Oil Extraction from Butter Fruit (*Dacryodes Edulis*) Seeds and its Optimization via Response Surface and Artificial Neural Network

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ABSTRACT: In this study, oil was extracted from butter fruit (*Dacrydes edulis*). To model and optimize the process conditions of oil extraction, Response Surface Methodology (RSM) and Artificial Neural Network (ANN) were used. Physicochemical analysis of the oil was carried out in order to determine the suitable of oil for industrial applications. Dacryodes edulis seeds were collected from Ikot Abasi Village in Eket Local Government of Akwa Ibom State, Nigeria. The seed was washed with clean water to remove dirt, and open with a sharp stainless knife to remove the seed from the pulp. The seeds were cut into small pieces and sundried for 5 days and were grinded into powder. Oil extraction from the powder seed was carried out using Sohxlet extraction method. The experiment was designed using Box-Behnken Design approach on three levels, three factors which generated 17 experimental runs. Independent factors considered were extraction time (X_1), solvent volume (X_2) and sample weight (X_3). The accuracy of the regression model obtained from the optimization software was determined using the co-efficient of determination (R^2). Results showed the highest oil yield of 17.878% (w/w) was obtained at solvent volume of 200 ml, sample weight of 50 g and extraction time of 55 min, respectively. However, response surface methodology predicted an oil yield of 17.826% (w/w), while the artificial neural network predicted 17.875% (w/w) at the same variables condition. The predicted values were validated in triplicate, and an average of 17.46% (w/w) and 17.72% (w/w) were obtained for RSM and ANN, respectively. The predicted values obtained were well within the range predicted. The coefficient of determination, which determines the model accuracy, was obtained to be 0.8454 for RSM and 0.8712 for ANN. Physicochemical analysis of the oil showed the oil is highly unsaturated with high saponification value and high iodine value. The study concluded that Dacryodes edulis seeds are found to be rich in oil and the oil can be applicable in industries as raw materials for products formation.

KEYWORDS: Dacryodes edulis seeds, response surface, artificial neural network, optimization, extraction, physicochemical properties.

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I. INTRODUCTION

The use of oil in Industries for production of cosmetic, biofuel, bio lubricant and other products has become greatly increase, which has necessitated the needs to search for alternative source through biomass (Agricultural oilseeds). To meet up with the needs of the incessant increase in the demand for oil by both domestic and industries have resulted in search for using underutilized seeds as sources of oil to supplement the already current traditional sources of oil (Ugbogu et al., 2014). In some nation, specifically Nigeria, different oil crops exist from the largely used and highly-utilized to under-utilized seed oils that have not been investigated for their potential uses.

Dacryodes edulis, an annual fruit known as African plum or African pear or Safou seed is one of the under-utilized seed oil. It is an indigenous fruit tree in the humid low lands and plateau regions of West, Central African and Gulf of Guinea countries. Usually, the trees are grown around homesteads and flowering always takes place from January to April, with fruiting season between May and October (Nwosuagwu et al., 2009). The pear is locally called 'Ube' among the Igbos in South eastern part of Nigeria belongs to the family of Burseraceae and genus Dacryodes (Ogunsuyi, 2015). It has the qualities of butter apart from the pulp outlook which contains 48% oil; the overall plantation can produce 7-8 tonnes of oil per hectare.

Generally, the extraction of oil from the seeds can be achieved via various methods such as mechanical, (Joglekar and May, 1987; Haidar and Pakshirajah, 2007; Schinas et al., 2009) pressurized solvent extraction (Mcginely, 1991), soxhlet extraction (Meher et al., 2006), ultrasonic extraction (Ramos et al., 2009; Rodríguez et al., 2012), Aqueous Enzymatic Oil Extraction (AEOE) (Perez-Serradilla et al., 2002; Pan et al., 2002), stirring and shaking extraction (Kim et al., 2004) to mention but few. These methods have been reported used for extraction of vegetable oils and other plant essential components, each with intrinsic advantages and shortcomings (Umer et al., 2011). Mechanical pressing is the oldest and easiest method used widely, but, the oil produced always with low value. Meanwhile, (Betiku and Adepoju,

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2013) reported the use of supercritical CO2 extraction; the oil yield obtained was higher than what was obtained from using solvent extraction and of high purity, but the high operating and investment costs make this method not suitable to be used. Solvent extraction has various advantages including high yield, less turbidity, environmentally friendly and cost effective (Adepoju et al., 2013; Betiku et al.,2012), which was why solvent extraction of oil from Jatropha curcus was carried out by (Kian et al., 2011), while (Umer et al., 2011) studied the solvent extraction of oil from Moringa oleifera. Betiku et al. (2012) extracted oil from sorrel seed using solvent extraction while solvent extraction of oil from Chrysophyllum albidium oilseeds and its quality characterization was carried out by (Adepoju et al., 2013). In another study, Betiku et al. (2012) worked on solvent extraction of oil from Beniseed (Sesamum indicum) oilseeds.

This work therefore employed the possibility of using Dacryodes edulis seed for oil production. For modelling and optimization, response surface methodology (RSM) and artificial neural network (ANN) were employed so as to generate the number of experimental runs, determine the predicted yields, compare the error values, and analyse the various variable factors responsible for optimum production of oil so as to increase the process efficiency. Lastly, the properties of oil were evaluated with a view to determining its suitability as edible or non-edible oil.

II. MATERIALS AND METHODS

A. Materials

The *Dacryodes edulis* seeds used in this work was collected from Ikot Abasi Village in Eket Local Government of Akwa Ibom State, Nigeria. The seed was washed with clean water to remove dirt, and open with a sharp stainless knife to remove the seed from the pulp. The seeds were cut into small pieces and sundried for 5 days. Finally, the cleaned seeds were grinded into powder with a Corona grinder**.** All the chemicals used in this study are of analytical grades.

B. Methods

1) Experimental design

To optimize the oil extraction from *Dacryodes edulis* seeds *powder*, three level-three factors were considered. Box-Behnken Design (BBD_{RSM}) was employed which generated 17 experimental runs used to study the effects of selected factors on oil yield. Table 1 showed the selected factors which are solvent volume (X_1), sample weight (X_2) and the extraction time (X_3) and their levels. These factors were also used for ANN Modeling and optimization. For the coefficient of the quadratic model of the response fitting, multiple regressions model was adopted using design expert software 10 version 15.5 (Stat Inc., Tulsa, OK, USA) and Neural Power _21356. Regression analysis and test of significance are the computationally intensive process that is best carried out via statistical software; hence the quality of the fitted model was evaluated using test of significance and regression analysis of variance (ANOVA) via Eq. 1. To compare the results of the model chosen and validate the coefficient of determination of the experimental result, artificial neural network (ANN) was incorporated.

$$R_F = \tau_0 + \sum_{i=1}^{k} \tau_i X_i + \sum_{i=1}^{k} \tau_{ii} X_i^2 + \sum_{i<j}^{k} \tau_{ij} X_i X_j + e \qquad (1)$$

where, R_F is the response (oil yield), τ_0 is the intercept term, τ_i, τ_{ii}, τ_{ij} are the coefficient terms for linear (X_i), quadratic (X_i^2) and interaction ($X_i X_j$), X_i is the selected factors, i= 1, 2, 3.

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

Variable	**Symbol**	**Coded factor levels**		
		-1	**0**	**+1**
SV (ml)	X_1	180	200	220
SW (g)	X_2	40	50	60
ET (min)	X_3	50	55	60

SV = Solvent volume, SW = Sample weight, ET= Extraction time

2) Description of oil extraction procedure

Based on the experimental design by Box Behken Design, seventeen experimental runs were generated and were carried out. Soxhlet extractor was used for the extraction process; the powdered sample was weighed and placed in the extractor through the muslin cloth. N-hexane was used as organic solvent for extraction. Three level three factors were considered, the excess n-hexane was recovered using rotary evaporator, and the yield of the oil was obtained using Eq. (2).

$$Oil\ yield\ (\%) = \frac{weight\ of\ oil}{weight\ Dacryodes\ edulis\ \ powder\ used} \qquad (2)$$

3) Physicochemical analysis of the extracted Dacryodes edulis seed oil

The properties of the oil were evaluated using standard of AOAC (1997), official methods of analysis and Wij's iodine method as described below:

0.52 g of oil sample was dissolved in 10 ml of cyclohexane. 20 ml of Wij's solution was added, the stopper flask was allowed to stand for 30 min in the dark at room temperature, and 20 ml of 10% potassium iodide solution was added. The resulting mixture was then titrated with 0.1 M $Na_2S_2O_3$ using starch as indicator. Iodine value was calculated using equation 3.

$$Iodine\ Value = \frac{[\rho_o - \rho]\ X\ MX12.69}{0.26} \qquad (3)$$

where M = concentration of sodium thiosulphate used; ρ_o = volume of sodium thiosulphate used as blank; ρ = volume of sodium thiosulphate used for determination.

4) Variable interactive effects

To study the effects of independent variables on oil yield, three factors were taking into consideration (Table 1). The interactive effects of the variables on the oil yield were considered by allowing for the linear (X_1, X_2, X_3), the interaction (X_1X_2, X_1X_3, X_2X_3) and the quadratic (X_1^2, X_2^2,

X_3^2) on the response. The variable interactive effects also lead to the formation of regression equation (Eq. 1) and explained vividly how the contour and three-dimensional plots are obtained.

C. Optimization of Dacryodes edulis Seed Oil Extraction

The important part of any regression analysis is to determine whether or not the standard assumptions of the simple linear regression model are satisfied. The relationship between the response variable (oil yield) and selected variables (X_1; X_2; X_3) are assumed to be in form of linear, interactions and quadratic (Eq. 1). To check the model accuracy and model estimation capabilities, the coefficient of determination (R^2) was determined by estimating these parameters using Eqns. (4). Their values were used together to juxtapose the Box Behnken design and genetic algorithm models by comparing the evaluated values for the models.

$$R^2 = 1 - \sum_{i=1}^{n} \frac{(\emptyset_{i,cal} - \emptyset_{i,exp})^2}{(\emptyset_{avg,exp} - \emptyset_{i,exp})^2} \quad (4)$$

where $\emptyset_{i,exp}$ is the experimental value, $\emptyset_{i,cal}$ is the calculated value and $\emptyset_{avg,exp}$ is the average experimental value.

IV. RESULTS AND DISCUSSION

D. Optimization of oil extraction

Table 2 depicts the 17 experimental generated, the oil yield results obtained together with the predicted oil yield and the residual values. It was observed that the lowest yield obtained was 8.1188% (w/w) and the predicted values for RSM and ANN couple with genetic algorithms were 8.0668% (w/w) and 8.5722% (w/w), respectively. Meanwhile, the highest yield obtained was 17.8780% (w/w), while the predicted values for RSM and ANN were 17.726% and 17.876% (w/w), respectively. The predicted values were validated in triplicate, and an average of 17.46 % (w/w) and 17.72% (w/w) were obtained for RSM and ANN, respectively.

Table 3 showed the results of test of significance for all coefficient of regression. The F-values of X_1, X_2, X_1X_2 and X_1X_3, implies the significant model terms. Values of "Prob > F" less than 0.0500 indicate model terms are significant. Table 4 showed the results of the Analysis of Variance (ANOVA). The analysis of variance is important to test significance and suitability of the model and to determine whether the variation from the model is significant when compared to the variation due to residual error at 95% confidence level. The "Lack of Fit" compares the residual error to the pure error from simulated design points. The lack of fit F-value of 11.37 with p-value 0.199 implies insignificant lack of fit relative to pure error.

The mathematical expression of the relationship between oil yield and the variable factors is given by the model equation 4. All negative values have reduction impact on the yield while positive values have ability to increase the yield (Table 5).

$$OY (\%) = +12.48 - 1.01X_1 + 1.28X_2 - 1.44X_3 - 1.78X_1X_2 - 0.98X_1X_3 - 1.00X_2X_3 - 0.59X_1^2 + 0.095X_2^2 - 1.861X_3^2$$

To test the fit of the model equation, the regression model was established using R^2 as a measure of how much variability in the observed response values can be explained by the experimental factors and their interactions (Sudamalla *et al*., 2012). The R^2 value is always between 0 and 100% (Haider and Pakshirajah, 2007; Schinas *et al.*, 2009). However, to create a good-fit model, it was recommended that R^2 should not be less than 80% (Joglekar and May 1987). The results in Table 4 indicated an R^2 value of 84.54% for RSM and 87.12% for ANN, which leaves only 15.46% and 12.87% of the variability, not explains by oil yield, this indicates that an unexplainable total variation could be caused by other factors, which were not included in the model. Figure 1 shows the relationship between the predicted plots (RSM and ANN) and the actual experimental oil yield.

A graphical drawing depicts a functional relation between two or three variables by means of a curve or surface containing only those points, whose coordinates satisfy

Table 2: Box-Behnken Experimental Design for Three Independent Factors.

Std. run	X_1	X_2	X_3	Oil yield %(w/w)	Predicted RSM	Predicted ANN	Residual RSM	Residual ANN
1	1	1	0	13.1102	13.0282	13.129	1.69	0.019236
2	0	0	0	17.8780	17.826	17.875	6.47	0.0030479
3	0	1	1	13.5602	13.5082	12.476	0.40	1.0838
4	0	-1	1	12.6760	12.624	12.476	-0.55	0.19955
5	1	-1	0	9.6400	9.588	9.7187	-1.77	0.078717
6	0	1	-1	8.8878	8.8358	8.9299	-1.85	0.042103
7	-1	0	-1	9.0790	9.027	9.0722	-0.16	0.0067995
8	0	0	0	12.1744	12.1224	12.476	0.55	0.30205
9	0	0	0	9.9718	9.9198	9.958	-2.25	0.013816
10	-1	0	1	11.4024	11.3504	12.476	-8.34	1.074
11	0	0	0	9.4003	9.3483	9.3893	-1.69	0.011029
12	-1	1	0	12.5876	12.5356	12.476	2.25	0.11115
13	1	0	1	8.1188	8.0668	8.5722	-3.29	0.4534
14	1	0	-1	9.2843	9.2323	8.7767	-0.40	0.50758
15	-1	-1	0	14.1445	14.0925	14.151	1.85	0.0062827
16	0	-1	1	11.3265	11.2745	11.305	0.16	0.021387
17	0	0	-1	10.0145	9.9625	10.034	-1.40	0.019628

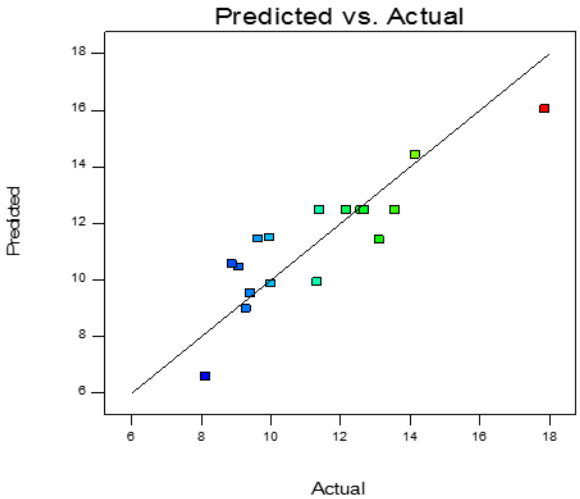
Table 3: Test of Significance for All Regression Coefficient Term.

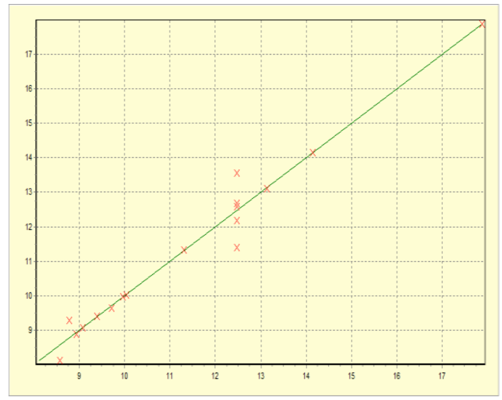
Source	Sum of squares	df	Mean Square	F-value	p-value
X_1	18.23	1	18.23	12.45	0.01617
X_2	13.10	1	13.10	13.89	0.0391
X_3	16.67	1	16.67	8.96	0.0613
X_1X_2	12.65	1	12.65	11.76	0.0437
X_1X_3	3.82	1	3.82	10.13	0.04600
X_2X_3	4.03	1	4.03	1.20	0.3101
X_1^2	1.49	1	1.49	10.44	0.05277
X_2^2	0.038	1	0.038	0.011	0.9186
X_3^2	14.63	1	14.63	4.35	0.0755

Table 4: Analysis of variance (ANOVA) of regression equation.

Source	Sum of squares	df	Mean Square	F-value	p-value
Model	175.16	9	19.46	12.48	0.0122
Residual	23.55	7	3.36		
Lack of fit	21.08	3	7.03	11.37	0.199
Pure error	2.47	4	0.62		
Correction for total sum	98.71	16			

R^2_{RSM}: 84.54% R^2_{ANN}: 87.12%





RSM **ANN**

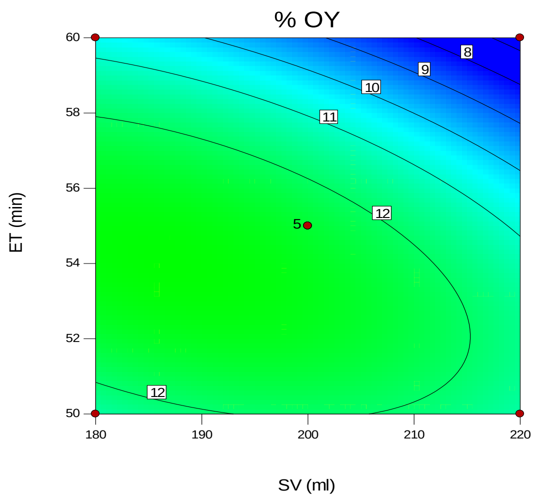
Figure 1: Actual yield Vs. Predicted values.

the relationship between the response and the experimental levels of each variable on the one side, and the type of interactions between the test variables, on the other, which allows for deducing the optimum conditions. The interaction effects of solvent volume (SV), sample weight (SW) and extraction time (ET) on the oil yield were studied using the contour plots and 3D surface plots of RSM (Figure 2 (a-d)). From the plots, it was observed that the oil yield increased with decreases in ET and SV while keeping SW constant at zero level. The curvature nature of the surface plots in Figures 2 (b-e) depict mutual interactions between ET and SV

Table 5: Regression Coefficients and Significance of Response Surface Quadratic

Factor	Coefficient estimate	df	Standard error	95% CI Low	95% CI High	VIF
Intercept	12.48	1	0.82	10.54	14.42	
X_1	-1.01	1	0.65	-2.55	0.52	1.00
X_2	1.28	1	0.65	-0.25	2.81	1.00
X_3	-1.44	1	0.65	-2.98	0.090	1.00
X_1X_2	-1.78	1	0.92	-3.95	0.39	1.00
X_1X_3	-0.98	1	0.92	-3.15	1.19	1.00
X_2X_3	-1.00	1	0.92	-3.17	1.17	1.00
X_1^2	-0.59	1	0.89	-2.71	1.52	1.01
X_2^2	0.095	1	0.89	-2.02	2.21	1.01
X_3^2	-1.86	1	0.89	-3.98	0.25	1.01

Figures 2 (b-e) depict mutual interactions between ET and SV on oil yield. It was observed that an increased in SW and SV favoured the high yield of the oil, while low SV and low SW reduced the oil yield. Figures 2 (c-f) showed the interaction between the SW and ET on the yield of oil. It was also observed that the high SW with low ET produced the highest oil yield. Further increase in ET reduced the yield of the oil.



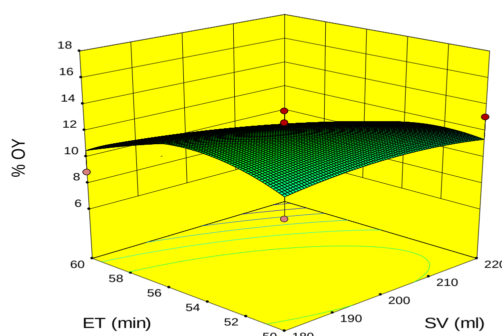


Fig. 2(a)

The RSM and ANN plots of contours and the 3 D's showed that the highest oil yield was obtained at high SW, low ET and low SV (Figures 2(b-e & c-f)).

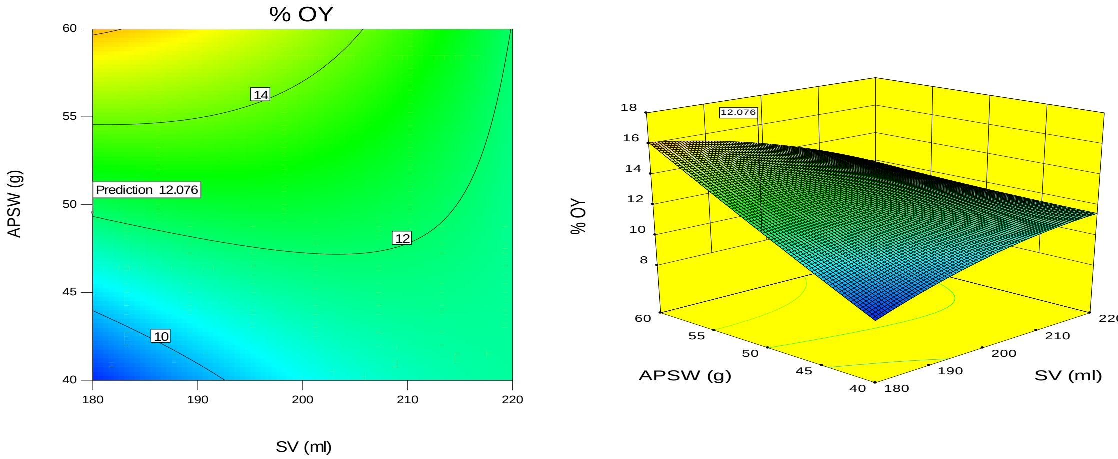


Fig. 2(b)

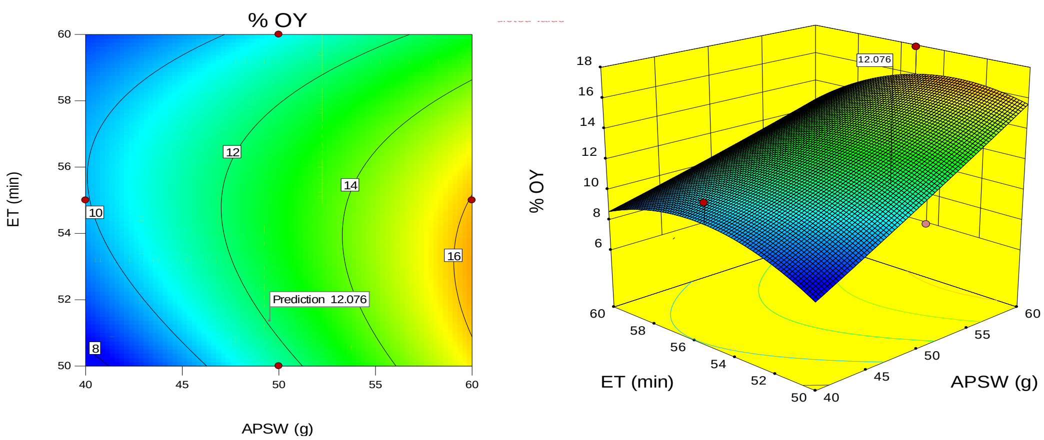


Fig. 2(c)

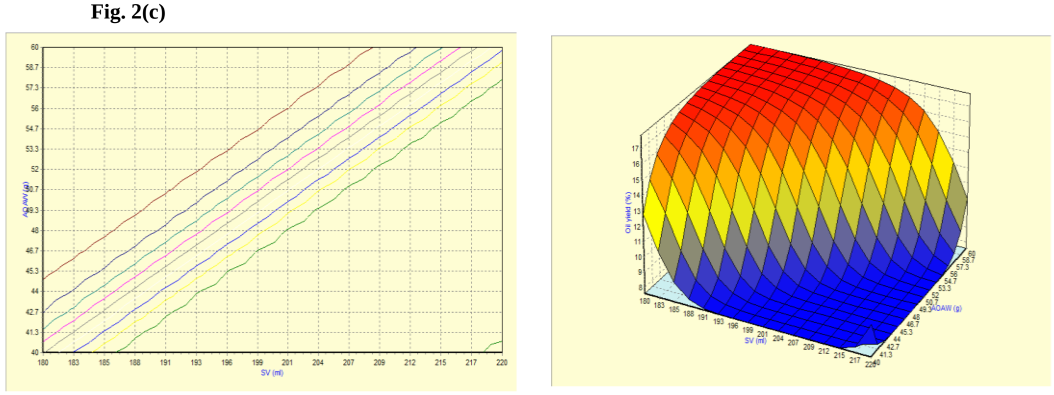


Fig. 2(d)

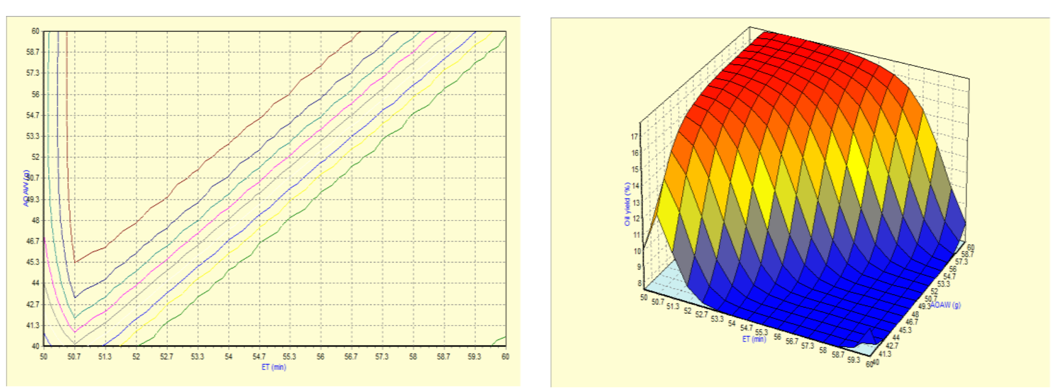
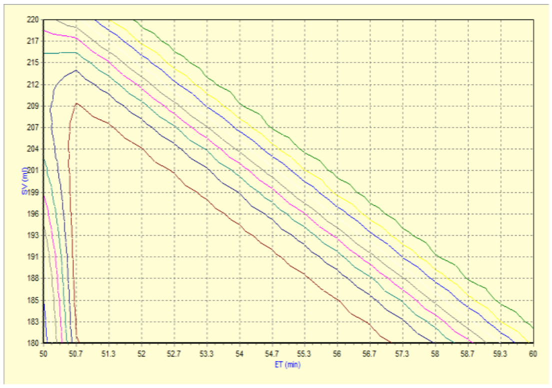


Fig. 2(e)



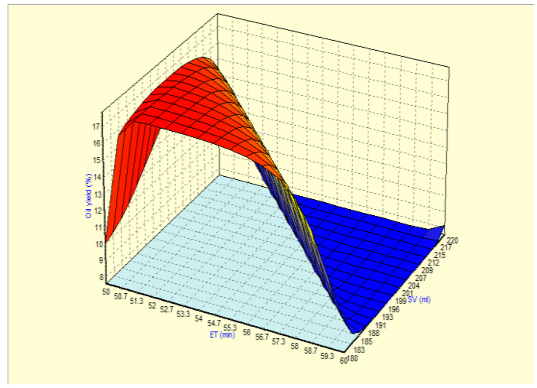


Fig. 2(f)

Figure 2(a-f): Contours and 3- Dimensional plots by RSM and ANN.

E. Physiochemical Properties of African Pear Oil (APO)

Table 6 showed the results of the physicochemical properties of the extracted oil obtained using (AOAC, 1997) standard methods. The oil obtained was liquid, brownish in colour with the moisture content of 0.04549% and specific gravity of 0.9217. The high acid value of 27.072 corresponding to high FFA of 13.036 obtained in this study indicated the good resistance of the oil to hydrolysis. Peroxide value measures the content of hydroperoxides in the oil (Mcginely, 1991) and its high value (37.60 meq. O_2/kg) indicates high resistance to oxidation.

High saponification value of 129.03 mg KOH/g with a high iodine value of 78.09 gI_2/100 g indicated a low concentration of triglycerides and the oil contained a substantial level of unsaturation. The high heating value of 42.968 MJ/kg takes into account the latent heat of vaporization of water in the combustion products. Fuel properties such as Cetane number, a measure of the fuel's ignition delay and combustion quality, and its fuel standard is a minimum of 40 (Meher *et al*., 2006; Ramos *et al*., 2008). The high value of 71.03 obtained in this study showed the oil has high fuel potential.

Table 6: Physiochemical Properties of oil.

Parameters	***Values***
Physical properties	
Absorbance @660ºC	2.032
Moisture content (%)	0.04549
Specific gravity	0.9217
Mean Molecular mass	434.008
Viscosity @34.2ºC (N.s/m^2)	1.282
Chemical Properties	
Free Fatty Acid	13.036
Acid Value (mg KOH/g oil)	27.072
Saponification Value (mg KOH/g oil)	129.03
Iodine Value (g I_2/100g oil)	78.09
Peroxide Value (meq O_2/kg oil)	37.60
Higher Heating Value (MJ/kg)	42.968
Other Properties	
Cetane number	71.0299

Dacryodes edulis seeds oil extraction showed the highest oil yield of 17.878% (w/w) at solvent volume of 200 ml, sample weight of 50 g and extraction time of 55 min, respectively. However, response surface methodology predicted an oil yield of 17.826 % (w/w), while the artificial neural network predicted 17.875 % (w/w) at the same variables condition. The predicted values were validated in triplicate, and an average of 17.46 % (w/w) and 17.72% (w/w) were obtained for RSM and ANN, respectively. The predicted values obtained were well within the range predicted. The coefficient of determination, which determines the model accuracy, was obtained to be 0.8454 for RSM and 0.8712 for ANN. Physicochemical analysis of the oil showed the oil is highly unsaturated with high saponification value and high iodine value.

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