



MODELLING AND OPTIMISATION OF OIL EXTRACTION FROM LOOFAH (*Luffa cylindrica*) SEEDS USING BINARY SOLVENT MIXTURE

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Abstract: Toxicity and safety concern coupled with the recent increase in its price has necessitated the need for finding alternative solvents to n-hexane. In this study, the effect of binary solvent (ethanol/n-hexane) composition at various extraction temperatures and times on the oil yield was investigated using response surface methodology (RSM). Artificial neural network (ANN) was used as a modelling tool for predicting the oil yield and the performance of both ANN and RSM models was compared. The optimum oil yield (27.67%) was obtained at extraction temperature (40 °C), extraction time (151.9 min) and binary solvent composition (98% ethanol /2% n-hexane). The predicted oil yield values from ANN model was more accurate than that of RSM when compared with experimental values. The fatty acid profile revealed that the refining process promoted saturation of the extracted oil with 67.75% of palmitic acid present in refined loofah seed oil (RLSO). This study demonstrated the feasibility of using a binary mixture of ethanol and n-hexane as a suitable replacement to the commonly used toxic n-hexane solvent for the extraction of oil from loofah seeds.

Keywords: Waste management, *Luffa cylindrica*, artificial neural network, optimisation, oil extraction.

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INTRODUCTION

The World is becoming more conscious of the environment with increasing replacement of synthetic products with naturally derived products (1). As a result, an increase in the demand for seed oils as raw materials for chemical industries is observed. Loofah oil-based derivatives could find wider markets worldwide with the recent industrial attention to its renewability and global friendliness, thereby increasing the number of research aimed at harnessing its seed oils for various applications (2-4).

Over the years, solvent extraction has been an efficient and reliable means of extracting oil from seeds or oil-bearing materials. Currently, n-hexane solvent usually obtained from petrochemical sources is used extensively for oil extraction. However, this solvent is identified as an air pollutant which has been known to react with other pollutants to produce ozone and some photochemical oxidants (5). Consequently, environmental and health concerns have increased interest in the search for alternative solvents that would lead to a decrease in the emissions of volatile organic compounds to the environment. The interest is also aimed at, avoiding the

challenge of potential traces of hexane in the oil after refining.

The most feasible alternative to hexane extraction appears to be the complete or partial replacement of this solvent by other organic solvents recognized as being more environmentally safe. A review of past studies carried out at both laboratory and pilot scales show the use of an alcohol such as ethanol and isopropanol for oil extraction to be very promising (5-7). Ethanol is being investigated for considering its suitability to serve as a substitute to n-hexane because of the economic advantage and the fact that, it could be produced from a large variety of biological items (including biological wastes) by employing simple technology. Moreover, natural alcohol can be gotten by fermentation and is recognized as non-toxic and has less handling risks than that of n-hexane. Replacement of n-hexane with ethanol as a solvent for extraction will also avoid potential toxicity of the residual cake which could be used as meals for animal feedstock (5). Some previous studies had been reported on the extraction of oil from loofah seed using hexane as a solvent (8-11). However, the use of ethanol or its binary mixture as a possible replacement to n-hexane solvent for the extraction of oil from loofah seeds has not been reported elsewhere.

Hence, this study was undertaken to investigate the feasibility of using ethanol solvent and its binary mixture with hexane as a suitable solvent for a complete or partial replacement to the commonly used, toxic hexane solvent for the extraction of oil from loofah seeds. The effect of process parameters such as binary solvent (n-hexane/ethanol) composition at various extraction temperature

and extraction time on the oil yield was investigated and optimisation was performed in order to propose a feasible binary solvent composition with better oil yield and low toxicity. Predictive models for oil yield as a function of the studied parameters were developed using RSM and ANN and the fatty acid constituent of the extracted oil was analysed.

MATERIAL AND METHODS

Sample Collection and Preparations

Dried and matured loofah fruits were collected and prepared by opening the dried fruits and removing the seeds from the spongy fruits. The seeds were air dried for easy removal of the shell after which the seeds were oven – dried at 60 °C to constant weight before grinding to increase the surface area for oil extraction. All the reagents used were analytical grade (BDH Chemical, England and Merck Chemical, Germany).

Design of experiments

Central composite face-centered design (CCFCD) under the response surface methodology (RSM) was used to study the individual and inter-relationships among the three studied factors (temperature, extraction time, and solvent composition) towards the response (oil yield). CCFCD is characterised by 2n axial runs, 2n factorial runs and six central runs. For a three-factor scenario where n is equal to three (3), it translates to 6 axial points, 8 factorial points and 6 replicates at the centre which gives a total of 20 experimental runs. The coded levels and ranges for the three studied factors are presented in Table 1. The solvent compositions are presented in terms of percentage composition of the binary solvent mixture with respect to n-hexane.

Table 1. Independent variables (factors) and their coded levels for CCFCD.

Independent Variable (Factors)	Coded Symbol	Units	Range and Levels		
			-1	0	+1
Temperature	x1	°C	40	50	60
Time	x2	min	150	210	270
% Binary solvent composition with respect to hexane*	x3	%	0	50	100

The optimum conditions for the response (oil yield) were determined using the optimal model predictor quadratic equation (12) given as:

$$Y = b_0 + \sum_{i=1}^n b_{ii}x_i + (\sum_{i=1}^n b_{ii}x_i)^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^n b_{ij}x_i x_j \tag{1}$$

Where Y is the predicted response (oil yield), bo is the constant coefficients, bii is the quadratic coefficients, bij is the interaction coefficients and xi, xj are the coded values of the variables considered. Design Expert software (version 6.0.8) was employed to test

the significance of the quadratic model generated and, analysis of variance (ANOVA) was used to determine the lack of fit and the effect of linear, quadratic and interaction terms on oil extraction (13). The characteristics of the reliability of the analysis carried out was measured by the variability in the observed response value expressed by the coefficient of determination R², the probability P – value (95% confidence level) and, Fisher’s test.

ANN modelling for loofah oil extraction

Levenberg-Marquardt Back Propagation Algorithm is widely used as the learning algorithm for artificial neural networks (ANN) in the multi-layered feed forward networks (14). This method verifies the predicted output using the learning result and continuously adjusts the difference between the target result and the result calculated by the model so as to minimize the error value. All neurons in the neural network model are divided into an input layer, a hidden layer, and an output layer depending on the function, and each layer is functionally connected. In this work, the hyperbolic tangent sigmoid (tansig) was used as the transfer function for both the input layer to hidden layer mapping and hidden layer to output layer mapping. The ANN architecture assembled contain an input layer with three neurons (extraction temperature, extraction time, and solvent composition) and an output layer with one neuron (oil yield). The number of neurons in the hidden layer of the MLP was determined experimentally by identifying the number of neurons with minimum error value from a range of neuron numbers tested. The experimental data set obtained from the design of experiments was divided into three subsets in order to obtain the desired ANN model. For this study, 70% of the data set was used for training, 15% was applied for validation, while the reliability of the model was tested with the remaining 15% of the data set.

Performance evaluation of RSM and ANN

The performance of RSM and ANN was compared using statistical parameters such as mean square error (MSE), average absolute deviation (AAD), correlation coefficient (R), and coefficient of determination (R^2).

Oil extraction

Extraction of loofah seed oil was carried out using a Soxhlet apparatus. 4 g of pulverized seed sample and 50 mL of solvent was used for each combination of the process variables based on the design of experiment for the CCFCD (Table 2). The extracted oil yield was expressed in percentage, which was evaluated as the ratio of the weight of oil extracted to the weight of the loofah seed powder sample used (Equation 2). Each test was repeated three times and the average value was determined and reported.

$$\text{Oil Yield (\%)} = \frac{\text{Weight of Extracted Oil}}{\text{Weight of the Seed}} * 100 \quad (2)$$

Prior to the physicochemical analyses, purification of the extracted crude loofah seed oil was carried out by degumming and de-

acidification according to the procedure reported by Audu et al. (11).

Physicochemical properties of the oil extract

The physicochemical properties such as viscosity, acid value, free fatty acid, saponification value, iodine value and peroxide value of the crude (CLSO) and refined (RLSO) extracted loofah seed oil were determined by standard methods (11, 15).

Fatty acid analysis of the extracted oil

The CLSO and RLSO were further characterised using Agilent 7890A Gas Chromatography (GC) coupled to Mass spectrometer (5975C) with triple axis detector and also equipped with an auto-injector (10 μ L syringe) for the analysis of the fatty acids in the extracted oil sample. Helium gas was used as the carrier gas. The chromatographic separation was performed on a capillary column having a specification (length 30 m, internal diameter 0.2 μ m, and thickness 250 μ m) and treated with phenyl methyl silox. The column temperature was set up from 35 $^{\circ}$ C to 250 $^{\circ}$ C for the total run time of 47.5 min. The injection volume was 1 μ L and, injection temperature was 300 $^{\circ}$ C for the GC. For the mass spectroscopy, the solution software provided by the supplier was used to control the system and acquired the data. Identification of the compounds was carried out by comparing the mass spectra obtained with standard mass spectra from the NIST library (NIST11).

RESULTS AND DISCUSSION

Design of experiments

The experimental design and results are given in Table 2. Run 15 – 20 at the center point were used to determine the experimental error and the reproducibility of the data. The highest oil yield of 30.0% was obtained at 100% ethanol solvent composition as shown in Table 2 (Run 2). The percentage oil yield obtained using 100% ethanol ranged from 19.0% to 30.0% with the mean value of 24.8%. As shown in Table 2 (Run 8), 100% n-hexane gave the highest oil yield of 18.0% and this value is slightly greater than the highest oil yield (17.0%) obtained from the mixture of hexane (50%) and ethanol (50%) under Run 10.

Several studies had shown that non – polar solvents (n-hexane) always gives high oil yield due to the absence of OH (13, 16, 17). However, this study revealed that the highest yield was obtained from polar solvent (ethanol) which may suggest that, some components in the LSO impaired the retardation of activities on the polar solvent or

other materials rather than oil was extracted thereby leading to higher yield. Some past studies also confirmed that extraction of

biocompounds such as phenolic compounds in solvent extraction process has been well favoured with polar solvents (6, 18, 19).

Table 2. CCFCD Experimental design matrix with coded and real values.

Run	Coded Values			Real Values			Oil Yield (%)		
	x1	x2	x3	Extraction Temp. (°C)	Time (min)	Binary solvent composition*(%)	Experimental	RSM	ANN
1	-1	-1	-1	40	150	0	28.000	28.430	27.999
2	1	-1	-1	60	150	0	30.000	29.130	29.990
3	-1	1	-1	40	270	0	22.000	21.330	21.997
4	1	1	-1	60	270	0	19.000	19.030	18.998
5	-1	-1	1	40	150	100	3.000	2.830	3.010
6	1	-1	1	60	150	100	11.000	11.530	11.424
7	-1	1	1	40	270	100	11.000	11.730	11.001
8	1	1	1	60	270	100	18.000	17.430	18.003
9	-1	0	0	40	210	50	15.000	14.670	15.624
10	1	0	0	60	210	50	17.000	17.870	17.000
11	0	-1	0	50	150	50	13.000	13.070	13.001
12	0	1	0	50	270	50	12.000	12.470	11.998
13	0	0	-1	50	210	0	24.000	25.070	25.614
14	0	0	1	50	210	100	12.000	11.470	12.004
15	0	0	0	50	210	50	15.000	14.820	14.999
16	0	0	0	50	210	50	15.000	14.820	14.999
17	0	0	0	50	210	50	15.000	14.820	14.999
18	0	0	0	50	210	50	15.000	14.820	14.999
19	0	0	0	50	210	50	15.000	14.820	14.999
20	0	0	0	50	210	50	15.000	14.820	14.999

* Composition of the binary (n-hexane/ethanol) solvent with respect to n-hexane.

Development of regression model equation using RSM

The development of the regression equation was performed using CCFCD under the RSM to describe the correlation between oil yield (%) and the studied factors (temperature, extraction time, and solvent composition). The quadratic model was developed using the Design Expert software version 6.0.8 (STAT-EASE Inc., Minneapolis, USA). The final empirical model in terms of coded factors for the oil yield (Y_{oil yield}) is given in Equation 3:

$$Y_{oil\ yield} = +14.82 + 1.60x_1 - 0.30x_2 - 6.80x_3 + 1.45x_1^2 - 2.05x_2^2 + 3.45x_3^2 - 0.75x_1x_2 + 2.00x_1x_3 + 4.00x_2x_3 \quad (3)$$

where x₁ = Extraction temperature, x₂ = Extraction time, x₃ = Solvent composition

The regression equation (Equation 3) indicates that the oil yield was negatively correlated with extraction time and solvent composition and positively correlated with the extraction temperature within the experimental range (Table 1) considered for the three studied variable. Thus, an increase in extraction time decreased the oil yield. Likewise, an increase

in percentage composition of hexane in the binary mixture decreased the oil yield and this is in agreement with oil yield range of 19.0% - 30.0% and 3.0% - 18.0% (Table 2) obtained experimentally for 0% hexane (100% ethanol) and 100% hexane (0% ethanol) respectively. It was also observed from Equation 3 that the magnitude of coefficients of solvent composition was larger than the coefficients for extraction temperature and extraction time, which indicated the solvent composition, had much significant effect on the oil yield than the individual or combined effect of extraction temperature and time.

The analysis of variance (ANOVA) for the non-linear regression equation (Equation 3) was used to also highlight the adequacy of the models and their significance. The Fisher's variance ratio F-value and Prob > F for the oil yield were 150.30 and < 0.0001, respectively (Table 3) which implied that the quadratic model suggested for the response (oil yield) is significant. In addition, Table 3 showed that x₁, x₃, x₁₂, x₂₂, x₃₂, x_{1x2}, x_{1x3}, and x_{2x3} were the significant terms while the x₂ (extraction time) was the only insignificant term in the oil yield model with "Prob > F"

greater than 0.05. In the same vein, the model term x3 (solvent composition) with F-value of 875.46 and Prob > F less than 0.0001 imposed the most significant effect on the oil yield. The relative predictive power of the quality of the model R2 was found to be 0.993 which is in

reasonable agreement with the adjusted R2 (0.986) and predicted R2 of (0.903). More so, a desirable ratio of 51.18 was obtained for the adequate precision which indicated an adequate signal, thus the model can be used to navigate the design space (12, 20-22).

Table 3. ANOVA for response surface quadratic model for loofah seed oil yield.

Source	Sum of Squares	Degree of Freedom	Mean Square	F - Value	Prob > F
Model	714.47	9	79.39	150.30	< 0.0001 ^a
x1	25.60	1	25.60	48.47	< 0.0001 ^a
x2	0.90	1	0.90	1.70	0.2210 ^b
x3	462.40	1	462.40	875.46	< 0.0001 ^a
x12	5.82	1	5.82	11.02	0.0078 ^a
x22	11.51	1	11.51	21.78	0.0009 ^a
x32	32.82	1	32.82	62.13	< 0.0001 ^a
x1x2	4.50	1	4.50	8.52	0.0153 ^a
x1x3	32.00	1	32.00	60.59	< 0.0001 ^a
x2x3	128.00	1	128.00	242.34	< 0.0001 ^a
Residual	5.28	10	0.53		

^asignificant at 95% confidence level, ^b not significant at 95% confidence level.

Process optimisation

Although the highest oil yield of 30.0% was obtained at 100% ethanol solvent composition as shown in Table 2 (Run 2), this study tends to accommodate the product separation challenges that might arise from the use of ethanol alone as a solvent and hence optimization study was carried out to propose a feasible binary solvent of ethanol and n-hexane. The California division of occupational safety and health reported the permissible exposure limit (PEL) as an eight-hour time-weighted average (TWA) for ethanol and n-hexane as 1000 ppm and 500 ppm, respectively (23). The PEL values confirm ethanol as far less toxic compared to hexane making ethanol to be a suitable replacement or a solvent that could serve to reduce the concentration of the much toxic hexane solvent from their binary mixtures. The Process optimization was carried out using design expert software to find the optimum

process parameters to maximize the percentage of oil extracted from loofah seed. The best solution for optimization within the experimental range of the studied independent variables is usually selected based on the highest desirability or its closeness to unity (6, 24). The maximum oil yield of 27.67% was obtained with the desirability of 1.0. The optimum conditions for the variables studied were obtained at extraction temperature (40 oC), extraction time (151.9 min) and solvent composition (2% n-hexane / 98% ethanol). This is an indication that, this new proposed binary solvent mixture could serve as a more efficient alternative solvent mixture with less toxic effect to achieve a better oil yield when compared with the widely used highly toxic n-hexane solvent. The model validation was carried out and the experimental oil yield (28.5%) was found to agree satisfactorily with the predicted oil yield (27.67%), with an error of 2.91% (Table 4).

Table 4 . Model validation.

Extraction Temperature, x ₁ (°C)	Extraction Time, x ₂ (min)	Binary solvent composition with respect to n-hexane, x ₃ (%)	Oil Yield, Yoil yield (%)		
			Predicted	Experimental	Error
40.00	151.90	2.00	27.67	28.50	2.91

The combined interaction effects of the three studied variables on the yield of loofah seed oil are shown on the three-dimensional surface response plots (Figure 1a – d). Figure 1a and 1b show the response surface plot for the combined effect of extraction temperature and time on oil yield at the fixed solvent composition of 100% ethanol and optimum

solvent composition of 2% n-hexane and 98% ethanol. The response surface plot in Figure 1c shows the combined effect of solvent composition and extraction temperature on oil yield at the fixed extraction time of 151.9 min while Figure 1d shows the response surface plot for the combined effect of solvent composition and extraction time on oil yield at

a fixed extraction temperature of 40 °C. The curvatures obtained from Figure 1c - d clearly indicates that solvent composition imposed the most significant effect on the oil yield.

Although the level of contributions may differ, all the studied variables contributed either individually or by way of interaction to the yield of loofah seed oil.

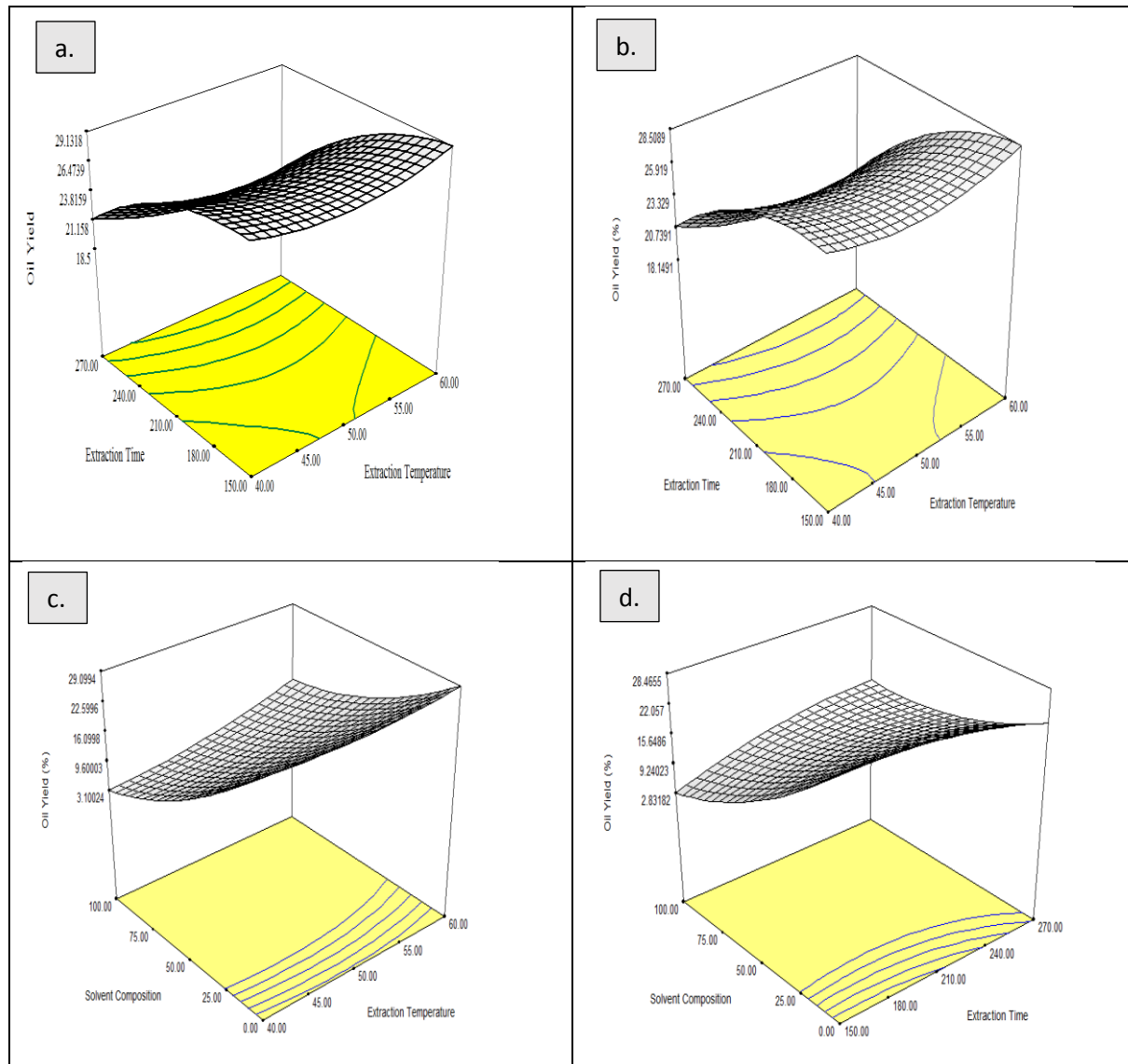


Figure 1. Response surface plot for the combined effect of (a) temperature and time on oil yield at fixed solvent composition of 100% Ethanol (b) temperature and time on oil yield at optimum conditions (c) solvent composition and temperature on oil yield at optimum conditions (d) solvent composition and time on oil yield at optimum conditions.

ANN modelling

The ANN model was developed using the MATLAB R2015a neural network toolbox (Math Works, Inc., Natick, MA, USA). The optimal number of neurons in the hidden layer of the MLP-ANN determined by trial and error was found to be neuron 12 and the best topology chosen for the ANN model was 3 - 12 - 1.

Hence, the ANN optimum architecture assembled contain an input layer with three neurons (extraction temperature, extraction time, and solvent composition), a hidden layer with 12 neurons and an output layer with one neuron (oil yield) as shown in Figure 2.

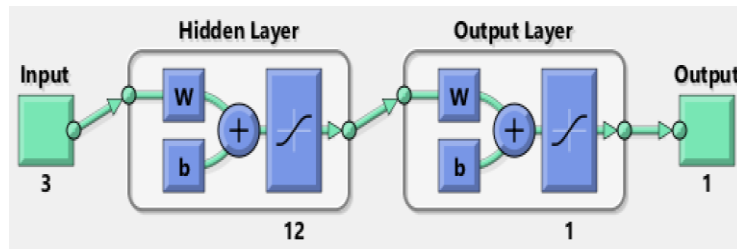


Figure 2. ANN architecture for prediction of oil yield.

The regression plots showing the coefficient of correlation for training, testing, validation and whole data sets are depicted in Figure 3. The predicted values obtained for oil yield from the

ANN model is presented in Table 2. The predicted oil yield from the ANN model was in the range of 3.01% to 29.99%.

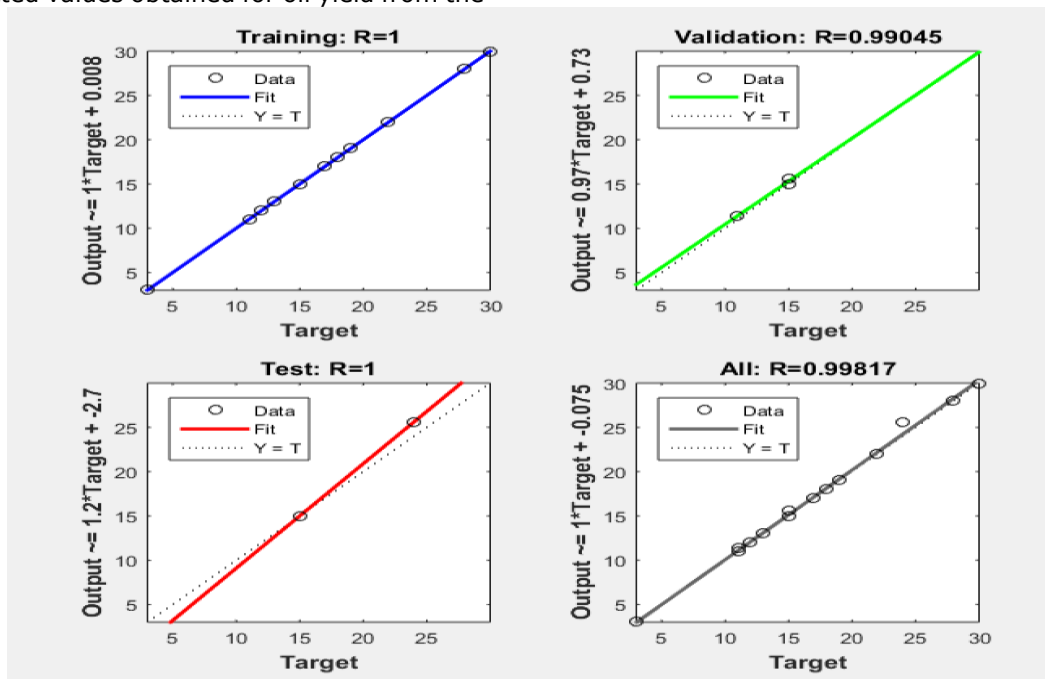


Figure 3. Regression plots for the ANN model.

Performance evaluation of RSM and ANN

The evaluation of the predictive capabilities of RSM and ANN for the oil yield was carried out and the results were compared. Statistical parameters such as MSE, AAD, R and R² were used to compare the performance of the two developed models. The results obtained (Figure 4) shows that the ANN model performed better than the RSM model, although the performance was satisfactory for both models. The mean square error (MSE)

values indicated that lower errors were obtained with the ANN model (0.1587) compared to the RSM model (0.2636). The average absolute deviation (AAD) of the ANN model (0.7632) was also found to be lower compared to the RSM model (2.7887). More so, the ANN model had higher precision and accuracy as shown with the values of R (0.9982) and R² (0.9964) compared with R (0.9963) and R² (0.9927) obtained from the RSM model.

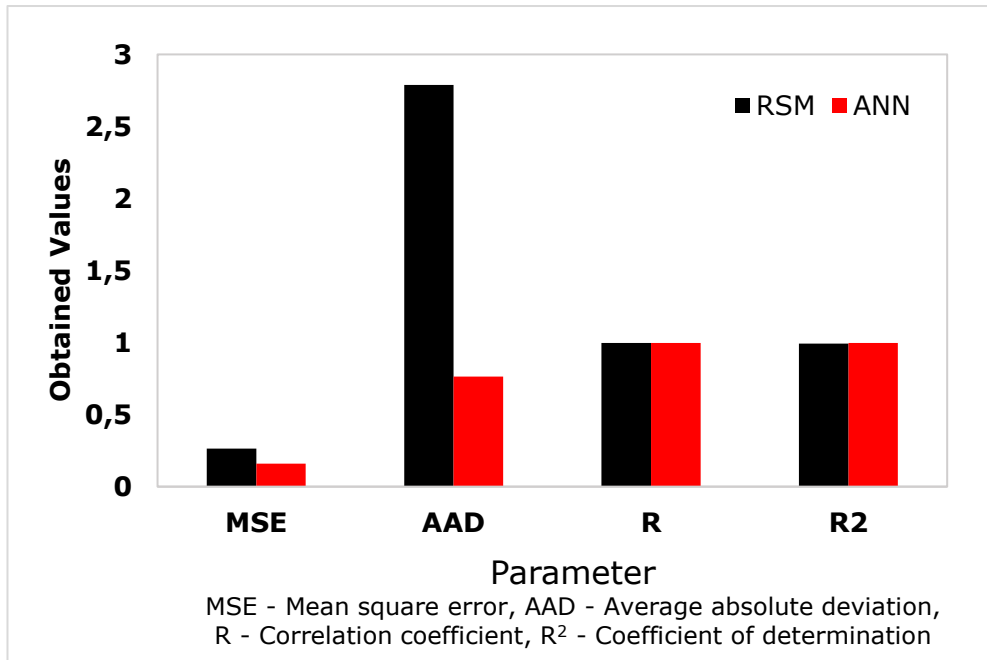


Figure 4. Comparison of statistical evaluation of the RSM and ANN models.

The plot of experimental and predicted oil yield values against the experimental run is shown in Figure 5. The figure confirms that the predicted values for the ANN model were much more closely aligned with the experimental values than the predicted values of the RSM model. The results from this study are in agreement with previous studies on the superiority of ANN over response surface methodology (25-27). Avramović et al. (26) in their study on predicting the biodiesel yield from ethanolsis of sunflower oil using calcium

oxide as catalyst showed that ANN is more superior to RSM. Betiku et al. (25) compared the performance of RSM, ANFIS and ANN for the predictive modelling of acid pre-treatment of palm kernel oil and their result showed that ANN was the best of the three modelling tools. Soji-Adekunle et al. (27) modelled the synthesis of waste cooking oil methyl esters using ANN and RSM and their findings also confirmed ANN as a better predicting tool than the RSM.

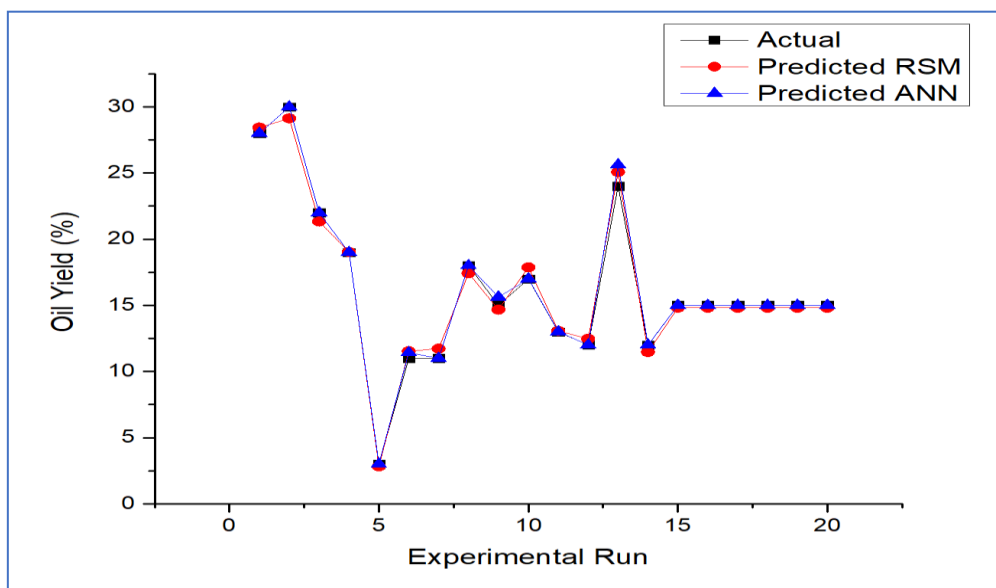


Figure 5. Actual and predicted oil yield from ANN and RSM against the experimental run.

Physicochemical properties of loofah seed oil

The physicochemical properties of the crude loofah seed oil (CLSO) and refined loofah seed oil (RLSO) were analysed and the results presented in Table 5. The result showed that the percentage oil yield (27.67%) obtained in this study using the proposed optimum binary solvent was found to be much higher compared to the oil yield of 14.08% reported by Audu et al. (11) for loofah seed oil extraction using only n-hexane solvent. Specific gravity and viscosity are important physical characteristics which indicate the handling, storage and operational conditions of oils and fuels (28). The specific gravity of both the crude and refined loofah seed oil was 0.86 and 0.84 respectively. These values are close to the standard range of 0.87 – 0.90 for biodiesel production (29). Moreover, the viscosity of crude oil extract was found to reduce from 0.109 N.s/m² to 0.078 N.s/m² which could be as a result of a degumming step in the refining process. These observed properties suggest that the refined oil could find good application in the manufacture of lubricants and biofuel industries (30). A similar trend was also observed from the viscosity results reported by Audu et al. (11).

From Table 5, the acid values for CLSO (3.67 mgKOH/g) and RLSO (3.33 mgKOH/g) obtained in this study were less than 10 mgKOH/g which is the recommended standard value for non-virgin edible oils (31). This is an indicator of the edibility of the extracted oil and its suitability for industrial purposes. In addition, the reduction in the acid value of the refined oil (RLSO) suggests that oil refining assisted in improving the quality of the oil and can easily be transesterified to biodiesel by acid catalysed system. The low acid values from this study also agree with values reported by Audu et al. (11) (3.72 mgKOH/g)

and Gafar et al. (8) (2.34 mgKOH/g). However, this observation is in contrast to the acid value of 34.15 mgKOH/g reported by Ozulu (9) for loofah seed oil extraction using only n-hexane solvent. The result of saponification values obtained from this study (Table 5) shows that the RLSO has a higher molecular weight than CLSO since saponification value is inversely proportional to the molecular weight of the oil. This is an indication that the refining process might have promoted saturation of the oil (32).

The iodine value measures the degree of unsaturation of fats and oils and can be used in predicting the drying property of oils. Usually, non-drying oils have iodine values between 80 – 120, while semi-drying oils have between 120 – 150 and drying oils have iodine values greater than 150 (10). Thus, the iodine values of 86.53 mgI₂/g and 80.15 mgI₂/g obtained for CLSO and RLSO, respectively can be classified as a non-drying oil. This iodine values obtained compare well with the iodine value of 82.29 mgI₂/g and 82.56 mgI₂/g reported by Ozulu (9) and Audu et al. (11), respectively for LSO. Oils with values less than 120 mgI₂/g according to EN 14214 (European committee for standardisation) may find use in surface coating applications like paints, resins, printing inks and also suitable as feedstock for biodiesel production. The peroxide value of the RLSO (5.3 meq/kg) was low when compared to that of the CLSO (8.01 meq/kg) which represented 34.5% reduction in the CLSO value. The low value of the refined oil could be as a result of gummy deposits removed during the refining process which indicates the stability of the refined oil over the crude oil. The value also suggests that the refined oil would have a longer shelf life than that of the crude loofah seed oil.

Table 5. Physicochemical properties of loofah seed oil (LSO).

Properties	Present Study		Audu et al. (11)	
	CLSO	RLSO	CLSO	RLSO
Oil yield (%)	-	27.67	-	14.08
Specific gravity	0.86	0.84	0.93	0.90
Viscosity @ 32 °C x10 ⁻¹ , (N.s/m ²)	1.09	0.78	1.045	1.045
Acid value (mgKOH/g)	3.67	3.33	3.82	3.72
Saponification value (mgKOH/g)	128	127.70	149	148.50
Iodine value (mgI ₂ /g)	86.53	80.15	-	82.56
Peroxide value (meq/kg)	8.10	5.30	-	5.43
Free fatty acid (%)	1.85	1.68	2.52	2.18

NB: CLSO – Crude loofah seed oil, RLSO – Refined loofah seed oil.

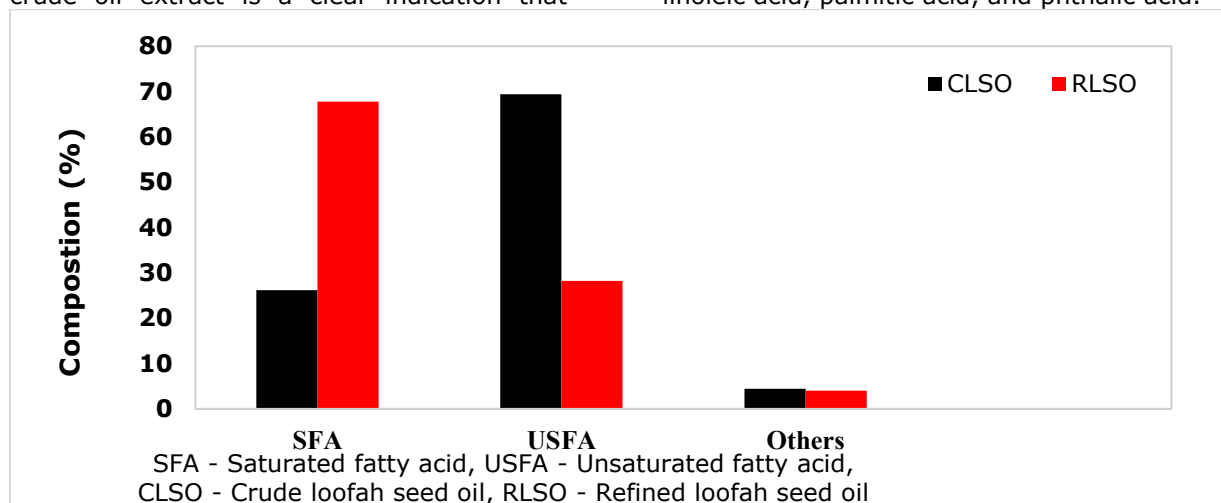
Fatty acid analysis of the extracted oil

A GC-MS analysis was conducted to investigate the fatty acids present in the

extracted oil. The summary of the fatty acid profile for crude loofah seed oil and refined loofah seed oil are presented in Figure 6. The

composition of saturated fatty acid predominantly palmitic acid was found to be 67.75% in the refined oil sample. This high value of saturated fatty acid obtained in the refined oil when compared with the 26.15% composition of saturated fatty acid in the crude oil extract is a clear indication that

refining of the loofah oil promoted saturation. More so, it could be inferred that the refined extracted oil is saturated oil which is in line with results of previous researches on loofah seed oil extraction (8, 9, 28). The fatty acid present in the refined loofah seed oil includes linoleic acid, palmitic acid, and phthalic acid.



CONCLUSIONS

This study has proposed a new binary mixture of ethanol/n-hexane as a suitable replacement to the commonly used, toxic n-hexane solvent for the extraction of oil from loofah seeds. The optimal condition was obtained at extraction temperature (40 °C), extraction time (151.9 min), and solvent composition (98% ethanol /2% n-hexane). Results of the statistical analysis showed that the solvent composition imposed the most significant effect while extraction time contributed the least effect on the yield of loofah seed oil (LSO). The performance evaluation of the developed predictive models shows that the ANN model performed better than the RSM model, although the performance of both models was satisfactory. Comparing the result of the properties of CLSO with RLSO, it is observed that, the refining process promotes saturation of the extracted oil with a high percentage of 67.75% palmitic acid found in RLSO.

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REFERENCES

1. Amoo I, Eleyinmi A, Ilelaboye N, Akoja S. Characterisation of oil extracted from gourd (*Cucurbita maxima*) seed. *Journal of Food Agriculture and Environment*. 2004;2(2):38-39.

2. Abayeh O, Garba I, Adamu H, Abayeh O. Quality characteristics of *Luffa aegyptiaca* seed oil. *International Journal of Scientific & Engineering Research*. 2013;4(4):11-15.

3. Sujatha D, Chithakari R, Raghuvardhan L, Prasad B, Khan G. In vitro plantlet regeneration and genetic transformation of sponge gourd (*Luffa cylindrica*). *African Journal of Plant Science*. 2013;7(6):244 - 252.

4. Ndukwe G, Amupitan J, Badmus N. Preliminary investigation on the antibiotic and fungicidal activities of the extracts from the seeds of *Luffa cylindrica*. *Journal of Engineering Technology*. 2001;8(2):3202 - 3207.

5. Ozulu O. Production and characterization of biodiesel from *Luffa Cylindrica* seed oil: Nsukka, University of Nigeria; 2015.

6. Bal K, Bal Y, Lallam A. Gross morphology and absorption capacity of cell-fibers from the fibrous vascular system of loofah (*Luffa cylindrica*). *Textile research journal*. 2004;73(4):241 - 247.

7. Gafar M, Itodo A, Warra A, Wyasu G, Usman J. Physico-chemical, cold saponification and GC-MS analysis of sponge gourd (*Luffa cylindrica* Linn.) seed oil. *International J Modern Chem*. 2012;3(2):98 - 107.

8. Obboh I, Aluyor E. *Luffa cylindrica*-an emerging cash crop. *African Journal of Agricultural Research*, 2009;4(8):684 -688.

9. Ajiwe V, Ndukwe G, Anyadiegwu I. Vegetable diesel fuels from *Luffa cylindrica* oil, its methyl ester and ester-diesel blends. *Chemistry Class Journal*. 2005;2(2):1-4.

10. Ferreira-Dias S, Valente D, Abreu J. Comparison between ethanol and hexane for oil extraction from *Quercus suber* L. fruits. *Grasas Aceites*. 2003;54(4):378 - 383.
11. Araromi D, Alade A, Bello M, Bakare T, Akinwande B, Jameel A, et al. Optimization of oil extraction from pitanga (*Eugenia Uniflora* L) leaves using simplex centroid design (SCD). *Separation Science and Technology*. 2017;52(8):1341-1349.
12. Sineiro J, Domínguez H, Núñez M, Lema J. Ethanol extraction of sunflower oil in a pulsing extractor. *Journal of the American Oil Chemists' Society*. 1998;75(6):753-754.
13. Auta M, Hameed B. Optimized waste tea activated carbon for adsorption of Methylene Blue and Acid Blue 29 dyes using response surface methodology. *Chemical Engineering Journal*. 2001;175(1):233 - 243.
14. Mani S, Jaya S, Vadivambal R. Optimization of solvent extraction of Moringa (*Moringa oleifera*) seed kernel oil using response surface methodology. *Food and Bioproducts Processing*. 2007;85(4):328 - 335.
15. Audu T, Aluyor E, Egualeona S, Momoh S. Extraction and characterization of *Chrysophyllum albidum* and *Luffa cylindrica* seed oils. *Food Chemistry*. 2013;8(3):201 - 208.
16. Horwitz W. Official methods of analysis of the AOAC International 2000.
17. Barchan A, Bakkali M, Arakrak A, Pagán R, Laglaoui A. The effects of sol-vents polarity on the phenolic contents and antioxidant activity of three *Mentha* species extracts. *Int J Curr Microbiol App Sci*. 2014;3(11):399 - 412.
18. Jayaprakasha G, Singh R, Sakariah K. Antioxidant activity of grape seed (*Vitis vinifera*) extracts on peroxidation models in vitro. *Food Chemistry*. 2001;73(3):285 - 290.
19. Lee Y, Huang G, Liang Z, Mau J. Antioxidant properties of three extracts from *Pleurotus citrinopileatus*. *LWT-Food Science and Technology*. 2007;40(5):823 - 833.
20. Ozsoy N, Can A, Yanardag R, Akev N. Antioxidant activity of *Smilax excelsa* L. leaf extracts. *Food Chemistry*. 2008;110(3):571 - 583.
21. Adewoye L, Mustapha S, Adeniyi A, Tijani J, Amoloye M, Ayinde L. Optimization of nickel (ii) and chromium (iii) removal from contaminated water using sorghum bicolor. *Nigerian Journal of Technology*. 2017;36(3):960-972.
22. Khuri A, Mukhopadhyay S. Response surface methodology. *Wiley Interdisciplinary Reviews: Computational Statistics*. 2010;2(2):128 - 149.
23. Rashid U, Anwar F, Ashraf M, Saleem M, Yusup S. Application of response surface methodology for optimizing transesterification of *Moringa oleifera* oil: Biodiesel production. *Energy Conversion and Management*. 2011;52(8):3034 - 3042.
24. Ibeto C, Okoye C, Ofoefule A. Comparative study of the physicochemical characterization of some oils as potential feedstock for biodiesel production. *ISRN Renewable Energy*. 2012:1- 5.
25. Jerković I, Rajić M, Marijanović Z, Bilić M, Jokić S. Optimization of supercritical CO₂ extraction of dried *Helichrysum italicum* flowers by response surface methodology: GC-MS profiles of the extracts and essential oil. *Separation Science and Technology*. 2016;51(18):2925 - 2931.
26. Ejikeme P, Egbounu C, Anyaogu I, Eze V. Fatty acid methyl esters of melon seed oil: Characterisation for potential diesel fuel application. *Leonardo Journal of Sciences*. 2011;18:75 - 84.
27. Audu TOK, Aluyor EO, Egualeona S, Momoh SS. Extraction and Characterization of *Chrysophyllum albidum* and *Luffa cylindrica* Seed Oils. *Petroleum Technology Development Journal*. 2013;3(1):1-7.
28. Onyeike E, Acheru G. Chemical composition of selected Nigerian oil seeds and physicochemical properties of the oil extracts. *Food Chemistry*. 2002;77(4):431 - 437.
29. Sayeed M, Ali M, Sohel F, Khan G, Yeasmin M. Physico-chemical characteristics of *Mesua ferrea* seed oil and nutritional composition of its seed and leaves. *Bulletin of the Chemical Society of Ethiopia*. 2004;18(2):157 - 166.
30. Kittiphoom S, Sutasinee S. Mango seed kernel oil and its physicochemical properties. *International Food Research Journal*. 2013;20(3):1145-1149.

