International Journal of Engineering and Information Systems (IJEAIS)
ISSN: 2643-640X
Vol. 5 Issue 9, September - 2021, Pages: 35-43

Maximum Extraction Of Palm Kernel Oil Using Ethanol In Response Surface Methodology And Artificial Neural Networks Methods.

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Abstract: *The nutritional and medical use of palm kernel oil is in high demand and has ignited the search for extraction of the oil at the optimum. The study investigated the use of ethanol as solvent, constituting one of the independent factors used in the research, which are; Sample Weight, Solvent Volume, Extraction Time to extract oil. An experimental design was employed to optimize the oil extraction in the Box-Behnken design, and the physiochemical characteristics of the extracted oil were obtained. 17 experimental runs were generated through Response Surface Methodology (RSM) and Artificial Neural Networks (ANN) methods. The results reflected that low solvent volume and high extraction time are required for an increase in the oil yield percentage for extraction of 40g weighted sample, 175 ml solvent volume, and 50 min extraction time. RSM predicted that the optimal yield of palm kernel seed to be 36.67 % while ANN predicted a yield of 37.693 % at the optimum sample weight of 40 g, the volume of 150 ml, and extraction time of 60 min. The results demonstrated that RSM and ANN software is effective for optimization oil extracted by ethanol, with a higher percentage of yields from ANN. The properties of the oil revealed yellowish-brown oil at room temperature, a fat content of 42%, and an Iodine value of 87.85(I2g/100g oil), the Saponification value of 140.125 (mg KOH/g Oil), with low acid and high FFA. Hence the extracted oil can be used for medical and cosmetic purposes.*

Keywords: Extraction; Oil Yield; Optimization; Ethanol; Box-Behnken design; Physiochemical properties**.**

1. INTRODUCTION

An average of 12 bunches of fruit weighing about 15-25 are produced by oil palm trees on per hectare basis [1]. The fruits produce both palm oil and palm kernel oil which is edible plant oils [2]. Palm oil is derived from the pulp of the fruit of oil palm (*Elaeis guineensis)* while palm kernel oil is gotten from Palm kernel seed PCSS) of the oil palm [3]. Palm oil is used for the production of margarine and compounds in cooking fats and oils for the production of; candles, detergents, soap, and cosmetic products. Meanwhile the Production of palm kernel oil is about 12% of the production of its palm oil [4], which is an important source of fat, used in food processing industries, soap making, cosmetics, pharmaceutical industries, and traditional medicine in many rural areas and as lubricant or biodiesel [5].

The Palm Kernel Oil can be extracted by using; hydraulic and screw presses, mechanical expression, solvent extraction, and others. The solvent extraction maximizes the remover of virtually all oil available has compare to other method from the seed [6]. Solvent extraction is both the polar and non-polar which are used for extracting oils. Hexane is a non-polar solvent used extensively for extracting oil from seed oils [7], because it has to low boiling point, though it is hazardous nature. It is a solvent gotten from petrochemical sources, and can be emitted during extraction and recovery; it is identified as air pollutant for it can react with other pollutants to produce ozone and photochemical oxidants [8, 9]. However, for safety in health and environment, interest to find alternative solvents instead of hexane is on increase.

The adequate replacement for hexane as solvent in extraction is adjudged to be the organic solvents recognized as environmentally friendly [10]. Researches carried out on the extraction of oil from the cottonseed have shown that hexane can be replaced with other hydrocarbons [11, 12] or alcohols [13, 14, 15] as solvents. For the hydrocarbon as a replacement, heptane and isohexane were investigated to be an adequate replacement for hexane to extract oil from cottonseed [8,12]. In the case of the use of alcohols, isopropanol and ethanol appropriate solvents for extracting oil from cottonseed [13], sunflower seed [15] and soybean [16, 14]. Out of the two organic substitutes ethanol was investigated as an alternative solvent since it is cost-effective, can be produced from varieties of biological matters. Moreover, this alcohol is known to be non-toxic and handling risks are less in comparing with hexane [14]. It can be gotten through fermentation, its use as a solvent without toxicity problems of meals for animal feedstuff [10]. Having applied ethanol as solvent with successes extraction of oil on some oilseeds, it can be also employed to extract oil from the palm kernel seed which is the proposition for this study.

The extracted oil is needed to be optimized. to maximize the oil production from the palm kernel seeds, therefore Response Surface Methodology (RSM) and Artificial Neural Networks (ANNs), is investigated to extract optimum oil from the Palm kernel seeds, these approaches are said to be important in modeling and optimization, the modeling evaluate the relationship between the output and input variable of the process using experimentally derived data [17]. As a result, the models are required to predict the optimum status of independent variables to minimize or maximize the target variable [18].

RSM is introduced in studies based on polynomial functions in the 1980s and it has been extensively and continually employed in modeling and optimization [17]. RSM is effective in determining response from independent variables and the interactions between them. It is very important in experimental runs to provide a statistically significant result. RSM analyses variables, generates a mathematical model to ascertain the optimum condition for investigating the influencing factors with graph perspective [19]. The shape of the surfaces of the graph generated by a mathematic model allows a visual understanding of the functional relationship that exist between the response and the variables [20] RSM provides efficient and accurate solutions even as a simple and efficient tool, it has, therefore, been successfully employed in proffering solution to different engineering problems over decades [21, 22].

ANNs are seemly new nonlinear statistical and computational technique developed to proffer solution to problems that are not easily solved by conventional statistical methods [17] and are developed based on neurons present in biological neural system [23]. It can handle obscure, complex, incomplete problem and execute modeling to produce predictions and generalizations at high speed. They can be used for predictions and data fitting once trained [17]. ANNs can be used to decode complicatedreal-world problems using statistical approaches without the need for complicated equations, and is capable of exploring regions that are otherwise omitted when using statistical approaches [24, 25, 26, 27, 28]. ANNs have been applied to some studies with success [29, 30].

Since both RSM and ANN techniques do not need the precise expressions or the physical meaning of the system under investigation [22]. And have being previously used successfully, this study investigates the suitability of both methods on maximizing the oil extracted using ethanol as solvent, and to determine the physical and the chemical properties of the oil extracted.

2. MATERIALS AND METHODS.

Palm kernel oil extraction and optimization from the s*eeds* collected from the fields follow the methods described as follows:

2.1 Equipment and Reagent

The equipment that was used includes: Muslim Bag, Soxhlet Extractor of 500 ml, Digital Weighing balance, Heating Mantle, Water Bath, and Oven. Flash Point Machine, Spectrometer, Viscometer, Glassware which includes beakers, round bottom flask, conical flasks,pycnometerr, Petri dish, Measuring cylinder and burettes. The analytical reagents used are; Ethanol, Potassium Iodide (KI), Phenolphthalein, Iodine, Chlorine, HCL, KOH, NaO,H and they are all obtained from BDH Chemical Ltd., Poole England

2.2 Seeds preparation

Palm kernel nuts collected from processed palm fruits in villages within Akwa Ibom state, were cracked from the shells manually, separated from the broken shells, they were sundried for 2 days, crushed and then grinded.

Figure 1. Palm Kernel Seeds inside a a Muslin Bag

Figure 2. A Crushed Palm Kernels Seeds Cracker

2.3 Extraction Procedures

A-500ml Soxhlet extractor was used for the work, with ethanol as solvent, and a known weight of palm kernel seed powder in a Muslim bag placed on the heating mantle. The temperature turned on slightly below the boiling point of the solvents. The solvents

International Journal of Engineering and Information Systems (IJEAIS)
ISSN: 2643-640X
Vol. 5 Issue 9, September - 2021, Pages: 35-43

are recycled and the oil was left in the round bottom flask which was later weight using a weighing balance. The oil yield was calculated as follows:

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

2.4 Experimental Design for the Extraction of oil from Palm Kernel Oilseed.

The experiment to optimize the palm kernel oilseeds extraction employed Box-Behnken experimental design, which was applied to produce 17 experimental runs. The factors for the extraction of oil from the palm kernel oilseeds are extraction weight (g): X_1, solvent volume (ml): $X_{,2}$ and sample time (min): X_3

2.5 Properties of the Extracted Kernel Oil

The physical and chemical properties of the palm kernel oil are analyzed as which includes: the moisture content, Viscosity, Acid Value, *Free Fatty Acid(FFA), and Saponification value,* Density, Specific gravity, Peroxide Value, Iodine value, Mean Molecular Mass**,** the procedural processes described in Association of Official Analytical Chemist [31, 32]. The Mean Molecular Mass was determined by the equation cited by Akintayo and Bayer [33]. The calculation of other properties 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* [34].

3. RESULTS AND DISCUSSION

3.1 Extraction and Optimization of Palm Kernel Seed Oil Extraction

Experimental design was employed in determination of results and values using design Expert Version 11.1.0.1 software for optimizing extraction of palm kernel oil from the seed using Ethanol. 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. The summary of the experimental design pattern of the variables is shown in Table 1a and 1b

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

Variable	Symbol	Coded factor levels		
		-1	0	+1
Extraction time (h)	X_1	30	40	50
Solvent volume (ml)	X_2	150	175	200
Sample weight (g)	X_3	40	50	60

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

Std run	X_1	X_2	X_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

It was discovered that the predicted values of RSM and ANN using ethanol as solvent was closely related to the experimented values obtained from the laboratory as seen in table 2.

Table 2a: Box-Behnken Experimental Design for Three Independent Factors for Ethanol, oil yield, predicted and residual values of RSM and ANN

Std	X_1	X_2	X_3	PKO Oil yield % (w/w)	Predicted value(RSM)	Residue (RSM)	Predicted value(ANN)	Residue (ANN)
1	30	150	50	26.01	26.21	-0.1975	26.008	0.0016445
2	50	150	50	32.55	32.55	0.0025	32.552	0.0016984
3	30	200	50	33.30	33.51	-0.2050	33.299	0.0010548
4	50	200	50	29.57	29.57	0.0000	29.568	0.0018724
5	30	175	40	26.70	26.50	0.2050	26.702	0.0024643
6	50	175	40	29.57	29.57	0.0000	29.57	0.00041155
7	30	175	60	26.70	26.50	0.2025	26.705	0.0051787
8	50	175	60	29.57	29.57	0.0000	29.57	5.8986E-5
9	40	150	40	36.67	36.47	0.1975	36.663	0.0068047
10	40	200	40	36.42	36.42	0.0050	36.429	0.0085301
11	40	150	60	35.00	35.00	-0.0025	34.999	0.00051272
12	40	200	60	33.61	33.62	-0.0050	33.611	0.001284
13	40	175	50	33.11	33.10	0.0073	29.581	3.5289
14	40	175	50	30.03	30.23	-0.2023	29.581	0.4489
15	40	175	50	29.57	29.37	0.0000	29.581	0.0111
16	40	175	50	25.63	25.64	-0.0073	29.581	3.9511
17	40	175	50	29.57	29.57	0.0000	29.581	0.0111

Table 2b: Regression coefficient and significance of response surface quadratic for Ethanol.

Factor	Coefficient estimate	Df	Standard error	95%CL Low	95%CL High	VIF
Intercept	29.57	1	0.0835	29.37	29.77	
X_1	1.55	1	0.0660	1.40	1.71	1.00
X_2	3.18	1	0.0660	3.02	3.34	1.00
X_3	-1.95	1	0.0660	-2.11	-1.80	1.00
X_1X_2	0.8825	1	0.0934	0.6616	1.10	1.00
X_1X_3	3.01	1	0.0934	2.79	3.23	1.00
X_2X_3	1.22	1	0.0934	0.9966	1.44	1.00

X_1^2	0.1888	1	0.0910	0.0315	0.3990	1.01
X_2^2	-0.2662	1	0.0910	-0.4815	-0.0510	1.01
X_3^2	3.25	1	0.0910	3.04	3.47	1.01

The coefficient of determination (R^2) was 99.89 while R^2 (adj) was 98.23% and 99.89%The two (R^2) shows a high consistency between the experimented values and the predicted values as seen in table 3(b). The R^2 for the solvent showed average stability between the experimented values and the predicted values. The analysis of variance of regression model in table 3(a) and (b), gives a clear significance due to the F-value for lack of fit which is 703.86. The results reflected high extraction time and low solvent volume is required for an increase in the oil yield percentage for an extraction of sample weight of 40g, and solvent volume of 175 ml, and extraction time of 50 min. as seen in table 1b.

Table 3(a): ANOVA for surface quadratic Model of Variance table for the solvent

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	221.04	9	24.56	703.86	< 0.0001	significant
A-SW (g)	19.28	1	19.28	552.61	< 0.0001	
B-SV (ml)	80.90	1	80.90	2318.50	< 0.0001	
C-ET (min)	30.50	1	30.50	874.05	< 0.0001	
AB	3.12	1	3.12	89.28	< 0.0001	
AC	36.18	1	36.18	1036.89	< 0.0001	
BC	5.93	1	5.93	169.93	< 0.0001	
A^2	0.1422	1	0.1422	4.07	0.0833	
B^2	0.2985	1	0.2985	8.55	0.0222	
C^2	44.58	1	44.58	1277.52	< 0.0001	

Table 3b: Analysis of Variance (ANOVA) of regression for Ethanol solvent.

Source	Sum of Squares	df	Mean Square
Model	221.04	9	24.56
Residual	0.2443	7	0.0349
Lack of Fit	0.2443	3	0.0814
Pure Error	0.0000	4	0.0000
Cor Total	221.28	16	
$R^2 = 0.9989$	$AdjR^2 =$ 0.9823	Predicted $R^2 = 0.9989$	

Figure 4 shows the graph of the predicted and the actual values for RSM for ethanol (ANN). It was noted that the predicted values and actual values are directly related.

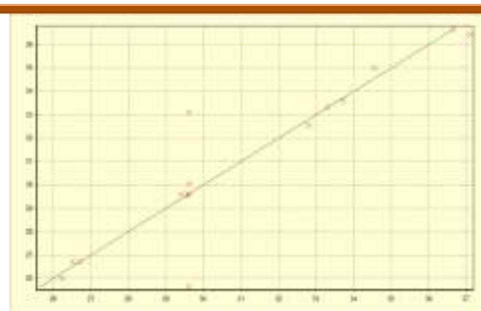

Figure 4: A graph of predicted VS actual of RSM for Ethanol (ANN)

The contour and 3D response surface plots are graphical representations of the interactive effects of the three variables. The contour and response surface plot showed the effect of keeping the palm kernel seed weight at the constant of 40g in Figure 5, which shows a reciprocal interaction with other variables. The equation for the response in terms of coded factors for the Box-Behnken surface quadratic model is given as:

$$\%OY\ ETHANOL = 29.57 + 1.55X_1 + 3.18X_2 - 1.95X_3 + 0.8825X_1X_2 + 3.01X_1X_3 + 1.22X_2X_3 + 0.1838{X_1}^2 - 0.2662{X_2}^2 + 3.25{X_3}^2 \text{ -----------2}$$

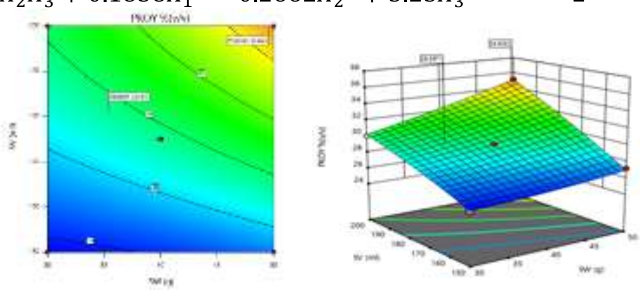

Fig 5: The Contour and 3D Response Surface Plots for the Effects of Solvent Volume, Sample Weight and their Reciprocal Interaction on oil Yield Keeping Sample Weight Constant at Zero Level

In Figure 6. The contour and 3D response surface graph at constant (zero) level of Ethanol; was observed as extraction time had so much effect on the oil yield, that is the lower the time the lower oil yield the higher the time the higher the oil yield.

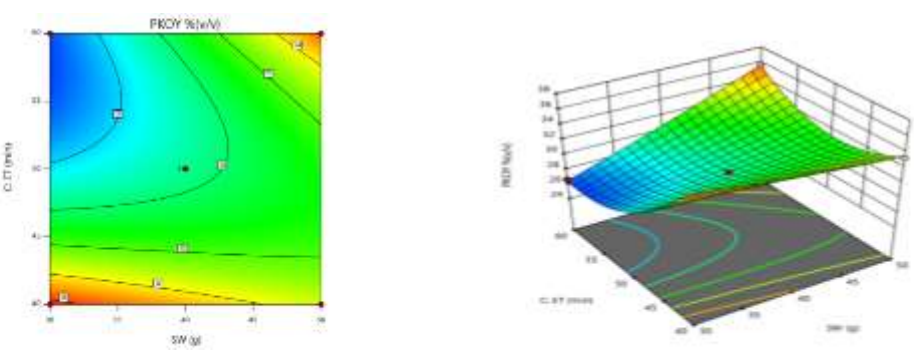

Figure 6: The Contour and 3D Response Surface plots for the Effects of Extraction Time, Sample Weight, and Their Reciprocal Interaction on Oil Yield Keeping Solvent Volume Constant at Zero LEVE.

The contour and 3D Response surface plots for the effect of extraction time and solvent volume has a reciprocal interaction on oil yield keeping sample weight at zero levels as seen in figure 7

International Journal of Engineering and Information Systems (IJEAIS)
ISSN: 2643-640X
Vol. 5 Issue 9, September - 2021, Pages: 35-43

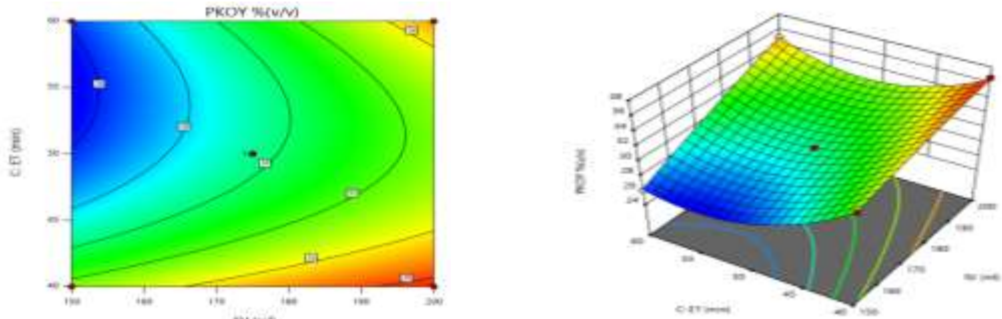

Figure 7: The Contour and 3D Response Surface Plots for The Effects of Extraction Time, Solvent Volume and Their Reciprocal Interaction On Oil Yield Keeping Sample Weight Constant at Zero Level for Ethanol

Altogether, the statistical model (RSM) predicted that the optimal yield of palm kernel seed was 36.67 % for Ethanol with the solvent volume of 175ml and extraction time of 50 min for the optimized conditions. Using these optimal factor values under experimental conditions in two independent replicates an average was 36.045% for ethanol, and this value was well within the range predicted by the model, and ANN predicted yield of 37.693 % at the following optimized conditions sample Weight (g) of 40 g, the solvent volume of 150 ml and extraction time of 60 min, as seen in table 8.

Fig 8: The Contour and 3D Response Surface Plots for the Effects of Independent Variables and their Relationships on Oil Yield at Constant at Zero level for Solvent Volume (ANN).

The results of this study demonstrate that RSM and ANN software can be effectively applied to the optimization of the process variables in oil extraction, with a higher percentage of yield from ANN.

3.2 Quality Characterization of Palm Kernel Oil

The physical and chemical properties of the oilseed were determined to be certain of the quality of oil to inform its possible uses, as seen in table 4.

Table 4: physical-chemical analysis and other properties Extracted 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
Iodine value (g l_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

4.2.1 Physical Properties of the Seed Oil

The color of 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%

4.2.2 Chemical Properties of the Seed Oil

The chemical properties of *Palm* kernel oil in table 4 shows; Low iodine value of 87.85g of I_2/100g of oil indicates a higher level of saturation. The high value for saponification 140.123 (KOH/g oil) shows that the oil can be used for soap production. 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 has a low shelf life.

4 CONCLUSION AND RECOMMENDATION

Using ethanol as solvent and RSM and ANNs to maximize oil yield from palm kernel seeds; the studied carried out discovered that the highest oil was extracted at the highest solvent volume, indicating that volume has so much significance in the percentage of oil yield. However, the low solvent is appropriate for the high yield of the oil because the extraction time and sample weight have reciprocal interactions on oil yield; keeping solvent volume constant at zero. It was also discovered that RSM and ANN software can be applied to the optimization of the process variables in oil extraction, while a higher percentage of yield gotten from ANN. The physical and chemical properties of the yellowish-brown extracted oil with good oxidative properties, high FFA, and low shelf life are valuable nutritionally and medically and can be used to meet domestic and industrial demands.

5 ACKNOWLEDGEMENTS

The authors acknowledge Okonkwo, Ezekiel. O for his efforts in collecting the sample seeds and commitment to assisting in laboratory works to carry out the research; the staff of Akwa Ibom State University's Laboratory is appreciated for their service.

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