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Research Article

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Optimization of Small Castor Seed (Ricinus Communis) Oil Extraction Yield Using Response Surface Methodology

Samuel Ayodeji Omotehinse[1,*] **Godwin Ovuworie**[1]

[1]Department of Production/Industrial Engineering, University of Benin, P.M.B 1154, Benin City, Edo State, Nigeria

[*]Corresponding Author: drsamayodeji@gmail.com

Abstract

The process conditions that affect the percentage yield of oil from small castor seed was optimized for maximum extraction. In this study, surface response methodology (RSM) that employed a two-factor, five-level factorial central composite design (CCD) was used. Thirteen (13) experiments with different combinations of reaction time (x_1) and reaction temperature (x_2) ranging from 1hr (60mins) to 10hrs (600mins) and 60oC to 100oC respectively were also performed. A quadratic model that is polynomial in nature was obtained to predict the percentage oil yield for the small castor seed. Within the experimental variables range, the optimal conditions were found to be 8.68hrs (520.92mins) and 94.14oC respectively. These values were fitted into the quadratic polynomial model that gave rise to the optimum value of small castor seed oil yield to be 55.76% with a p-value less than 0.05. The coefficient of determination R2 was obtained as 0.9530 representing 95.30% of variation in the original data. Our result also gave an adjusted R2 value of 91.95% and predicted R2 value of 66.59% which indicate that the model explains 67% variation in predicting original observations. The result of this study showed clearly the percentage yield of oil that could be extracted from small seeded varieties of castor seed and the optimum yield value possible at a certain reaction time and temperature. The result also established the reliability of response surface methodology to model and optimize the expression of oil from small seeded varieties of castor seed.

Keywords: Response surface methodology, Central composite design, Castor seed oil, Optimization, Oil yield

Introduction

Some concern has been expressed over the measure of oil that could be gotten from various varieties of castor seed. This is as a result of the various varieties and sizes of castor seed we have available. This research gap has necessitated this study to know the percentage oil yield from small seeded varieties of castor seed and as well optimize the yield to achieve the maximum oil extraction at a particular reaction temperature and time. Oil extraction yield is referred to as the amount of oil that can be derived from an oil seed. The yield is usually being represented as a percentage (%). In arid and semi-arid regions where little or no maintenance is applied, Castor bean (Ricinus Communis) usually produces up to 350-650 kg of oil per hectare. The oil from the seed is the principal product of the castor bean plant. This oil can be extracted either by the use of cold-pressing (mechanical) or by using soxhlet extraction method with solvent. The content of the oil ranges from 35% to 55% of the weight of the beans depending on the variety (Kulkarni and Sawant, 2003; Oluwole *et al.,* 2014). It is instructive to note that castor seed oil has many industrial uses. Some of these uses are found in internal combustion engines, in processing and manufacturing of products like rubber, inks, flypaper, linoleums, artificial leather, varnish and medical preparations as well as being a plasticizer in plastic production.

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It can also be used as a sulfonated oil to produce Turkey red oil which is a compound that is widely engaged in the dyeing and cloth printing industry. The latest advances in castor seed oil dehydration makes it a substitute to sunflower oil or tung oil as a drying oil. Apart from the various uses of castor seed oil, recent investigations have shown that the pulps can also be used as fertilizers (Lima et al., 2011; Mello et al., 2018).

Research interest in the percentage oil extraction yield from castor seed and its application has built up over the years. Notable among them are Abdulrasheed *et al.* (2015) who characterized and investigated the use of castor seed oil extract in soap manufacture. Yusuf *et al.* (2015) extracted oil from castor seed in Katsina, Nigeria and went ahead to checked the physicochemical properties of the oil extracted to assess its industrial potentials. The result showed oil extraction yield to be 39.43% while the physicochemical characterization of the purified oil revealed low acid value of 2.07, low iodine value of 84.18, low peroxide value of 38.00, but relatively high specific gravity of 0.959, hydroxyl of 163.64 and saponification of 175.31 values. These values compare favourably well with the general standards and specification of the American standard for testing and materials (ASTM) for grade castor oil (WHC, 2012) suggesting that the oil has good industrial potential. Nangbes *et al.* (2013) Extracted and Characterized Castor (Ricinus Communis) Seed Oil native to Kapil-Lankan area of Pankshin Local Government Area of Plateau State, Nigeria. The outcome of this analysis revealed percentage oil extraction yield of 48.32. The result that show case the commercial value of the seed in Nigeria. The result also confirms the castor seed oil to be a good quality oil that can be useful as food additives in food industry as well as in industrial application for the manufacturing of products like soaps, paint, varnishes, cosmetics etc. Shridhar *et al.* (2010) optimized the dilution level as well as the agitation time for castor seed oil yield using response surface methodology technique. The author also investigated the percentage recovery of castor seed oil in relation to dilution level (x_1) and agitation time (x_2). The maximum extraction was found to be 48.75% while the optimal value for dilution and agitation time were established as 7.3 and 2.38 hr respectively. Other outstanding research in this area includes: Akpan *et al.,* (2006), Kyari (2008), Montoya *et al.* (2011), Mosquera-Artamonov *et al.* (2016). The aim of this study is geared towards optimizing small castor seed oil extraction yield using response surface methodology. To achieve this, central composite design (CCD) in response surface methodology (RSM) that is seen as a proficient statistical technique used for optimizing multiple variables was adopted to maximize the best reaction conditions with least possible number of experiments.

Materials and Methods

Materials and equipment

The castor seeds used in this research include the small castor oil bean seeds- Ricinus Communis obtained from a farmland in Ikokogbe, Omhen in Ewossa Community, Igueben Local Government Area of Edo State, Nigeria (9.0820° N, 8.6753° E). The seeds were sown in plots having a size of 3 x 4m, 1metre between plants and 1metre between rows. The experiment was laid out in a completely randomized design replicated two times. Germination was observed at the emergence of the cotyledons above the soil surface and monitored until the seed matured. The harvested ripe and dried castor fruits were carefully cleaned and dried in the sun for about 5 days at ambient conditions. This was done until the capsules of the fruit split open to release encased seeds. Seed pod removal as well as tray-winnowing followed to separate the shells from the beans. The castor beans were then oven dried to a constant weight of 100g per sample at 80^0C for 9hrs with a moisture content of 0.32%. Prior to extraction, mortar and pestle was used to grind the dried beans to a paste. All the chemicals and reagents used was analytical grade obtained from Sigma Aldrich without further purification. Reagents were prepared using distilled water while the laboratory apparatuses washed with detergent, cleaned with distilled water and oven-dry before use.

Method

Extraction

Soxhlet extraction method was used. A Soxhlet extractor is a piece of laboratory apparatus designed in 1879 by Franz von Soxhlet. The typical soxhlet extraction set up for the small castor seed oil is shown in Figure 1. Soxhlet extractor with three main sections was used. The first is a percolator that circulate the solvents. The second is a thimble made of thick filter paper that help retain the solid to be extracted. The third is a siphon mechanism which empties the thimble. The compound to be extracted is positioned inside the thimble while the thimble is placed in the main chamber. The solvent being used for the extraction is placed in the distillation flask with the flask positioned on the heating element. The soxhlet extractor is however put on the flask and the reflux condenser placed on the extractor. Extraction solvent is taken in the round bottom flask and heated by using heating source like heating mantle. The heating temperature is built on the solvent employed to extraction. Due to heat the solvent in the bottom flask vaporizes into the condenser and then drip back to the sample thimble. When liquid content reaches the siphon arm, the liquid contents emptied into the bottom flask again which is the end of the process indicated by the clear solution in the siphon tube.

Thirteen (13) experimental runs were carried out to dig out maximum oil yield in small castor seed where optimum process conditions were sustained. The controllable factors considered are reaction time as well as reaction temperature. The reaction time varies from 1hr (60mins) to 10hrs (600mins), and the reaction temperature ranges from 60^{O}C to 100^{O}C.

The percentage yield of the oil was determined by using the expression described in equation 1.

$$\%\,\text{Oil yield} = \frac{y_1 - y_2}{y_1} \times 100 \qquad (1)$$

where, y_1 and y_2 represent the weights of small castor beans prior to and after extraction.

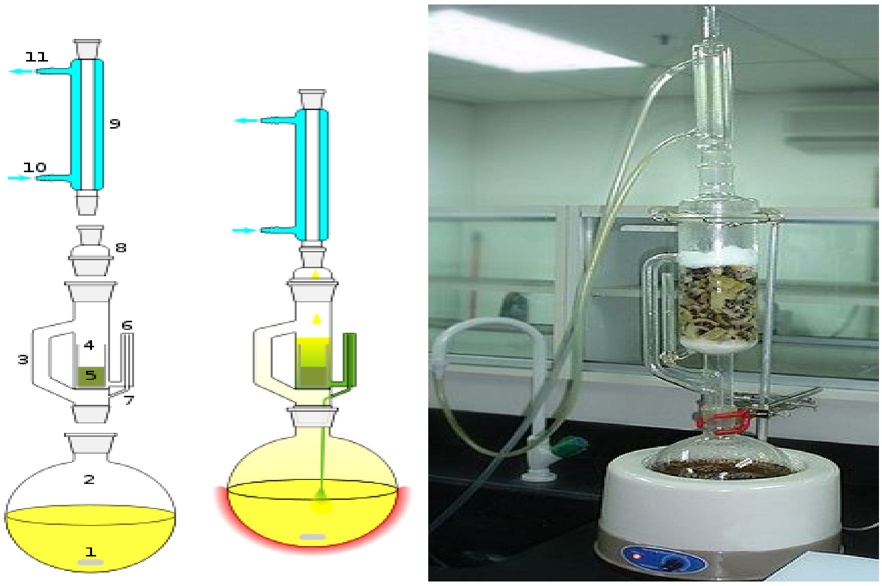

Figure 1. A schematic representation of a Soxhlet extractor
1- The Stirrer bar, 2 - The Still pot, 3 - The Distillation path, 4 - The Thimble, 5 -The Solid to be extracted, 6 - The Siphon top, 7 - The Siphon exit, 8 - The Expansion adapter, 9 - The Condenser, 10 - The Cooling channel.

Experimental Design and Data Analysis

Two-factor, five-level factorial of Central Composite Design (CCD) in surface response methodology (RSM) was employed with replicates at the centre point and star points. Reaction time (x_1) and reaction temperature (x_2) are the variables used in this study with each at low (-1) and high (+1) coded levels. The initial levels selected as the center points were based on the actual levels of the variables. Thirteen (13) experimental trials which consist of 4 trials for axial points – two for each variable, 4 trials for factorial design as well as 5 trials for replication of the central points were executed. The oil yield responses Y (%) adopted in this study refers to the average of triplicate representing yield for the small castor seed. Table 1 illustrate the CCD experimental conditions for the extraction process.

Table 1. CCD Experimental Conditions for Small Castor Seed oil Extraction Process

Independent variable	Unit	Symbol	Levels		
			-1	0	+1
Reaction time	Mins	x_1	60	330	600
Reaction temperature		x_2	60	80	100

The analysis of the above experimental data was carried out based on the response surface regression system to accommodate the second-order polynomial equation. The level of significance of the coefficients was less than 0.05. Statistical software package design-expert® (version 8.0.6; stat-ease, Inc., Minneapolis, USA) was used to determine the regression coefficient which help to predict the process response as a function of the independent variables as well as their interaction that aid the understanding of the system behavior. The relationship that exist between the response and the process variables was calculated mathematically using the quadratic polynomial expression in equation 2.

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i x_i + \sum_{i=1}^{n} \beta_{ii} x_i^2 + \sum_{i=1}^{n} \sum_{j>1}^{n} \beta_{ij} x_i x_j \quad (2)$$

Y is the percentage oil yield (response), X_i and X_j are the coded independent variables. β_0 - constant, β_i - linear term coefficient, β_{ii} - the quadratic term coefficient, β_{ij} - interaction (cross-term) coefficient while *n* denote the number of process variables studied and optimized. Analysis of variance was used to estimate the effects of process variables including their interaction effects on the maximum yield of oil in the response surface regression procedure. Regression coefficient R^2 was also used to estimate the best of fit and goodness of the model. The fitted quadratic polynomial equation generated from regression analysis helped to obtain the response surface and contour plots by holding one of the independent variables at central value (0) and changing the other two.

Results and Discussion

The percentage (%) yield of oil for the small seeded varieties of castor seed was determined and the results are presented in Table 2. The in depth analysis involving the interaction of reaction time and reaction temperature was carried out on the percentage (%) oil yield. The Design-Expert (Stat-Ease, Inc., Minneapolis USA) software was employed for regression analysis and graphical analysis of the data obtained. The optimum values of the reaction time and reaction temperature were reached by solving the regression equation. This was also achieved by analyzing the response surface and the contour plots. Table 2 shows the coded and actual design matrix as well as the results for the combination of the variables for extraction process.

Figure 2 represent the correlation that stuck between the predicted % oil yield and the actual (experimental) % oil yield plot for small castor seed oil. The 45° straight line shows a perfect fit.

Analysis of Variance Table result for percentage oil yield of small castor seed is depicted in Table 3.

Table 2. Coded and Actual values and Experimental responses for CCD experimental combination of variables for small castor seed oil extraction process.

Run	Coded values		Actual values		$\text{Yield}_{\text{Small seed}}$ (%)	
	X_1	X_2	Reaction time (mins)	Reaction temperature (C^0)	Observed values	Predicted values
1	-1.000	1.000	139.08	94.14	44.14	44.29
2	-1.414	0.000	60.00	80.00	41.36	42.43
3	0.000	0.000	330.00	80.00	50.80	50.82
4	1.000	-1.000	520.92	65.86	53.90	52.98
5	0.000	1.414	330.00	100.00	52.60	51.54
6	0.000	0.000	330.00	80.00	50.83	50.82
7	1.000	1.000	520.92	94.14	53.90	55.02
8	1.414	0.000	600.00	80.00	53.90	53.60
9	-1.000	-1.000	139.08	65.86	49.81	47.92
10	0.000	-1.414	330.00	60.00	50.83	52.66
11	0.000	0.000	330.00	80.00	50.83	50.82
12	0.000	0.000	330.00	80.00	50.83	50.82
13	0.000	0.000	330.00	80.00	50.83	50.82

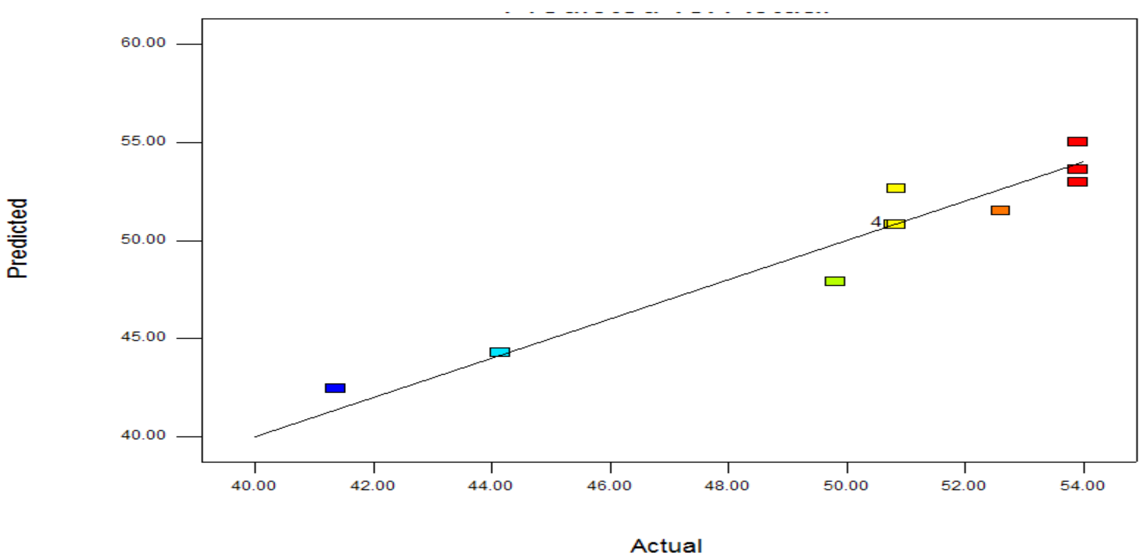

Figure 2. Predicted % oil yield vs the actual (experimental) % oil yield for small castor seed

Table 3. Analysis of Variance Result for Small Castor Seed Percentage oil yield

Source	Sum of Squares	df	Mean Square	F value a = 0.05	p-value Prob > F
Model	152.490	5	30.50	18.72	0.0006 significant
A–Reaction time	124.70	1	124.70	76.52	<0.0001
B-Reaction temperature	1.25	1	1.25	0.77	0.4095
AB	8.04	1	8.04	4.93	0.0618
A^2	13.75	1	13.75	8.44	0.0228
B^2	2.82	1	2.82	1.73	0.2298
Residual	11.41	7	1.63		
Lack of fit	11.41	3	3.80	21123.01	<0.0001 significant
Pure Error	7.200E-004	4	1.800E-004		
Cor total	163.90	12			

R-sq. = 93.50 % R-sq. (adjusted) = 88.07% R-sq. (predicted) = 50.51%

Table 3 show that the model F-value of 18.72 is small as well as the corresponding P-value of 0.0006. The implication is that the model employed is highly significant. It also implies that the chances of having "Model F-Value" this large could be 0.06%. The values of "Prob > F" less than 0.500 show that the model terms are significant which infers that A, A^2 are significant model terms. If the values are greater than 0.1000, it means that the model terms are not significant. It was observed from the Table also that effect of reaction temperature is insignificant on the response variable (yield) studied. The response variable had a quadratic response, where both reaction time and reaction temperature (AB) exhibited an effect, with a quadratic relationship. Adequacy precision was used to measure the signal to noise ratio involving the average prediction error and the design points. The adequacy precision of 14.524 was obtained in this study. This result show adequate signal because a ratio that is greater than 4 is required for the model to be adequate. And since the ratio obtained in this study is greater than 4, the model developed could be used to guide the design space. The "Lack of fit F-value" of 21123.01 shown in Table 3 denotes that the "Lack of Fit" is significant. We have only 0.01% chance in this case for the Lack of Fit F-value to occur due to noise. Second order polynomial regression model was obtained from the experimental data to predict optimum oil yield from the small castor seed using design expert software. The empirical model in-terms of coded variables for percentage oil yield (Y, %) for small castor seed is expressed in Equation 3.

$$Y_{Small\ seed} = 50.82 + 3.95x_1 - 0.40x_2 + 1.42x_1x_2 - 1.41x_1^2 + 0.64x_2^2 \quad (3)$$

where, Y is the yield (response variable)

x_1 and x_2 are the reaction time and reaction temperature respectively.

The negative sign indicates the antagonistic relationship whereas the positive sign point to the synergistic relationship. The terms with positive coefficient have positive effect on the yield (i.e. increase in these terms leads to corresponding increase in small castor seed oil yield). Reaction time (), interaction term () and the quadratic term ²) has significant influence on the yield of small castor seed oil. On the other hand, terms with negative coefficient (and ²) do not significantly influence the yield of small castor seed oil.

The coefficient of determination R^2 for small castor seed oil extraction was obtained to be 0.9350. The result point to the model been effective in describing 93.50% of variation in the original data. The value of 0.8807 was obtained for the respective adjusted R^2. The R^2_{pre} value gotten through cross-validation advocated that the model is capable of explaining about 51% variation in predicting novel observations. Figure 3 (a-d) shows residuals based on the empirical model developed for the small castor seed.

To fully comprehend the relationship between the variables studied, the response surface curves was plotted as it also helped us to evaluate the optimum level of both the reaction time and the reaction temperature for maximum response. The contour plots for small seed oil extraction is shown in Figure 4 while the response surface plot displaying the interactive effect of reaction time and reaction temperature is shown in Figure 5.

The achieved maximum desirability of 0.935 obtained point out that it is possible to reach maximum castor oil yield target. The maximum small castor seed oil yield of 55.02% was attained at the reaction time of 520.92 mins (8.68 hrs) and reaction temperature of 94.14 respectively.

Conclusion

This study has been able to revealed the percentage yield of oil that could be extracted from small seeded varieties of castor seed as well as the optimum yield value possible at a certain reaction time and reaction temperature. The reliability of central composite design in response surface methodology was also demonstrated in determining the process conditions such as reaction time and reaction temperature leading to optimum oil extraction yield of small castor seed. This study discovered the range of percentage oil extraction yield in small seeded varieties of castor seed to be 41.36 - 53.90. The outcome of the study compared favourably well with 48% oil extraction yield obtained by Abitogun *et al.* (2009), 48.75% discovered by Shridhar *et al.* (2010), 50.16% reported by Muzenda *et al.* (2012) as well as 48.32% obtained by Nangbes *et al.* (2013). Others include Akande *et al*. (2012) and Yusuf *et al.* (2015). The optimal conditions to achieve the maximum oil extraction yield of 55.02% in small seeded varieties of castor seed was reached at a reaction time of 520.92 mins (8.68 hrs) and reaction temperature of 94.14. The result also suggest that the response surface methodology adopted could effectively be utilized in any reaction process that involves so many experimental factors to study their various effects, the optimum values and their interaction.

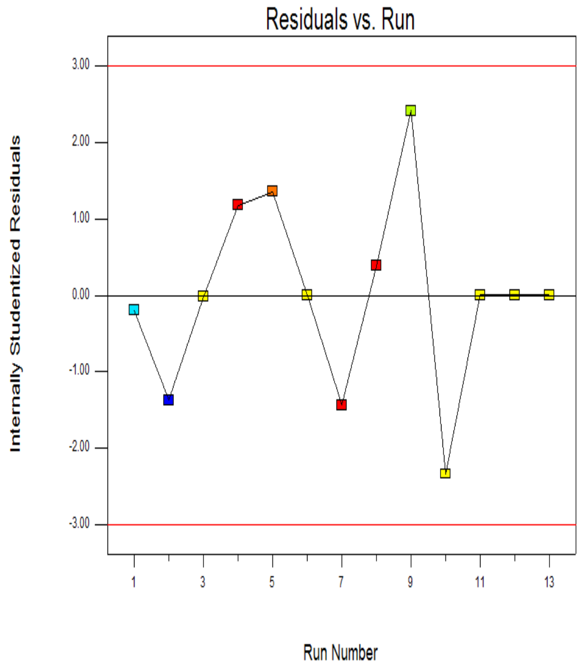

Figure 3(a). Residuals based on the run order for small castor seed oil yield

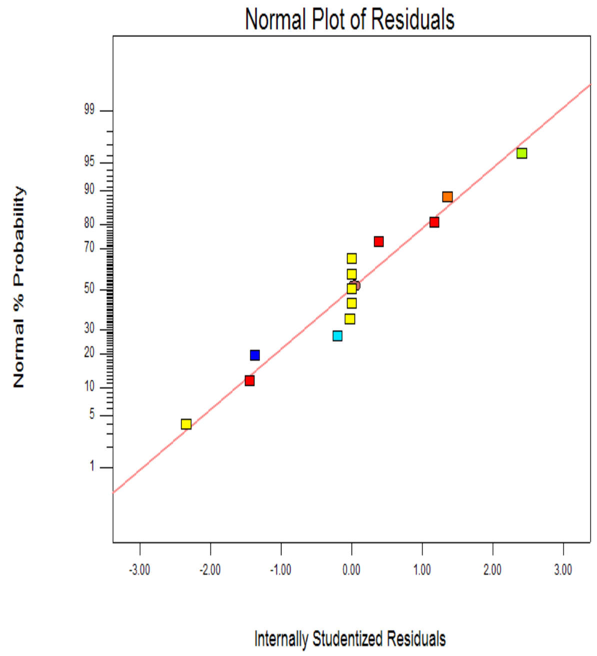

Figure 3(b). Normal probability plot of raw residuals for small castor seed oil yield

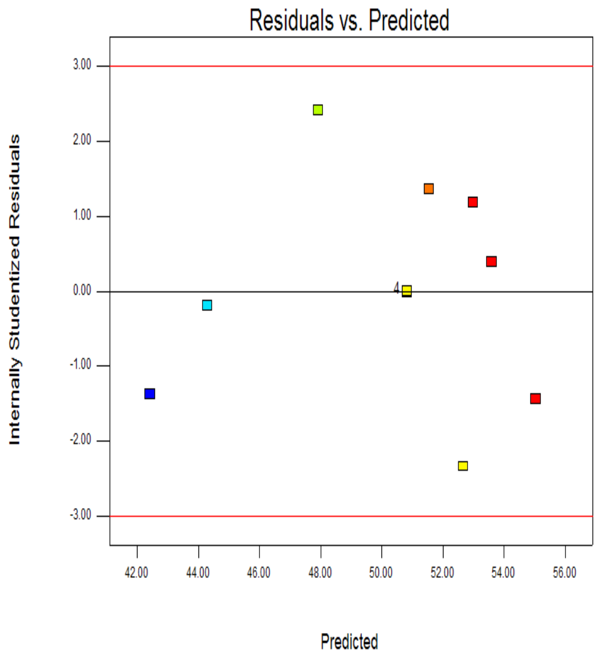

Figure 3(c). Residuals based on predicted order for small castor seed oil yield

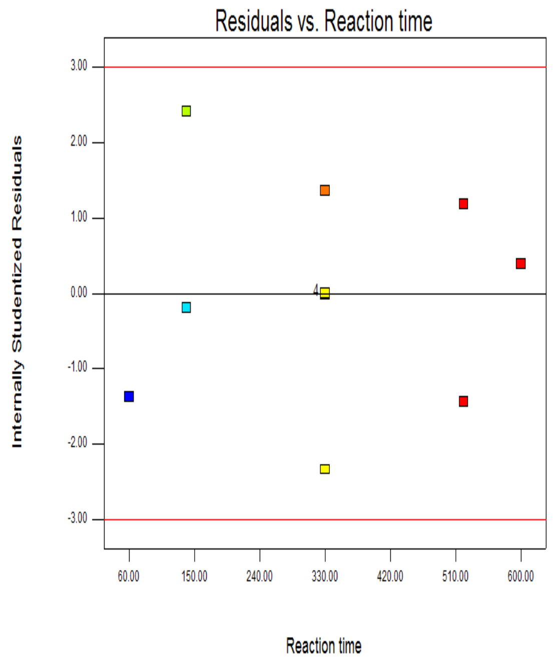

Figure 3(d). Residuals based on experimental order for small castor seed oil yield

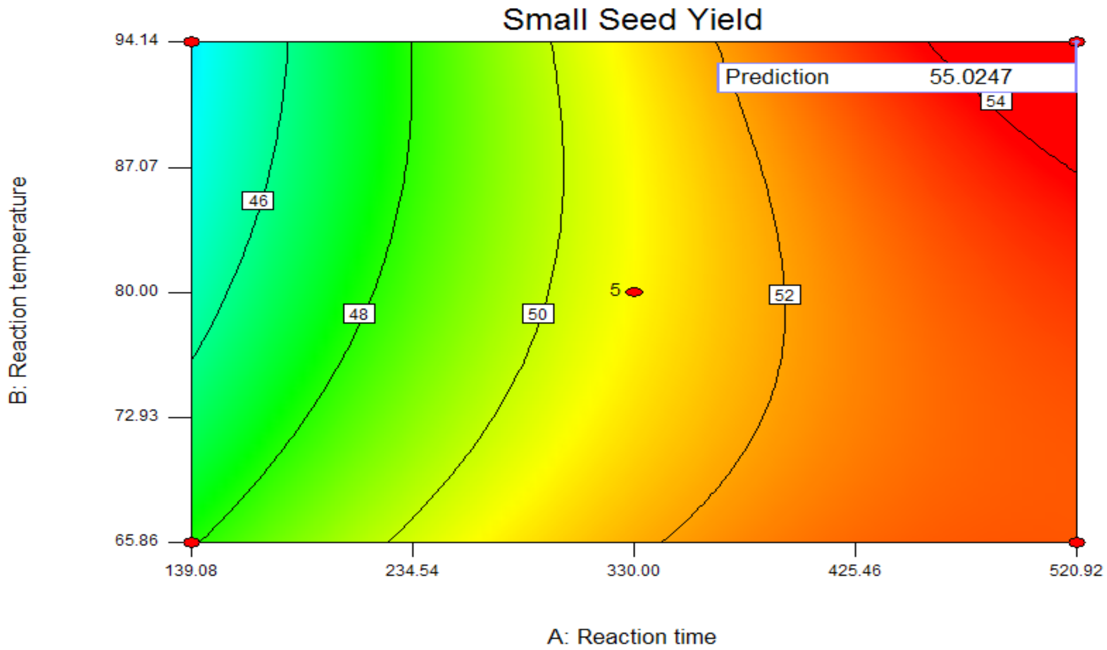

Figure 4. Isoresponse contour plots displaying the influence of reaction time and reaction temperature and their interaction effect on the % yield of small castor seed oil.

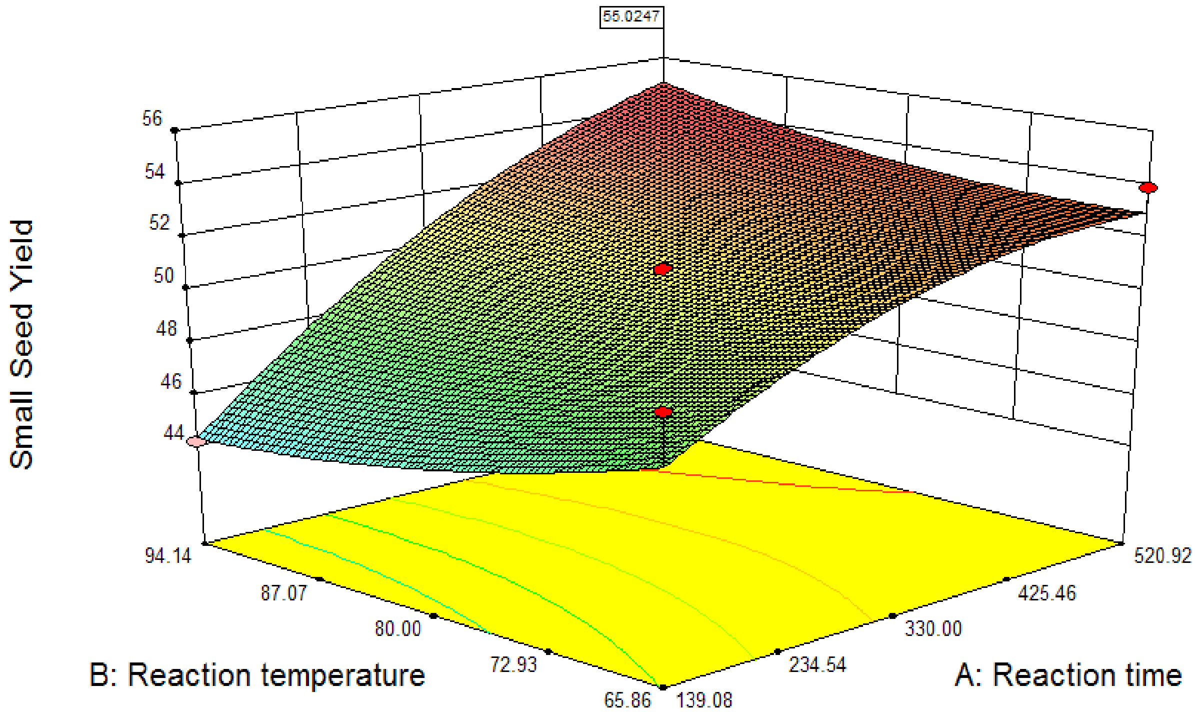

Figure 5. Response surface plots displaying the influence of reaction time and reaction temperature and their interaction effect on the % yield of small castor seed oil.

Compliance with Ethical Standards

Conflict of interest

The authors declared that for this research article, they have no actual, potential or perceived conflict of interest.

Author contribution

The contribution of the authors to the present study is equal. All the authors read and approved the final manuscript. All the authors verify that the Text, Figures, and Tables are original and that they have not been published before.

Ethical approval

Not applicable.

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Data availability

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Consent for publication

Not applicable.

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