	2.36	8.87	174.96	15.35
25	15	7.5	125.0	45	4.14	0.44	10.35	200.46	14.28
26	10	5.0	107.5	30	4.26	2.40	9.28	151.44	13.73
27	10	7.5	125.0	30	4.10	0.64	9.09	144.72	12.67
28	15	7.5	90.0	15	4.00	1.26	9.10	156.72	15.10
29	20	7.5	125.0	30	3.20	0.65	8.20	150.84	16.54

Model Fitting and Response Surface Analysis: Experimental values obtained were fitted to the second-order polynomial models (Eq. **Error! Reference source not found.**–**Error! Reference source not found.**), and multiple regression coefficients were obtained for all answers using a statistical method. The optimization used a quadratic design model. A multiple regression approach with the lowest residual achievable was developed. The regression coefficients of the model for the different response are presented in **Error! Reference source not found.**. According to particularly high value of coefficient of multiple determinations, R^2 for the five different responses obtained are reported in the last row of **Error! Reference source not found.**. The model expressed in Eq. **Error! Reference source not found.**–**Error! Reference source not found.**, provides good model and representation of the experimental values. Moreover, for the response, the mathematical model was statistically acceptable due to significant regression for the model ($p < 0.05$ for FFA, moisture content and peroxide value; The p values for acid values and saponification values were however greater than 0.05). Lack of fit testing confirmed how adequate it was in fitting experimental data to a second-order polynomial model; p values for lack of fit was insignificant for the FFA test ($p > 0.05$). Therefore, as suggested by ANOVA it can be inferred that Eq. **Error! Reference source not found.**–**Error! Reference source not found.** can adequately describe the behaviour of PKO bleaching process.

Table 6: Regression coefficient values for PKO bleaching

Regression Coefficient	FFA (%)	Moisture Content (%)	Acid Value (mg NaOH g^{-1})	Sap Value (meq NaOH mg^{-1})	Peroxide Value (meq)
Intercept	4.15	2.50	9.04	159.32	15.38
A-Temperature	-0.0475	-0.4233*	0.2942	11.58*	-0.1917
B-H_2O_2 concentration	-0.4133**	0.0258	-0.1350	-3.48	2.23***
C-Clay amount	0.1025	0.5142*	0.3817	4.40	0.0408
D-Contact time	0.1150	0.0850	0.2442	4.55	-0.1508
AB	0.0800	-0.2575	-1.00	13.14	
AC	-0.2925	0.0100	-1.24*	15.66	
AD	-0.0550	-0.2925	0.2500	-4.27	
BC	0.1875	0.0875	0.2125	-5.19	
BD	-0.0475	-0.1125	0.3450	1.89	
CD	0.2675	-0.1900	0.6175	-10.32	

A^2	-0.0466	-1.15***	-0.1943	-3.90	
B^2	-0.5328**	0.0294	-0.9881*	2.59	
C^2	0.0559	0.3119	0.7269	12.52	
D^2	0.1322	-0.1493	0.8057**	20.76	
R^2	0.7437	0.7674	0.6663	0.6672	0.8045

$$\text{FFA}(\%) = -1.95739 + 0.0727034A + 0.364948B + 0.186514C - 0.049054D + 0.000914286AB - 0.00668571AC - 0.000209524AD + 0.015BC - 0.000633333BD + 0.00713333CD - 0.000152109A^2 - 0.0213133B^2 + 0.00894667C^2 + 0.000587407D^2 \quad (1)$$

$$\text{Moisture content} = -48.5284 + 0.862056A + 0.383724B - 0.310505C + 0.225775D - 0.00294286AB + 0.000228571AC - 0.00111429AD - 0.007BC - 0.0015BD - 0.00506667CD - 0.00376925A^2 + 0.00117667B^2 + 0.0499067C^2 - 0.000663704D^2 \quad (2)$$

$$\text{Acid value} = -31.0214 + 0.509097A + 2.12484B + 0.705924C - 0.493448D - 0.0114571AB - 0.0283429AC + 0.00095238AD + 0.017BC + 0.0046BD + 0.0164667CD - 0.000634558A - 0.0395233B^2 + 0.116307C^2 + 0.00358074D^2 \quad (3)$$

$$\text{Sap value} = 529.219 - 1.05072A - 17.59B - 52.2841C - 1.79642D + 0.150171AB + 0.357943AC - 0.0162857AD - 0.4152BC + 0.0252BD - 0.2752CD - 0.0127249A^2 + 0.10362B^2 + 0.10362B^2 + 2.00328C^2 + 0.09228D^2 \quad (4)$$

$$\text{Peroxide value} = 10.0381 - 0.0109524A + 0.446333B + 0.0163333C - 0.0100556D \quad (5)$$

Effect of process variables on the bleaching process: In this study, three-dimensional (3D) response surface plots were created using the 29 experimental data and established model to show the interacting effects of independent factors on the response. Fig. 1 shows the relation of temperature and H_2O_2 concentration to the amount of FFA for the bleaching process of PKO.

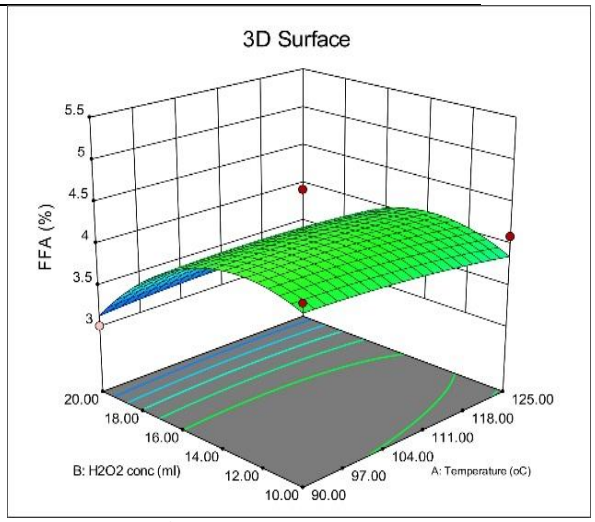

Fig. 1: 3D plot of temperature and H_2O_2 amount in relation to % FFA of PKO

The plot shows that the variation in temperature did not significantly affect the FFA content owing to the non-sloppiness of the temperature axis from 90 ℃ to 125 ℃. However, a reduced FFA content is what will be a more desirable outcome (Anakhu and Ibrahim, 2020; Salawudeen *et al., 2014*); hence we can deduce that the change in temperature from 90 ℃ to 125 ℃ had little or no effect on the change in the FFA content of the PKO. The FFA content initially increased as H_2O_2 concentration rose from 12-14 mL, but beyond 14 mL, FFA levels steadily decreased, reaching a minimum of 3.1% at 20 mL H_2O_2. Fig. 2 illustrates the impact of clay amount and contact time on FFA content. The results show that FFA increases with longer contact times, rising from 3.8% to 4.75% as time increases from 15 to 45 mins. Conversely, higher clay amounts (above 5 g) lead to a slight decrease in FFA. Fig. 3 reveals a strong correlation between predicted and actual values, with points clustering closely around the line, indicating high accuracy.

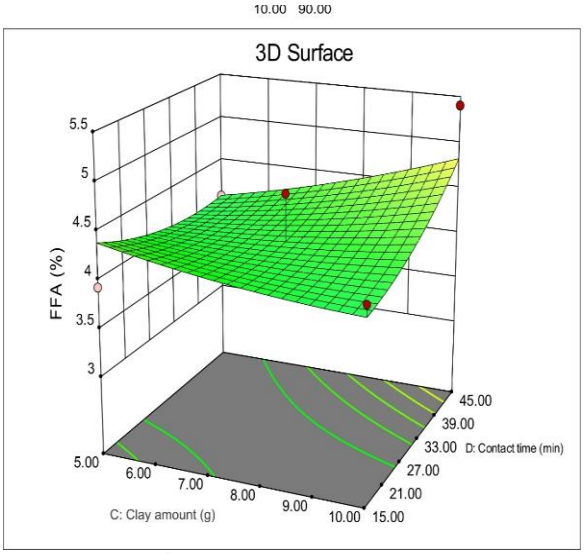

Fig. 2: 3D plot of clay amount and contact time in relation to % FFA of PKO

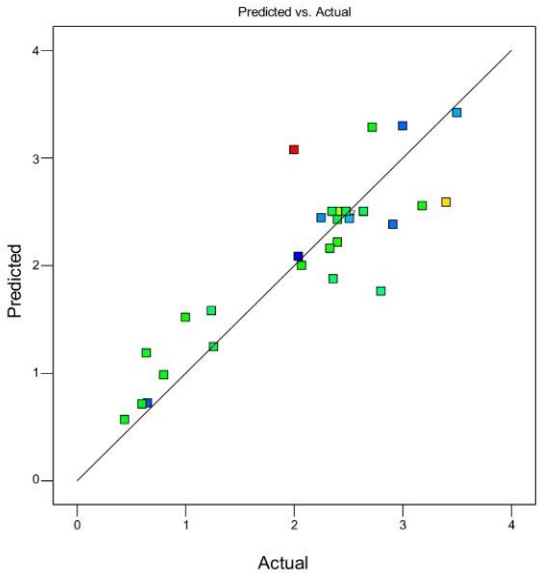

Fig. 3: 3D plot of predicted vs actual value for FFA

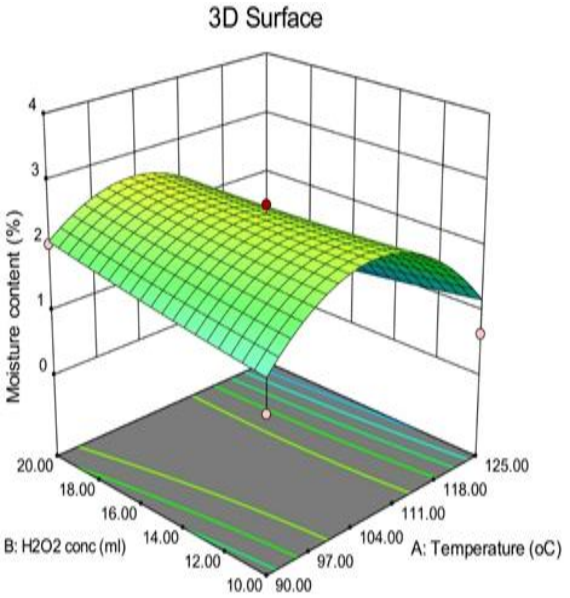

Fig. **4** illustrates the combined effects of temperature and H_2O_2 concentration on the moisture content of PKO. Notably, moisture content increased linearly with H_2O_2 concentration rising from 1 % at 10 mL to 2 % at 20 mL. Temperature also played a significant role, with moisture content peaking at 2.5 % at 107.5 ℃.

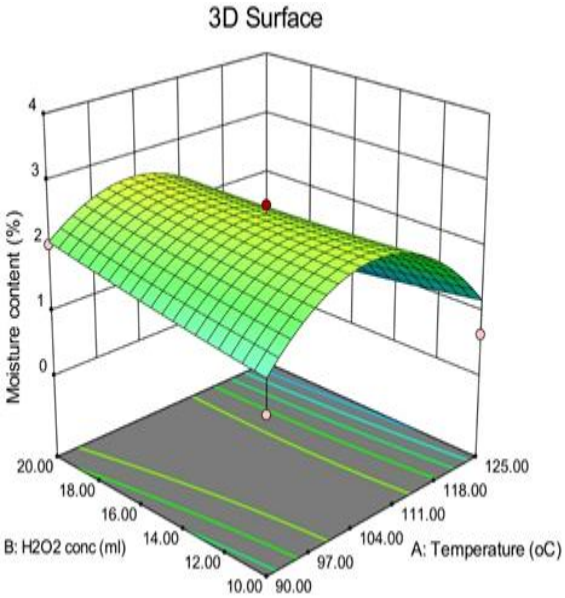

Fig. 4: 3D plot of temperature and H_2O_2 amount in relation to moisture content of PKO

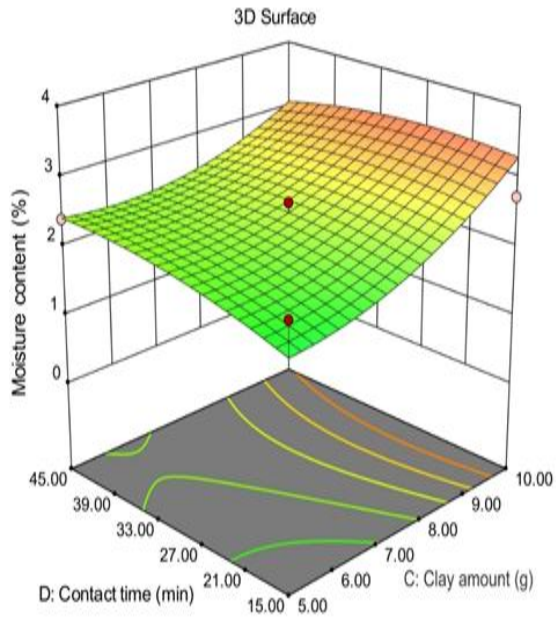

Fig. 5: 3D plot of clay amount and contact time in relation to moisture content of PKO

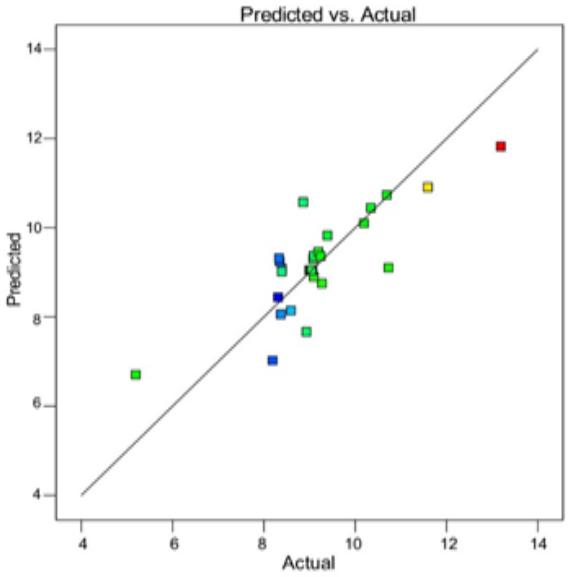

Fig. 6: 3D plot of predicted vs actual value for moisture content

This optimal temperature marked a turning point, as further increases in temperature (up to 125 ℃) led to a decline in moisture content, dropping to 1 %. This optimal temperature threshold is crucial as excessive moisture can accelerate oil rancidity (Anakhu and Ibrahim, 2020). Consequently, minimizing moisture content is desirable to ensure oil stability. Fig. 5 illustrates the impact of contact time and clay amount on PKO's moisture content during bleaching. The plot reveals that moisture content increases significantly with moisture content, rising from 2 % to 3.2 % as clay concentration increases from 5 to 10 g. Additionally, moisture content exhibits a moderate increase from 2 % to 2.2 % as contact time increases from 15 to 45 mins. This phenomenon can be attributed to the prolonged interaction between H_2O_2 and oil, leading to H_2O_2 decomposition into H_2O molecules. Furthermore, **Error! Not a valid bookmark self-reference.** demonstrates the robustness of the predictive model for moisture content. The plot shows a strong correlation between predicted and actual values, with points clustering closely around the line, indicating high accuracy.

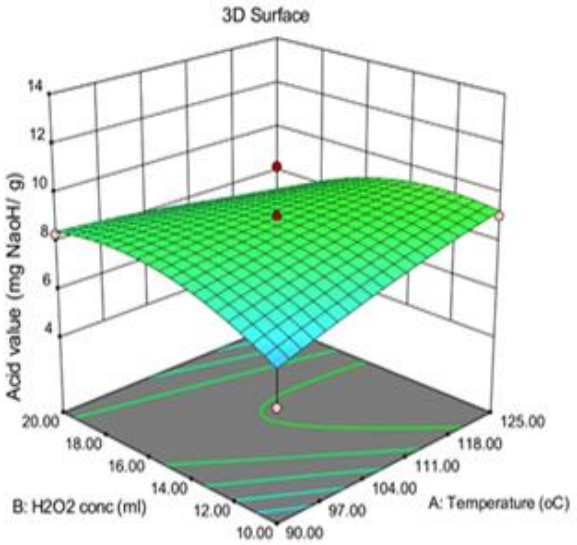

Fig. 7 illustrates the impact of temperature and H_2O_2 amount on the acid value of PKO during bleaching. The plot reveals a positive correlation between temperature and acid value, increasing from 6 to 9 mg NaOH g^{-1} as temperature rises from 90 to 125 ℃. Similarly, acid value increases with H_2O_2 amount rising from 6 to 8 mg NaOH g^{-1} as H_2O_2 increases from 10 to 16mL. Notably, acid value stabilizes at 8 mg NaOH g^{-1} beyond 16 mL H_2O_2, indicating a

threshold effect.

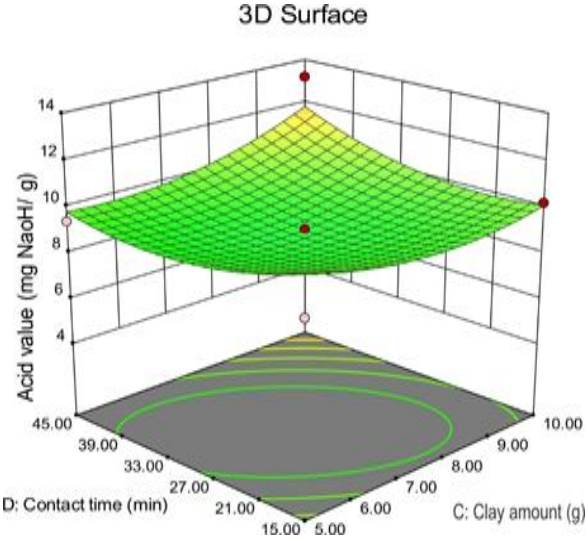

Fig. ***8*** illustrates the impact of clay amount and contact time on PKO's acid value during bleaching. Notably, a mid-range clay amount of 7.5 g yields the lowest acid value, below 10 mg NaOH g^{-1}. Deviations from this optimal clay amount (5 – 10 g) result in higher acid values, around 10 mg NaOH g^{-1}.

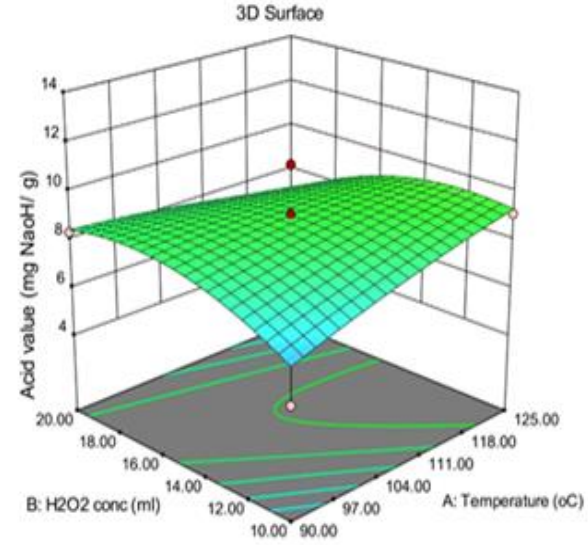

Fig. 7: 3D plot of temperature and H_2O_2 amount in relation to acid value of PKO

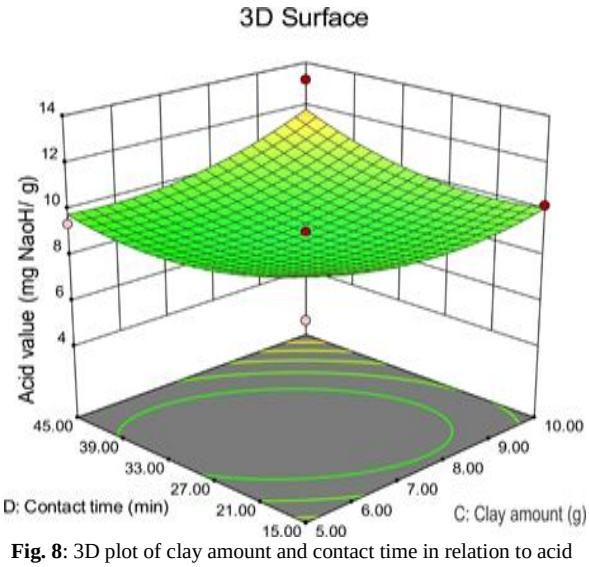

Fig. 8: 3D plot of clay amount and contact time in relation to acid value of PKO

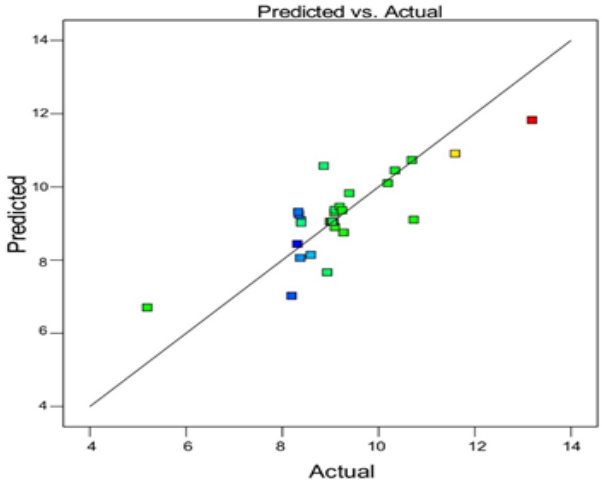

Fig. 9: 3D plot of predicted vs actual acid value

Contact time also significantly influences acid value. Shorter times (15 mins) lead to higher acid values (>10 mg NaOH g^{-1}), while longer times (>15 mins) yield lower values (<10 mg NaOH g^{-1}). Specifically, contact time between 20 – 35 mins achieve the lowest acid values, approximately 9 mg NaOH g^{-1}. Furthermore, Fig. 9 demonstrates the robustness of the predictive model for acid values. The plot shows a strong correlation between predicted and actual values, with points clustering closely around the line, indicating high accuracy.

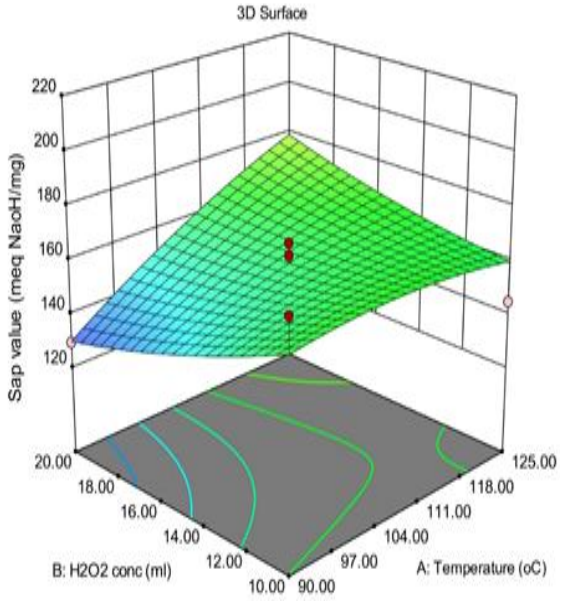

Fig. 10: 3D plot of temperature and H_2O_2 amount in relation to saponification value of PKO

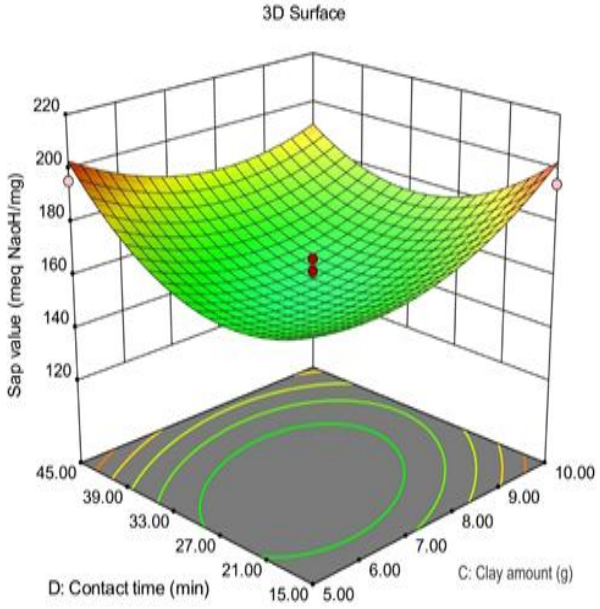

Fig. 11: 3D plot of clay amount and contact time in relation to saponification value of oil

Saponification value: Fig. 10 illustrates the impact of temperature and H_2O_2 amount on PKO's saponification value during bleaching. The results show that saponification value increases linearly with temperature, rising from 145 to 160 meq NaOH mg^{-1} as temperature increases from 90 to 125 ℃. Conversely, saponification value decreases from 145 to 130 meq NaOH mg^{-1} as H_2O_2 amount increases from 10 to 20mL. This trend aligns with expectations, as higher temperatures typically enhance saponification reactions (Eze *et al., 2015*). Meanwhile, increased H_2O_2 amounts likely facilitated the saponification of unsaturated fatty acids in the oil, which led to decreased saponification values. Fig. 11 illustrates the impact of clay amount and contact time on PKO's saponification value during bleaching. The saponification value increases significantly with increasing clay amount, rising from 180 to 200 meq NaOH mg^{-1} as clay amount increases from 5 to 10 g. Contact time exhibits a non-linear effect, with saponification value initially decreasing from 180 to less than 160 meq NaOH mg^{-1} between 15 – 30 mins, then increasing to 200 meq NaOH mg^{-1} between 30 – 45 mins. This inverse U-shaped relationship suggests that optimal contact time and clay amounts are crucial for minimizing saponification values. Notably, higher clay amounts and prolonged contact time tend to increase saponification values (Shahidi *et al., 2015*; Shemsu and Ababa, 2017). Additionally,

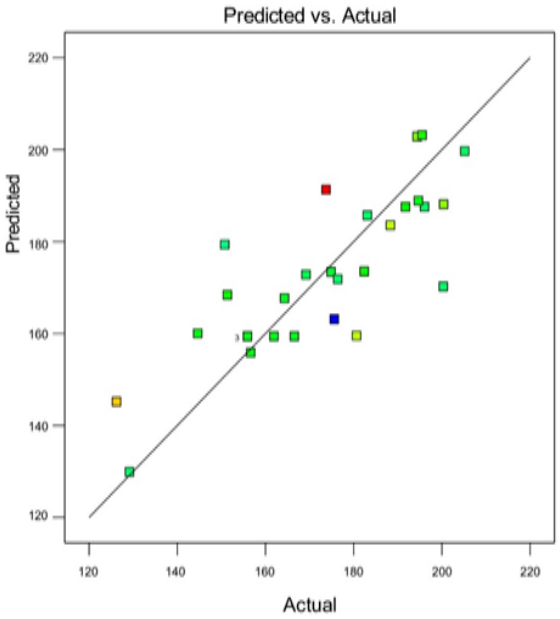

Fig. ***12*** demonstrates the robustness of the predictive model for saponification values. The plot reveals a strong correlation between predicted and actual values, with points clustering tightly around the line, indicating high accuracy.

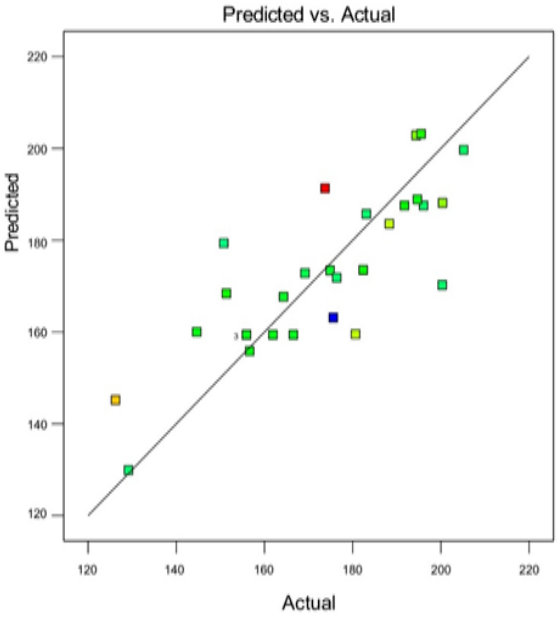

Fig. 12: 3D plot of predicted vs actual for saponification value

Peroxide Value: Fig. 13 reveals the linear relationship between temperature, amount of H_2O_2, and peroxide value of PKO during bleaching. While temperature exhibits a minimal impact, with a slight decrease in peroxide value from 90 to 125 ℃, the amount of H_2O_2 emerges as the dominant factor. The peroxide value increases substantially from 12 to 18 meq as the amount of H_2O_2 rises from 10 to 20 mL, indicating a direct correlation between H_2O_2 amount used and peroxide value obtained. Notably, clay amount and contact time have negligible effects on peroxide value, suggesting that H_2O_2 amount is the key factor. Fig. 14 demonstrates the predictive model's accuracy for peroxide value, with a compact plot showing strong correlation between predicted and actual values.

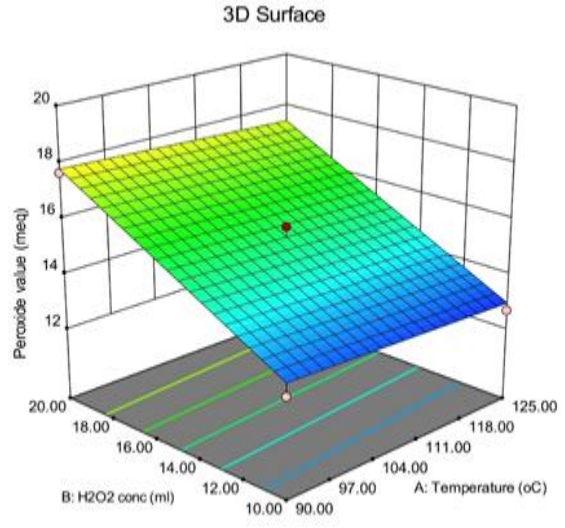

Fig. 13: 3D Plot showing effect of temperature and H_2O_2 amount in relation to peroxide value

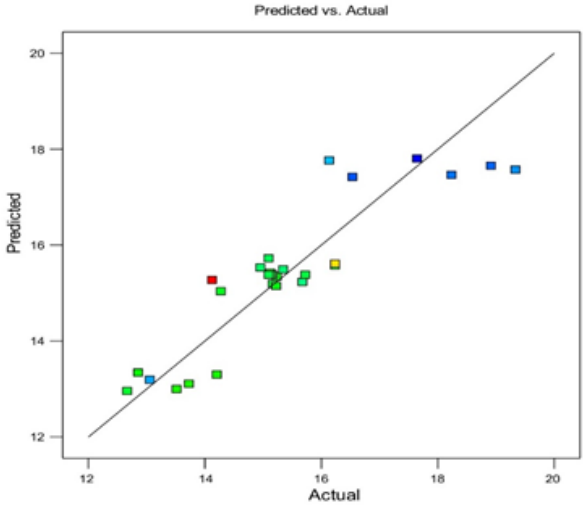

Fig. 14: 3D Plot showing effect of temperature and H_2O_2 amount in relation to peroxide value

Model verification and optimization: Following the response surface methodology, Box-Behnken Design (BBD), coded levels are calculated using the Eq. $Z=Z_0-\frac{Z_c}{\Delta Z}$ (6):

$$Z = Z_0 - \frac{Z_c}{\Delta Z} \qquad (6)$$

Where Z and Z_o indicate coded and real levels of independent variables, respectively. ΔZ represents step change while Z_c indicates actual value at central point. The Z_o values are the optimal values predicted by the model, the Z_c values are central values of the variables, while ΔZ is the range from the central value of the variables to the extreme of the variable. The coded values of each of the variables, and the actual levels of optimization which were predicted was calculated and tabulated in

Table 7.The coded and actual levels of optimization which are predicted are seen in

Table 7. For the responses, the predicted values are obtained, same as the actual values which are within the range of experimental values. The model is thus fitted for this analysis.

Table 7: Optimum Conditions, experimental and predicted value of response at optimized conditions

Optimum Conditions	**Coded Levels**	**Actual levels**
Temperature (℃)	-0.503	98.697
H_2O_2 amount (mL)	0.9526	19.763
Clay amount (g)	0.9472	9.868
Contact time (min)	-0.4879	22.681
Response	**Predicted**	**Experimental**

	Values	Values
% FFA	3.567	3.99 ± 1.41
Moisture Content (%)	3.272	2.1 ± 1.66
Acid Value (mg NaOH g^{-1})	9.784	9.19 ± 3.99
Sap Value (meq NaOH/mg)	154.068	172.56 ± 46.26
Peroxide Value (meq)	17.11	15.38 ± 3.96

Conclusions: This study optimized palm kernel oil bleaching using hydrogen peroxide (H_2O_2) and activated clay. Combining H_2O_2 and clay yielded better efficiency than clay alone. Process variables, including temperature, contact time, H_2O_2 amount, and clay amount, significantly impacted the bleaching process. Optimization revealed a temperature range of 90-100°C and a 2:1.5 H_2O_2-to-clay ratio produced the best results. H_2O_2 pre-treatment removed more impurities but increased moisture content. Response surface methodology (RSM) predicted optimal values for key responses, demonstrating the potential for industrial application, further research, and contributing to sustainable development goals.

Declaration of Conflict of Interest: The authors declare no conflict of interest.
Data Availability Statement: Data are available upon request from the first author or corresponding author or any of the other authors.

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