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MODELING AND OPTIMIZATION OF EXTRACTION OF OIL FROM *SESAMUM INDICUM* SEEDS: A CASE STUDY OF RESPONSE SURFACE METHODOLOGY VS. ARTIFICIAL NEURAL NETWORK

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Abstract

In this work, response surface methodology (RSM) and artificial neural network (ANN) was used to optimize of oil from Sesamum indicum seeds. ANN predicted optimal condition for extraction was Sesamum indicum powder weight (SIPW) = 54.71 g, extraction time (ET) = 44.88 min and solvent volume (SV) = 165.8 mL. The predicted Sesamum indicum oil yield (SIOY) was validated as 85.70% (w/w) while RSM predicted optimal condition was Sesamum indicum powder weight (SIPW) = 60.00 g, Extraction time (ET) = 44.48 min and solvent volume (SV) = 150 mL. The predicted SIOY under this condition was validated as 83.20% (w/w). The result obtained showed that ANN was superior and more effective optimization tool than RSM owing to its value of RMSE, AAD, R^2, R^2_{Adj}. Meanwhile, the qualities of Sesamum indicum oil yield (SIOY) as compared to the earlier researched works indicated that the oil produced is of good qualities and needs no further purification. Fatty acids profile reflected that the oil is highly unsaturated. The study concluded that the oil is not only edible, but also could have an industrial application.

Keywords: Optimization, Response surface methodology, Artificial neural network, Fatty acid profile, *Sesamum indicum* oil.

Contribution/ Originality

The paper's primary contribution is finding that Sesamum *indicum* seed is not only for food consumption but could also be used as oil source for industrial application. This study documents the supremacy of ANN over RSM.

1. INTRODUCTION

Literature survey showed that the use of statistical software tools to optimize process conditions is receiving more attention day by days. A classical modeling technique, such as

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response surface methodology (RSM) is a statistical software tool which has been used extensively in laboratory as well as industries. It major advantage is the capability to minimize the number of standard experimental runs, required to provide classically acceptable results [1]. In view of this, Betiku and Adesina [1] worked on methanolysis optimization of sesame oil to biodiesel and fuel quality characterization using RSM. Jeong, et al. [2] carried out research on optimization of transesterification of animal fat ester using RSM. In the same vein, Fan, et al. [3] worked on the biodiesel production from crude cottonseed oil using RSM. Statistical approach to the optimization of citric acid production using filamentous fungus *Aspergillus niger* grown on sweet potato starch hydrolyzate using RSM was carried out by Betiku and Adesina [1]. Adepoju, et al. [4] applied the same tool in optimization of oil extraction from Chrysophyllum albidium oilseeds and its quality characterization. Artificial neural network (ANN) a similar statistical software, is a learning system based on a computational technique that can simulate the neurological processing ability of the human brain and can be applied to quantify a non-linear relationship between connecting factors and actual responses by means of iterative training of data obtained from a designed experiment Achanta, et al. [5]. ANN shows superiority as a modeling technique for data sets showing non-linear relationships, and thus for both data fitting and prediction abilities [6, 7]. It has been used to solve myriads of problems in the field of medicine, metrology, neurology, biology, phycology, science, mathematics and engineering [8]. Based on this findings, Ghaffari, et al. [9] worked on performance comparism of neural network training algorithms in modeling of biomodal drug delivery, Gueguim Kana, et al. [10] optimized biogas production from sawdust using ANN, whereas Rajendra, et al. [11] applied the same tool to predicts the pretreatment process parameters for biodiesel production, Adepoju, et al. [12] optimize transesterification of *chrysophyllum albidium* seed oil to *chrysophyllum albidium* oil biodiesel using artificial neural network. However, Ghorbani, et al. [13] compared the performance of ANN and RSM in prediction and optimization of biodiesel production and reported that ANN offers a promising outlook in the estimation of the optimum variables for biodiesel production, Adepoju and Olawale [14] also compared the performance of ANN and RSM for achieving desire benzene alcohol in the biotransformation of benzaldehyde using free cells of *Saccharomyces cerevisae* and the effect of β-cyclodextrin and reported that ANN methodology presents a better alternative than the RSM model. In this study, an effort was made to optimize the extraction conditions of oil extraction of *Sesamum indicum* seeds while comparing the performance of ANN and RSM.

2. MATERIALS AND METHODS

2.1. Materials

The seeds of *Sesamum indicum* were collected from Kabba, Nigeria. The dirty seeds were washed to remove the adherent dirty, sundried for six days and then winnowed to remove the chaffs. The cleaned seeds were then grinded into powdery form via grinding machine. All chemical and reagents used were of analytical grades factory-made by GFS Chemicals, Inc., 867

McKinley Ave., Columbus OH 43223 (99.7-100%) and BDH Analar Ltd., Poole England (99%) and supplied by FINLAB Nig. Ltd.

2.2 Methods

2.2.1. Sesamum *Indicum* Oil Extraction

A 250 ml Soxhlet extractor apparatus with n-hexane as a solvent was used for oil extraction. At first, the thimble of apparatus was charged with a known weight of the powder seed placed in a muslin cloth. The solvent containing part of the apparatus was filled with a known volume of n-hexane, fixed to the end of the thimble, and a condenser was tightly fixed at the bottom end of the extractor. The whole set up was placed in a temperature controlled heating mantle and heated up at temperature of 68-70 °C. Excess solvent in the extracted oil was recycled and recovered by distillation. This extraction was based on the box-behnken experimental design an allied of response surface methodology. The yield of *Sesamum indicum* oil yield (SIOY) was determined using Eq. (1).

$$SIOY = \frac{weight\ of\ oil\ produced}{SIPW\ used} \qquad (1)$$

2.2.1.1. Experimental Design for Sesamum *Indicum* Oil Extraction.

A three-level- three factors box-behnken design was employed in these modeling and optimization studies, which generated 17 experimental runs and were carried out. The independent variables considered for the optimization include *Sesamum indicum* powder weight (SIPW), extraction time (ET) and solvent volume (SV). The coded level independent variables are shown in Table 1, while the box-behnken and ANN experimental results are displaced in Table 2. The experimental results obtained were analyzed by the functional relationship between the response variable and the explanatory variable plus error term using the second-order polynomial (Eq. (2)), and artificial neural network model equations (Eq. (3), Eq. (4) and Eq. (5)).

$$Y = \beta_o + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \in \quad (2)$$

Y is the value of response corresponding (oil yield) to the value of X_1, X_2, X_3 of the explanatory variable, β_o is the intercept, $\beta_1, \beta_2,$ and β_3 are the linear coefficients, $\beta_{12}, \beta_{13}, \beta_{23}$ are interaction coefficients, while $\beta_{11}, \beta_{22}, \beta_{33}$ are the quadratic coefficients. $\in$ is the error term.

$$R^2 = 1 - \sum_{i=1}^{n} \frac{(X_{i,cal} - X_{i,exp})^2}{(X_{avg,exp} - X_{i,exp})^2} \qquad (3)$$

$$ADD = \left\{ \frac{1}{n} \left[\sum_{i=1}^{n} \left(\frac{X_{i,exp} - X_{i,cal}}{X_{i,exp}} \right) \right] \right\} \qquad (4)$$

$$RMSE = \sqrt{\frac{\sum(X_{i,cal} - X_{i,exp})}{n}} \tag{5}$$

Where n is the number of data set, $X_{i,cal}$ is the calculated values, $X_{i,exp}$ is the experimental values and $X_{avg,exp}$ is the average experimental values.

Table-1. Factors and their levels for Box-Behnken design.

Factor	Symbol	Coded factor levels		
		-1	0	+1
Sesamum *indicum* powder weight (SIPW) (g)	X_1	30	45	60
Extraction time (ET) (min)	X_2	40	50	60
Solvent volume (SV) (mL)	X_3	150	200	250

Table-2. Experimental results by Box-Behnken and

Std. run	X_1	X_2	X_3	Exp.SIOY% (w/w)	Pred. RSM % (w/w)	Res. RSM	Pred. ANN	Res. ANN
1	30	40	200	40.00	38.37	1.63	40.001	0.0014905
2	45	50	200	52.00	51.74	0.26	51.735	0.26456
3	60	50	150	40.40	39.10	1.30	40.474	0.9259
4	60	40	200	36.67	35.50	1.17	36.682	0.011603
5	45	50	200	52.00	51.74	0.26	51.735	0.26456
6	45	40	250	56.62	56.94	-0.32	56.618	0.001588
7	30	60	200	52.67	53.84	-1.17	52.67	0.00015391
8	30	50	150	25.00	24.16	0.84	25	7.652E-6
9	60	60	200	30.56	32.19	-1.63	30.48	0.9201
10	60	50	250	48.67	49.51	-0.84	48.666	0.0037221
11	45	50	200	51.56	51.74	-0.18	51.735	0.17544
12	45	40	150	28.67	31.14	-2.47	28.669	0.0007581
13	45	50	200	51.56	51.74	-0.18	51.735	0.17544
14	45	60	250	77.30	74.83	2.47	77.302	0.001852
15	30	50	250	87.67	88.97	-1.30	87.672	0.002052
16	45	50	200	51.56	51.74	-0.18	51.735	0.17544
17	45	60	150	25.72	25.40	0.32	25.721	0.0010908

2.3. Modeling and Optimization Studies by ANN and RSM

In this study, a 2.5 CPC-X Software NeuralPower new version was used. Multilayer Normal Feed Forward (MNFF) and Multilayer Full Feed Forward (MFFF) networks were used for predictive accuracy of oil and biodiesel yields. Search approach used was force approach. The two networks were trained by the QuickProp (QP) learning algorithms and default stopping of 1,187,300 iterations. For the input layer, three total layer numbers were used and the node number of input layer was three (3). For the output layer, Node Number was one (1), the transfer function was Tanh and the slope of transfer function and the hidden layer was also one (1). Meanwhile, the node number of output layer was sixteen (16) with Tanh transfer function. The slope of transfer function was also one. Similarly, for the RSM, a new design expert version 9.0.1 was used. Based on box-behnken data was divided into training and testing data sets (Table xxx).

Thirteen (13) out of seventeen (17) experimental obtained data were used as the training set while the remaining four (4) were used for the testing set.

2.4. Analysis of Qualities of the Oil and Free Fatty Acid Compositions

Oil qualities such as refractive index, moisture content, viscosity at 40 °C, acid value, saponification value, peroxide value, specific gravity, % FFA, cetane number etc. of the SIOY was carried out by AOAC [15] methods, the mean molecular mass was obtained by the method of Akintayo and Bayer [16], the higher heating value was determined by Demirbas [17] method, iodine value was obtained by Wijs method. The free fatty acid of the SIOY was determined using gas chromatography (HP 6890 powered with HP ChemStation Rev. A 09.01 [1206] Software). 50 mg oil was esterified for five minute at 95 °C with 3.4 ml of the 0.5 M KOH in dry methanol. The mixture was neutralized using by adding 0.7 M HCl and 3 ml of 14% boron triflouride in methanol. The whole mixture was heated for 5 min at the temperature of 90 °C to achieve complete process. The fatty acids were thrice extracted from the mixture with redistilled n-hexane. The content was concentrated to 1 µl for gas chromatography analysis (GC) with 1 µl was injected into the injection port of GC.

3. RESULTS AND DISCUSSION

3.1. Modeling and Optimization of *Sesamum Indicum* Oil Extraction by RSM

Presents in Table 2 are the results of experimental runs carried out, these results include the actual variables, the experimental, the predicted as well as residual value obtained. Fig. 2, showed the graph of RSM experimental against the predicted values.

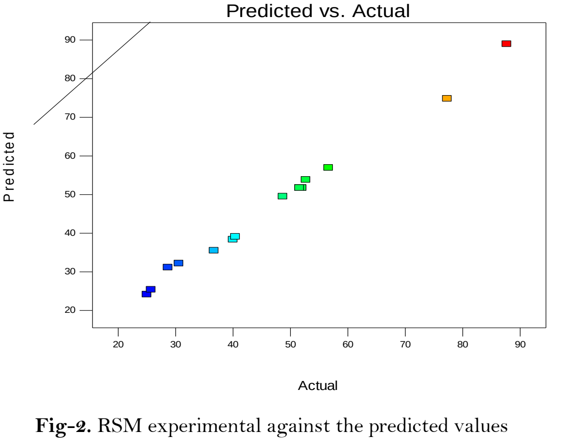


Fig-2. RSM experimental against the predicted values

Based on the constraints and the single objective function, the model equation that correlate the controllable variables and the single objective function (response) is given in Eq. (1).

$$SIOY = 51.74 - 6.13X_1 + 3.04X_2 + 18.81X_3 - 4.70X_1X_2 - 13.60X_1X_3 + 5.91X_2X_3 - 4.40X_1^2 - 7.56X_2^2 + 2.90X_3^2 \quad (6)$$

The usefulness of Eq. (6) is to carry out the ANOVA test, test of significant as well as determining the simple linear regression model parameters such as coefficient of determination (R^2) and ($R^2_{adj.}$)). The results of test of significance and ANOVA test were presented in Table 3. Based on the results, all the variables are significant at 95% CI level (p<0.05). However, the variable X_3 with *F-test* value = 777.66, p<0.0001, is the most significant variable. The results of regression coefficient were also presented in Table 4. Observation shows that all the factors has negative coefficient of estimation effects on the response except X_1, X_2 and X_3^2 with positive effects on the model response (SIOY).

All the degree of fredom (df) associated with sum of the squares are unity (1), the standard error are all lesser than one, and the variance inflation factor ranges from 1.00 to 1.01 implied a high significance for the regression model [18]. The coefficient of determination (R^2) and $R^2_{Adj.}$ were evaluated as 99.44% and 98.72%, respectively. The variance activity that varies from a norm was determined as 1.858, while the square root of the variance called the standard deviation was obtained as 1.363.

Table-3. Test of significance for all regression coefficient terms

Source	Sum of squares	df	Mean Square	F-value	p-value
X_1	300.62	1	300.62	82.60	< 0.0001
X_2	73.75	1	73.75	20.26	0.0028
X_3	2830.15	1	2830.15	777.66	< 0.0001
X_1X_2	88.17	1	88.17	24.23	0.0017
X_1X_3	739.84	1	739.84	203.29	< 0.0001
X_2X_3	139.59	1	139.59	38.36	0.0004
X_1^2	74.34	1	74.34	20.43	0.0027
X_2^2	240.60	1	240.60	66.11	< 0.0001
X_3^2	35.43	1	35.43	9.74	0.0168
Analysis of variance (ANOVA) of regression equation					
Model	4523.44	9	502.60	138.10	< 0.0001
Residual	25.48	7	3.64		
Lack of fit	25.24	3	8.41	144.87	0.0002
Pure error	0.23	4	0.058		
Cor total	4548.92	16			

R^2 = 99.44%, R^2adj. = 98.72%

Table-4. Regression coefficients and significance of response surface quadratic

Factor	Coefficient estimate	df	Standard error	95% CI Low	95% CI high	VIF
Intercept	51.74	1	0.85	49.72	53.75	-
X_1	-6.13	1	0.67	-7.72	-4.54	1.00
X_2	3.04	1	0.67	1.44	4.63	1.00
X_3	18.81	1	0.67	17.21	20.40	1.00
X_1X_2	-4.70	1	0.95	-6.95	-2.44	1.00
X_1X_3	-13.60	1	0.95	-15.86	-11.34	1.00
X_2X_3	5.91	1	0.95	3.65	8.16	1.00
X_1^2	-4.20	1	0.93	-6.40	-2.00	1.01
X_2^2	-7.56	1	0.93	-9.76	-5.36	1.01
X_3^2	2.90	1	0.93	0.70	5.10	1.01

Fig. 2, showed the graphical representation of the 3D's. The curvature nature of the graphs proved that there are perfect interactions among the controllable variables. To establish the optima condition, twenty seven (27) variables combination was generated, the optimal variables condition for this process was established at twenty three (23) variable combinations. This was obtained by minimizing the controllable variables while maximizing the response variable (SIOY). The condition was established at X_1= 60.00 g, X_2 = 44.48 min cm and X_3= 150 mL. The predicted SIOY under this condition was 83.52 % (w/w). Using these optimal condition values for three independent replicates, a mean of 83.20 % (w/w) SIOY was achieved, which was within the range predicted by the model.

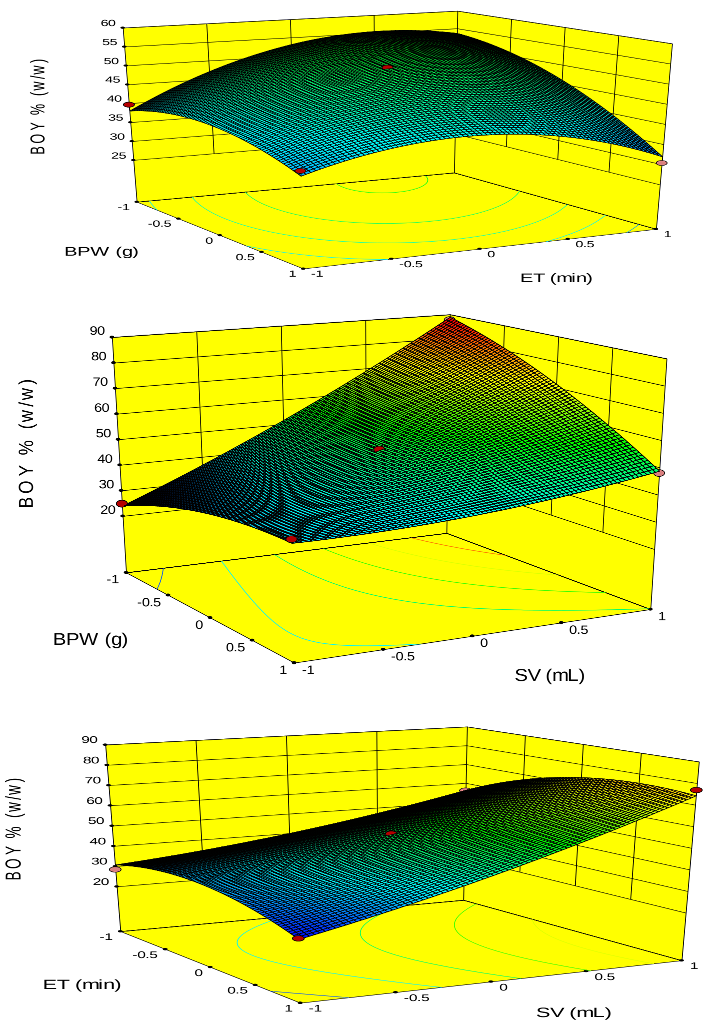


Fig-3. Graphical representation of the 3D's by RSM

3.1.1. Modeling and Optimization of *Sesamum Indicum* Oil Extraction by ANN

Similarly, the results of the experimental, the predicted values and the residual values are presented in Table 3. Fig. 3 shows the graph of predicted ANN against the experimental yield.

Based on the force approach, the connection value of N3L1-N3L2 [3.9554], with the lower [2.9554] and upper value [4.9554] proved suitable for determination of the root mean square error (RMSE) as well as average coefficient of determination (R^2) and $R^2_{Adj.}$ The RMSE, R^2 and $R^2_{Adj.}$ were evaluated to be 0.2200, 99.99% and 99.98%, respectively. The variance activity that varies from a norm was determined to be 2.26, while the square root of the variance called the standard deviation was obtained to be 1.503.

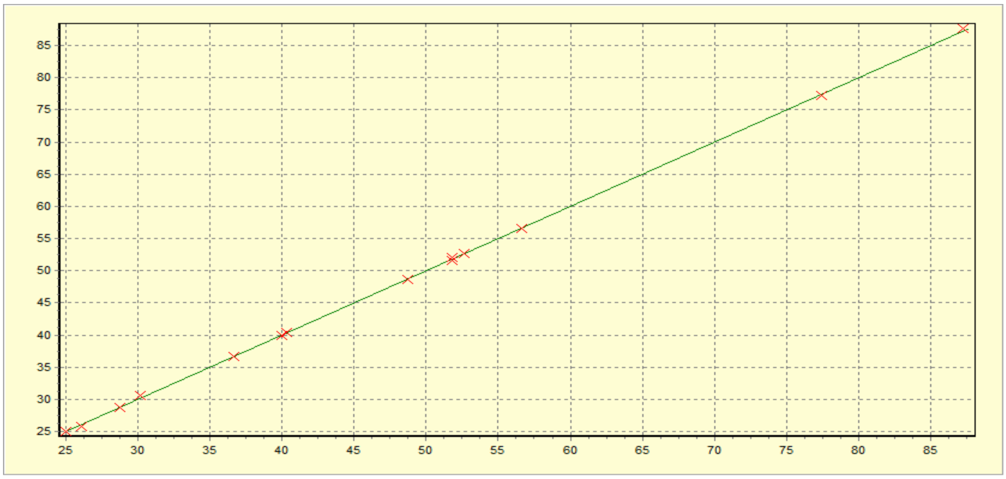
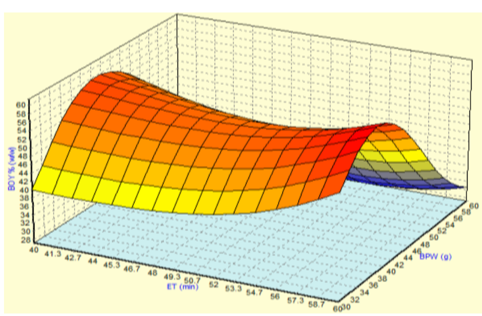


Fig-3. Graph of predicted against the experimental yield by ANN

Fig. 4 also showed the graphical representation of the 3-dimensional by ANN software. The nature of the graphs indicated that there are mutual interactions among the variables. To establish the optima condition, twenty seven (27) variables combination were generated, the optimal variables condition for this process was established at twenty three (23) variables combination. This was obtained by minimizing the controllable variables while maximizing the response variable (SIOY). The conditions was established at X_1= 54.71 g, X_2 = 44.88 min and X_3= 165.8 mL. The predicted SIOY under this condition was 86.20 % (w/w). Using these optimal condition values for three independent replicates, a mean of 85.70 % (w/w) SIOY was achieved, which was within the range predicted by the model.



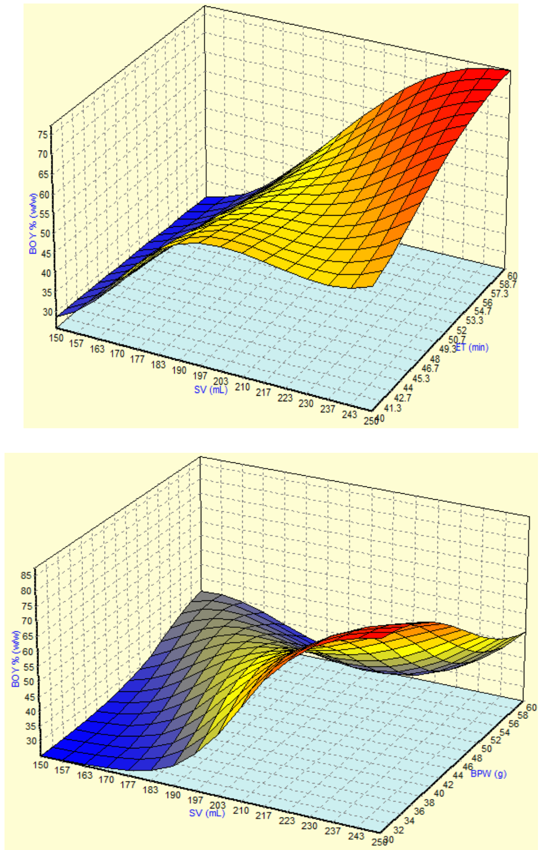


Fig-4. Graphical representation of the 3D's by ANN

3.2. Assessment of ANN and RSM

The accuracy of the obtained models (ANN and RSM) was checked by evaluating the predicted, AAD, RMSE, R^2 and $R^2_{Adj.}$ variance and standard deviation. The results revealed that the ANN and RSM are good optimization tools with accurate predictions (Table 5). However, the results showed the superiority of ANN over RSM because of predicted values (85.70%w/w, ANN; 83.20 %w/w, RSM) R^2 (99.99%, ANN; 99.44%, RSM) and $R^2_{Adj.}$(99.98%, ANN; 98.72%, RSM). In conclusion, ANN proved to better than RSM in the modeling and optimization of extraction of oil from *Sesamum indicum* seeds.

Table-5. Assessment of RSM and ANN SIOY

Data	Values	
	RSM	**ANN**
Predicted %(w/w)	83.20	85.70
AAD	0.1600	0.1300
RMSE	0.8450	0.2200
R^2	0.9944	0.99992
$R^2_{Adj.}$	0.9872	0.99983
Variance	1.3630	2.2600
Standard deviation	1.2767	1.5030

3.3. Qualities of the Oil and its Free Fatty Acid Compositions

The SIOY produced was tested for its qualities and the results obtained were compared with previous researchers. The results obtained were well within the ranges previously reported by the same seed and other oil seeds (Table 6). Values obtained for the fatty acid composition revealed that the oil contained both saturated and unsaturation fatty acids.

Table-6. Quality and free fatty acids of SIOY

Parameters	Values	Previous researchers	References
Moisture content %	0.008	0.09	Betiku and Adepoju [19]
Specific gravity	0.90	0.88	Betiku and Adepoju [19]
Viscosity (cP) @ 40 °C	28. 70	50-100	Elleuch, et al. [20]
Refractive index @ 25 °C	1.465	1.470	Betiku and Adepoju [19]
Acid value (mg KOH/g oil)	0.72	0.50	Betiku and Adepoju [19]
% FFA	0.36	0.25	Betiku and Adepoju [19]
Saponification value (mg KOH/g oil)	185	190	Betiku and Adepoju [19]
Peroxide value (meq O_2/kg oil)	9.20	7.80	Betiku and Adepoju [19]
Iodine value (mg KOH/g oil)	202	190	Betiku and Adepoju [19]
Cetane No	30.35	29.30	Betiku and Adepoju [19]
Higher heating value (MJ/kg)	38.82	40.02	Demirbas [17]
Fatty acid profile			
Parameters	**Composition**	**Early researchers**	**References**
Palmitic acids (C16:0)	11.58	17.80	Betiku and Adepoju [19]
Stearic acids (C18:0)	5.34	7.41	Betiku and Adepoju [19]
Oleic acids (C18:1)	48. 60	43.74	Betiku and Adepoju [19]
Linolenic acids (C18:2)	27.20	24.01	Betiku and Adepoju [19]
Myristic acids (C14:0)	2.01	1.11	Betiku and Adepoju [19]
Others	5.27		

4. CONCLUSION

This study demonstrated that *Sesamum indicum* seeds are potential candidate for edible oils production. Extraction of oil from the seeds was successfully optimized by ANN and RSM. The study showed that ANN was a superior and more effective optimization tool than RSM owing to its value of predicted, RMSE, AAD, R^2, R^2_{Adj}. Meanwhile, the qualities of extracted oil as compared to the earlier research works indicated that the oil produced is of good qualities. Fatty acids profile reflected that the oil is highly unsaturated and is suitable for human consumption.

5. ACKNOWLEDGEMENTS

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