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Optimization of Bleaching Crude Palm Oil with Activated Snail Shell using Central Composite Design [CCD]

Ojewumi Modupe Elizabeth[*1] and Ezeocha Roosevelt Chizurum[1]
[1]* Chemical Engineering Department, Covenant University, P.M.B. 1023, Ota. Nigeria.

Corresponding author: Modupe Elizabeth Ojewumi
Email: modupe.ojewumi@covenantuniversity.edu.ng
[1]*Orcid: 0000-0002-9254-2450

Abstract

Agricultural wastes have posed as a treat to the environment over the years and they are found in large quantities due to domestic and industrial utilization of such materials in under developed and developing countries. The inability to recycle this waste has led to researches on how to use them in carrying out productive industrial activities. The aim of this study is to use Central Composite Design (CCD) to optimize the bleaching effects by snail shell as adsorbents on crude palm oil. The predictive ability of the model was close to accurate using MINITAB 19 software with the design application for the process simulation for % FFA yield to have 75.856% for experimental and 77.587% for predicted yield with just 1.731% residual. The saponification value increased with adsorption, and it indicates that palm oil can be used for soap making.

Keywords: adsorbent, palm oil, central composite design, bleaching, snail shell

1. Introduction

The increase in agricultural waste has resulted in endangering the life of livestock's and man. Environmental pollution can be of positive relevance if the waste can be recycled and re-used to ensure a cleaner and healthier environment [1, 2]. Its recent discovery that some industries has helped in curbing environmental pollution, as some waste materials are now being recycled and re-used instead of being wasted [2-4]. Environmental conservation and limiting the production waste materials by recycling it to useful products can never be over-emphasized [5, 6]. Solid waste does not only occupy valuable land but also contributes largely towards environmental pollution [6]. Other organic waste materials such as garden waste, kitchen waste and agricultural waste also contribute to solid waste generation which researchers are working on their conversion to useful products [7-10].

Crude palm oil also known as red palm oil, it is an edible vegetable oil and has high inactive vitamin A content [6]. It consists of saturated and unsaturated fatty acid such as palmitic acid, oleic acid and 10% linoleic acid which is unsaturated [3]. Other minor components of crude palm oil are phytosterols which are very beneficial in pharmaceutical industries [4] and carotenes which are used in the prevention of cancer, cataracts and heard disorder [5]. It differs from kernel oil or coconut oil. Palm oil is a common ingredient used in cooking, frying and soap making. In homes, it serves as a first aid treatment when a poisonous substance in ingested and used as a remedy for cough [3]. Presently, in industries Palm oil is used to manufacture wax, detergents, ink, biofuels and cosmetics. Bleaching of oil is vital for producing light coloured oil of improved stability, acceptable and sensory quality. The process involves the removal of impurities, pigments, trace metals and high molecular component of fats and oil [4,7].

Bleaching of can be achieved either by heat treatment or adsorption methods. Heat treatment method involves simply heating the oil to a high temperature of 1200 ^{o}C to 1500 ^{o}C, the temperature is maintained for a period of time before the colour changes. The adsorption method is the most effective and widely used form of bleaching; this involves mixing the oil with an adsorbent such as activated alumina, activated clay, carbon, silica gel etc then heating the mixture for a particular period of time at temperature between 80 ^{o}C to 150 ^{o}C after which particles are removed by filtration. Researchers investigated that the most widely used adsorbent is the activated clay because it doesn’t alter the chemical properties of the oil and also very efficient. Although, attention is presently drawn to the agricultural waste which can serve as a substitute adsorbent [7,8].

Mathematical model is regarded as a decision tool that assists decision makers in effectively dealing with complex issues such as oil spillage on soil surfaces [11-13].

However, this study aims to comparing the efficacy of activated groundnut hull, snail shell and rice husk as adsorbent in bleaching of crude palm oil.

2. Materials and methods

Snail shells were obtained from Umuahia, Abia state, Nigeria. Crude palm oil was obtained from Covenant university farm, Ota, Ogun state. The adsorbents were chemically activated to improve their bleaching ability.

2.1 Proximate analysis: Method by [14, 15] were used.

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Plate 1: Snail shell

2.2 Methodology

The distinctive examination was done on the adsorbents, snail shell powder (SSP) and palm oil. The moisture content and debris content were completed utilizing the AOCS Recommended Practice Ca 2f-93 technique, and the peroxide value was done using the methodology proposed by [16] and modified by [17].

2.3 Physico-Chemical Analysis of the Adsorbent

2.3.1 Moisture content

One gram of the dried adsorbents was weighed and put in a dry crucible. The crucible was set in a spray dryer at 105℃ for around 2 hours [2].

Calculations

$$\text{Moisture\%}= \frac{\text{loss in weight on drying}}{\text{initial weight}} \times 100\% \qquad [1]$$

2.3.2 Ash content

One gram of the adsorbent sample was put into a crucible and reweighed. The sample was then positioned in a furnace with temperature of 1000ºC for about 1 hour 30 minutes. After which it was cooled in a desiccator.

Calculations

$$\text{Ash content}= \frac{\text{Wc\&s} - \text{Wc}}{\text{Wo}} \qquad [2]$$

Wc&s = Weight of the crucible and sample after ash testing

Wc = Weight of the adsorbent before ash testing

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3. Physico-Chemical Analysis of Palm oil

3.1 Moisture Content

As described by [18], the crucible was washed, dried using spray dryer and cooled in the desiccator before weighing. 2 g of the sample was stored in the crucible, and the weight recorded [16]. It was later dried at 80 °C for 2 hours and 135 °C for 4 hours and cooled in the desiccator, after which the dry load of the sample in addition to crucible was taken.

Calculation:

% Moisture: $\frac{W2-W3}{W2-W1} \times 100$ [3]

Where: W1 = Initial weight of the empty crucible

W2 = Weight of crucible plus sample before drying

W3 = Final weight of crucible plus food after drying

3.2 Peroxide Value

The technique described by [19] in their work was utilized. 5 ml of oil was measured and pour in a dry 250ml conical flask. 10ml chloroform was added, and the oil-chloroform mixture was properly mixed. Potassium iodide was later added to the mixture. The flask with stopper was shaken for one minute and put in a dark locker for one minute. 75ml of water was included and titrated with 0.002M sodium thiosulphate arrangement utilizing solvent starch arrangement (1%) as a pointer. The titre value was recorded as (V), and the precise worth (VO) was additionally recorded.

Calculations

Peroxide value (mEq/kg) = $\frac{(V-VO)}{M} \times 10^3$ [4]

Where: T = exact molarity of sodium thiosulphate solution.

3.3 Acid value and free fatty acid content

The technique announced by [16, 19] was utilized. 10 ml of n - propanol was blended with 10 ml of diethyl ether and, and 1ml of Phenolphthalein (1%) was added. 5 ml of oil was disintegrated in the solvent and titrated with 0.1 M KOH, shaking continually until a pink shading which endured for 15sec was obtained. The measure of KOH utilized was recorded.

Calculation

Acid value (mg KOH/g) = $\frac{\text{Titre value} \times 5.61}{\text{sample weight}}$ [5]

Free fatty acid (mg KOH/g) = $\frac{\text{Acid value}}{2}$ [6]

3.4 Ester value

The ester value was obtained by [19, 20], this was done by finding the contrast between the saponification value and acid value.

3.5 Saponification value

5 ml of oil were measured accurately and placed into a conical flask containing 25 ml of 0.5 M KOH. The reflux condenser was fitted to the jar containing the ionic solution and warmed in a water shower for an hour, whirling the flask continually. KOH was titrated with 0.5 M HCl utilizing 1ml of phenolphthalein (1%) solution. The technique was repeated for the blank [2, 20].

Saponification value (mg KOH/g) = $\frac{(b-a) \times 28.05}{\text{sample weight}}$ [7]

4. Results and Discussions

4.1 Design of Experiment (DOE)

Table 1: Design of the Variables with FFA as response

Factors	Notation
Mass	X1
Temperature	X2
Time	X3

Central Composite Design

Factors: 3 Replicates: 1
Base runs: 20 Total runs: 20
Base blocks: 1 Total blocks: 1

Two-level factorial: Full factorial

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Cube points: 8
Center points in cube: 6
Axial points: 6
Center points in axial: 0

α: 1.68179

Three operating factors viz. X1 (Mass), X2 (Temperature) and X3 (Time) were taken into consideration, to yield 20 runs.

The data obtained were analyzed using CCD for the quadratic polynomial in order to fit the equation generated to correlate the DOE variables [21-23].

$$F = \beta 0 + \beta 1X1 + \beta 2X2 + \beta 3X3 + \beta 1,1X1X1 + \beta 1,2X1X2 + \beta 1,3X1X3 = \beta 2,2X2X2 + \beta 2,3X2X3 + \beta 3,3X3X3 \quad [8]$$

F is the response generated as a function of 3 variables: Mass (g), Temperature (ºC) and Time (mins). The response was fitted by a 2^{nd} order polynomial in order to correlate the set variables. F is associated with each factor level combinations. β0, β1, β2, β3, β1, 2 ….. β3, 3 are the regression coefficients while X1, X2 and X3 are the factors.

Table 2: Physicochemical properties of the unrefined palm oil before adsorption

Analysis	Result
FFA (mg KOH/g)	7.43
Peroxide value (mEq./kg)	12.5
Moisture content	0.4
Saponification value (mg KOH/g)	195
Density (kg/m^3)	921
Acid value (mg KOH/g)	14.9
Specific gravity	0.9
Ester value	173
Molecular weight (g/mol)	968

Table 3: Randomized Central Composite Design of the experiment using MINITAB 19

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Run Order	Mass (g) [X1]	Temperature (ºC) [X2]	Time (mins) [X3]
1	1.8	100	60
2	0.8	110	90
3	3.5	100	60
4	2.8	110	90
5	1.8	100	60
6	0.8	110	30
7	1.8	100	110
8	0.8	90	30
9	0.8	90	90
10	1.8	100	60
11	1.8	100	60
12	2.8	110	30
13	1.8	117	60
14	2.8	90	30
15	1.8	83	60
16	0.1	100	60
17	1.8	100	60
18	2.8	90	90
19	1.8	100	9
20	1.8	100	60

Regression Equation in Uncoded Units

%FFA=16.6 − 3.46 (Mass) − 0.052 (Tempt) −0.017 (Time) −0.077 (Mass×Mass 0.00012) (Tempt×Tempt) + 0.000178 (Time×Time) + 0.0115 (Mass×Tempt) + 0.0053 (Mass×Time) − 0.00037 (Tempt×Time) [9]

Table 4: POV and FFA analysis on the bleached palm oil using snail shell as adsorbent

Mass (g)	Temperature (ºC)	Time (mins)	FFA (mg KOH/g)	POV (mEq/kg)
1.8	100	60	5.042	10.041
0.8	110	90	6.740	13.432
3.5	100	60	0.532	9.634
2.8	110	90	0.837	12.837
1.8	100	60	5.042	10.041

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0.8	110	30	6.943	13.213
1.8	100	110	4.872	8.241
0.8	90	30	7.023	9.042
0.8	90	90	6.924	8.82
1.8	100	60	5.042	10.041
1.8	100	60	5.042	10.041
2.8	110	30	1.043	12.673
1.8	117	60	4.973	13.631
2.8	90	30	1.320	4.370
1.8	83	60	5.410	8.403
0.71	100	60	7.084	8.638
1.8	100	60	5.042	10.041
2.8	90	90	0.956	7.63
1.8	100	9	7.352	6.07
1.8	100	60	5.042	10.041

Table 5: Plan application for the cycle simulation on the % FFA yield of snail shell (SSP)

Std Order	Run Order	PtType	Blocks	Mass (g)	Temperature (ºC)	Time (mins)	%FFA [Response]		
							Experimental	Predicted	Residual
17	1	0	1	1.8	100	60	5.042	4.69615	-0.34585
7	2	1	1	0.8	110	90	6.740	6.96678	0.22678
10	3	-1	1	3.5	100	60	0.532	0.02945	-0.50255
8	4	1	1	2.8	110	90	0.837	1.47654	0.63954
19	5	0	1	1.8	100	60	5.042	4.69615	-0.34585
3	6	1	1	0.8	110	30	6.943	7.71216	0.76916
14	7	-1	1	1.8	100	110	4.872	4.07500	-0.797
1	8	1	1	0.8	90	30	7.023	7.91577	0.89277

5	9	1	1	0.8	90	90	6.924	7.17039	0.24639
18	10	0	1	1.8	100	60	5.042	4.69615	-0.34585
16	11	0	1	1.8	100	60	5.042	4.69615	-0.34585
4	12	1	1	2.8	110	30	1.043	2.22192	1.17892
12	13	-1	1	1.8	117	60	4.973	4.52308	-0.44992
2	14	1	1	2.8	90	30	1.320	2.42553	1.10553
11	15	-1	1	1.8	83	60	5.410	4.86923	-0.54077
9	16	-1	1	0.17	100	60	7.084	7.68833	0.60433
15	17	0	1	1.8	100	60	5.042	4.69615	-0.34585
6	18	1	1	2.8	90	90	0.956	1.68015	0.72415
13	19	-1	1	1.8	100	9	7.352	5.32973	-2.02227
20	20	0	1	1.8	100	60	5.042	4.69615	-0.34585
			TOTAL				92.261	92.26096	-4E-05

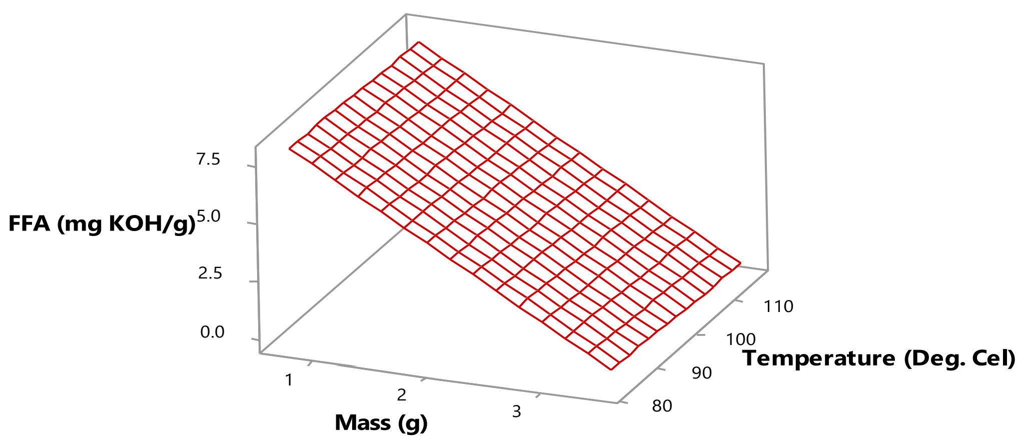

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Fig 1. 3D surface plot of FFA for snail shell vs. mass and temperature

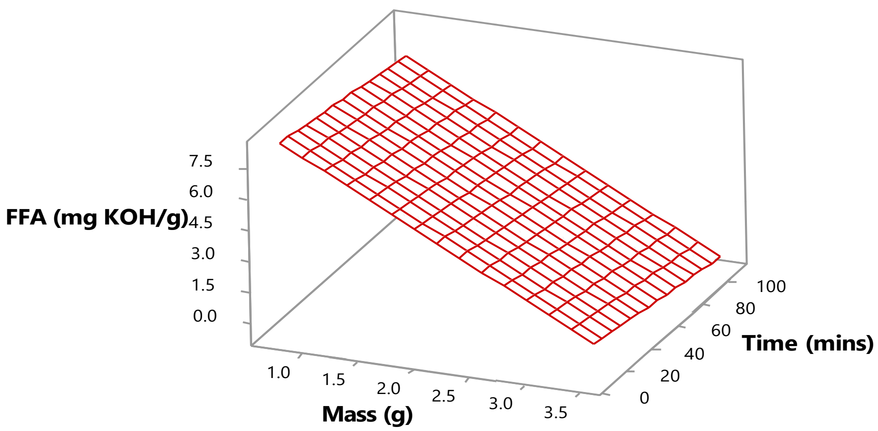

Fig 2. 3D surface plot of FFA for snail shell vs. mass and time

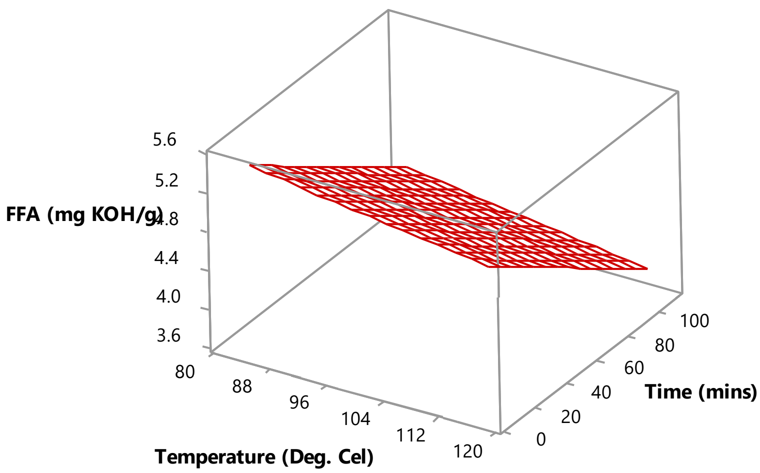

Fig 3. 3D surface plot of FFA for snail shell vs. temperature and time

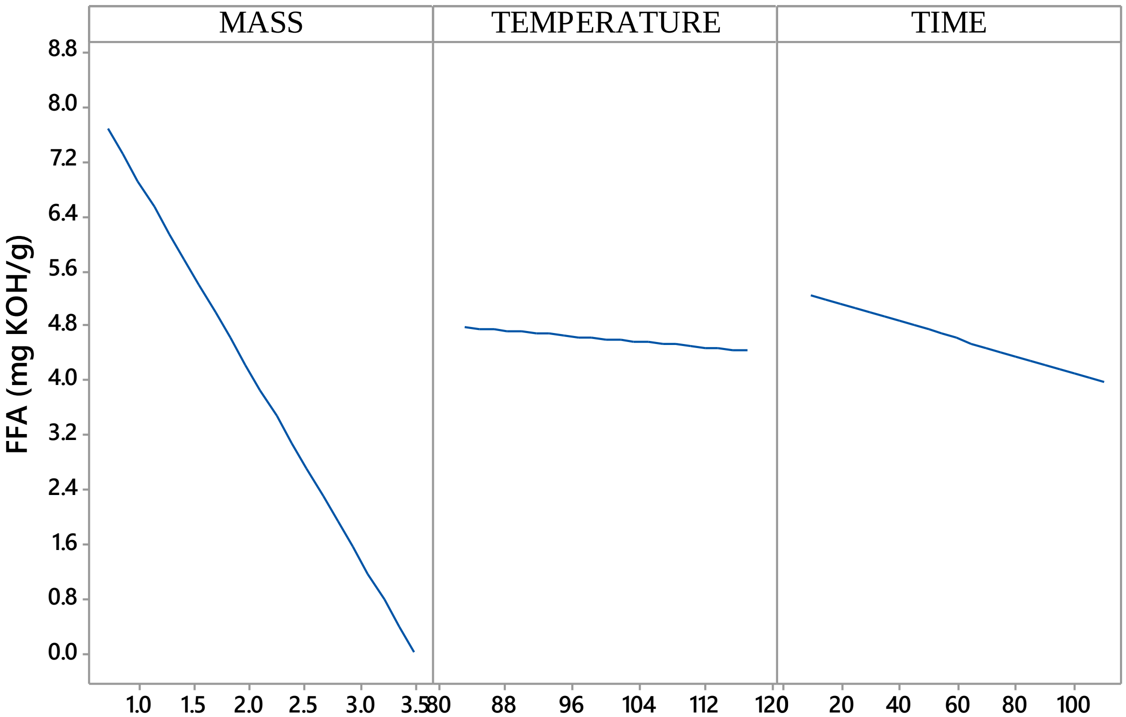

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Fig 4. Factorial plot of FFA for snail shell vs. time, temperature and mass

Regression Equation

Peroxide value = -12.91 - 0.501 ×mass + 0.2710× temperature – 0.0202× time [10]

Table 6: Plan application for the cycle simulation on the POV of snail shell (SSP)

Std Order	Run Order	PtType	Blocks	Mass (g)	Temperature (ºC)	Time (mins)	Peroxide value [Response]		
							Experimental	Predicted	Residual
17	1	0	1	1.8	100	60	10.041	9.8601	-0.1809
7	2	1	1	0.8	110	90	13.432	13.1480	-0.284
10	3	-1	1	3.5	100	60	9.634	9.0087	-0.6253
8	4	1	1	2.8	110	90	12.837	12.1463	-0.6907
19	5	0	1	1.8	100	60	10.041	9.8601	-0.1809
3	6	1	1	0.8	110	30	13.213	12.0995	-1.1135
14	7	-1	1	1.8	100	110	8.241	10.7339	2.4929
1	8	1	1	0.8	90	30	9.042	7.5740	-1.468
5	9	1	1	0.8	90	90	8.820	8.6225	-0.1975
18	10	0	1	1.8	100	60	10.041	9.8601	-0.1809
16	11	0	1	1.8	100	60	10.041	9.8601	-0.1809
4	12	1	1	2.8	110	30	12.673	11.0978	-1.5752
12	13	-1	1	1.8	117	60	13.631	13.7068	0.0758
2	14	1	1	2.8	90	30	4.370	6.5723	2.2023
11	15	-1	1	1.8	83	60	8.403	6.0135	-2.3895
9	16	-1	1	0.17	100	60	8.638	10.4061	1.7681

15	17	0	1	1.8	100	60	10.041	9.8601	-0.1809
6	18	1	1	2.8	90	90	7.630	7.6208	-0.0092
13	19	-1	1	1.8	100	9	6.070	8.9689	2.8989
20	20	0	1	1.8	100	60	10.041	9.8601	-0.1809
			TOTAL				196.88	196.8797	-0.0003

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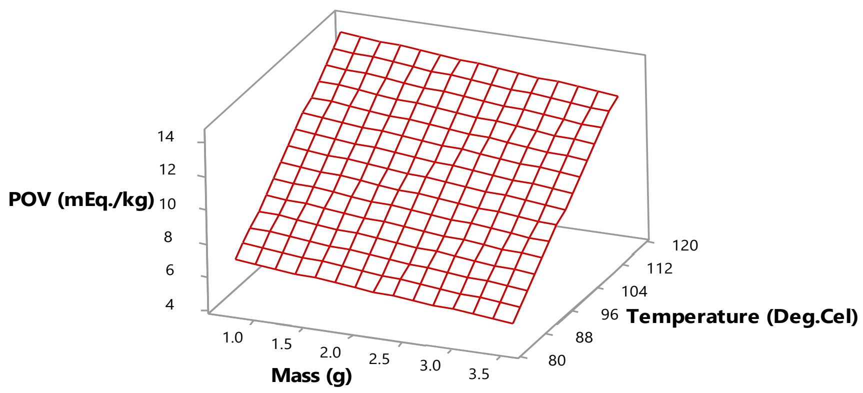

Fig 5. 3D surface plot of POV for snail shell vs. mass and temperature

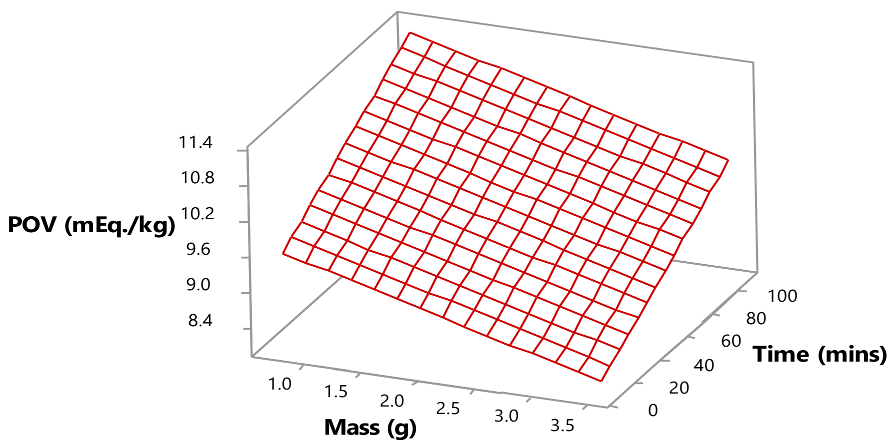

Fig 6. 3D surface plot of POV for snail shell vs. mass and time

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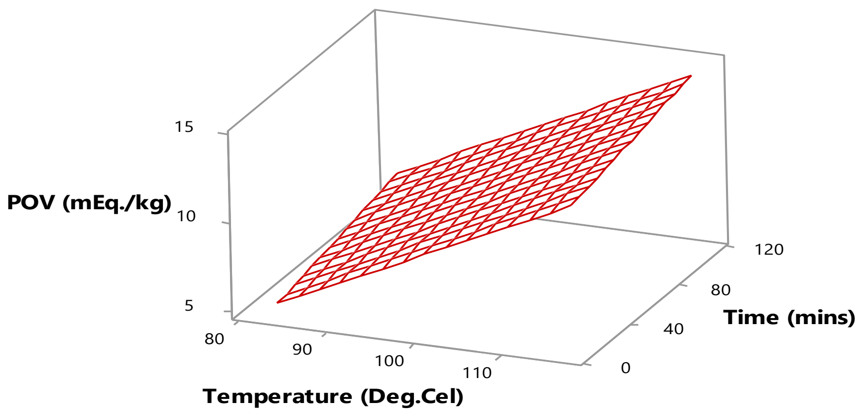

Fig 7. 3D surface plot of POV for snail shell vs. temperature and time

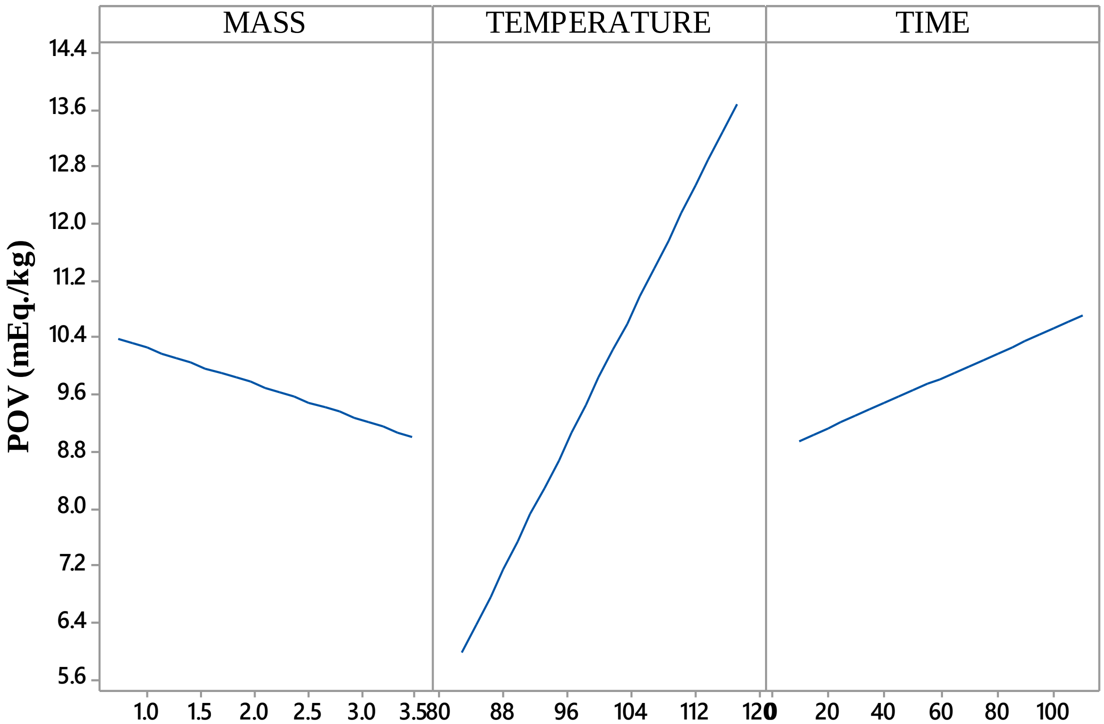

Fig 8. Factorial plot of POV for snail shell vs. time, temperature and mass

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Table 7: Physicochemical properties of the palm oil after adsorption for 0.8g

1.8g of bleached palm oil at 100ºC for 60 mins

Analysis	SSP
FFA (mg KOH/g)	5.042
Peroxide value (mEq./kg)	10.041
Acid value (mg KOH/g)	10.084
Saponification value (mg KOH/g)	230

Table 8: Physicochemical properties of the palm oil after adsorption for 2.8g

2.8g of bleached palm oil at 110ºC for 90 mins

Analysis	SHP
FFA (mg KOH/g)	0.837
Peroxide value (mEq./kg)	12.837
Acid value (mg KOH/g)	1.674
Saponification value (mg KOH/g)	247

Table 9: Physicochemical properties of the palm oil after adsorption for 3.5g

3.5g of bleached palm oil at 100ºC for 60 mins

Analysis	SHP
FFA (mg KOH/g)	0.532
Peroxide value [POV] (mEq./kg)	9.634
Acid value (mg KOH/g)	1.064
Saponification value (mg KOH/g)	272

5. Discussion of Result

5.1 Activation of adsorbents

Snail shell was subjected to an activation reaction using a strong acid (H_2SO_4) so as to effectively bleach the palm oil. Previous work done has shown a high reduction in the impurities present in palm oil as a result of contact with bleaching adsorbents, such as used in this work. From the research work carried out by [24-26] on activation of oyster shell, the time of activation was modified in this present work for the activation (using H_2SO_4) on the adsorbent samples for 5hrs to analyze the influence of time on the surface area increment.

5.2 Analysis of SSP on the response (FFA) data

Table 1 shows the DOE of the variables on the %FFA yield of Snail shell powder. Table 3 is the Randomized Central Composite Design of the experiment using MINITAB 19 while table 4 is the POV and FFA analysis on the bleached palm oil using snail shell as adsorbent that were obtained from the laboratory. Predicted values that was obtained from the software (MINITAB 19) using a regression equation represented by equation 8 were presented as the plan application for the cycle simulation on the % FFA yield of snail shell (SSP) in table 5.

5.3 Effect of SSP dosage on the FFA response

Figure 1 to 3 show the 3D effect of the predictor variable (mass) on the response while time and temperature are held constant respectively. The surface plots show that as the mass of the SSP increases, the response reduces and this can be attributed to the increasing active sites available for adsorption.

5.4 Effect of temperature on the FFA response

Figure 1 reveals the 3D effect of temperature on the response. It comprises the relationship between the response variable (FFA) and the two predictor variables (mass and temperature) while time is held constant. It is evident that the increase in the temperature at which the process was subjected to increase the FFA rate declined.

5.5 Effect of time on the FFA response

The time variable is another factor taken into consideration in this segment. Figure 1 revealed that the longer the process time, the more the process reaction occurs. The software's surface response plot shows that as the reaction time increases, the FFA decreases. Likewise, figure 2 and 3 shows the 3D effect of the predictor variables (mass and time / temperature and time) on the response respectively. There is a decrease in the FFA as the time of adsorption increases. The difference between the figures mentioned above is their surface representation.

5.6 Analysis of snail shell on the response (POV) data

Figures 5 to 8 reports the effect of mass, temperature, and time on the response (POV). Table 6 shows the experimental data, predicted values, and the design of the experiment. The experimental values were obtained from the laboratory using the experiment's design as a basis/guide. The predicted data were obtained from the MINITAB 19 software by setting the confidence level to 95% and using a regression model as indicated by equation 10.

According to [27, 28] FFA content is the most common property for determining palm oil quality, as it must not exceed 5% alongside the peroxide value.

5.7 Effect of SSP dosage on the POV response

The surface response plots in figures 5 to 7 shows the 3D effect of the predictor variables on the response variables. Figure 5 shows the relationship between the response and the predictor variables (mass and temperature) while time is held constant. From the plot, it is evident that the increase in the adsorbent mass decreases the POV in the palm oil. The same results were observed in figures 6 and 7, where the effect of the predictor variables (mass and time / temperature and time) respectively and the response was monitored. The factorial plots of the snail shell increase in mass, which might probably be due to the rise in the active sites, which led to the reduction of peroxide value.

5.8 Effect of temperature on the POV response

Peroxide value is susceptible to heat and excess temperature. Figures 5 and 6 shows the 3D effect of the variables on the response. From the predictor variables (figure 5) are mass and temperature while they are mass and time for figure 6. The effect of temperature is observed, and it is deduced that an increase in temperature increases the POV when snail shell is used. Figure 7 also shows a similar result.

5.9 Effect of time on the POV response

An increase in the time of a process increases the process reaction. From the data outlined in table 6 to the plot represented by Figures 6 and 7, the increase in process time reduced the peroxide value, and it might have resulted from the rise in the period at which the adsorbent spent in the oil. Figure 6 shows the relationship between the response variable and two predictor variables (mass and time), while the variable of temperature and time is shown in figure 7. Figure 8 illustrates a factorial plot that summarizes the response surface plots. It makes interpretation easy for readers. From the model, which consists of two negative and one positive slope, we can denote that as the mass and time increase, the POV reduces but as the temperature increases to the maximum, the POV increases.

5.10 Physicochemical properties of the palm oil after adsorption

Tables 7 to 9 show the palm oil's physicochemical properties after adsorption, varied at different masses. This is to record the effect of different adsorbent mass on the properties. From these tables, it is evident that the adsorbent has some effect on palm oil. Referring to table 2, there is a vast difference between the palm oil properties before and after adsorption. The free fatty acid (FFA) was reduced upon an adsorbent addition, and this is expected. According to [29-31] free fatty acid in cooking oil lies within the limits of 0% to 3%. Although this range was not achieved by the 1.8g mass, the FFA values obtained for the others, 2.8g and 3.5g, show proximity to the set range. Hence, we can attribute the reduction of FFA to mass. The low value of free fatty acid indicates that palm oil is fresh and will be prolonged.

The peroxide value test is used to determine the oxidative rancidity of the oil. The lower the peroxide value, the longer it stays without deterioration. Likewise, oil with high peroxide value quickly becomes rancid. The peroxide value decreased drastically with the increase in mass and time. However, there was a significant increase in the POV as the process temperature increased to certain levels, but the response decreases as the mass increases which are directly proportional to the increase in time and temperature. The increase in temperature can be related to the work of [31-34] who stated that the peroxide value of palm oil is highly sensitive to heat and high temperature [35].

6. Conclusion

There is no need for disposing Snail shell since this work concluded that it is a valuable material for the palm oil industries. The saponification value increases with bleaching this indicates that the oil is very suitable for soap making. The FFA decreased with bleaching, the reduction in FFA prolongs the oil shelf life. It was observe that adsorption process made the palm oil lighter. The oil's peroxide value decreased for mass and time and increased for temperature. This is as a result of the sensitivity of oil peroxide value to high temperature and heat.

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