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Optimizing *Elaeis guinesis* Kernel Oil Extraction Using N-hexane and Physicochemical Characterization of the Extracted Oil

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ABSTRACT

The importance of *Elaeis guinesis* kernel oil is germane to the increasing global demand for the utilization of vegetable oil; this prompts optimizing the extraction of the oil. The study investigated the palm seeds' oil extraction that was obtained from the cracked nuts after separating the nuts from the seeds. The oil was extracted from the powdered seed using a Soxhlet extractor making use of n-hexane as a solvent. The experimental design used, the oil extraction employed a three-level-three-factors Box-Behnken design. And 17 experimental runs were generated through Response Surface Methodology (RSM). The independent factors considered include Sample Weight (g), Solvent Volume (ml), and Extraction time (min). The physicochemical characterization of the oil was obtained according to standard methods to determine its quality. The results showed that the statistics model predicted the optimal oil yield of *Elaeis guinesis* kernel to be 34.9232% at optimized conditions for sample weight of 40 g, Solvent volume 175 ml, and the extraction time to be 50 (min). Physicochemical properties of the oil showed that the extracted oils are distinct in color variation at room temperature, and an acid value of 22.1mgKOH/g oil, Iodine value of 87.85(I_2g/100g oil), the Cetane number of 65.449, Saponification value 140.125 (mg KOH/g Oil). It is discovered that an optimum *Elaeis guinesis* kernel oil yield is dependent on some factors like the solvent and the extraction time. It was observed that low solvent volume favored high extraction and oil yield for global use.

Keywords: Oil Yield, Box-Behnken Design, RSM, Soxhlet Extractor, Oilseed crops

1. INTRODUCTION

An increase in the global utilization of oil has led to increasing demand for oilseeds to produce oils and their by-products for domestic and industrial usage. The vegetable oil has been obtained from many oilseeds one of which is

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Elaeis guinesis seed (commonly known as oil palm seed) to obtain useful oil to meet global utilization demand. The demand for sustainable *Elaeis guinesis* is products (Palm oil and Palm Kernel oil) is driven by the growing focus of developed nations on the production of green fuel, application of feedstock in biofuel production where the products are valuable, and their global use in the food sector as; cooking oil, margarine, frying fat, bakery fat, and biofuel where it has the highest yield couple with the fact that it is the cheapest among all vegetable oil (Dublin, 2021). Because of this importance, the global demand for the products e has an impact on the global economy; for *Elaeis guinesis* oil is estimated at US$42.8 Billion in the year 2020, while that of *Elaeis guinesis* kernel oil is estimated at US$10.2 Billion in 2020 and with both having the compounded annual growth rate (CAGR) of 5% and 5.4% respectively over the analysis period. (Dublin, 2021)

Elaeis guinesis is one of the species of oil palm that produces the fruit for the said products. It is a widely cultivated oil-bearing tropical palm tree that originated from West African (originally in the rain forest of Guinea) and was initially illustrated by Nicholas Jacquin in 1973, as reported by Ng *et al*, (2003). In the late 1400s, the European visitors who were dwelling around the coast of Guinea discovered the oil palm trees (Poku, 2002). By 1508, palm groves are recognized in nearby countries such as Liberia and Nigeria (Poku, 2002). The belts in Africa initially were running through Ghana, Cameroon, Cote d' Ivoire, Liberia, Nigeria, Sierra Leone, Togo, Angola, and the Republic of Congo. It later spread to other parts of Africa and regions of the world including South America and Asia between the fourteenth and seventeenth centuries (Henderson and Osborne 2000; Poku, 2002). The spread of oil palm makes it a global tree. *Elaeis guinesis* is is found to thrive very well in areas of high rainfall within the isohyets of 1200 mm rain per year with the topography of about 400m altitude and tropical climates within 70^{o}north and south from the equator (Schmidt, 2007).

The tree gained international acceptance when the demand for *Elaeis guinesis* is oil as lubricants for steam engines, machinery, and as major raw material for soap manufacturing rose in the late nineteenth century. The oil yields and quality of the *Elaeis guinesis* is cultivated in Asia are still superior to those produced in other parts of the world. South America also contributes to a significant share in the global production of *Elaeis guinesis*is presently. The oil palm trees produces averagely 12 fresh fruit bunches (FFB) annually with a bunch weighing about 15-25 kg and containing 1000-1300 fruitlets depending on the plantation management and establishment method. On a per hectare basis, and *Elaeis guinesis* plantation can yield an average of 35 tons of FFB and 8.6 tons of palm oil (Schmidt, 2007). The two types of oil produced from the fruit which are palm oil and palm kernel oil, are obtained from squeezing fleshy mesocarp, and crushing endosperm of the fruits respectively

Palm kernel seeds contain about 75-80% oil (Jin, 2008). The oil extracted from nuts of palm fruit is yellowish when modern methods is used, but it is dark brown when processed or extracted traditionally (Ibiam *et al.,* 2014). Palm kernel oil is inedible oil derives from *Elaeis guinesis* (Poku, 2002). It attracts much attention globally in food industries, it is solid at room temperature, and this makes its transportation and storage. The oil can also be stored longer than other vegetable oils. (Bjorklund, 2010: Dublin, 2021) Its oxidative stability after frying is superior, making it attractive for restaurants. The oil is used in; cosmetic, pharmaceutical industries, and traditional medicine, and as lubricant or biodiesel (Alander, 2004). Remold Rajan (2019), states the oil benefit as being; zero cholesterol, loaded with antioxidants, unsaturated fats rich in vitamin k, zero trans-fat, it contains vitamin A, and filled with nutrients, he further mentioned that it improves hair growth, and provides naturally soft skin.

Elaeis guinesis kernel oil is extracted using solvent extraction in this work, among many methods of extraction like mechanical screw-press, and traditional methods (Jin, 2008). The need to optimize extraction of the oil using solvent extraction is very important because it is time effective, it is cost effective and it yields maximum oil. However, the conventional optimization method of–factor-at-a-time consumes time and it is burdensome; it does not provide room for interactive effects of the factors under investigation. In solving these problems, the response surface methodology (RSM) is employed. RSM is a computational tool for describing the impact of the independent variables and their interactions. It has been applied to various investigations on the extraction of seed oil from *Sesamuumindicum* (Betiku *et al*., 2012), *Hibiscus sabdariffa* (Betiku and Adepoju 2013), *Piper nigrum* (Bagheri et al., 2014), *Nitrariatangutorum* (Liu *et al*., 2014). The quality of the extracted oil is determined by its physicochemical composition, which its variation has been attributed to environmental factors (Sonau *et al.,* 2006).

2. MATERIALS AND METHODS

The suitable method of extraction is applied to optimize the oil extracted from *Elaeis guinesis Seed* which was collected from the fields.

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2.1. Chemicals

The chemicals and the reagents that were used in this work were of Analytical Reagents (AR) grade that was obtained from BDH Chemical Ltd., Poole England; n-Hexane, Iodine Chlorine, Potassium Iodide (KI), Phenolphthalein, HCL, KOH, NaOH.

2.2. Equipment

The equipment that was used includes Muslim Bag, Soxhlet Extractor of 500 ml, Digital Weighing balance, Heating Mantle, Water Bath, Oven. Flash Point Machine, Spectrometer, Viscometer, Glassware which includes beakers, round bottom flask, conical flasks, pycnometer, Petri dish, Measuring cylinder and burettes.

2.3. Seeds preparation

Elaeis guinesis kernel nuts were collected from processed palm fruits in the village of IkotAkpaden, with thin mesocarp. It was cracked from the shells and manually separated from the broken shell; they were sundried for 2 days and crushed before grinded

Fig. 1-Cracked Elaeis guinesis Seeds

Fig. 2-A Crushed Elaeis guinesis Seeds

2.4. Extraction Procedures

A 500-ml Soxhlet extractor was used for the work, with n-hexane as solvent. The apparatus was filled with palm kernel seed powder in a Muslim bag and then placed in a thimble of Soxhlet apparatus, containing a known volume of the solvent and was placed on the heating mantle which was turn on to temperature slightly below the boiling point of the solvents (70%). After the process, the solvents are recycled and the oil was left in the round bottom flask which was later weighed using a weighing balance. The oil yield was evaluated as the ratio of the weight of the extracted oil to the weight of *Elaeis guinesis seed ground* sample as expressed in 3.1; oil obtained was stored in a freezer at 4^0 C for further characterization.

$$\%\text{Oil yield}(w/w) = \frac{\text{Weight of extractedoil in grams}}{\text{Weight of grinded samples in grams}} \times 100 \quad ....1$$

2.5. Experimental Design for the Extraction of oil from Elaeis guinesis Seed

Box-Behnken experimental design was employed to optimize the palm kernel oilseeds extraction. A Three-level-three-factors design was applied, which produced 17 experimental runs. Certain factors for the oil extraction from the palm kernel oilseeds are extraction weight (g): Y_1, solvent volume (ml): $Y_{2,}$ and sample time (min): Y_3

2.6. Physicochemical characterization of the Extracted Elaeis guinesis Kernel Oil

The physical and chemical characteristics of palm kernel oil are analyzed as follows:

2.6.1 AOAC methods

The method of Association of Analytical Chemists was used to determine: the moisture content, Viscosity, Acid Value, *Free Fatty Acid (FFA) (AOAC, 1990), and Saponification value (AOAC, 1984)*

2.6.2 Density

Density is calculated thus: $Density = \frac{mass\ of\ sample\ kg}{volume\ of\ sample\ m^3}$ 3

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2.6.3 Specific gravity

A cleaned and dried and weighed bottle (w_0) was filled with the oil, the stopper was inserted and reweighed (w_1). The oil was substituted with water after washing and drying the bottle and weighed to give as follows:

$$Sp.gr = \frac{w_1 - w_0}{w_2 - w_1} = \frac{weight\ of\ the\ oil}{weight\ of\ equal\ volume\ of\ water} \quad \ldots\ldots\ldots\ldots\ldots\ldots 4$$

2.6.4 Peroxide Value

2g of the oil was weighed into a 250 ml of pyrex flask of the solvent mixture (2:1 glacial acetic acid/ chloroform) and 2g KI powder was added. The mixture was boiled briskly in a water bath for 1 min at a temperature of 70^0 C. The boiled mixture was added to the flask containing 40ml of 5% KI; the resulting mixture was washed twice with 20ml of distilled water in the flask. The content of the flask was then titrated with 0.004M sodium thiosulphate (NaS_2O_3) solution by using a starch indicator. The peroxide value was calculated as indicated:

$$Peroxide\ value(meqO_2/kgoil) = \frac{Volume\ of\ Na_2S_2O_2 \times Normality\ of\ Na_2S_2O_2}{weight\ of\ the\ sample\ in\ kg} \ldots\ldots\ldots 5$$

2.6.5 Iodine value

An oil sample of 0.26g is dissolved in 5ml of cyclohexane. 10ml of Wij's solution is then added, the stoppered flask was allowed to stand for 30mins in the dark at room temperature, and 20ml of 10% potassium iodide solution was added. The resulting mixture is then titrated with 0.1M NaS_2O_3 using starch as an indicator using Wij methods and the Iodine was calculated:

$$Iodine\ Value = \frac{[B-S] \times N \times 12.69}{Weight\ of\ the\ sample} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 6$$

Where N = Concentration of sodium thiosulphate used; B = Volume of Sodium thiosulphate used for blank; S = Volume of sodium thiosulphate used for determination.

2.6.6 Mean Molecular Mass

This was determined by the equation cited by Akintayo and Bayer (2002) which was given as

$$Mean\ molecular\ mass = \frac{56}{Saponification\ Value} X\ 1000 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 7$$

2.6.7. Other Calculated properties

The properties determined by calculation like Cetane number and Higher Heating Value (HHV**)** were calculated according to ASTM D2015, also API and Aniline point were determined using the equations as described by Haldar *et al*., (2009)

3. RESULTS AND DISCUSSION

3.1. Optimization of Palm Kernel Seed Oil Extraction

The experimental design was used to determination of results and values using design Expert Version 11.1.0.1 software for optimizing the extraction of palm kernel oil the seed using n-hexane. The experiment was designed on three levels of factors that generated 17 experimental runs. The three independent factors were sample weight, solvent volume, and extraction time. Tables 1a and 1b summarized the experimental design pattern of the variables.

Table 1a-Factors and their Levels for Box - Behnken Design

Variable	**Symbol**	**Coded factor levels**		
		-1	**0**	**+1**
Extraction time (h)	Y_1	30	40	50
Solvent volume (ml)	Y_2	150	175	200
Sample weight (g)	Y_3	40	50	60

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Table 1b-Box-Behnken Experimental Design for Three Independent Factors

Std run	Y_1	Y_2	Y_3
1	30	150	50
2	50	150	50
3	30	200	50
4	50	200	50
5	30	175	40
6	50	175	40
7	30	175	60
8	50	175	60
9	40	150	60
10	40	200	40
11	40	150	40
12	40	200	60
13	40	175	60
14	40	175	50
15	40	175	50
16	40	175	50
17	40	175	50

Tables 2a and b shows that the predicted values for the solvent were close to the experimented values obtained from the laboratory.

Table 2a-Box-Behnken Experimental Design for Three Independent

Std	Y_1	Y_2	Y_3	PKO Oil yield % (w/w)	Predicted value	Residue
1	30	150	50	30.92	30.88	0.0425
2	50	150	50	36.86	36.86	-0.0037
3	30	200	50	35.97	35.92	0.0463
4	50	200	50	35.72	35.76	-0.0350
5	30	175	40	31.56	31.52	0.0350
6	50	175	40	37.47	37.48	-0.0060
7	30	175	60	37.60	37.68	0.0812
8	50	175	60	37.47	37.48	-0.0060
9	40	150	40	33.08	34.91	-0.0425
10	40	200	40	36.31	33.12	-0.0463
11	40	150	60	34.84	36.36	-0.0812
12	40	200	60	34.62	34.92	0.0038
13	40	175	50	33.07	34.58	0.0038
14	40	175	50	37.47	33.07	-0.0060
15	40	175	50	37.47	37.48	-0.0240
16	40	175	50	37.50	37.48	0.0240
17	40	175	50	32.99	33.03	-0.0387

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Table 2b-Regression coefficient and significance of response surface quadratic

Factor	Coefficient estimate	df	Standard error	95%CL Low	95%CL High	VIF
Intercept	15.23	1	0.2615	14.61	15.84	1
Y_1	1.92	1	0.0947	1.70	2.15	1
Y_2	2.19	1	0.0947	1.97	2.41	1
Y_3	26.26	1	0.3528	25.42	27.09	1
Y_1Y_2	1.17	1	0.0312	1.09	1.24	1
Y_1Y_3	-1.60	1	0.0609	-1.74	-1.46	1
Y_2Y_3	-1.26	1	0.0609	-1.41	-1.12	1
Y_1^2	-2.65	1	0.0304	-2.72	-2.58	1
Y_2^2	0.1445	1	0.0304	0.0725	0.2165	1
Y_3^2	-7.64	1	0.1157	-7.91	-7.36	1

3.2. Analysis of Regression of Oil Yield

The coefficient of determination ($R^{2)}$ measures the percentage of variation in the value of the dependent variable which is explained through the variation of independent variable. The R^2 was 99.97% while R^2 (adj) was 99.92%, the two (R^2) shows a high consistency between the experimented values and the predicted values as seen in table 3(a) and (b). The R^2 for the solvent showed average stability between the experimented values and the predicted values with 0.0013 the lack of fit. The variance analysis of the regression model in tables 4(a) and 4(b), gives a clear significance due to the F-value for lack of fit which is *2384.79*. The results seen disclosed that high extraction time and low solvent volume are ideal for an increase in the oil yield percentage for extraction of sample weight of 40g, and solvent volume of 175 ml, and 50 min of extraction time as seen in the table

Table 3a-ANOVA for surface quadratic Model of Variance

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	82.43	9	9.16	2348.79	< 0.0001	significant
A-SW (g)	1.61	1	1.61	412.11	< 0.0001	
B-SV (ml)	2.08	1	2.08	534.70	< 0.0001	
C-ET (min)	21.61	1	21.61	5541.20	< 0.0001	
AB	5.45	1	5.45	1398.26	< 0.0001	
AC	2.69	1	2.69	689.77	< 0.0001	
BC	1.68	1	1.68	430.09	< 0.0001	
A^2	29.52	1	29.52	7571.59	< 0.0001	
B^2	0.0879	1	0.0879	22.55	0.0021	
C^2	16.98	1	16.98	4353.90	< 0.0001	

Table 3b-Analysis of Variance (ANOVA) of regression

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	82.43	9	9.16	2348.79	< 0.0001	Significant
Residual	0.0273	7	0.0039			
Lack of Fit	0.0266	3	0.0089	49.21	0.0013	Significant
Pure Error	0.0007	4	0.0002			
Cor Total	82.45	16		**Cor Total**	82.45	16
$R^2 = 0.9997$	AdjR^2 = 0.9992	Predicted R^2 = 0.9948				

The contour and response surface plot showed the effect of keeping the palm kernel seed weight at the constant of 40g in Figure 3. The curves show the interaction between the variables, the elliptical curve indicates good interaction of the two variables and a

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circular curve indicates no interaction between the variables. The equation for the response in terms of coded factors for the Box-Behnken surface quadratic model is given as

$\%OY\ \ N-HEXANE = 15.23 + 1.92X_1 2.19X_2 + 26.26X_3 + 1.17X_2 - 1.60X_1X_3 - 1.26X_2X_3 - 2.65{X_1}^2 + 0.1445X_2^2 - 7.64{X_3}^2$ ---------------- 8

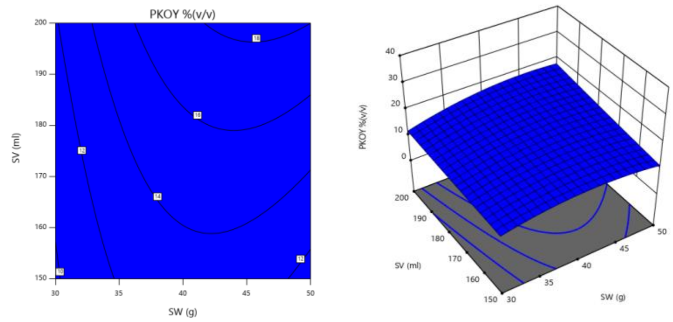

Fig. 3-The Contour and 3D Response Surface Plots for the Effects of Solvent Volume, Sample Weight and Their Reciprocal Interaction on Oil Yield at Constant Solvent Volume

The actual values for the solvent- n-hexane and the predicted values are seen in the graph of Fig. 4, and it shows the predicted value of the RSM was close to the percentage of oil yield gotten.

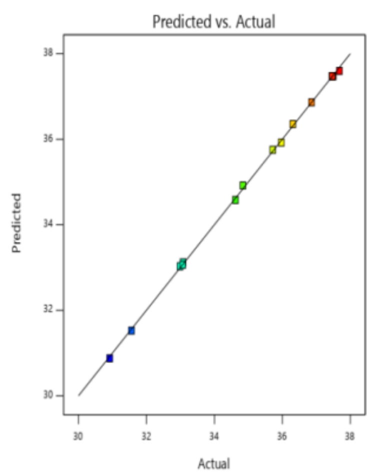

Fig. 4-A graph of predicted VS actual of RSM

The contour and 3D response surface plots are graphical representations of the interactive effects of the three variables. The highest oil yield was observed at the lowest sample weight in Fig. 5. It reveals that the highest solvent volume was when the highest oil yield was gotten, meaning that solvent volume has so much significant in the percentage of oil extracted.

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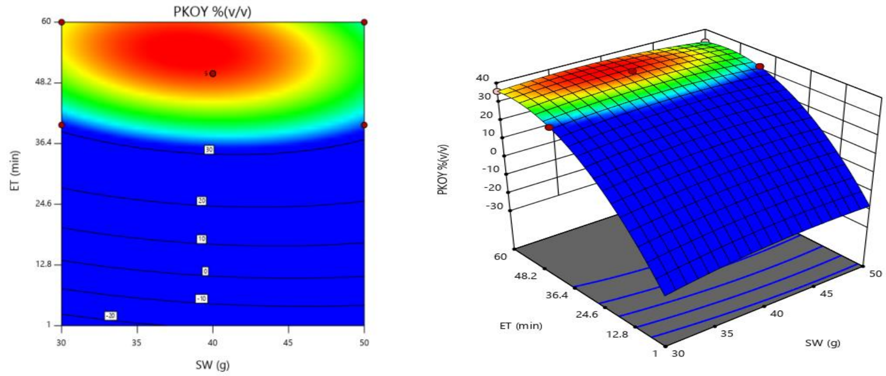

Fig 5-The Contour and 3D Response Surface Plots for the Effects of Extraction Time, Sample Weight and their Reciprocal Interaction on Oil Yield at Constant Solvent Volume

The impact of extraction time and solvent volume and their inverse interaction on oil yield keeping sample weight at zero levels is indicated in Figure 6. A statistical model (RSM) predicted that the optimal yield of palm kernel seed would be 37.68% following optimized conditions: sample weight 40g, the solvent volume of 175ml, and extraction time of 50 min. The results of this study also demonstrate that RSM software can be used for optimization of the process variables in oil extraction.

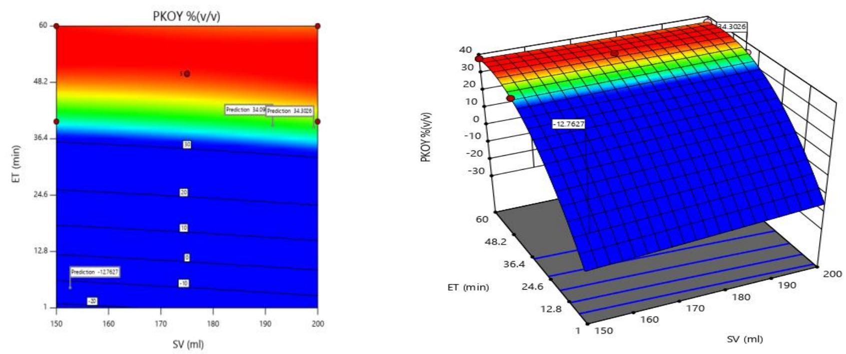

Fig 6-The Contour and 3D Response Surface Plots for the Effects of Extraction Time, Solvent Volume and Their Reciprocal Interaction on Oil Yield at Constant Sample Weight

3.3. Quality Characterization of Palm Kernel Oil

The physiochemical properties of the oilseed were carried out to determine the quality of oil yield as seen in table 4

.

Table 4-Physiochemical analysis and other properties of *Elaeis guinesis* kernel seed oil

Parameters	Mean values
Physical properties	
Physical state	yellowish-brown
Density	912.5
Moisture content (%)	0.043
Specific gravity	0.9125
Viscosity (mPa.s)	0.3631
Mean molecular mass	399.65
Chemical properties	
%FFA	**11.08**
Acid value (mg KOH/g oil)	22.16
Saponification value (mg KOH/g oil)	140.123

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Iodine value (g I_2/100g oil)	87.85
Peroxide value (meq o_2/kg oil)	69
Higher heating value (MJ/Kg)	30.51
Other properties	
API	23.56
Cetane number	65.449
Aniline point	326.86

3.4. Physical Properties of the Seed Oil

The oil extracted is yellowish-brown, with low protein content indicating that the nut is not suitable for animal feed or to improve nutritional values. The moisture content of 0.043, specific gravity of 0.9125, and fat content of 42% conform to the result of (Akpanabiatu *et al.*, 2000) who reported 7.2% - 6.0% range moisture content, 49 - 42% range for fat

3.5. Chemical Properties of the Seed Oil

The results in table 4. include the chemical properties of *Elaeis guinesis*is kernel oil. A low iodine value of 87.85g of I_2/100g of oil indicates a low level of unsaturation in the oil or a higher level of saturation. The high value for saponification 140.123(KOH/goal) shows that the oil can be used for soap production (i.e. High concentration of triglycerides). The peroxide value of 69(me of O_2/kg) in table 4 detected in this analysis is a good property that is more impervious to oxidation, with low shelf life. It measures the contents of hydroperoxide in the oil. High FFA content of (11.08%) and the high acid value of 22.16 showed that the oil is not edible and also has low shelf life.

5. CONCLUSION

Elaeis guinesis kernel oil is extracted and optimized using n-hexane and RSM. It was discovered that at the highest solvent volume, the highest oil yield was gotten, meaning that solvent volume has so much significance in the percentage of oil extracted, and the sample weight and the extraction time have reciprocal interaction with oil yield keeping the solvent volume constant. Likewise, the RSM software can be used for the optimization of the process variables in oil extraction. The physicochemical analysis of the yellowish-brown oil that is low in iodine and high in saponification, couple with good oxidative properties, the high FFA, and low shelf life, makes it valuable for commercial, (soap-making) medical use and domestic consumption. Since Oil palm is globally available, Palm Kernel oil can be extracted and optimized through this method to meet its global demand.

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Conflict of Interest

The author declares that there are no conflicts of interests.

Data and materials availability

All data associated with this study are present in the paper.

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