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ORIGINAL RESEARCH ARTICLE

EFFECT OF PROCESSING PARAMETERS ON SOLVENT OIL EXPRESSION FROM LOOFAH SEEDS (LUFFA CYLINDRICA L.) USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT

Luffah cylindrica plant grows in the wild, around uncompleted buildings and fenced walls. The percentage oil composition of its seeds is about 30% oil. The research focused was the extraction oil from loofah seed using a solvent extraction methodology. Optimum conditions for oil extraction were determined using Response Surface Methodology of Central Composite Rotatable Design. A total of 20 experimental runs were used to investigate the optimum condition considering three independent variables at five levels each: extraction temperature (55, 60, 65, 60, 75°C), seed/solvent ratio (0.04, 0.05, 0.06, 0.07, 0.08 g/ml) and extraction time (4, 5, 6, 7, 8 hr.). An empirical model equation that could be used to forecast oil yield as a function of the independent variables was developed. The optimum oil yield obtained was 27.43% at the extraction temperature (74.05°C), seed/solvent ratio (0.05 g/ml) and extraction time (5.35hr). The analysis of variance showed that extraction temperature and time had significant effect on oil yield ($p = 0.05$). The interaction of the independent variables with oil yield gave R^2 and R^2 adj. values of 0.98 and 0.93, respectively. The result showed that the selected independent variables had a significant effect on oil yield, thus an optimum condition was established.

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1.0 Introduction

Luffa cylindrical is a sub-tropical plant, which requires warm summer temperatures and long frost-free growing season when grown in temperate regions. It is an annual climbing plant which produces fruit containing fibrous vascular system. Loofa is derived from the cucumber and marrow family and originates from America (Mazali and Alves, 2005; Oniya et al., 2017). Luffa commonly called bath sponge or dishcloth gourd, sponge gourd, loofa, vegetable sponge, is a member of the cucurbitaceous family. The number of species in the genus Luffa varies are about seven, but only two species, L. cylindrica and L. acutangula, are domesticated (Rowell et al., 2002). Fruits, leaves, flowers, and seeds of Cucurbitaceae have considerable economic value (Bamgboye and Oniya, 2012). Luffa cylindrica linn, commonly known as Loofah is an annual plant commonly found in the tropics including Nigeria. The oil extracted from *L. cylindrica* seed can be used in the biodiesel production which is now gaining wide acceptance because of low CO_2 emission and other considerations. In Nigeria, *Luffa cylindrica* plant grows in the wild and on abandoned building structures and fenced walls in towns and villages (Ndukwe et al., 2001; Ajiwe et al., 2005). Consumption of fresh *L. cylindrica* fruit and use of fibre have been widely reported (Onelli et al., 2001; Italo and Alves, 2005). The seed which contains about 30 % oil content is often discarded as waste and literature is sparse on extraction and quality of its seed oil. Therefore, optimization of solvent extraction of oil from *Luffa cylindrica* using response surface methodology was the focus of this study.

Response surface methodology (RSM) is a collection of statistical and mathematical techniques useful for developing improving and optimising processes. The approach is useful for optimizing, designing, developing, formulating and improving processes where a response or responses are affected by several variables (Montgomery, 2005; Baş and Boyacı, 2007). The main disadvantages of this method are the interactive effects among the variables, which are not considered and there is a lack of explanation of the complete effect of the factors on the response. In addition, this method increases the number of experiments needed to conduct the research, which leads to increased cost and time (Baş and Boyacı, 2007; Bezerra et al., 2008). RSM is useful in three different techniques or methods (i) statistical experimental design, in particular two level factorial or fractional factorial design, (ii) regression modeling techniques, and (iii) optimization methods (Myers and Montgomery, 2002). The state-of-the-art applications of Response surface methodology (RSM) in the optimization of different food processes in the food industries include extraction, drying, blanching, enzymatic hydrolysis and clarification, production of microbial metabolites, and formulation. In biological and clinical sciences RSM is used in biometric systems, while in the social sciences and wastewater treatment, RSM is used in person-environment fit research, self-other agreement studies in leadership research and perceptual distance studies when comparing leader’s and their subordinates ratings of their work context and optimize the process of chemical coagulation based on a central composite rotatable design (CCRD) respectively (Edwards, 2002; Mosaddeghi et al., 2018).

2.0 Materials and Methods

2.1 Materials

The matured dry *Luffa cylindrica* seeds were harvested from bush and uncompleted buildings around Ilorin, Kwara State, Nigeria. The envelope brownish parts of the harvested seeds were peeled, and the seeds were shaken out into a bow bowl and winnowed to remove the matured seeds. The cleaned seeds were pre-cooked to soften the shell to ease decortication. The seeds were eventually sundried so as to remove the moisture absorbed during precooking and the shells were removed manually, dried and ground with the hammer mill to flour.

2.2 Experimental design

The experimental matrix was designed using Design Expert version 6.0.6, where a Response Surface Methodology (RSM) in a Central Composite Design (CCD) was employed to optimize the extraction of oil from loofah seeds. The experiment was designed on five levels, ($-\alpha$, -1, 0, +1 and $+\alpha$) three independent variables, (seed/solvent ratio, extraction temperature and extraction time) that generated 20 experimental runs for optimal oil yield. The summary of this is as shown in Table 1.

Table 1: Experimental design

Variable	Parameter	Unit	Code	Level				
				- α	-1	0	+1	+ α
A	Seed/solvent ratio	g/ml	X1	0.04	0.05	0.06	0.07	0.08
B	Extraction Temperature	°C	X2	55.00	60.00	65.00	70.00	75.00
C	Extraction Time	hr.	X3	4.00	5.00	6.00	7.00	8.00

2.2.1 Extraction of oil from loofah seed using solvent extraction

The extraction of oil was carried out at the Industrial Chemistry Laboratory, Kwara State University Malete, Kwara state, Nigeria, using the method described by Oniya et al. (2017). The extraction was done with a Soxhlet apparatus of 250 cm^3 capacity using n-hexane of analytical grade as the solvent. A prepared sample of 15 – 25 g loofah seed, extraction temperature ranges of 55 and 75°C, seed/solvent ratio range between 0.04 – 0.08 g/ml while the extraction time ranges between 4 – 8 hours as given in Table 1. The experiments were carried out in batches according to the experimental layout generated from Design Expert software. The solvent used was recovered at every batch through a distillation process, and the actual oil obtained was weighed. The experiment was repeated for other runs and the percentage oil yield was determined as the ratio of the weight of oil recovered to the weight of the loofah seed sample before extraction. Oil yield was mathematically expressed using the expression adopted by Adeeko and Ajibola (1989), Olaniyan and Oje (2011) and Oniya et al. (2017) and presented in equation (1) as:

$$O.Y = \frac{M_{OR}}{M_{SS}} \qquad (1)$$

where;

O. Y = Oil yield (%),

MOR = Mass of oil recovered (g),

MLE = mass of the loofah seeds extracted (g)

3.0 Results and Discussion

3.1 Result of oil yield from the extraction of oil from loofah seed

The result of the solvent extraction of oil from loofah seeds is presented in Table 2. The yield for each batch of the experiment was determined using Equation 1. The differences in oil yield values are indications that the independent variables had considerably affected the yield. The predicted oil yield from the Design Expert software compared well with the yield obtained. The optimum oil yield of 27.43% was obtained from the extraction at corresponding seed/solvent ratio of 0.05g/ml, extraction temperature of 74.05°C, and extraction time of 5.35 h.

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Table 2: Experimental design layout using Response Surface Methodology

Std.	Run	Factor 1 A: Seed/solvent ratio (g/ml)	Factor 2 B: Extraction Temperature (°C)	Factor 3 C: Extraction Time (hr.)	Response 1 Oil Yield (%)	Predicted Value (%)
7	1	0.04	75.0	8.0	27.30	27.28
3	2	0.04	75.0	4.0	26.90	26.81
5	3	0.04	55.0	8.0	24.60	24.70
13	4	0.06	65.0	5.0	24.90	25.43
2	5	0.08	55.0	4.0	23.50	23.51
11	6	0.06	60.0	6.0	25.50	25.30
16	7	0.06	65.0	6.0	26.00	25.93
9	8	0.05	65.0	6.0	25.90	26.10
18	9	0.06	65.0	6.0	26.00	25.93
8	10	0.08	75.0	8.0	27.40	27.46
4	11	0.08	75.0	4.0	26.80	26.70
17	12	0.06	65.0	6.0	26.00	25.93
1	13	0.04	55.0	4.0	24.00	23.93
14	14	0.06	65.0	7.0	26.20	25.81
6	15	0.08	55.0	8.0	24.50	24.58
20	16	0.06	65.0	6.0	26.00	25.93
10	17	0.07	65.0	6.0	26.10	26.04
15	18	0.06	65.0	6.0	26.00	25.93
12	19	0.06	70.0	6.0	26.40	26.74
19	20	0.06	65.0	6.0	26.00	25.93

3.2 Mathematical Model Equation for Oil Expression

The optimum conditions for oil expression were performed according to the central composite design experimental plan and the observed responses (oil yield) were presented in Table 2. The regression model equation developed for oil yield with single factors, quadratic factor, and interactive factors observed B, C, A2, B2, AB, and AC are directly proportional to the oil yield while A, C2, and BC with negatively coefficient indicates an indirect proportionality with the oil yield. The model obtained is given in Equation 2 as

$$Y = 25.93 - 0.059A + 1.44B + 0.38C + 0.56A^2 + 0.36B^2 - 1.24C^2 + 0.075AB + 0.075AC - 0.075BC \tag{2}$$

where,

Y = oil yield measured (%),

A = Seed solvent ratio (g/ml),

B = extraction temperature (°C), and

C = extraction time (hr.)

3.3 Statistical analysis of extracted loofah seed oil

The design of the experiment (DOE) and the responses (oil yield) of batch laboratory experiments were shows in Table 3. From the Table, the fitness of the model was expressed by the coefficient of determination (R^2) and adjusted coefficient of determination (Adj. R^2), which were obtained as 0.9656 and 0.9346, respectively; the values indicate that the regression model is acceptable. However, the determination coefficient (R^2 = 0.9656) indicates that the sample variation of 96.56% for oil expression is attributed to the independent variables and only 3.44% of the total variations are not explained by the model. The value of the adjusted coefficient of determination (Adj. R^2 = 0.9346) is also very high which advocate for a high significance of the model (Yuan et al., 2008). Akintunde et al. (2015) reported that the fitness of a model is determined by the R^2 and the value should not be less than 0.80. Also, it can be deduced from the F-value of 31.19 with a correspondingly low probability value of 0.0001, which is less than 0.05, that the model terms are significant. More so, the standard deviation of 0.27, mean of 25.80, C.V. of 1.03, and adequate precision of 21.051 were obtained for oil expression. In this case, the adequate precision value (P adeq. = 21.051) is greater than 4, which is desirable for the model (Bamgboye and Oniya, 2012; Oniya et al., 2017). Table 4 shows the assessment of experimental errors and the confidence interval (CI) of the experimental variables. The standard errors are analyzed based on the differences between the predicted oil yield and the experimental values.

3.4 Effect of Operating Parameters

The significance and adequacy of the model were tested using ANOVA. It was observed from the table that only two factors; extraction temperature and extraction time (B, C) are significant in the model, corresponding to seed/solvent ratio, which is non- significant factor. The quadratic effects of seed solvent/ratio (A2), quadratic effects of extraction temperature (B2) quadratic effects of extraction time (C2), seed/solvent ratio and extraction temperature (AB), seed/solvent ratio and extraction time (AC) and extraction temperature and extraction time (BC) are all non-significant being higher than 0.05, while the values less than 0.05 means significant. The interaction effects of seed/solvent ratio, temperature, and time on the yield were studied using the interaction plot and 3D surface plot of surface response methodology.

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Table 3: Analysis of variance for oil extraction from loofah seeds (partial sum of squares)

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	19.74	9	2.19	31.19	< 0.0001	Significant
A	0.029	1	0.029	0.42	0.5324	
B	17.65	1	17.65	251.08	< 0.0001	
C	1.24	1	1.24	17.67	0.0018	
A2	0.059	1	0.059	0.84	0.3810	
B2	0.025	1	0.025	0.35	0.5679	
C2	0.29	1	0.29	4.06	0.0715	
AB	0.045	1	0.045	0.64	0.4423	
AC	0.045	1	0.045	0.64	0.4423	
BC	0.045	1	0.045	0.64	0.4423	
Residual	0.70	10	0.070			
Lack of Fit	0.70	5	0.14			
Pure Error	0	5	0			
Cor. Total	20.44	19				
Std. Dev.	0.27					
Mean	25.80					
C.V.	1.03					
PRESS	4.01					
Adeq. Precision	21.051					

Table 4: ANOVA analysis of experimental errors and confidence interval

Factor	Coefficient Estimate	DF	Standard Error	95% CI Low	95% CI High	VIF
Intercept	25.93	1	0.08	25.75	26.11	
A-Seed/solvent ratio	-0.059	1	0.091	-0.26	0.14	1.00
B-Extraction Temperature	1.44	1	0.09	1.24	1.64	1.00
C-Extraction Time	0.38	1	0.09	0.18	0.59	1.00
A2	0.563	1	0.614	-0.81	1.93	24.18
B2	0.363	1	0.614	-1.01	1.73	24.18
C2	-1.24	1	0.61	-2.61	0.13	24.18
AB	0.075	1	0.094	-0.13	0.28	1.00
AC	0.075	1	0.094	-0.13	0.28	1.00
BC	-0.075	1	0.094	-0.28	0.13	1.00

3.5 Effect of seed/solvent ratio and extraction temperature on the oil yield from loofah seed

The effect of seed/solvent ratio and extraction temperature was studied using the 3D surface plot (Figure 1), which depicts the interaction between seed/solvent ratio and extraction temperature. It was observed that as temperature increases and a slight increase in seed/solvent ratio improved the oil yield. This implied that increase in seed/solvent ratio and extraction temperature favours the extraction rate as much solvent is needed to ensure that vapour rose through the vertical monitor tube into the condenser at the top just as the solvent boiled (Oniya et al., 2017).

3.6 Effect of the seed/solvent ratio and extraction time on the oil yield from loofah seed

The 3D surface plot interaction from Design Expert presented in Figure 2 represents the effects of seed/solvent ratio and extraction time. Their interactions on the oil yield for extraction were studied. It was observed that increase in seed/solvent ratio favors the extraction rate as much solvent is needed for extraction, while the oil yield increases gradually as extraction time increases until the extraction time was above 7hr. when the increased in extraction time does not have any significant effect on the oil yield. The curvature nature of Figure 2 depicts a mutual interaction between extraction time and seed/solvent ratio.

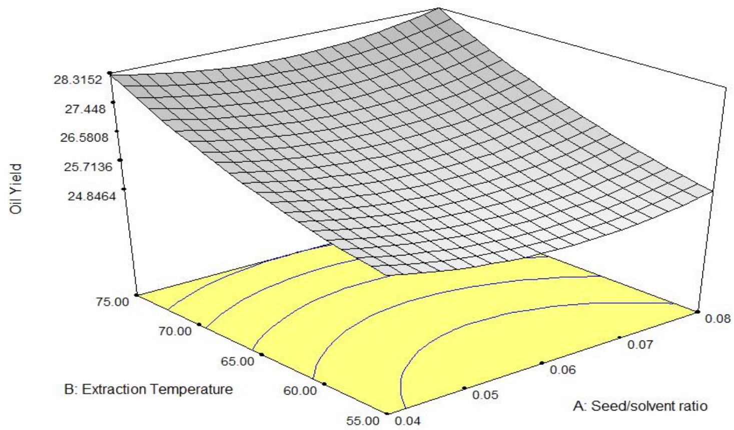

Figure 1: 3D Response surface plot showing the effect of extraction temperature and seed/solvent ratio on the oil yield.

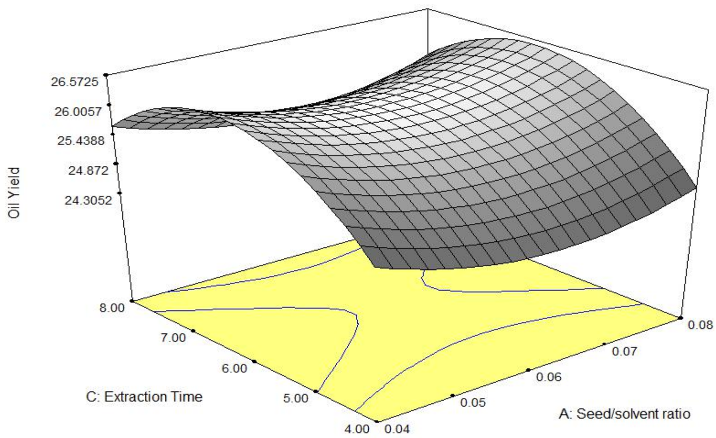

Figure 2: 3D Response surface plot showing the effect of extraction time and seed/solvent ratio on the oil yield.

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3.7 Effect of extraction time and temperature on the oil yield from loofah seed

From Figure 3, the elliptical nature of the contour plots indicated that interaction between the extraction temperature and time had no significant effect on the yield of oil. But the two factors individually had (extraction temperature and extraction time) a significant effect in the model. It was noticed that increase in time favoured the extraction of oil until it the reaction time was above 7 hrs. Increased reaction time above this level tends to have no significant effect on the oil yield. The oil yield was observed to increase with higher temperature even at the temperature higher than the boiling point of the solvent. This is because at this temperature higher than the boiling temperature of the solvent, evaporation of the solvent will take place as a result of the increased temperature leaving the extracted oil. The combined effect of these two independent variables at high-level experimental process will obviously result in a decrease in oil yield. However, Afolabi et al. (2015) and Yuan et al. (2008) who worked on optimization of solvent extraction of oil from Parinari polyandra and reported that the oil yield was observed to decrease with higher temperature, especially at the temperature higher than the boiling point of the used solvent. The effect of the decrease oil yield at a higher temperature is more prominent in hexane than when another solvent is utilized. This may be due to its low boiling point of 67ºC when compared with other solvents (Afolabi et al., 2015).

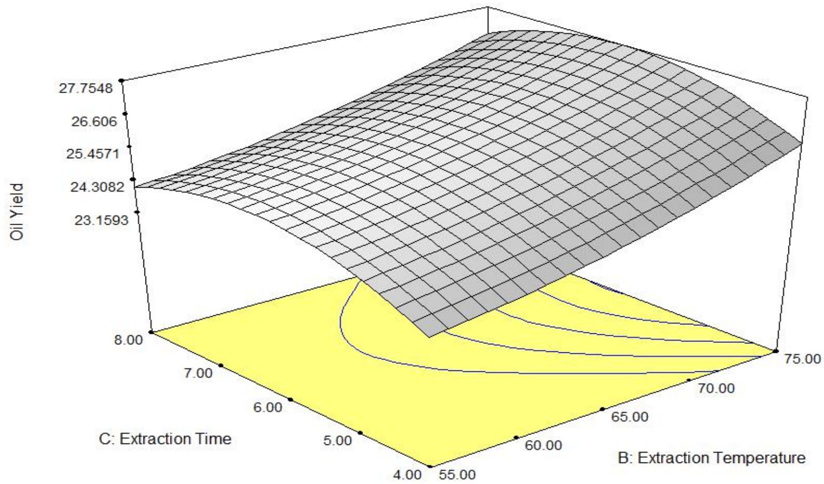

Figure 3: 3D Response surface plot showing the effect of extraction time and extraction temperature on the oil yield.

3.8 Validation of the model

The relationship between predicted and experimental values of oil yields is shown in Figure 4. It can be seen that there is a high correlation (R^2 = 0.9656) between the predicted and experimental oil yields. It means that the data fit well with the model and give a convincingly good estimate of response for the system in the range studied. The percentage error was calculated to be 0.88 which confirmed the validity of the model equation.

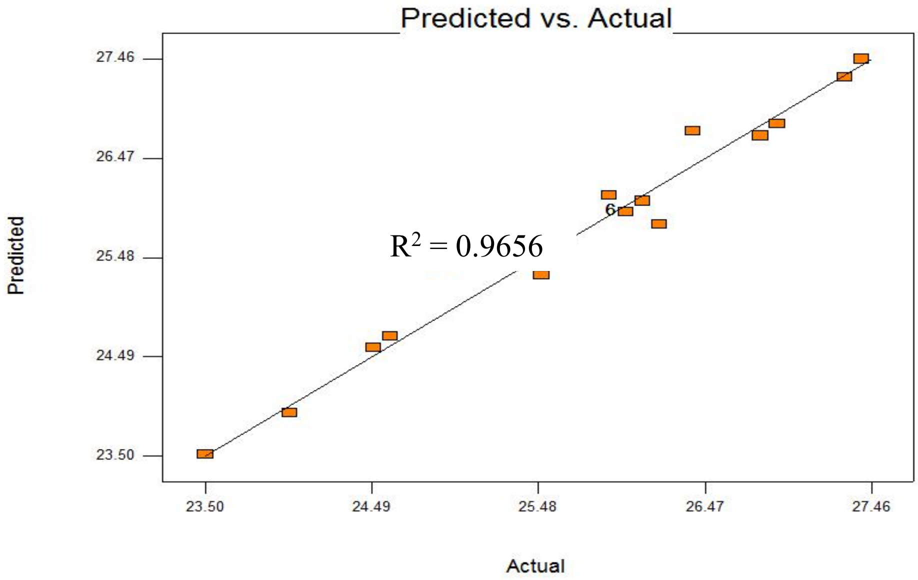

Figure 4: Predicted oil yield versus experimental oil yield.

4.0 Conclusion

The study revealed that the extraction temperature has the highest influence in the regression model with highest F-value followed by extraction time and solvent/seed ratio. This implies that extraction temperature plays a significant role in solvent oil expression from Luffa cylindrical seeds. Based on the findings of this research work, the optimal conditions for oil extraction from Luffa cylindrica were determined as a seed/ solvent ratio of 0.05 g/ml, extraction temperature of 74.05ºC, and extraction time of 5.35 hr. at corresponding oil yield of 27.43%

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