Optimization Of Process Parameters of Soxhlet Extraction Methods for African Locust Bean Oil Yield Using Response Surface Methodology

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Abstract- ***African locust bean Oil was extracted using soxhlet extraction method by varying some of the process parameters that affect the yield of oil extracted from oil bearing seeds. The parameters varied include seed moisture content, extraction temperature and extraction time. Response surface methodology was used to design the experimental matrix by using Box-Behnken design in other to achieve the optimum oil yield from African locust bean seed. It was observed that oil yield ranges from 13.58% to 27.98%. The highest percentage oil yield of 27.98% was obtained at moisture content of 12%, temperature of 60^0C and extraction time of 6hrs. The result obtained was subjected to statistical analysis, a quadratic model was develop and found to be significant ($p< 0.0001$). The developed model shows predicted R^2 of 0.8789, which is in reasonable agreement with the adjusted R^2 of 0.9827 indicating that the model satisfactorily predicted of the oil yield. The optimization result reveals optimum oil yield of 28.556% obtained from Locust Beans at the combination of 6.31706% moisture content, 59.9528^{0}C extraction temperature and extraction time of 5.89658hr. The predicted variables obtained were subjected to experimental validation and oil yield of 28.58% was obtained. The validation shows a close value with the predicted value. Results obtained in this study revealed that response surface methodology helps to optimize oil extraction process parameters to achieve optimum oil yield from oil bearing seeds.***

Indexed Terms- ***African locust bean,Box-Behnken Optimization, Process Parameters, Response surface methodology.***

I. INTRODUCTION

The benefits that accrue from oilseed processing and sales to individual or grouped farmers, companies, and even to national economies cannot be overemphasized because two of its major products oils and meal or cake are both of great commercial value [1]. Vegetable oils account for 80% of the world's natural oils and fat supply [2]With an ever increasing demand for vegetable oils for food and industrial applications, there is need for considerable expansion of oilseed crop production [3]. This expansion is possible by exploring other sources of vegetable oils, especially underutilized oilseeds [4] such as African locust bean. The African locust bean tree is a perennial tree, which belongs to sub-family, Mimosodee and family Leguminosae (now Fabaceae) [5]. Locust bean tree is a leguminous crop peculiar to the tropics. The tree is not normally cultivated but can be seen in population of two or more in the savannah region of West Africa [6].

Over the last decades researchers have focussed lots of efforts on the design of efficient and more sustainable methods of oil extraction from oil bearing seeds which are less detrimental to the environment [1]. Soxhlet extraction is the most commonly used method of extracting oil from oil bearing seeds [7], as this extraction method has various advantage of total recovery of extracts and less time and solvent consuming [8]. The optimization of oil from oil bearing seeds require the application of statistical software to optimize the extraction process parameters that seek reduce cost and increase performance [1]. Hence, this study was carried out to extract and optimise oil yield from African locust bean using Box-Behnken of response surface methodology.

II. MATERIALS AND METHODS

African locust bean was collected manually by hand from Igboora, Ibarapa central local Government, Oyo state. The Sample was cleaned thoroughly,

sorted to remove the deteriorated ones and oven dried at a temperature of 50^0C before milling into fine particle size before variation of moisture content. The extraction process was conducted at the teaching and research laboratory of Oyo State College of Agriculture and Technology, Igboora according to the method describe by [9-10] with some modifications. n-hexane of analytical grade was used as solvent.

The design of experiment and optimization was done using Box-Behnken of Response Surface Methodology. The factors considered were the moisture content, extraction temperature and extraction time while the response of the experiment was chosen to be the percentage oil yield obtained African locust bean seed. The maximum and minimum level used for the factors is given in Table 1. Seventeen experimental runs were obtained and carried out using soxhlet apparatus. A quadratic model was developed for the oil yield and the effect of each of the factors considered from analysis of variance (ANOVA), predict oil yield base on the experimented value and determining the optimum extraction conditions.

Table 1 Experimental Matrix

Variables	Symbol	Levels	
		Maximum	Minimum
Moisture content (%)	A	6	12
Extraction temperature (^{0}C)	B	40	60
Extraction time (hr)	C	4	6

III. RESULTS AND DISCUSSION

Table 2

	Process Parameters			Oil Yield (%)		
Run Order	Moisture Content (%)	Extraction temperature (^{0}C)	Extraction Time (hr)	Actual Value	Predicted Value	Residual
1	9	50	5	18.80	18.80	0.0000
2	9	50	5	18.80	18.80	0.0000
3	9	50	5	18.80	18.80	0.0000
4	6	50	4	18.82	19.29	-0.4700
5	6	40	5	21.22	20.84	0.3813
6	9	40	4	13.58	13.49	0.0888
7	9	50	5	18.80	18.80	0.0000
8	9	60	4	21.09	20.48	0.6087
9	9	60	6	25.96	26.05	-0.0888
10	6	50	6	27.98	27.75	0.2275
11	9	40	6	20.19	20.80	-0.6087
12	12	50	4	17.60	17.83	-0.2275
13	9	50	5	18.80	18.80	0.0000
14	6	60	5	24.76	24.90	-0.1388
15	12	40	5	15.43	15.29	0.1387
16	12	50	6	22.71	22.24	0.4700
17	12	60	5	23.09	23.47	-0.3813

Table 3 ANOVA for Quadratic model of Oil yield

Model	213.85	9	23.76	101.95	< 0.0001	Significant
A-moisture content	24.33	1	24.33	104.37	< 0.0001	
B-extraction temperature	74.91	1	74.91	321.41	< 0.0001	
C-extraction time	82.88	1	82.88	355.63	< 0.0001	
AB	4.24	1	4.24	18.21	0.0037	
AC	4.10	1	4.10	17.59	0.0041	
BC	0.7569	1	0.7569	3.25	0.1145	

A^2	15.99	1	15.99	68.61	< 0.0001
B^2	0.5961	1	0.5961	2.56	0.1538
C^2	4.46	1	4.46	19.12	0.0033
Residual	1.63	7	0.2331		
Lack of Fit	1.63	3	0.5438		
Pure Error	0.0000	4	0.0000		
Cor Total	215.49	16			

Std. Dev.	0.4828	R^2	0.9924
Mean	20.38	Adjusted R^2	0.9827
C.V. %	2.37	Predicted R^2	0.8789
		Adeq Precision	38.5166

Soxhlet extraction method using n-hexane as solvent was used to investigate the effect of extraction process parameters on oil yield of African locust beans. The result of the experimental runs with oil yield as the response is presented in Table 2. It was observed that oil yield ranges from 13.58% to 27.98%. The highest percentage oil yield of 27.98% was achieved at moisture content of 12%, temperature of 60^0C and extraction time of 6hrs. While, the least percentage yield of 13.58% was obtained at the moisture content of 9%, extraction temperature of 40^0C and extraction time of 4hr. The variation in the oil yield as the extraction process parameters changes indicates the extraction process strongly depends on the extraction process parameters. The oil yield from African locust beans obtained in this study was higher than the value reported by [11-13].

The ANOVA results of the analysis describes the relationship between the independent variables (moisture content, extraction temperature and extraction time) and the dependent variable (oil yield) as presented in table 3. The P-value of <0.0001 obtained, shows that the model was significant at 95% confidence level and the model terms A,B,C,AB,AC,A^2 and C^2 are significant.

Final Equation in Terms of Coded Factors

$$Oil\ yield = +18.80 - 1.74A + 3.06B + 3.22C + 1.03AB - 1.01AC - 0.4350BC + 1.95A^2 + 0.3762B^2 + 1.03C^2 \quad (I)$$

Final Equation in Terms of Actual Factors

$$Oil\ yield = +34.68875 - 4.50792A - 0.161750B - 1.85625C + 0.034333\,AB - 0.337500AC - 0.043500BC + 0.216528\,A^2 + 0.003763B^2 + 1.02875C^2 \quad (II)$$

The developed model was used to predict the oil yield and the results obtained were compared with the experimental values as shown in table 2 and figure 1. The predicted R^2 of 0.8789 is in reasonable agreement with the adjusted R^2 of 0.9827 with a difference less than 0.2 indicating that the model satisfactorily predicted of the oil yield.

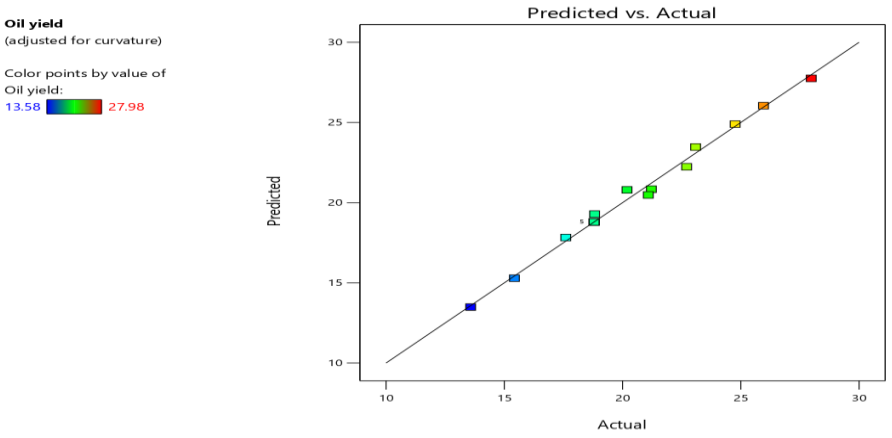

Figure 1 Experimented and predicted value for African locus bean oil yield as affected by process parameters

A Effect of Processing Parameters on Oil Yield of African Locust Bean Seeds

The three-dimensional (3D) surface graphs and contour plots in figure 2 shows the effect of the process parameters (moisture content, extraction temperature and extraction time) on oil yield obtained from African locust bean seeds. The interaction of moisture content and extraction temperature has a significant effect on oil yield from African locust bean as presented in table 2 with a P-value of 0.0037. It was observed that, oil yield increased with increase in temperature and increased with decrease in moisture content. The nature of the plots indicated that, interaction between the moisture content and extraction time had a significant effect (p-value of 0.0041) on the oil yield. The combined effect of extraction temperature and extraction time resulted to an increase in oil yield. Increase in extraction temperature and time favoured oil yield from oil bearing seeds as reported by Ogusola, *et al.*, *(*2022), Siyanbola, *et al.*, *(*2020), *Juliet et al.*, (2017).

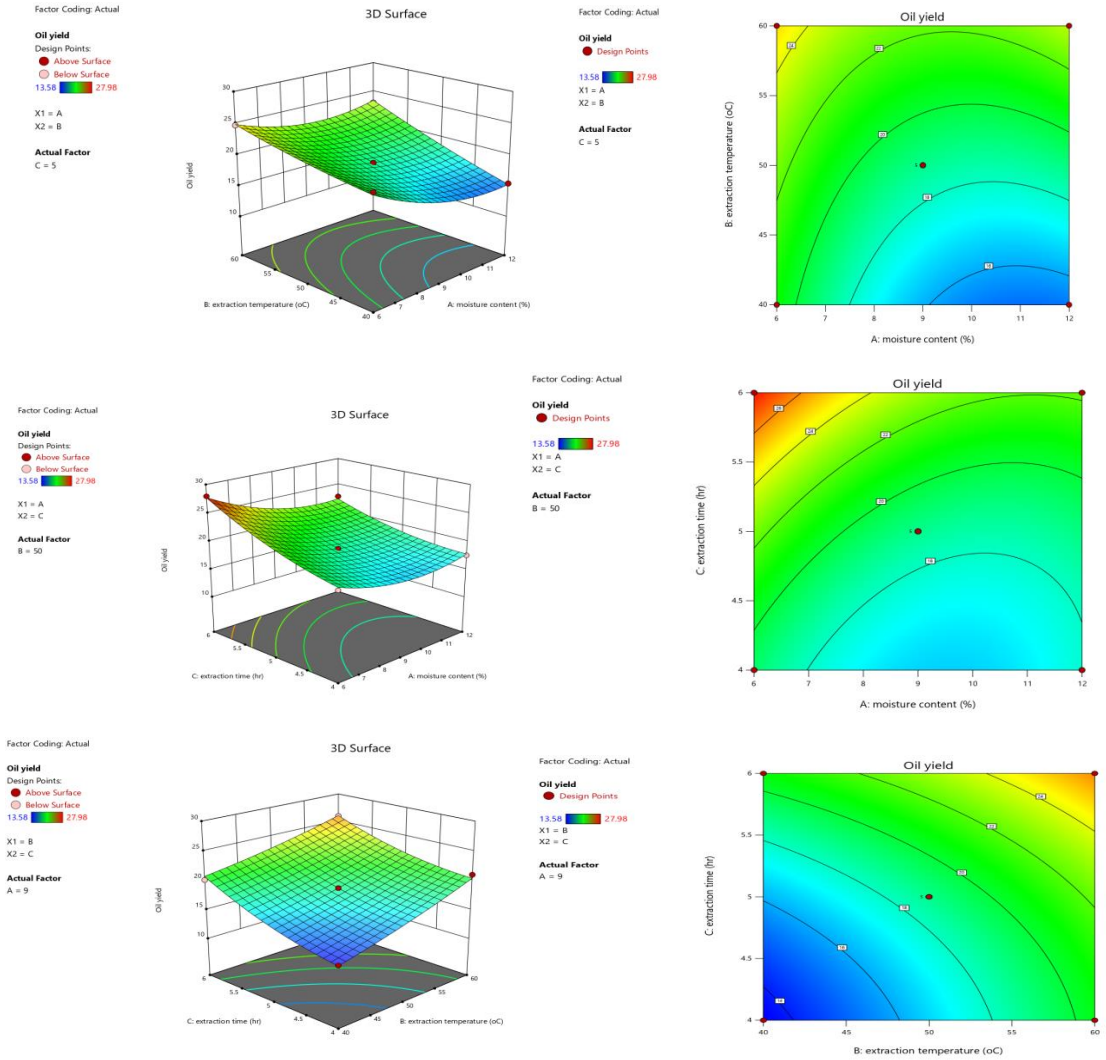

Figure 2 Surface plot for the effect of process parameters on African locust bean oil yield

Table 4 Optimization of Oil Yield from African locust bean seed

Name	Goal	Lower limit	Upper limit	Lower weight	Upper Weight	Importance
A:moisture content	in range	6	12	1	1	3
B:extraction temperature	in range	40	60	1	1	3
C:extraction time	in range	4	6	1	1	3
Oil yield	maximize	13.58	27.98	1	1	3

Response surface methodology was used to optimize the oil yield set to maximum level. It was revealed that the combination of 6.31706% moisture content, 59.9528^{0}C extraction temperature and extraction time of 5.89658hr gave an oil yield of 28.556% as presented in figure 8. The predicted variables obtained were subjected to experimental validation. Oil was extracted using the combined variables predicted and a percentage oil yield of 28.58% was obtained. The validation shows a close value with the predicted value.

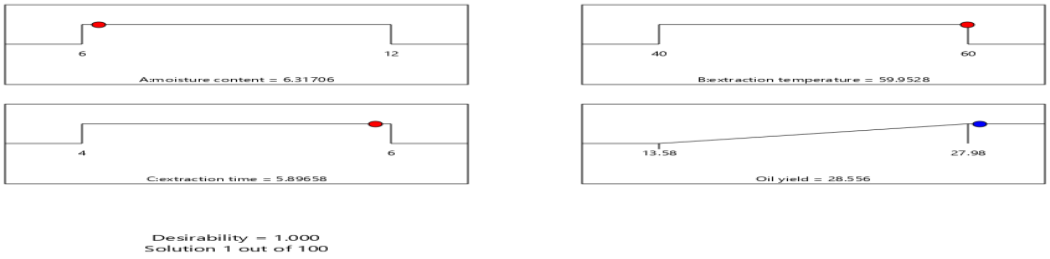

Figure 3 Response optimization of African locus bean oil

CONCLUSION

This study was conducted to optimize the process parameters of soxhlet extraction methods for African locust bean seeds using response surface methodology. African locust bean seeds were obtained and conditioned to various moisture content achieved through experimental design, using Box-Behnken of Response Surface Methodology. Three process parameters were considered (moisture content, extraction temperature and extraction time) while the response of the experiment was chosen to be the yield of the oil obtained from the seed. It was observed that oil yield ranges from 13.58% to 27.98%. The highest percentage oil yield of 27.98% was obtained at moisture content of 12%, temperature of 60^{0}C and extraction time of 6hrs. While, the least percentage yield of 13.58% was obtained at the moisture content of 9%, extraction temperature of 40^{0}C and extraction time of 4hr. The result obtained was analysed to the develop a quadratic model which indicated that, the process parameters has a significant effect (P-value of <0.0001) on the oil yield and was used to predict oil yield value found to be in reasonable agreement with the experimented value. Response surface methodology was used to optimize the oil yield set to maximum level. It was revealed that oil yield of 28.556% was achieved at the combination of 6.31706% moisture content, 59.9528^{0}C extraction temperature and extraction time of 5.89658hr. The predicted variables obtained were subjected to experimental validation. Oil was extracted using the combined variables predicted and a percentage oil yield of 28.58% was obtained. The validation shows a close value with the predicted value.

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