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Optimization of Solvent Extraction of Oil From Wild Bush Mango Seed (Irvingia Gabonensis) Using Rsm

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Abstract

Four operating parameters of the solvent extraction of oil from seed of wild bush mango plant (irvingia gabonensis) using normal hexane as the solvent have been optimized by a response surface method using central composite design. A linear model was developed, and validated with diagnostic plots.

The model was optimized numerically and the optimum conditions established were; temperature of 40^{o}c, particle size of 2.05MM, time of 4hours and solvent volume of 300mls at a constant value of the sample dosage of 10grams. The predicted oil yield was 42.4167% at 0.767 desirability. There was a good correlation between the predicted and actual values.

Keywords: ANOVA, Normal Hexane, Oil extraction, Optimization, RSM,

Introduction

Oil extraction is the process of recovering oil from oil bearing agricultural product through manual, mechanical, or chemical extraction (Ibrahim and Onwualu, 2005).

Nut oil, seed oils and oils of fruits and vegetables are receiving growing interest due to their high concentration of bioactive lipid components, such as polyunsaturated fatty acids and phytosterols, which have show various health benefits (Maria et al.,2012). Fats and oils, and their several lipid components are extensively used in the food and also in cosmetics, pharmaceuticals, oleochemicals and other industries.

The extraction of oil from oil bearing product could be done in two major ways; traditional and improved mehods.The traditional method is usually a manual process and involves preliminary processing and hand pressing. The improved method consists of chemical extraction and mechanical expression.

The chemical extraction method required the use of organic solvents to recover the oil from the product (Ibrahim and Onwualu, 2005). Mechanical method involves the application of pressure to already pre-treated oil bearing products. It employs the use of device like screw and hydraulic presses as a means of applying the pressure (Gunstone and Morris, 1983). Whichever method that is employed, researchers(Norris, 1964; Ward, 1976; Khan and Hanna, 1983; Adekola, 1992) reported that the yield and quality of the oil extracted depends on the content adjustment, heating time, pressure application, operating temperature etc.

Solvent extraction method which was used in this work involves the use of organic solvent such as straight chain hydrocarbons, chlorinated hydrocarbons, alcohols, and ketones to recover the oil from the sources. Solvent extraction is capable of removing nearly all the available oil from oil seed or nuts. About 98% of the oil is being extracted by solvent method (Ihekoronye and Ngoddy, 1985; Cecoco, 1988)

Response surface methodology (RSM) is a relatively new method of optimization of experimental conditions. It is suited for solving nonlinear data processing issues. It has many advantages like relatively simple calculation, less number of experiments, short cycle and regression equation with high accuracy (Weixu et al, 2011).It is an effective method to reduce development costs, optimize process conditions, improve product quality, and solve practical problem in the product process by analyzing the resulting graph. While the tradition method of mathematical statistics often employ orthogonal or uniform designs that can simultaneously consider several factors to optimize factor level. They cannot find the regression equation based on the given factors of the entire region between the values and the response function to choose the best combination (Weixu et al, 2011).

The sample used for this study was the seed of a wild bush mango plant called the African mango (Irvingia gabonensis). Traditionally in Igbo land, it is used in preparing a protein rich delicacy called Ogbono soup. This plant grows freely in the tropical rain forest of African, and its fruit, the African bush mango is eaten all across this region. The seed of sample can be obtained by collecting the bush mango's seed, split this to obtain a pearly white ovoid kernel that is sun dried.

The aim of this work was to optimize the process factor used in solvent extraction of African bean seed oil. The process factor considered were temperature, particle size, volume of solvent and time.

Materials and Methods

African Bean Seeds

The dehulled bean African seeds were bought from local Abakpa market in Enugu state Nigeria. The seeds were stored in dark air-tight containers to prevent photo-oxidation as well as to minimize moisture adsorption.

Extraction Method

The solvent extraction method using normal hexane was used to optimize the extraction parameters for maximal production of oil. The important factors of extraction process considered for this study were; temperature of extraction, particle size of the seed and the volume of solvent used. The experimental runs were based strictly on the design matrix obtain from central composite design. Each extraction run was set up by measuring 10g of the sample into a known volume of solvent in a corked conical flask. The mass of the sample was held constant thought the experimental run. The mixture was placed in a thermostatic water bath operating at a predetermined temperature. At the end of each time interval, the sample was taken, centrifuged for 20mins to separate the solid fraction from the solution. The extracts were heated and evaporated using heating mantle to obtain solvent free oil.

The percentage oil yield was calculated for all runs using the expression below

$$Y = \frac{W_O(100)}{W_m}$$

Where Y is the oil yield (%), W_o is the weight of oil expressed in grams and W_m is the weight of the sample of milled seed used in the experiments in grams.

Experimental Design

A centre composite design of the RSM is the most commonly used in optimization experiments. The method includes a full or fractional factorial design with center points that are augmented with a group of star points (extreme values) to allow the estimation of the curvature (Bhattacharjee, 2007). As the distance from the design space to a factorial point is defined as ±1 unit for each factor, the distance from the center of the design space to a star point is ±α with |α|>1 (Luu et al, 2009). The important parameters optimized were extraction temperature (°C), extraction time (hours), volumes of Solvent (mls) and the particle size (MM). The operation conditions were varied at 5 levels as in Table 1.

Table1.Levels of Independent Variables

	Variables	Units	Levels				
			-x	-1	0	1	x
A:	Time	Hours	1	2	3	4	5
B:	Particle size	MM	1.5	2.05	2.6	3.15	3.7
C:	Temperature	^{0}C	35	40	45	50	55
D:	Solvent Volume	mls	150	200	250	300	350

The design required 30 experiments with sixteen factorial points, eight extra points (star points) and six replication of the central point. The experimental design layout used for the experimental runs is shown on the table 2 below.

Experimental yields were analyzed by a response surface method to fit a second order Polynomial equation

$$Y = ß_0 + \sum_{i=1}^{n} ß_i x_i + \sum_{j=i+1}^{n} ß_{ii} x_i^2 + \sum_{i=1}^{n-1} \sum_{i=1}^{n} ß_{ij} x_i x_j$$

Where Y represents the response, oil yield, $ß_0$ is a constant, $ß_i$, $ß_{ii}$ and $ß_{ij}$ are the linear, quadratic and interactive coefficients respectively, n is the number of independent variables. x_i and x_j are the coded variables which are related to the original variable as follow (Luu et al, 2009)

$$X = \frac{\text{Original Variable - midpoint of Original interval}}{\text{Interval of original range}}$$

Table 2. Design Layout with the Experimental and Predicted Values.

Std order	Run order	Time (HRS)	Particle size (MM)	Temp (^oC)	Solvent Volume (mls)	Experimental Value (%)	Predicted Values (%)
30	1	3.00	2.60	45.00	250.00	36.00	37.33
25	2	3.00	2.60	45.00	250.00	37.00	37.33
6	3	4.00	2.05	50.00	200.00	33.00	36.25
16	4	4.00	3.15	50.00	300.00	43.00	41.08
26	5	3.00	2.60	45.00	250.00	36.00	37.00
21	6	3.00	2.60	35.00	250.00	36.50	36.92
23	7	3.00	2.60	45.00	150.00	34.00	30.75
7	8	2.00	3.15	50.00	200.00	30.00	32.25
9	9	2.00	2.05	40.00	300.00	43.50	40.17
24	10	3.00	2.60	45.00	350.00	46.00	43.92
2	11	4.00	2.05	40.00	300.00	353.50	35.83
14	12	4.00	2.05	50.00	300.00	45.00	42.83
11	13	2.00	3.15	40.00	300.00	35.00	38.42
3	14	2.00	3.15	40.00	200.00	34.00	31.83
4	15	4.00	3.15	40.00	200.00	34.00	34.08
1	16	2.00	2.05	40.00	200.00	38.50	33.58
29	17	3.00	2.60	45.00	250.00	39.00	37.33
18	18	5.00	2.60	45.00	250.00	46.00	39.58
17	19	1.00	2.60	45.00	250.00	36.00	35.08
5	20	2.00	2.05	50.00	200.00	35.50	34.00
13	21	2.00	2.05	50.00	300.00	36.50	40.58
8	22	4.00	3.15	50.00	200.00	30.50	34.50
27	23	3.00	2.60	45.00	250.00	35.00	37.33
22	24	3.00	2.60	55.00	250.00	42.00	37.75
19	25	3.00	1.50	45.00	250.00	35.00	39.08
12	26	4.00	3.15	40.00	300.00	36.00	40.67
15	27	2.00	3.15	50.00	300.00	40.00	38.83
28	28	3.00	2.60	45.00	250.00	30.00	37.33
10	29	4.00	2.05	40.00	300.00	45.00	42.42
20	30	3.00	3.70	45.00	250.00	38.50	35.58

Results and Discussion

Model Fitting and Summary Statistics.

Four key factors, namely extraction time (hrs), extraction temperature (^oc), solvent volume (mls) and particle size (MM) were chosen to study the extraction process using the CCD design. Multiple regression analysis was done for the data of the yield of African seed oil in the table 2 using Design expert software.

According the to summary statistics the model was selected based on the highest order polynomials where the additional terms were significant and the models were not aliased, no lack of fit (p-value >0.10) and reasonable agreement between adjusted R-squared and predicted R- square(within 0.2 of each other).

Table 3. Summary Statistics

Source	Sequential p-value	Lack of Fit p-value	Adjusted R-Squared	Predicted R-Squared	
Linear	0.0013	0.3586	0.4181	0.2682	Suggested
2FI	0.5273	0.3281	0.4010	-0.0595	
Quadratic	0.5170	0.2934	0.3811	-0.5276	
Cubic	0.2864	0.3221	0.5230	-51472	Aliased

Sequential Model Sum of Squares [Type 1]

Source	Sum of Squares	df	Mean Square	F Value	p-value prob>F	
Mean vs Total	41813.33	1	41813.33			
Linear vs Mean	309.83	4	77.46	6.21	0.0013	Suggested
2FI vs Linear	67.88	6	11.31	0.88	0.5273	
Quadratic vs 2FI	44.96	4	11.24	0.85	0.5170	
Cubic vs Quadratic	127.42	8	15.93	1.56	0.2864	Aliased
Residual	71.58	7	1023			
Total	42435.00	30	1414.50			

Lack of Fit Tests

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob>F	
Linear	266.33	20	13.32	1.46 ,	0.3586	Suggested
2FI	198.46	14	14.18	1.56	0.3281	
Quadratic	153.50	10	15.35	1.69	0.2934	
Cubic	26.08	2	13.04	1.43	0.3221	Aliased
Pure Error	45.50	5	9.10			

Model Summary Statistics

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	3.53	0.4984	0.4181	0.2682	454.91	Suggested
2FI	3.58	0.6076	0.4010	-0.0595	658.65	
Quadratic	3.64	0.6799	0.3811	-0.5276	949.68	

For this study, the linear model was suggested based on the summary statistics in table 3 above. The obtained linear model in terms of coded factors is shown below

$Y = +37.33 + 1.12A – 0.88B + 0.21C + 3.29D$

The final equation in terms of actual factors is

Yield = +19.69553 + 1.12500 Time – 1.590991 Particle size + 0.041667 Temperature + 0.65833 Solvent Volume.

Where Y is the oil yield in (%), A is the time in hrs, B is the particle size in (MM), C is the Temperature in (^{0}c) and D is the solvent volume in (mls). The positive sign in front of the term

indicates synergistic effect whereas the negative sign indicates antagonistic effect (Tan et al, 2011).

Analysis of the variance

When the model was selected, an analysis of variance was calculated to asses how well the model represented the data. The analysis of variance for the responses is presented on table 4.

Table 4 : ANOVA TABLE

Source	Sum of squares	df	Mean squares	F value	p-value probe >F
Model	309.83	4	77.46	6.21	0.0013
A-Time	30.38	1	30.38	2.44	0.01312
B-particle size	18.38	1	18.38	1.47	0.2362
C-Temperature	1.04	1	1.04	0.084	0.7750
D-solvent	260.04	1	260.04	20. 85	0.0001
Residual	311.83	25	12.47		
Lack of fit	266.33	20	13.32	1.46	0.3586
Pure Error	45.50	5	9.10		
Cor Total	621.67	29			

The model F- value of 6.21 implied the model was significant. There was only a 0.13% chance that a "model F- value" this large could occur due to noise. The lack of fit F-value of 1.46 implied that the lack of fit was not significant relative to the pure error. There was a 35.86% chance that a "lack of fit F- value" this large could occur due to noise.
The "pred R- squared" of 0.2682 was in reasonable agreement with the Adj R-squared" of 0.4181. ''Adeq precision'' measures the signal to noise ratio. A ratio greater than 4 is desirable. The obtained ratio of 9.132 implied an adequate signal. The model can be used to navigate the design space.

Model Diagnostic Plots

The model diagnostic plots were used to observe how well the model satisfied the assumptions of the analysis of variance. Most of the splots displayed residuals.

1. **Normal plot of residuals**. The normal probablility plot indicates whether the residuals followed a normal distribution, in which case the points will follow straight line, even though a moderate scatter can be observed even with normal data.

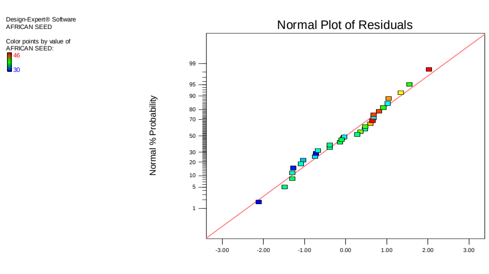

Fig 1. Normal plot of residuals

If a definite pattern like "S- shape" curve is observed, definitely, a transformation of the responses will provide a better analysis.

2. **Residual vs predicted plot**. This is a plot of the residuals versus the ascending predicted response values. It tested the assumption of constant variance. The plot should have a random scatter (constant range of residual across the graph).

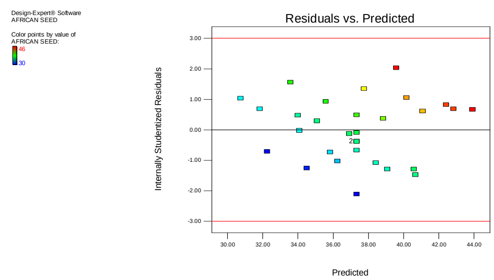

Fig 2. Residuals vs. predicted plot

3. **Residual vs. Run**. This is a plot of the residuals versus the experimental run order. It allowed one to check for lurking variables that may have influenced the response during the experiment. The plot should show random scatter. Trends indicate a time-related variable lurking in the backgrounds.

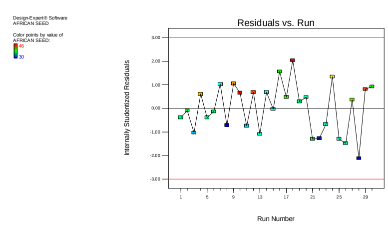

Fig 3: Residuals vs. Run plot

Blocking and randomization provided insurance against trend ruining the analysis.

4. **Produced vs. Actual**. A graph of actual response values versus the predicted response values helped to detect a value or group of values that were not easily predicted by the model. The data point should be split evenly by the 45 degree line.

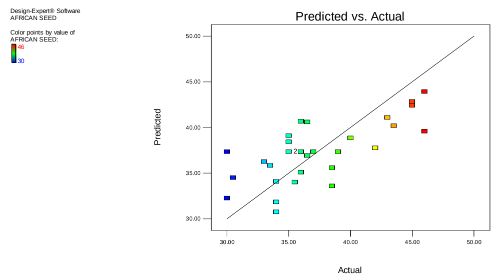

Fig 4: Predicted vs. Actual plot

The diagnostic plots showed above indicated the models satisfied the assumptions of ANOVA.

Interaction effects

The interaction effects of the process variables were analyzed by contour plots and 3D surface plots.

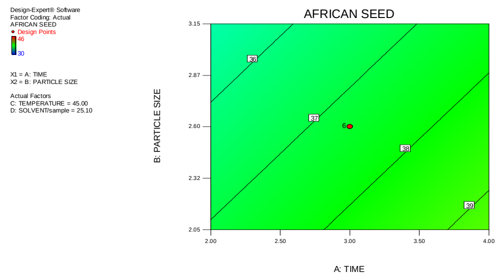

Fig 5a: Contour plot of particle size with time

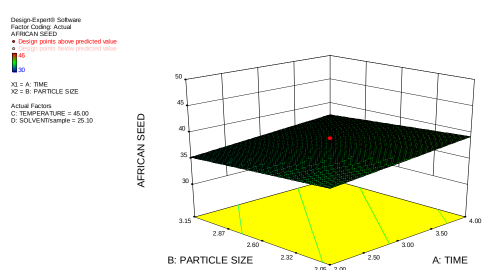

Fig 5b: 3D surface plot of particle size with time.

The 3D surface and contour plots of the interaction effects of particle size with time in figure 5 above showed that as particle size was decreased with increase in time, the oil yield increased.

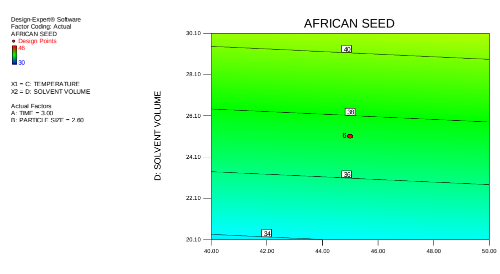

Fig 6a: Contour plot of solvent volume with temperature.

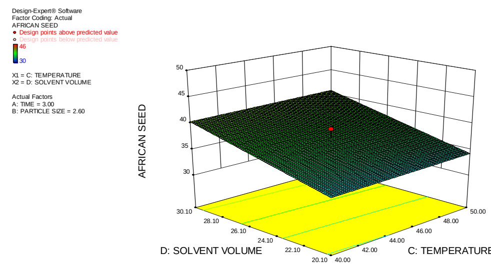

Fig 6b: 3D surface plot of solvent volume with temperature.

The 3D surface and contour plots of the interaction effects of solvent volume and temperature in figure 6 above showed that as the solvent volume was increased with increase in temperature, the oil yield also increased.

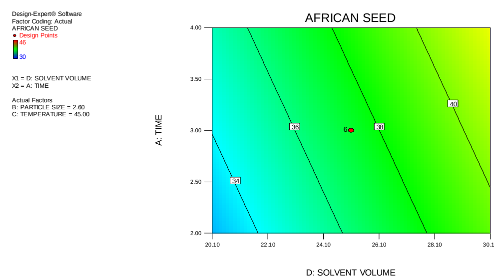

Fig 7a: Contour plot of solvent volume with time.

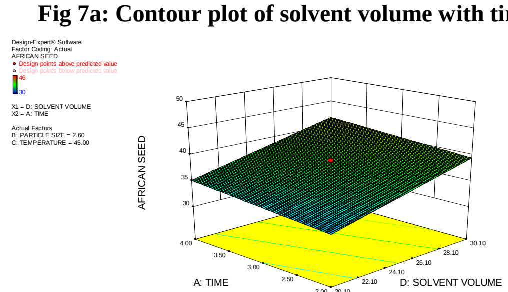

Fig 7b: 3D surface plot of solvent volume and time.

The 3D surface and contour plots of interaction effects of Time and solvent volume in figure 7 above showed that the oil yield increased as time and solvent volume were increased.

Optimization

Once the model has been developed and validated as a good and useful one, it can be optimized. Optimization searches for a combination of factors levels that will give the best response. The goal here was to maximize the oil yield within the lower and upper limits of 30 and 46 respectively. This goal was combined into an overall desirability function. The program seeks to maximize the function. The goal seeking begins at a random starting point and proceeded up the steepest slope to a maximum. With a weight of one and important of + + +, 45 solutions were found. The optimum condition obtained within the specified conditions were time of 4hours, particle size of 2.05MM, temperature of 40^0c, solvent volume of 300mls with the predicted yield of 42.4167 at 0.767 desirability.

Conclusion

The optimization of process conditions for the extraction of oil from the seed of a wild bush mango plant has been done using Response Surface Methodology. The solvent extraction method using normal hexane as the extracting solvent was used. The process parameters studied were temperature (^{0}c), particle size (MM), time (hours) and solvent volume (mls). A linear model was developed, diagnosed and optimized. The optimal conditions obtained were temperature of 40^0c, time of 4hrs, particle size of 2.05MM and solvent volume of 3000ml at a constant dosage of the sample. The produced oil yield was 42.4167% at desirability of 0.767.

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