



D-Optimal Optimization of Desert Date Oil Extraction

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Date Submitted: 10/02/2022

Date Accepted: 11/03/2022

Date Published: 18/03/2022

Abstract: This work was carried out to investigate the optimum conditions of temperature, time, solvent to biomass ratio and particles size on n-hexane extraction efficiency measured in terms of amount of oil extracted in g. D-Optimal Design of Response Surface Methodology was used to obtain 28 experimental runs, according to which oil was extracted from ground desert date seed kernel with the aid of Soxhlet apparatus. The particle size was used as a categorical factor at two different levels (0.6 mm and 1 mm) and other factors were numeric in nature. The temperature, time and solvent to biomass ratio were in the range of 50-70 °C, 1-4 h and 10 - 20 mL/g respectively. After the runs, obtained experimental data were fitted to cubic model and statistical significance of the model was evaluated using analysis of variance (ANOVA). The results obtained showed that the reduced cubic equation obtained was significant statistically with p-value of 0.001, which was found to be less than 5% at 95% confidence level. Also, based on the same criterion, the significant terms in the model were discovered to include time, solvent to biomass ratio and particle size. It was, however, noticed that the effect of temperature was insignificant on the model. The optimum operating conditions found was 60 °C, 4 h, 15 (150 mL/10 g) and 0.6 mm. The maximum amount of oil extracted experimentally at these conditions was 6.08 g, which compared well with the predicted value of 6.117 g.

Keywords: Categorical factor, desert date oil yield, cubic model, numeric factor, Response Surface Methodology (RSM).

1. INTRODUCTION

Desert date (*Balanite aegyptiaca*) trees are commonly found in Borno, Yobe States [1] and northwest region of Nigeria [2]. Locally, various parts of this plant are used for different purposes ranging from consumption especially the fruit and oil, and use as medicine. In addition to saturated fatty acid, the seed oil of *Balanite aegyptiaca* contains saponins, sapogenins and diosgenins which are used as raw materials for industrial production of contraceptive pills and sexual hormones [1,3]. The seed kernel of desert date fruit is known for its oil among the locals, most of whom use it for cooking and treating fungal related skin infections. Oil yield as high as 50% has been extracted from the seed kernel [4]. In another source, the seed kernel has been reported to contain 40-87% of edible oil [5]. Although the oil is consumed among the locals [5], it is said to contain toxic anti-nutritional compounds like oxalate, tannin, saponin and phytic acid [6-9]. This means that long time consumption of the oil can have health implication. This confirms the reason why the oil has been described to be a good raw material for various industrial applications. Researches have shown that desert date oil could be used for production of cosmetic products such as liquid soap, shampoo and body lotion [10]. The oil can also be used as a biodiesel feedstock [2,11-12]. Its use for soap formulation has been associated to its abundance and presence of saponin [10]. Thus, any study on the extraction of this golden oil is really of good importance as cosmetic products and energy are necessities.

Seed oils are usually obtained using wet (hot water or steam extraction), mechanical and solvent methods. Also, techniques such as microwave-assisted, ultrasonic-assisted and supercritical fluid extractions are used for seed oil extraction. N-hexane extraction has been widely used [13] due to high yield of extraction, easy recoverability, non-polarity, high selectivity to other solvents, low latent heat of vapourisation, excellent oil solubility, and narrow range of boiling point (63 – 69 °C) [14-16]. Several factors, such as solvent to biomass ratio, agitation speed, temperature of extraction, extraction duration, biomass particle size and solvent type influence the oil yield of solvent extraction process [17-19]. Since oil extraction process is a multi-factor one, it is necessary to obtain the optimum parameters required for its operation.

Classical optimization involves varying one factor at a time while keeping other factors constant. This approach hardly gives true optimum of the process under study as it normally ignores interaction occurring among the factors. Also, conducting experiments in this manner takes more time and can lead to higher cost of the project due to large number of experiments involved. Response surface methodology (RSM), on the other hand, gives a mathematical model that describes the relationship between the experimental factors and a response; and it reveals the true optimum factors with minimum number of experiments compared to classical optimization method. This approach has been recently used to statistically optimize the oil extraction process. Many researchers study the optimization of oil extraction process using design such as Box-Behnken design [17, 20], central composite design (CCD) [19, 21-23] and full factorial design [18].

This present study has been carried out to find the optimum value of temperature, time, solvent to biomass ratio and particle size that would give the maximum yield of desert date oil using D-optimal design of response surface methodology. The particle size was taken as categorical factor while others were numeric in nature. The objective function that of the optimisation problem was taken to be the maximisation of the yield of oil extracted using Soxhlet apparatus.

2. MATERIALS AND METHODS

Some desert date fruits were purchased from a Sunday market in Damaturu, Yobe State. Since the seeds of the fruits were only needed for the study, the obtained fruits were soaked in water for at least 12 hours to obtain the seeds. Thereafter, desert date seeds were sundried and their coats (shells) were separated using hammer. The obtained kernels were ground using blender and sieved into two different particle sizes (≤ 0.6 and ≤ 1 mm).

2.1 Design of Experiment and Optimization

A total of twenty-eight experiments were generated according to D-optimal design of response surface methodology with the aid of Design Expert 7.0. The design matrix consisted of three (3) numeric factors namely, temperature ($^{\circ}\text{C}$), time (h), and solvent to biomass ratio (mL/g) and particle size as the categorical factor at two different levels while the amount of oil extracted in g was the response. The ranges of the factors used were as given in Table 1.

Table 1. Range of the factors

Factor	Symbol	Minimum	Maximum
Temperature ($^{\circ}\text{C}$)	A	50	70
Time (h)	B	1	4
Solvent to biomass ratio (mL/g)	C	10	20
Particle size (mm)	D	0.6	1

Soxhlet extraction experiments were carried out according to the condition specified in a design matrix. For each experimental run, 10 g of the ground desert date kernel of the specified particle size was wrapped in a preweighed filter paper and placed in the extractor chamber of the Soxhlet apparatus. The extraction chamber coupled with a reflux condenser circulating chilled water to prevent escape of solvent was attached to a 250 ml round-bottom containing a specific amount of normal hexane (in mL) as indicated in solvent to biomass ratio, and the entire system was placed on a digital intelligent heating mantle (ZNCL-TS500ML). After the extraction, the resulting mixture of n-hexane and oil was distilled to recover the solvent and oil separately. The weight of extracted desert date oil for each run was measured and recorded. The oil yield was calculated as the weight ratio of extracted oil and the ground desert date kernel.

In order to model the extraction process for optimization purpose, the obtained experimental results were entered into the design matrix earlier created using Design Expert 7.0 and the data containing the operating conditions and the respective response values were fitted to a cubic model, which was later modified to improve its performance. The significance of the model was evaluated using result of analysis of variance. After the statistical importance of the model had been ascertained, it was optimized numerically using the same software.

3. RESULTS AND DISCUSSION

The D-Optimal design matrix comprising the different operating conditions used and the corresponding values of the response is given in Table 2. As it can be seen from the table, combinatory variation of the factors based on D-optimal method led to attainment of different amounts of desert date oil, which was an indication that n-hexane extraction of desert date oil was also affected by various operating conditions considered. The maximum weight (6.52 g) of extracted oil that was equivalent to 65.2% yield was achieved at 70 $^{\circ}\text{C}$, 2.58 h, 15 (150 mL/10 g) and 0.6 mm of temperature, time, solvent to biomass ratio and particle size respectively. In a similar manner, the minimum yield of 18.1% was obtained at 50 $^{\circ}\text{C}$, 1 h, 10 (100 mL/10 g) and 0.6 mm. The results demonstrated the effects of variation of time, temperature and amount of n-hexane on the efficiency of the extraction process in terms of yield despite the fact that fine particles usually give high yield. The increase in temperature, time and volume of the solvent must have been responsible for this high yield obtained. The contribution of each of these factors can be explained based on the fact that the solubility of the solute (oil in the biomass) is increased as a result of higher kinetic energy attained by the solvent molecule at high temperature, which is also responsible for breaking of bond holding the solute together in biomass. Also, increasing the amount of solvent can make more solvent available for enhanced mass transfer [28]. Moreover, increasing time of extraction gives room for more mass transfer to take

place until the extraction was complete. Looking at the table again, it can be noticed that the maximum amount of oil extracted with particle size of 1 mm was 6.46 g at 58 °C, 4 h, 20 (200 mL/10 g) of temperature, time and solvent to biomass ratio respectively. This is fairly close to the overall maximum oil yield of 65.2%. This comparison is suggesting that the statistical optimization might give the maximum yield around this value.

Consequently, the extraction data in Table 2 were fitted to a cubic model. The reason behind choosing this polynomial model was because the trial analyses done with linear and quadratic models gave no good result. Thereafter, the model was modified to remove the aliased and some insignificant non-hierarchical terms so as to improve its statistical significance. Given in Equation (1) is the resulting empirical model obtained for amount (in g) of oil in actual terms. In the equation the constant K is 375.66143 and 380.99724 respectively for 0.6 mm and 1 mm.

$$Y = K - 13.49826A - 35.34595B - 15.77265C + 1.20859AB - 0.63768AC + 0.11286A^2 + \dots \quad (1)$$

$$\dots + -0.086293C^2 - 0.010038A^2B - 0.00533293A^2C$$

Table 2. The D-optimal Design Matrix

Run No	A, °C	B, h	C, mL/g	D, mm	Y _{exp} , g	Y _{exp} , %
1	57.35	3.33	13.75	0.6	4.510	45.10
2	60.00	2.50	15.00	1.0	4.500	45.00
3	68.47	2.88	19.64	1.0	3.940	39.40
4	70.00	1.00	20.00	1.0	3.540	35.40
5	50.00	1.00	20.00	0.6	3.360	33.60
6	57.50	1.05	19.69	1.0	4.380	43.80
7	50.00	4.00	20.00	0.6	4.600	46.00
8	70.00	1.00	10.00	0.6	2.570	25.70
9	50.00	4.00	10.00	1.0	2.965	29.65
10	70.00	4.00	20.00	0.6	4.070	40.70
11	60.00	2.50	15.00	0.6	3.330	33.30
12	60.90	1.00	15.47	0.6	2.720	27.20
13	70.00	1.00	10.00	0.6	2.065	20.65
14	70.00	4.00	10.00	0.6	2.868	28.68
15	70.00	2.58	15.00	0.6	6.520	65.20
16	64.25	2.55	10.00	1.0	2.295	22.95
17	50.00	4.00	20.00	0.6	3.590	35.90
18	60.00	2.50	15.00	0.6	5.230	52.30
19	50.00	2.44	16.97	1.0	5.650	56.50
20	70.00	4.00	10.00	0.6	2.745	27.45
21	58.36	4.00	20.00	1.0	6.460	64.60
22	60.00	2.50	15.00	1.0	6.420	64.20
23	50.00	1.00	10.00	0.6	1.810	18.10
24	50.00	4.00	10.00	1.0	5.065	50.65
25	58.96	2.50	19.85	0.6	6.420	64.20
26	51.43	1.00	10.00	1.0	2.920	29.20
27	50.00	1.00	10.00	0.6	2.738	27.38
28	70.00	4.00	14.17	1.0	6.140	61.40

The results of analysis of variance obtained for the model at the two levels of biomass type are given in Table 3. As it can be seen from the table, the empirical cubic model was obtained to be significant with probability of error value (p-value) of 0.001 that is less than maximum permissible probability of error value of 0.05 since the analysis was done at 95% confidence level. Based on the same criterion, the significant model terms are B, C, D, CD, C², A²B, A²C. This implies that variation of time, solvent to biomass ratio, particle size singly affected the extraction system. Also, interaction effect of solvent to biomass ratio and particle size as well as square of temperature and time, and square of temperature and solvent

to biomass ratio had significant influence on the model. It can be observed from Table 3 that the linear effect of temperature was statistically insignificant and so also did the effect of square of temperature. On contrary, Yahaya *et al.* [20] reported that the cubic model developed for n-hexane extraction of jatropha was singly and interactively (with other considered factors) affected by temperature. This reported trend corroborated the results of Yusuff [26]. Also noticeable from Table 3 is the insignificant Lack of Fit obtained for the model, which implied that the chosen model adequately represented the experimental data. Also, the coefficient of variation (CV), which is the ratio of standard deviation to the mean, obtained for the model was 0.2251 (20.51%). This is an acceptable CV value as explained by Alam [24], suggesting the precision of the model. The coefficient of determination obtained for the model was 0.8218, and this was found to be close to 1. But the predicted R² was observed not to be close to the adjusted R² as normally expected. This can be attributed to problem with the experimental data [25]. However, the R-squared value of 0.8218 was found to be close to its adjusted value by 0.142, which was observed to be approximately equal to the difference of 0.0881 between the R-squared and adjusted R-squared reported by Yusuff [26]. This implied that the developed model fitted the experimental data well.

Table 3: Analysis of variance for the oil weight model

Source	Sum of Squares	Df	Mean Square	F Value	p-value (Prob > F)	
Model	47.72	12	3.98	5.76	0.001	significant
A-Temperature	0.33	1	0.33	0.47	0.5022	
B-Time	7.02	1	7.02	10.17	0.0061	
C-solvent:bio	16.21	1	16.21	23.49	0.0002	
D-D	3.25	1	3.25	4.71	0.0465	
AB	0.048	1	0.048	0.07	0.7949	
AC	0.15	1	0.15	0.21	0.6501	
AD	0.015	1	0.015	0.021	0.8861	
CD	3.8	1	3.8	5.5	0.0332	
A ²	1.94	1	1.94	2.8	0.1147	
C ²	15.63	1	15.63	22.65	0.0003	
A ² B	4.61	1	4.61	6.68	0.0207	
A ² C	8.66	1	8.66	12.55	0.003	
Residual	10.35	15	0.69			
Lack of Fit	3.42	8	0.43	0.43	0.8686	not significant
Pure Error	6.93	7	0.99			
Cor Total	58.07	27				
Std. Dev.	0.83		R-Squared	0.8218		
Mean	4.05		Adj R-Squared	0.6792		
C.V. %	20.51		Pred R-Squared	0.4333		
PRESS	32.91		Adeq Precision	8.732		

Given in Figures 1 and 2 are the contour plots illustrating the interactive effects of the two numeric factors. These are followed by interactions plots of numeric factor with categorical factor. The iso-response values obtained at various values of temperature and time range between 3.5 g and 5.6 g. The trend of the isoline shows that the response values increased with increase in time while temperature was just around the center point (60 °C). Yahaya *et al.* [20], Yusuff [25] and Ntalikwa [16] also found that the yield of jatropha oil increased with increase in time of extraction. The low range of the isoline might be the reason for the insignificance of the interaction term on the response as shown in the ANOVA results.

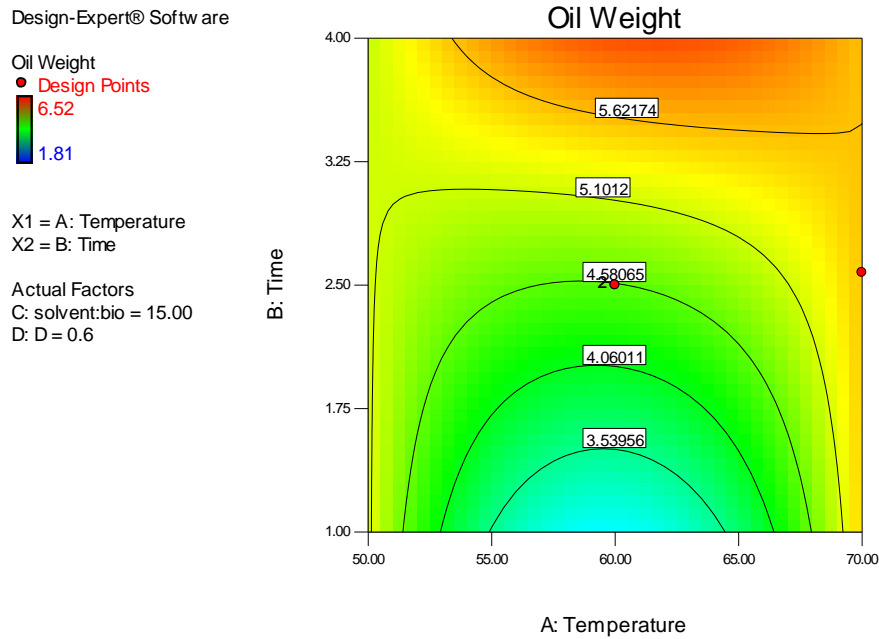


Figure 1: The effect of temperature and time on amount of oil extracted

The effect of variation of temperature and solvent to biomass ratio are illustrated in Figure 2. The contours within the region of (50, 10) and (70, 17.5) show significant variation in the values of the response (0.082 and 4.81). Beyond this region, the contours are not very close. Nonetheless, the obtainable maximum yield of 4.81 can be noticed from the figure at high solvent to biomass at both low (close to 50 °C) and high (close to 70 °C) temperatures. This may be the reason for insignificant nature of this term as shown in Table 2. The statistical insignificance of AB and AC can be understood from the small values obtained for the coefficient in the model [27].

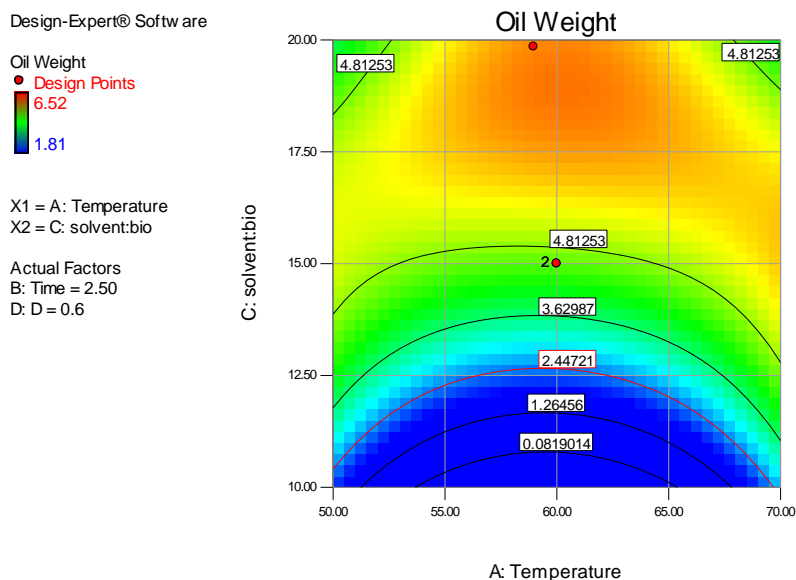


Figure 2. The effect of temperature and time on amount of oil extracted

The interaction graph for the temperature and particle size is given in Figure 3. The effect was evaluated at 3.19 h as time, and solvent to biomass ratio of 15 (150 mL/10 g). As it can be seen from the figure, the oil weight achieved when the particle size was 1 mm was higher than that obtained at 0.6 mm, which was contrary to the usual inverse relationship between particle size and yield of extracted oil. However, the variation of temperature for both particle sizes was insignificant as almost the same value of response was obtained at both 50 °C and 70 °C. This slight increase in amount with respect to particle size

might be attributed to slight irregularity in the temperature. Based on the ANOVA results, this term too was found to be of less or no statistical importance for this model.

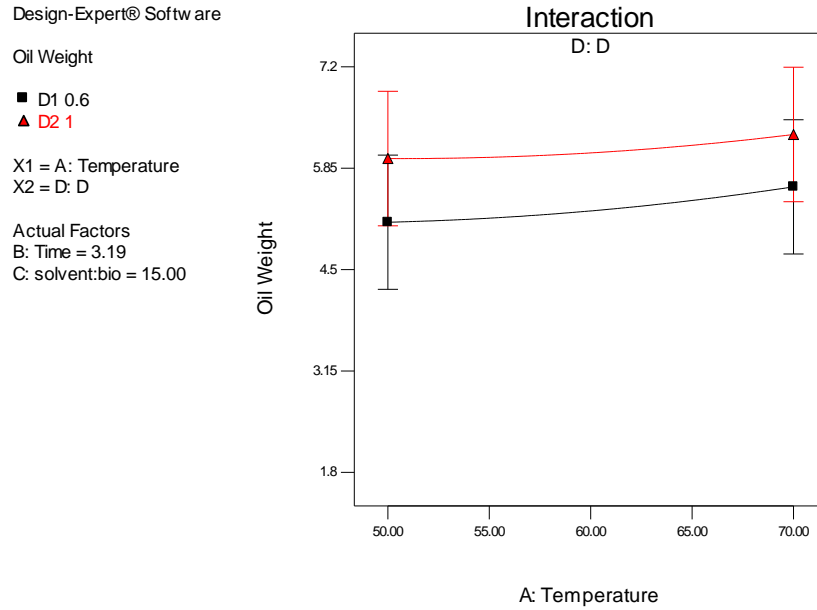


Figure 3. The effect of temperature and solvent to biomass ratio on amount of oil extracted

Also, in order to validate the model, the actual (experimental) values of the response (oil weight) were compared with the simulated values graphically as given in Figure 4. It is obvious from the plot that the model accurately represented the system under consideration as the predicted values are reasonably close to the empirical ones with the residual in the range of -1.26 and 1.08 g (Figure 5).

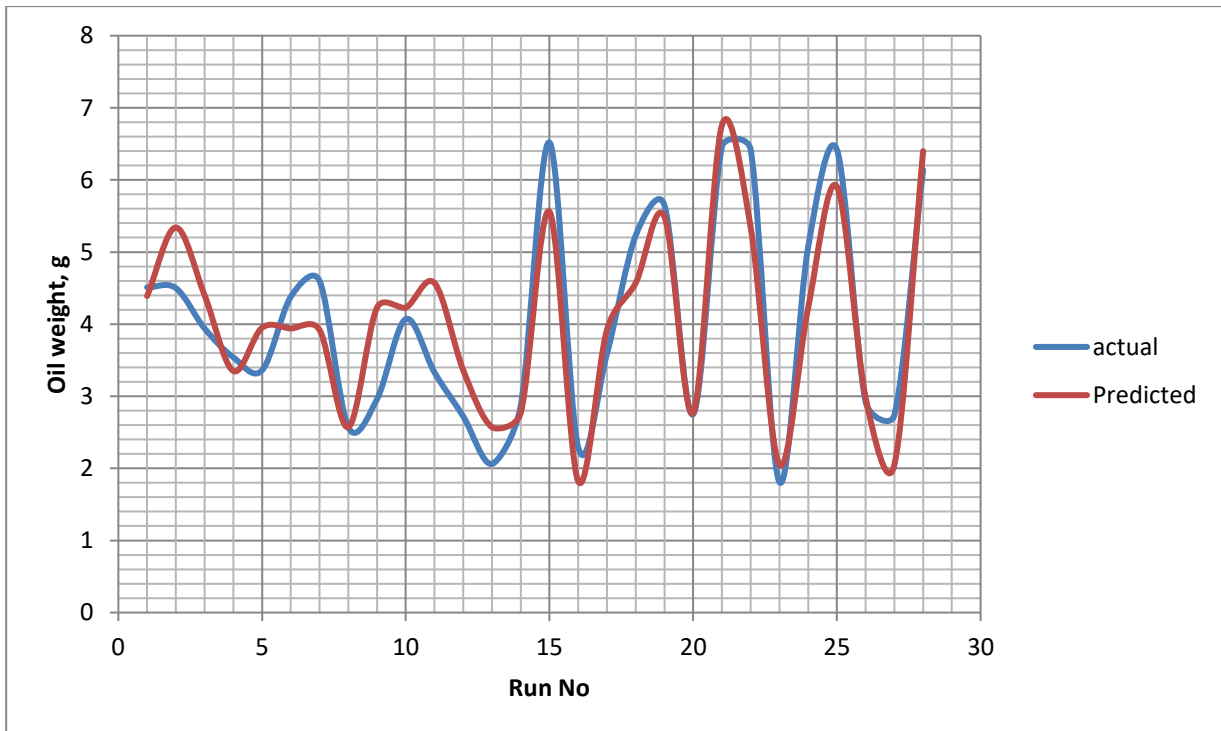


Figure 4: Actual and predicted oil weight at various run

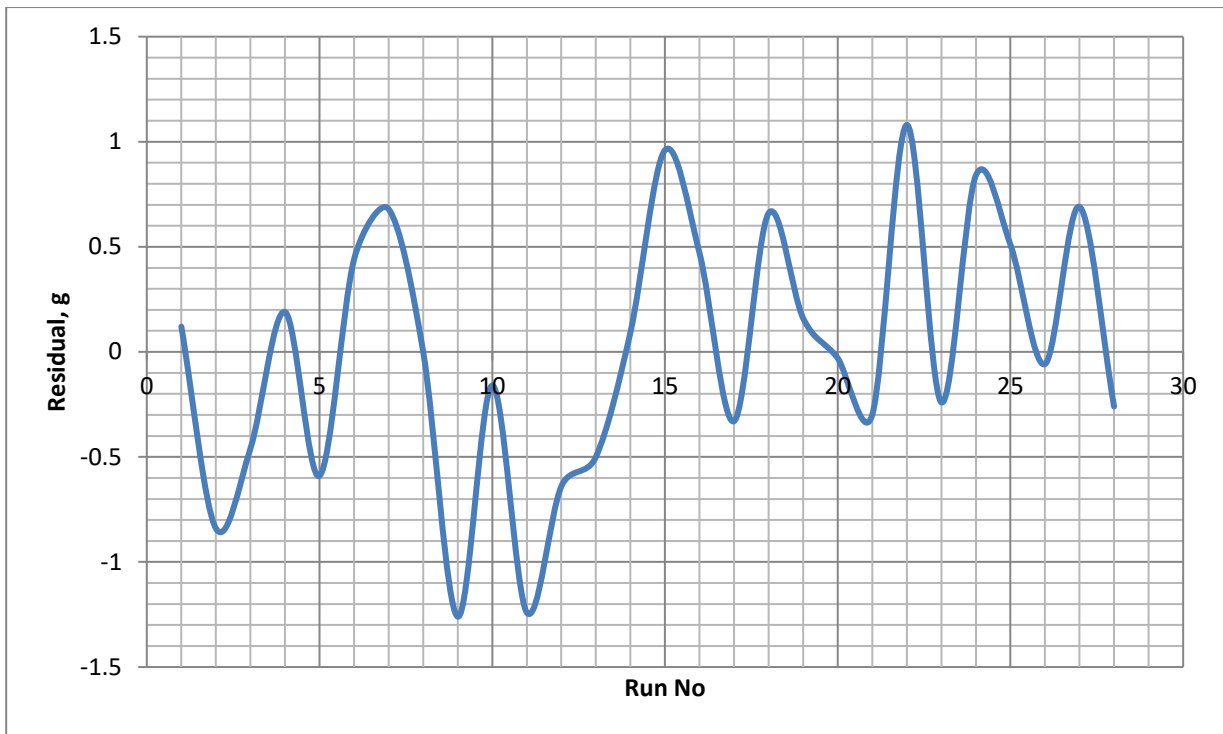


Figure 5. Response error at various runs

Sequel to ascertainment of statistical significance of the developed model, a numerical optimization was carried out by setting the optimization criteria based on the experimental results given in Table 2. It can be noticed that 60 °C, 2.5 h and 15 (150 mL/10 g) gave yield of 5.23 and 6.42 g of oil respectively for 0.6 mm and 1 mm. Using these operating conditions as the targets for independent variables yielded predicted maximum of 5.34 g and 4.8 g with 1 mm and 0.6 mm, and desirability of 0.93 and 0.87 respectively. Maintaining the temperature (60 °C) and solvent to biomass (15 mL/g) and setting the time criterion to be in range yielded optimum conditions of 60 °C, 3.65 h, 15 and 1 mm with maximum yield of 65.2%. Also, 60 °C, 4 h, 15 (150 mL/10 g) and 0.6 mm (with maximum yield of 61.18%) were also among optimization solution obtained. validating the latter conditions resulted to 60.8%. These obtained optimum operating conditions were found to be close to the ones (54.19 °C, 5.24 h and 224.23 mL of n-hexane) reported by Mas’ud *et al.* [27] when mango seed oil extraction was optimized using response surface methodology. Also, the yield obtained in this study was higher than 36.5% (at extraction conditions of 150 mL of n-hexane/100 g of desert date kernel powder, 70 °C and 4 h) reported by Ogala *et al.* [5] and 45.32% obtained by Zang *et al.* [10] at 250 mL petroleum ether/200 g of desert date kernel powder, 80 °C and 8 h. The improved yield of desert date oil observed here should be due to parametric optimization of the extraction carried out.

Table 4: Summary of desert date kernel oil extraction results reported in previous and present study

Optimum /selected operating conditions	Oil yield (%)	Reference
150 mL of n-hexane/100 g, 70 °C, 4 h	36.50	[5]
Not stated	40.32	[9]
250 mL of petroleum ether/200 g, 80 °C, 8 h	45.32	[10]
250 mL of n-hexane/10 g, 55 °C, 4 h, 0.6 mm	45.20	[11]
150 mL of n-hexane/10 g, 60 °C, 4 h, 0.6 mm	60.80	Present study

Moreover, the achieved maximum yield obtained in this work was found to be within the desert date seed kernel oil range of 40-87% reported by Ogala *et al.* [5].

4. CONCLUSION

In this study, optimization of desert date oil extraction has been carried out using response surface methodology. The Results of Analysis of variance performed on the developed reduced cubic model showed that the model adequately represented the n-hexane extraction system with p-value of 0.001. ANOVA results also indicated that time, solvent to biomass ratio and particle size significantly affected the process. The optimum conditions of extraction found were 60 °C, 4 h, 150 mL/10 g and 0.6 mm. The maximum yield of the oil obtained experimentally under these conditions were 60.8 % which compared well with the predicted value of 61.18%. More work needs to be done on this system so that effect of important factor such as temperature can be adequately represented by a model. Nonetheless, the model is useful in predicting the seed oil yield when the effect of temperature is insignificant or the constant temperature cannot be ascertained.

ACKNOWLEDGMENT

Saidat Olanipekun GIWA and Kabir Abogunde ABDULYEKEEN appreciate the financial support received from TETFund under the Institution Based Research (IBR) with Reference number ATBU/DVC/Acad/076 for the study.

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