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FINDING THE OPTIMUM PARAMETERS FOR OIL EXTRACTION FROM SESAME SEED USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT

Optimization of sesame oil extraction using response surface methodology (RSM) was carried out in this study. The effects of three factors (particle size, extraction temperature and extraction time) on the oil yield obtained from the seed were considered. With the aid of Design Expert and using the central composite design of the response surface methodology, twenty (20) experiments were generated and run. Using n-hexane as the solvent, each experimental run was carried out in 250 ml Soxhlet extraction apparatus. The results obtained from the experiments together with the factors considered during the experiments were modelled and analysed using a quadratic and a cubic model that were modified based on observations made during the model analysis. The results obtained thereafter revealed that the performance of the cubic model developed was better than that of the quadratic model for this extraction because the R-squared, the adjusted R-squared and the predicted R-squared values of the developed and modified quadratic model were 0.8907, 0.8402 and 0.5481 while those of the cubic model developed and modified were 0.9979, 0.9950 and 0.9089, respectively. Also, with the aid of the Design Expert, the optimum parameters of the process were estimated to be particle size of 0.91 mm, extraction temperature of 88.53°C and extraction time of 6.58 hr. It was finally concluded that the optimum values of the parameters given by the software were genuine ones because the validation of the optimum values obtained experimentally showed a very good conformity between the predicted optimum oil yield and the experimental one. The modified quadratic and cubic models developed predicted 55.8% and 51.15% respectively as the maximum oil yield while the one obtained from the experiment was found to be 55.5%. Therefore, the application of response surface methodology to optimize oil extraction from sesame seed has been successfully demonstrated.

KEYWORDS: *Sesame seed, extraction, response surface methodology, oil yield, Design Expert.*

I. INTRODUCTION

Sesame (*Sesamum indicum*) from Pedaliaceae, is an important oil seed crop being cultivated in the tropics and the temperate zone of the world (Biabani and Pakniyat, 2008). It is one of the oldest oil crops and is widely cultivated in Asia and Africa (Ali *et al.*, 2007). It was a highly prized oil crop of Babylon and Assyria at least 4000 years ago (Ross, 2005). This plant is called "sesame" internationally, while it is called "benniseed" in West Africa, "simsim" in East Africa and "Till" in India. Within Nigeria, it is called different names in different localities. It is generally called "ridi" in the Northern States. The Igalas, Idomas and Tivs of Benue State call it "igogo", "ocha" and "ishwa" respectively. The Ibos call it "isasa" and Yorubas call it "ekuku" or "eeku" in parts of Ogun, Ondo and Oyo States and Ilorin in Kwara State (Aboje, 2011; Warra, 2011). This crop is grown mostly for the oil extracted from its seed which is edible and use for industrial and pharmaceutical purposes (Ogbonna and Ukaan, 2013). It ranks ninth among the top thirteen oilseed crops which make up 90% of the world production of edible oil (Adeola *et al.*, 2010). Sesame seed has higher oil content (around 50%) than most of the known oil seeds (Hwang, 2005). The seed has 40-60 per cent of oil with almost equal levels of oleic (range of 33-50%, typically 41%) and linoleic acids (range of 33-50%, typically 43%) and some palmitic acid (range of 7-12%, typically 9%) and stearic acid (range of 3-6%, typically 6%) (Gunstone, 2004).

Natural sesame oil derived from good quality seed has a very pleasant flavour and can be consumed without further purification. The natural sesame oil has excellent stability due to the presence of high level of natural antioxidants (Lyon, 1972).The oil is also useful in the industrial preparation of perfumery, cosmetics (skin conditioning agents and moisturizers, hair preparations, bath oils, hand products and make-up), pharmaceuticals, insecticides and paints and varnishes (Warra, 2011).

Generally, oil is extracted from sesame seed using methods like Soxhlet extraction, supercritical extraction, etc. Soxhlet method is one of the most popular methods used for lipid extraction from natural products and foodstuffs. According to this method, lipids are extracted from solids by multiple rinsing out with an organic solvent like hexane or any low-boiling petroleum ether (Carvalho *et al.*, 2012). However, the efficiency of Soxhlet extraction, usually evaluated using oil yield has been discovered to be affected by operating conditions such as temperature of extraction, seed size and extraction time. So, in order to obtain good yield of oil from this seed, there is the need to obtain the optimum values of these parameters affecting the extraction of oil from the seed.

Conventionally, the optimization of a process being affected by more than one factors is carried out by varying one factor at a time while others are kept constant. This method normally ignores the interactions occurring among the factors. Thus, it may not actually give the best conditions that will give the optimum efficiency of the process. Consequently, response surface methodology (RSM) has been discovered to be an effective statistical method of optimizing a process using designs such as central composite design (CCD), Box-Behnken design and D-optimal design. Apart from giving the optimum conditions with minimal number of experiments compared to the conventional method, this method (response surface methodology) gives the mathematical expression(s) showing the relationship(s) between the response(s) and the factors (Giwa *et al.*, 2013).

Actually, it has been discovered that some researchers have worked on oil extraction from seeds as well applied response surface methodology. One of them was Lawson et al. (2010) who used Soxhlet extractor to obtain oil from soybean seed and discovered that temperature and flake thickness had great effects on oil yield. Also, Date *et al.* (2010) applied supercritical method to extract oil from sesame seed and discovered that composition of lignan obtained was dependent on the conditions for the extraction. Moreover, Bimakr *et al.* (2013) used response surface methodology to carry out supercritical extraction of oil from winter melon seed and found out that the method was effective and promising for obtaining high oil yield. Finally, Awolu *et al.* (2013) applied response surface methodology to achieve the extraction of oil from neem seed successfully.

Therefore, in this work, with the aid of Design Expert, response surface methodology has been applied to obtain the optimum values of the parameters required for the extraction of oil from sesame seed using n-hexane as the solvent. In carrying out the optimization, the response variable was chosen to be the yield of the oil obtained from the seed while the factors affecting the oil yield were selected to be the particle size of the seed, the extraction temperature and the extraction time.

After this introductory part of the work, the methods used followed by the discussion of the results obtained and, then, the conclusion derived are given.

II. METHODOLOGY

2.1 Sesame Seed Preparation

The sesame seed that was used in this work was obtained from Kawo Market, Kaduna, Kaduna State of Nigeria. After the seed was obtained, it was unshelled and later grinded using a hand grinder to reduce the seed into a size of range 0.5-2.5 mm.

2.2 Experimental Design

After the sesame seed was obtained and prepared, the experiments to be carried out were designed with the aid of Design Expert 7.0.0 (Stat-Ease, 2005) using the central composite design of the response surface methodology. In designing the experiments, the factors considered were the particle size of the seed, the temperature of extraction and the time used for the extraction while the response of each of the experiments was chosen to be the yield of the oil obtained from the seed. The maximum and the minimum levels used for the factors considered in this work are as given in Table 1. Using the levels of the three factors given in the Table 1 and an alpha value of the design of 1.6818, twenty (20) experiments were designed and carried out using Soxhlet extractor.

Table 1. Factors and their levels for central composite design

Variable	Symbol	Levels	
		Minimum (-1)	Maximum (+1)
Particle size (mm)	X_1	0.91	2.09
Temperature (°C)	X_3	80.07	94.93
Extraction time (hr)	X_2	2.42	6.58

2.3 Extraction Procedure

To implement each of the experimental runs designed with the aid of Design Expert using the central composite design of the response surface methodology, the sesame seed (5 g per sample) was packed inside a thimble bag (see Figure 1) and placed inside the thimble chamber of the 250 ml Soxhlet extractor shown in Figure 2. The extractor itself was placed inside a thermostatic water bath (shown in Figure 3). A round bottom flask containing n-hexane as well as a condenser was fixed to the extractor. The flask was heated to a temperature above the boiling point of the solvent as indicated in the design matrix. The solvent then vaporized and passed through the prepared seed to remove its oil. The mixture obtained (solvent and oil) moved directly into the round bottom flask. The process was allowed to continue for the specified time, as obtained from the experimental design. Thereafter, the oil extracted was recovered by distilling the solvent using the same apparatus.

Figure 1. Thimble bag

Figure 2. Soxhlet extractor

Figure 3. Thermostatic water bath

At the end of each experiment, the yield of the oil was obtained using the following relationship.

$$Oil\ yield = \left(\frac{w_3 - w_1}{w_2}\right) \times 100\% \qquad (1)$$

where
W_1 is the weight of flask
W_2 is the weight of sample
W_3 is the weight of flask and extracted oil

2.4 Statistical Analysis and Optimization

After obtaining the responses (oil yield) from each of the experiments, the values (the responses) were entered into the appropriate column in Design Expert, and the response together with the factors considered were analysed, and a quadratic and a cubic models relating the percentage oil yield to the factors considered were developed for the extraction. In order to get a model that would best fit the experimental results and take care of effects of all the factors, the developed models were modified. The modifications carried out resulted in improvements in the performances of the models.

Using the modified models, a numerical optimization was carried out and the optimum conditions of the three factors investigated for the extraction were obtained. For the quadratic model, the goals for the factors were in a range while that of the response was "maximize". In maximizing the response using cubic model, the goal of each of the factors was a target of 0.91 mm, 88.53 ^oC and 6.58 hr for particle size, temperature and extraction time respectively.

III. RESULT AND DISCUSSION

The responses, together with the values of the factors used, that were obtained from the experiments carried out are as given in Table 2 shown below.

Table 2. The central composite design matrix and responses

	Factor 1	Factor 2	Factor 3	Response
Run	X_1: Particle size, mm	X_2: Temperature, ^oC	X_3:Time, hr	Oil yield, %
1	1.5	87.5	4.5	44.8
2	0.91	80.07	6.58	52
3	0.5	87.5	4.5	59.2
4	1.5	100	4.5	48
5	1.5	87.5	4.5	44.6
6	1.5	87.5	4.5	44.8
7	2.09	80.07	2.42	31.8
8	2.09	94.93	2.42	33.4
9	0.91	80.07	2.42	49.6
10	0.91	94.93	6.58	52.6
11	1.5	87.5	8	54
12	1.5	87.5	4.5	45
13	0.91	94.93	2.42	52.6
14	1.5	75	4.5	45
15	2.09	94.93	6.58	41
16	2.09	80.07	6.58	40
17	1.5	87.5	4.5	44.6
18	2.5	87.5	4.5	10.6
19	1.5	87.5	1	42.2
20	1.5	87.5	4.5	44.4

As shown in Table 2, the results obtained showed that variation of particle size and extraction time affected the oil yield most. For instance, looking at Run 3 and Run 18 in which particle size was increased from 0.5 mm to 2.5 mm, oil yield was noted to decrease from 59.2 to 10.6%. However, comparing Run 19 with Run 11, similar variation in extraction time led to increase in oil yield from 42.2 to 54%. Although, varying the extraction temperature in this manner also affected oil yield but the effect was not that significant compared to those of particle size and extraction time.

After the results of the experiments were obtained and considered, they were, first, used to develop a quadratic model equation for the extraction. The developed quadratic model equation is as shown in Equation (2) while the analysis of variance (ANOVA) of the model is given in Table 3. It was observed from the results of the analysis of variance that the model contained some terms that were not significant. As a result of this, the model was modified by reducing the terms it contained, and the modified model obtained is given in Equation (3). Just as it was done to the developed full quadratic model of the process, the modified model was as well analysed, and the results obtained from its analysis of variance are as seen in Table 4.

$$\begin{aligned} Y_{quad} &= 150.0869 + 6.3731X_1 - 2.4565X_2 - 1.5295X_3 - 0.0283X_1X_2 + \cdots \\ &\cdots + 1.3536X_1X_3 - 0.0242X_2X_3 - 9.1725X_1^2 + 0.0155X_2^2 + 0.3288X_3^2 \end{aligned} \tag{2}$$

Table 3. Analysis of variance (ANOVA) results for full quadratic model

Source	Sum of squares	Degree of freedom	Mean Square	F-value	p-value
Model	1833.67	9	203.74	12.29	0.0003
X_1-Particle size	1483.45	1	1483.45	89.47	< 0.0001
X_2-temperature	9.26	1	9.26	0.56	0.4721
X_3-extraction time	105.99	1	105.99	6.39	0.03
X_1X_2	0.13	1	0.13	7.54E-03	0.9325
X_1X_3	22.45	1	22.45	1.35	0.2717
X_2X_3	1.12	1	1.12	0.068	0.7998
X_1^2	151.56	1	151.56	9.14	0.0128
X_2^2	10.62	1	10.62	0.64	0.4422
X_3^2	29.22	1	29.22	1.76	0.2139
Residual	165.81	10	16.58		
Lack of Fit	165.59	5	33.12	752.69	< 0.0001
Pure Error	0.22	5	0.044		
Cor Total	1999.48	19			
R-squared = 0.9171;	Adj R-squared = 0.8424;		Pred R-squared= 0.3705		

$$\begin{aligned} Y_{quad\mathrm{mod}} &= 130.5084 + 13.6640X_1 - 2.1177X_2 + 1.3386X_3 - 0.02828X_1X_2 + \cdots \\ &\cdots - 9.5724X_1^2 + 0.01298X_2^2 \end{aligned} \tag{3}$$

Table 4. Analysis of variance (ANOVA) results for reduced quadratic model

Source	Sum of squares	Degree of freedom	Mean square	f-value	p-value
Model	1812.54	5	362.15	27.15	0.0001
X_1-particle size	1483.54	1	1483.45	111.10	<0.0001
X_2-Temperature	9.26	1	9.26	0.55	0.4712
X_3-extraction time	105.99	1	105.99	7.94	0.0137
X_1X_2	22.44	1	22.44	1.68	0.2158
X_1^2	161.22	1	161.22	12.07	0.0037
X_2^2	26.08	1	26.08	1.95	0.1840
Residual	186.94	14	13.35	-	-
Lack of fit	186.72	9	20.75	471.51	< 0.0001
Pure error	0.22	5	0.044		
Core total	1999.48	19	-		
R-squared = 0.8907 ;	Adj R-squared = 0.8402;		Pred R-squared= 0.5481		

Comparing the results of analyses of variance (ANOVA) given in Tables 3 and 4 for the full and the modified quadratic model equations of the extraction, respectively, and considering the R-squared and the adjusted R-squared values of the models, it was noted that the modification did not result into improving the model relating the factors to the oil yield. However, it was discovered that there was an improvement in the prediction capability of the model because its Pred R-squared value was found to increase from 0.3705 to 0.5481.

In order to clearly see the performances of the developed models, they were simulated and the results of their simulations were compared with the experimental values of the oil yield (see Figure 5). From Figure 5, it was discovered that there were good comparisons between the results of the simulation of the models and the experimental ones. The good conformities of the two quadratic models with the experimental results have confirmed the high values of R-squared (given in Tables 3 and 4) obtained for the full and the modified quadratic models because, as can be seen on Tables 3 and 4, the R-squared value of the full quadratic model was 0.9171 while that of the modified quadratic model was found to be 0.8907.

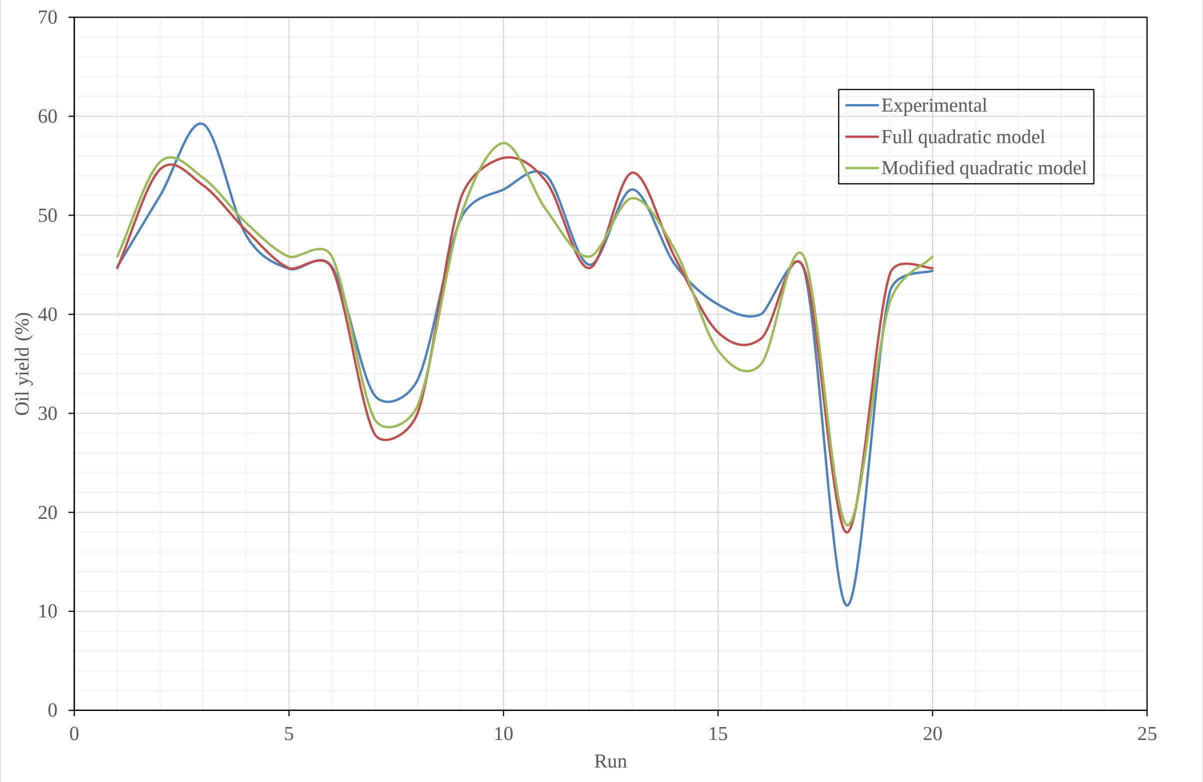

Figure 5. Experimental and quadratic model simulated oil yields

In an attempt to obtain a model with better performance than the one given by the quadratic model of the process, a cubic model equation was developed, but owing to the fact that the developed cubic model was found to be aliased in nature, its model expression in terms of the actual factors was not given by the Design Expert. However, the aliased cubic model, the mathematical expression of which was not available, was still analysed for variance to see if actually increasing order of model had any effect on the response statistically. The results obtained from the analysis are as given in Table 5. As it can be seen in the table, an increase in the order of the model was discovered to improve the performance of the model, because the R-squared, the Adj R-squared and the Pred R-squared values of the full quadratic model were 0.9171, 0.8424 and 0.3705 while those of the full cubic model have been obtained to be 0.9981, 0.9941 and 0.6107, respectively.

Table 5. Analysis of variance (ANOVA) results for full cubic model

Source	Sum of square	df	Mean square	F-value	p-value
Model	1995.73	13	153.52	245.62	< 0.0001
X_1-Particle size	1180.98	1	1180.98	1889.51	< 0.0001
X_2-Temperature	4.5	1	4.5	7.2	0.0364
X_3-Extraction time	69.62	1	69.62	111.39	< 0.0001
X_1X_2	0.13	1	0.13	0.2	0.6704
X_1X_3	22.45	1	22.45	35.91	0.001
X_2X_3	1.13	1	1.13	1.8	0.2283
X_1^2	151.56	1	151.56	242.49	< 0.0001
X_2^2	10.62	1	10.62	16.98	0.0062

X_3^2	29.22	1	29.22	46.75	0.0005
$X_1X_2X_3$	0.4	1	0.4	0.65	0.4515
$X_1^2X_2$	0.045	1	0.045	0.072	0.7968
$X_1^2X_3$	5.04	1	5.04	8.06	0.0296
$X_1X_2^2$	156.57	1	156.57	250.51	< 0.0001
$X_1X_3^2$	0	0			
$X_2^2X_3$	0	0			
$X_2X_3^2$	0	0			
X_1^3	0	0			
X_2^3	0	0			
X_3^3	0	0			
Residual	3.75	6	0.63		
Lack of Fit	3.53	1	3.53	80.23	0.0003
Pure Error	0.22	5	0.044		
Cor Total	1999.48	19			

R-squared= 0.9981; Adj R-squared= 0.9941; Pred R-squared= 0.6107

The better performance of the full cubic model over the quadratic ones has actually been observed, but it should be recalled that it was found that the model (cubic model) contained some terms that were aliased with one another. Of course, that was the reason why its model expression in terms of the actual factors was not available. As such, the developed cubic model was also subjected to modification and the modified model obtained with the aid of Design Expert is as given in Equation (4).

$$\begin{aligned} Y_{cubicmod} &= -2221.6364 + 1579.1677X_1 + 52.4758X_2 - 4.9533X_3 - 36.6498X_1X_2 + \cdots \\ &\cdots + 6.3816X_1X_3 - 0.0242X_2X_3 - 1.6306X_1^2 - 0.2984X_2^2 + \cdots \\ &\cdots + 0.3288X_3^2 - 1.6760X_1^2X_3 + 0.2093X_1X_2^2 \end{aligned} \tag{4}$$

Analysing the modified cubic model equation developed, the results of which are given in Table 6, it was discovered that even though there were still some terms in the model that were not significant, its performance was found to be very good because its R-squared, Adj R-squared and Pred R-squared values were estimated to be 0.9979, 0.9950 and 0.9089, respectively.

Table 6. Analysis of variance (ANOVA) results for reduced cubic model

Source	Sum of square	df	Mean square	F-value	p-value
Model	1995.278	11	181.3889	345.469	< 0.0001
X_1-Particle size	1180.98	1	1180.98	2249.267	< 0.0001
X_2-Temperature	9.259712	1	9.259712	17.63583	0.0030
X_3-Extraction time	69.62	1	69.62	132.5966	< 0.0001
X_1X_2	0.125	1	0.125	0.238072	0.6387
X_1X_3	22.445	1	22.445	42.74822	0.0002
X_2X_3	1.125	1	1.125	2.142649	0.1814
X_1^2	151.5609	1	151.5609	288.6593	< 0.0001
X_2^2	10.61539	1	10.61539	20.21782	0.0020
X_3^2	29.22044	1	29.22044	55.65256	< 0.0001
$X_1^2X_3$	5.03911	1	5.03911	9.597372	0.0147
X_1 X_3^2	156.5729	1	156.5729	298.205	< 0.0001
Residual	4.200408	8	0.525051		
Lack of Fit	3.980408	3	1.326803	30.15461	0.0013
Pure Error	0.22	5	0.044		
Cor Total	1999.478	19			

R-Squared = 0.9979; Adj R-Squared = 0.9950; Pred R-Squared = 0.9089

The high R-squared value obtained from the analysis of variance of the model was, actually, found to be evident from the good closeness of the simulated values of the oil yield and the experimental ones (Figure 5).

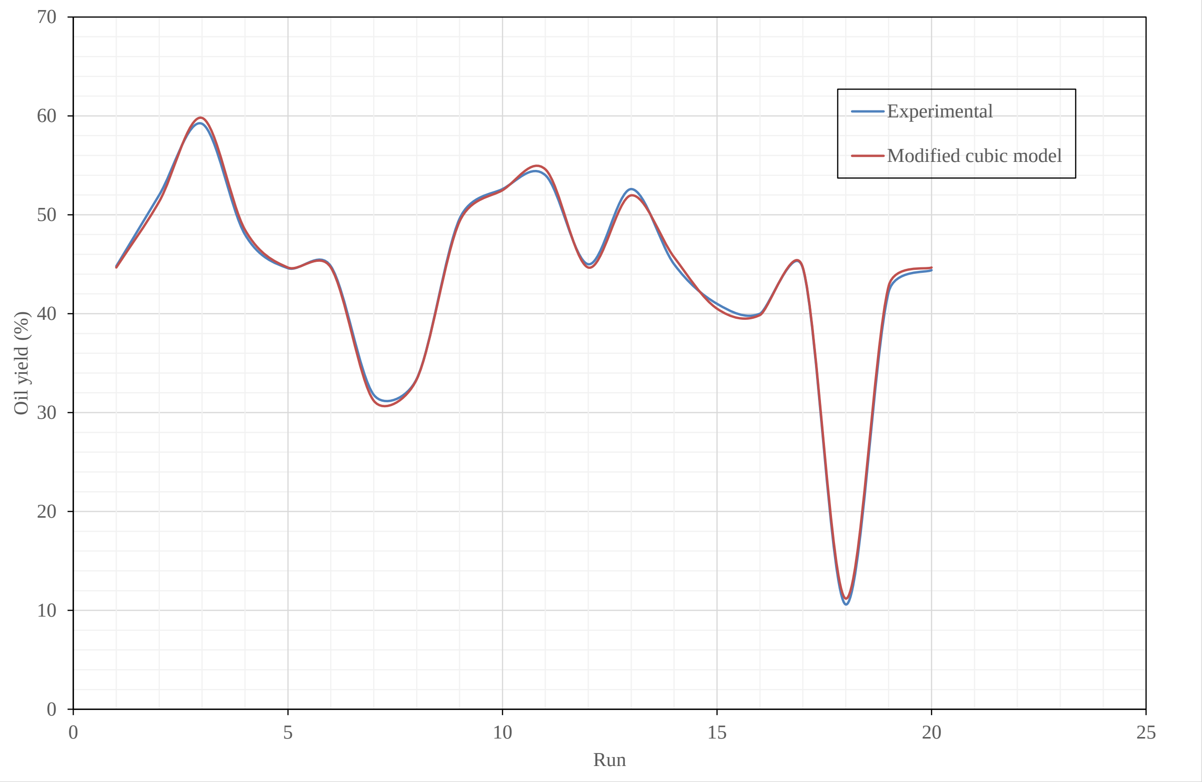

Figure 5. Experimental and cubic model simulated oil yield

Shown in Figures 6 and 7 are the variations of particle size and extraction temperature with oil yield for the modified quadratic and cubic models, respectively. According to the figures, it was discovered that the variation of particle size with temperature affected oil yield and it could be seen that at the temperature of approximately 87.5 °C, increasing the particle size was discovered to lead to decrease in oil yield value obtained from the sesame seed.

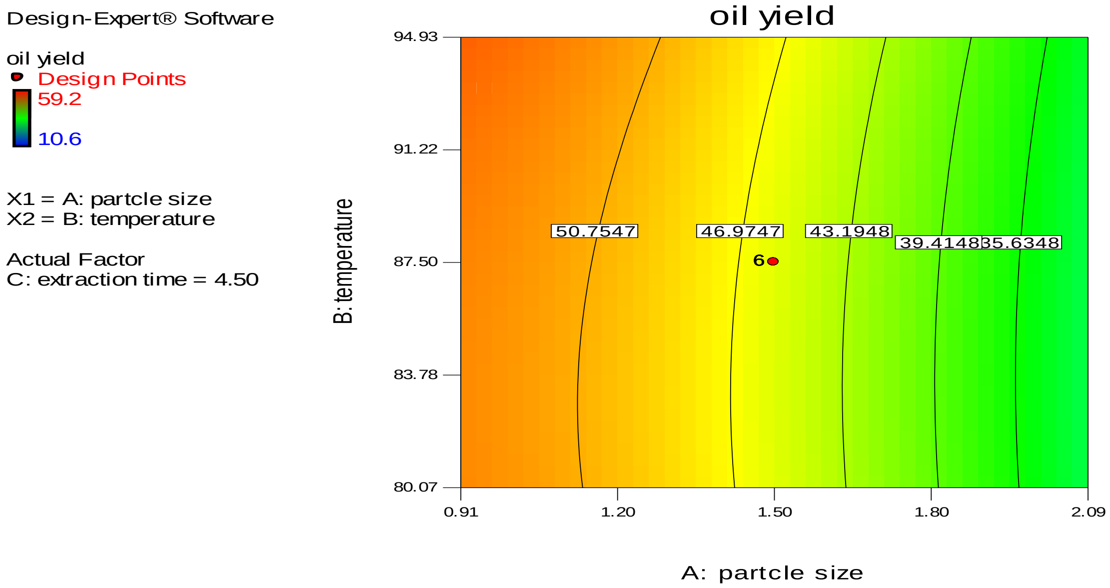

Figure 6. Contour plot for effect of particle size and extraction temperature on oil yield based on modified quadratic model

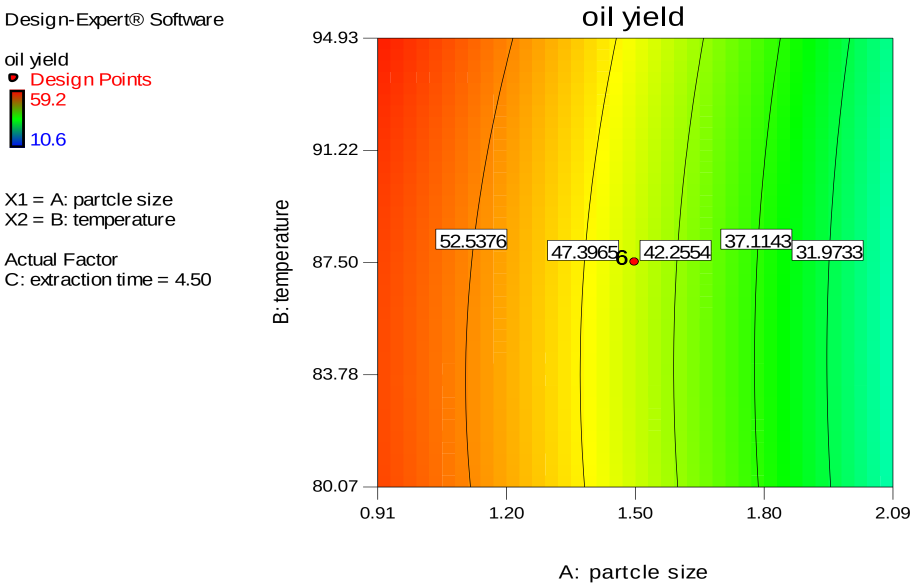

Figure 7. Contour plot for effect of particle size and extraction temperature on oil yield based on modified cubic model

Looking at Figures 8 and 9, the effects of the three factors (particle size, extraction temperature and extraction time) considered on the oil yield can be easily seen. From these figures, it was discovered that the variation of particle size was found to significantly affect the oil yield obtained from sesame seed as the extraction time was varied. For instance, increasing the particle size while slightly varying the extraction temperature and extraction time as shown in Figures 7 and 8 led to decrease in oil yield. However, the oil yield was not significantly affected by the interaction of temperature and extraction time; this is obvious from Figure 9.

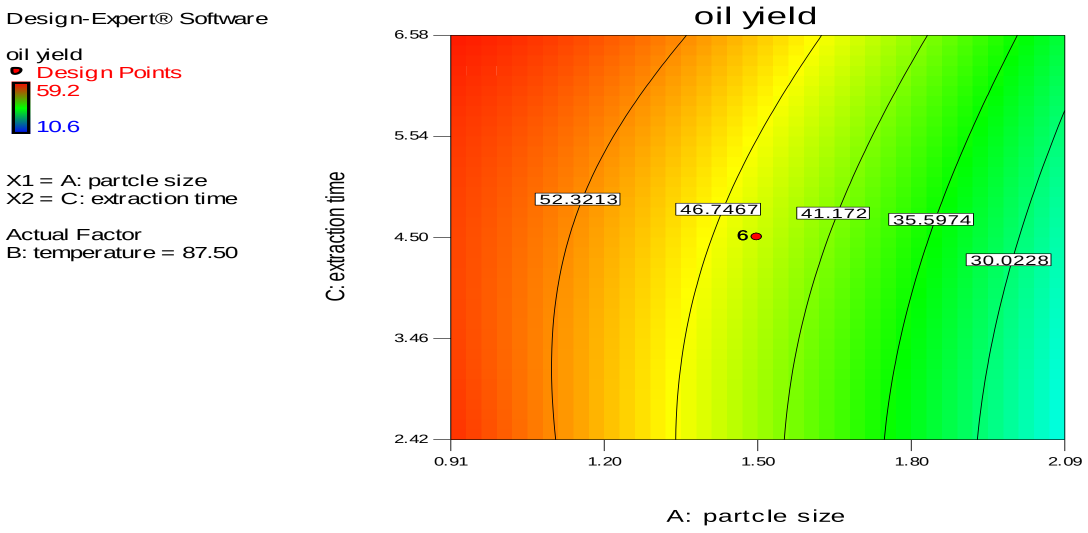

Figure 8. Contour plot for effect of particle size and extraction time on oil yield based on modified cubic model

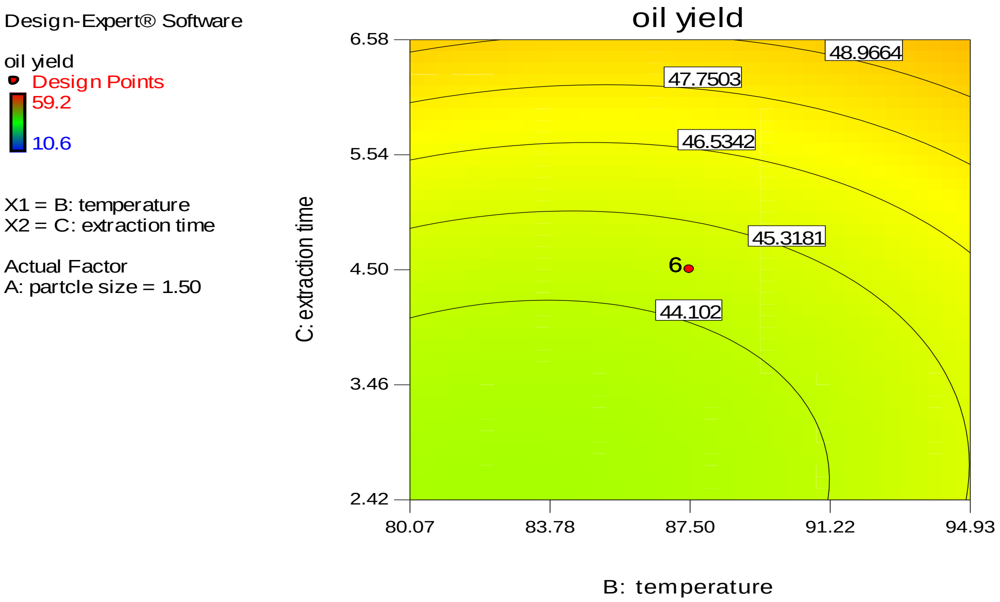

Figure 9. Contour plot for effect of temperature and extraction time on oil yield based on modified cubic model

From the numerical analysis carried out for both the quadratic and the cubic models developed for oil extraction from sesame seed, particle size of 0.91 mm, extraction temperature of 88.53 °C and extraction time of 6.58 hr were found to be the optimum operating conditions, which led to optimum oil yield of 55.80% and 51.15% for modified quadratic and cubic models, respectively. Furthermore, using the optimum parameters obtained from the response surface methodology employed carried out to perform a validation experiment, oil yield of 55.5% was obtained. The value obtained was found to compare well with the predicted optimum yield. Therefore, the result obtained has revealed that the optimum values of the parameters given by response surface methodology with the aid of Design Expert were valid ones.

IV. CONCLUSION

The results obtained from this work have revealed that the performance of a cubic model was better than that of a quadratic model for this extraction of oil from sesame seed because the R-squared, the Adj R-squared and the Pred R-squared values of the developed and modified quadratic model were 0.8907, 0.8402 and 0.5481 while those of the cubic model developed and modified were 0.9979, 0.9950 and 0.9089, respectively. Furthermore, the results of the optimization carried out revealed that the optimum values of the particle size, the extraction temperature and the extraction time were 0.91 mm, 88.53 °C and 6.58 hr, respectively. Validating the obtained optimum values of the parameters experimentally, it was discovered that there was a good agreement between them because the optimum oil yields predicted by the statistical method employed were 55.80% and 51.15% for modified quadratic and cubic models, respectively while that obtained from the validation experiment was found to be 55.5%. This has demonstrated that the optimum values obtained from the Design Expert were valid ones. Thus, it can be concluded that response surface methodology has been successfully applied to optimize sesame seed oil yield.

NOMENCLATURE

Adj	Adjusted
Pred	Predicted
X_1	Particle size
X_2	Extraction temperature (°C)

X_3	Extraction time (hr)
$Y_{cubicmod}$	Oil yield from modified cubic model
Y_{quad}	Oil yield from full quadratic model
$Y_{quadmod}$	Oil yield from modified quadratic model

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