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Optimization of Process Conditions affecting Percentage Oil Yield from Big Seeded Varieties of castor-oil plant, (*Ricinus communis*) Seed

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ABSTRACT: The objective of this study is to optimize reaction time, reaction temperature and their interactive effects on the oil yield of big seeded varieties of castor-oil plant, (*Ricinus communis*) seed using response surface methodology (RSM). 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 performed. A quadratic polynomial model was obtained to predict the percentage oil yield for the big seeded varieties of castor seed. Our result show that the optimal conditions for the oil yield within the experimental range of the variables studied were 8.68hrs (520.92mins) and 94.14ºC respectively. These values of the optimum process conditions were then fitted into the quadratic polynomial model to predict the optimum value of big seeded varieties of castor seed oil yield found to be 55.76%. The model has a p-value less than 0.05 having a coefficient of determination R^2 value of 0.9530 indicating that the model explained 95.30% of variation in the original data. The result also presented an adjusted R^2 value of 91.95% and predicted R^2 value of 66.59% which suggest that the model could explain 67% of variation in predicting novel observations. The optimization process employed in this study, clearly aid us to know the percentage yield of oil that could be extracted from big seeded varieties of castor seed and the optimum yield value possible at a certain reaction time and reaction temperature.

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Castor oil, like other seed oils, is extracted from the ripe or mature seeds of the castor plant after sun drying, and following a sequence of seed processing operations that may include dehulling, pod or seed coat removal, winnowing, sorting, cleaning, grinding or milling, preheating etc. (Akpan *et al*., 2006; Alirezalu *et al.*, 2011; Hussen, 2021). The oil extraction is usually achieved by mechanical expression or solvent extraction. Average oil content for all castor seed varieties has been reported to be between 30-55% oil by weight (Aldrich, 2003; Arawande, 2018). However, the actual yield depends particularly on the seed variety, geographical origin/climatic conditions, and on the oil extraction method(s) used. Response Surface Methodology (RSM) has been very popular for optimization studies in recent years. Some research examples of the applications performed for optimization of oil yield includes: Shridhar *et al.* (2010) who used response surface methodology to optimize the dilution level and agitation time for castor oil extraction. The optimal dilution level and agitation time obtained were 7.3 and 2.38 hr respectively while the maximum extraction was found to be 48.75%. Mosquera-Artamonov *et al.* (2016) also use RSM to optimize the extraction process of oil from hydraulic pressed *Ricinus communis* L. Olaoye and Busari (2017) optimized the effect of processing parameters of castor seed on its oil yield by applying Central Composite Rotatable Design of Response Surface Methodology. The optimal conditions within the experimental range of the studied variables; moisture content 7%, roasting temperature 110°C and roasting duration 20 min were used to predict optimum value of oil yield to be 25.77%. According to Mwithiga and Moriasi (2007), "the yield from oil-bearing seeds is dependent on the quality of the oilseeds and no method of extraction can compensate for this. The extraction process needs to be well managed in order to extract as much oil from the seed. Besides, there are a number of factors that can be considered during extraction process in order to increase the oil yield. These controllable factors include the size of particles and roasting temperature, the moisture content of oil seeds, the roasting duration and the pressure applied during extraction". The effect of these controllable factors has been investigated by (Ajibola *et al.,* 1990; Baryeh, 2001; Mwithiga and Moriasi 2007). These authors have all established that there exists an optimum value of moisture content for each product at which the oil yield is highest when other variables are kept constant. Danlani *et al.* (2014) optimized the percentage oil yield extracted from castor seed using response surface methodology and Box-Behnken design. However, the linear and quadratic effects of two variables studied were most significant in affecting the oil yield. In Dairo *et al.* (2013) optimization of in- situ biodiesel production from raw castor oil-bean seed was carried out from raw castor bean oil seed (37.9% oil content) by alkaline catalyzed in situ trans-esterification with sodium hydroxide as catalyst and ethanol as the solvent in a laboratory batch processor. Response surface methodology and central composite experimental design was also applied to evaluate the effects of reaction time (30-120min), alcohol/seed weight ratio (0.5-2.0), catalyst amount (0.3-1.5%) and reaction temperature (40-70^{0}C). Jeong and Pack (2009) applied response surface methodology in order to optimize the reaction factors for biodiesel synthesis from inedible castor oil. The study evaluated the effects of multiple parameters and then reciprocal interactions using a five-level, three-factor design. In a total of 20 individual experiments, the authors optimized the reaction temperature, oil – to – methanol molar ratio and quantity of catalyst. The objective of this study is to optimize reaction time, reaction temperature and their interactive effects on the oil yield of big seeded varieties of castor-oil plant, (Ricinus communis) seed.

MATERIALS AND METHODS

Materials and equipment: The castor seeds used in this research are the big seeded varieties of castor oil bean - *Ricinus Communis* obtained from a farmland in Ikokogbe, Omhen in Ewossa Community, Igueben Local Government Area of Edo State, Nigeria. The harvested ripe castor fruits were cleaned and sun-dried for 5 days, until fruit capsules split open to discharge encased seeds. This was followed by seed pod removal and tray-winnowing to separate shells from beans (cotyledons). The castor beans were later dried (per 100g sample) at 80^{0}C to constant weight for 9hrs in a hot air oven. The beans were then grinded to a paste using mortar and pestle, prior to extraction. All chemicals and reagents used in this study was analytical grade from Sigma Aldrich which was used without any further purification. Distilled water was used for the preparation of reagents. Laboratory apparatuses were washed with detergent, rinsed with distilled water and oven-dry before use.

Extraction: Soxhlet extraction method was used. The typical soxhlet extraction set up for the castor seed oil is shown in Figure 1. The Soxhlet extractor has three main sections: a percolator (boiler and reflux) which circulates the solvent, a thimble (usually made of thick filter paper) which retains the solid to be extracted, and a siphon mechanism, which periodically empties the thimble. In the assembly, the source material containing the compound to be extracted is placed inside the thimble; the thimble is loaded into the main chamber of the Soxhlet extractor; the extraction solvent to be used is placed in a distillation flask; the flask is placed on the heating element; the Soxhlet extractor is placed atop the flask and a reflux condenser is placed atop the extractor.

Thirteen (13) experimental runs were conducted to find the maximum oil yield from the big castor seed where the optimum process condition was maintained. The controllable factors considered are reaction time and 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.

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

Where y_1 and y_2 are the weights of castor beans before and after the extraction.

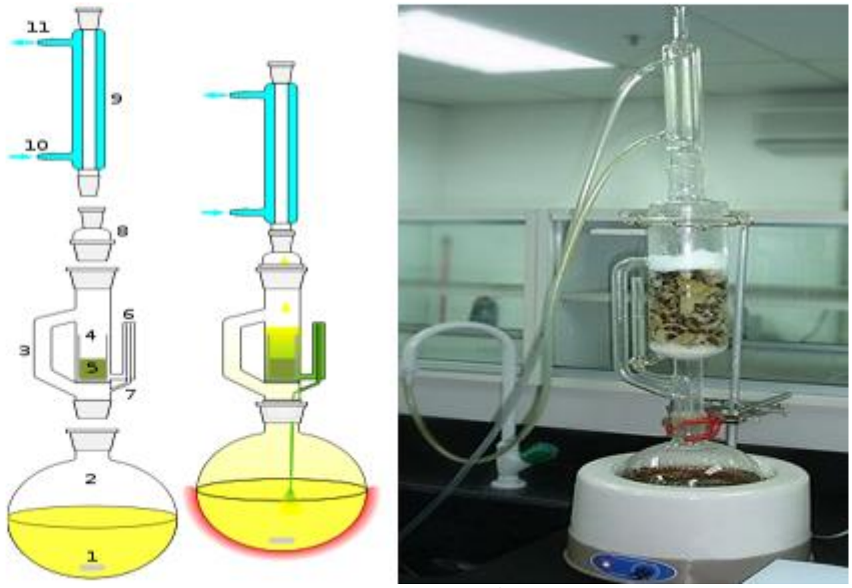


Fig 1: A schematic representation of a Soxhlet extractor

***1:** Stirrer bar **2:** Still pot (the still pot should not be overfilled and the volume of solvent in the still pot should be 3 to 4 times the volume of the soxhlet chamber) **3:** Distillation path **4:** Thimble **5:** Solid **6:** Siphon top **7:** Siphon exit **8:** Expansion adapter **9:** Condenser **10:** Cooling*

Experimental Design and Optimization: A two-factor five-level factorial Central Composite Design (CCD) under surface response methodology (RSM) with replicates at the centre point and star points was used in this study. The variables used were reaction time (x_1) and reaction temperature (x_2) each at low (-1) and high (+1) coded levels. The actual levels of the variables for CCD experiments were selected based on the initial levels as the center points. A total of 13 experimental trials that included 4 trials for factorial design, 4 trials for axial points (two for each variable) and 5 trials for replication of the central points were performed. The responses of oil yield; Y (%) is the average of triplicate representing yield for the big castor seed. Table 1 shows the CCD experimental conditions for the extraction process.

Table 1: CCD Experimental Conditions for the 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 experimental data were analyzed according to the response surface regression procedure to fit the second-order polynomial equation in which the level of significance (p-value) of all coefficients was < 0.05. Based on experimental data shown in Table 1, the regression coefficient was determined by the statistical software package Design-expert® (version 8.0.6; stat-ease, Inc., Minneapolis, USA) to predict the process response as a function of independent variables and their interactions were used to understand the system behaviour. The mathematical relationship between the process variables and response was calculated by the following quadratic polynomial expression:

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

where Y is the response, i.e. the percentage oil yield (%), X_i and X_j represent the coded independent variables, β_0 is constant, β_i is linear term coefficient, β_{ii} is the quadratic term coefficient, β_{ij} is interaction (cross-term) coefficient and 'n' is the number of process variables studied and optimized during the study. The matrix notation of the model is given in Equation 3:

$$y = X\beta + \in \quad (3)$$

$$\underbrace{\begin{bmatrix} y_1 \\ y_2 \\ \vdots \\ y_n \end{bmatrix}}_{y} = \underbrace{\begin{bmatrix} 1 & x_{11} & x_{12} & \cdots & x_{1k} \\ 1 & x_{21} & x_{22} & \cdots & x_{2k} \\ \vdots & \vdots & \vdots & & \vdots \\ 1 & x_{n1} & x_{n2} & \cdots & x_{nk} \end{bmatrix}}_{x} \underbrace{\begin{bmatrix} \beta_0 \\ \beta_1 \\ \vdots \\ \beta_k \end{bmatrix}}_{\beta} + \underbrace{\begin{bmatrix} \varepsilon_1 \\ \varepsilon_2 \\ \vdots \\ \varepsilon_n \end{bmatrix}}_{\varepsilon} \quad (4)$$

ANOVA was carried out to estimate the effects of process variables and their possible interaction effects on the maximum oil yield in the response surface regression procedure. The goodness and best fit of the model was evaluated by a regression coefficient R^2. The response surface and contour plots were obtained using the fitted quadratic polynomial equation generated from regression analysis by keeping one of the independent variables at central value (0) and varying the other two.

RESULTS AND DISCUSSION

The optimization of process conditions affecting the percentage (%) yield of oil from big seeded varieties of castor seed were carried out for the maximum extraction using central composite design (CCD) method. Thirteen (13) experiments with different combinations of controllable factors: reaction time and reaction temperature were performed. The percentage (%) yield of oil was determined and the results shown in Table 2. A detailed analysis on the interaction of reaction time and reaction temperature on the percentage (%) yield of oil from big castor seed was carried out. The 'Design-Expert (Stat-Ease, Inc., Minneapolis USA) software was used for regression and graphical analysis of the data obtained. The optimal values of the experimental conditions were achieved by solving the regression equation and also by analyzing the response surface and contour plots. Table 2 shows the coded and actual design matrix and results for the combination of the variables for extraction process. Figure 2 represent the correlation between the predicted % oil yields and the actual (experimental) % yields plot for big seeded varieties of castor seed oil. The 45° straight line illustrates a perfect fit. **Table 3**. Represents the Analysis of variance table for big castor seed percentage oil yield

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

Run	Coded values		Actual values		Yield$_{Big\ seed}$ (%)	
	X_1	X_2	Reaction time (mins) X_1	Reaction temperature (℃) X_2	Experimental values	Predicted values
1	-1.000	1.000	139.08	94.14	45.55	45.51
2	-1.414	0.000	60.00	80.00	42.45	43.47
3	0.000	0.000	330.00	80.00	52.60	52.60
4	1.000	-1.000	520.92	65.86	54.80	54.22
5	0.000	1.414	330.00	100.00	53.40	52.62
6	0.000	0.000	330.00	80.00	52.60	52.60
7	1.000	1.000	520.92	94.14	54.80	55.76
8	1.414	0.000	600.00	80.00	54.80	54.40
9	-1.000	-1.000	139.08	65.86	50.60	49.01
10	0.000	-1.414	330.00	60.00	52.60	54.01
11	0.000	0.000	330.00	80.00	52.60	52.60
12	0.000	0.000	330.00	80.00	52.60	52.60
13	0.000	0.000	330.00	80.00	52.60	52.60

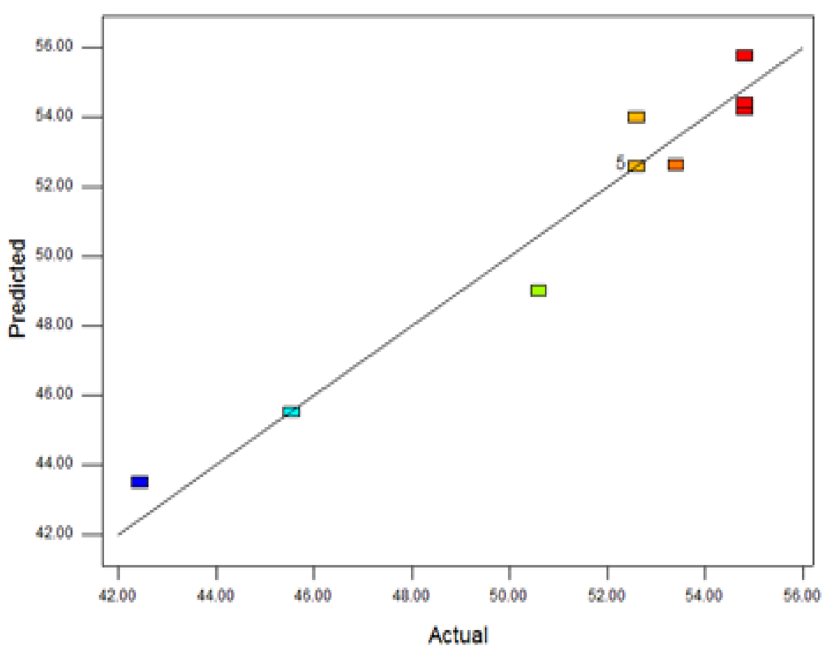


Fig 2: Predicted oil yield vs Experimental (actual) oil yield for big castor seed oil

Table 3: Analysis of Variance table for Big Castor Seed Percentage oil yield

Source	Sum of Squares	df	Mean Square	F value $\alpha = 0.05$	p-value Prob > F	
Model	153.60	5	30.72	28.40	0.0002	Significant
A–Reaction time	119.47	1	119.47	110.43	<0.0001	
B-Reaction temperature	1.92	1	1.92	1.77	0.2246	
AB	6.38	1	6.38	5.89	0.0456	
A^2	23.33	1	23.33	21.56	0.0024	
B^2	0.88	1	0.88	0.82	0.3964	
Residual	7.57	7	1.08			
Lack of fit	7.57	3	2.52			
Pure Error	0.000	4	0.000			
Cor total	161.17	12				

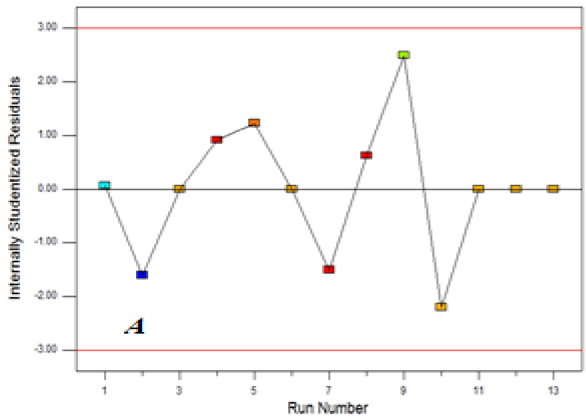
R-sq. = 95.30 % R-sq. (adjusted) = 91.95% R-sq. (predicted) = 66.59%

The Model F-value of 28.40 implies the model is significant. There is only a 0.02% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, AB, A^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. It was observed from the table that reaction temperature had no significant effect on the response variable (yield) studied. The response variable had a quadratic response (p-value 0.0055), in which both reaction time and reaction temperature showed an effect, and had a quadratic relationship. The signal to noise ratio is measured by adequacy precision, which comprised the predicted value at the design points and the average prediction error. In this present study, adequacy precision ratio was obtained to be 17.392 which indicate an adequate signal. A ratio greater than 4 is desirable for the model to be adequate. And since the ratio obtained in this study is greater than 4, the developed model can be used to guide the design space. The application of response surface methodology offers, in the basics of parameter estimate, an empirical relationship between the response variable and the test variables under consideration. Multiple regression analysis of the experimental data (using design expert software) gave the following second order polynomial equation on terms of castor oil extraction.

$$Y_{Big\ seed} = 52.60 + 3.86x_1 - 0.49x_2 + 1.26x_1x_2 - 1.83x_1^2 + 0.36x_2^2$$

Where, Y is the response variable (yield) in terms of castor oil extraction and x_1 and x_2 are the coded values of the independent variables i.e., reaction time and reaction temperature respectively. The negative sign indicates the antagonistic effects whereas the positive sign indicates the synergistic effects.

The independent and the dependent variables were fitted to the second-order model equation to predict the optimum yield of oil from the big castor seed. They were examined in terms of the goodness of fit. The goodness of fit of the regression equation Y was evaluated by the coefficient of determination (R^2) and the coefficient of relation (R). The coefficient of correlation (R) explains the correlation between the experimental and predicted values from the model. The coefficient of determination (R^2) is a measure of total variation of observed values of extracted oil about the mean explained by the fitted model. A good model equation explains most of the variations in the response. The determined coefficient of determination R^2 for big castor seed oil extraction value was 0.9530 indicating that the model explained 95.30% of variation in the original data. The respective adjusted R^2 value was 0.9195. The R^2_{pre} value determined by cross-validation suggested that the model could explain 67% of variation in predicting novel observations. The optimal reaction time and reaction temperature were found to be 520.92 mins (8.68 hrs) and 94.14ºC respectively. These values of the optimum process conditions were then fitted into the quadratic polynomial model described in equation 4 to predict the optimum value of big seeded varieties of castor seed oil yield to be 55.76%. As illustrated in Figure 2, the model described the data reasonably well and was able to correctly predict approximately 4 out of 5 observations in the original design. Since the final model has been determined for big seed as seen in equation 4, it is now useful to look at the residuals. Figure 3 (a-d) shows residuals based on the final model. Both response surface and isoresponse contour plots are parts of data presentation from response surface methodology. The isoresponse contour plots are shown in the Figure 4. The model equation, $Y_{Big,seed}$, illustrates the two dimensional relationship for the effects of reaction time and reaction temperature respectively, on big castor seed oil extraction. From the contour plot shown below, it was observed that oil yield increases when the levels of both controllable factors reaction time and reaction temperature are raised.



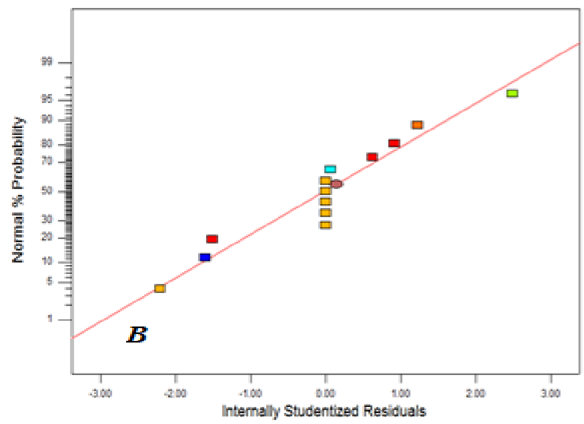
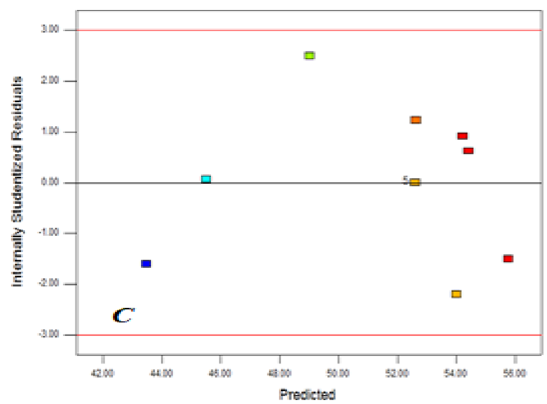


Fig. 3(a): Residuals based on run order for big castor seed oil yield **Fig. 3(b):** A normal probability plot of the raw residuals for big castor seed oil yield



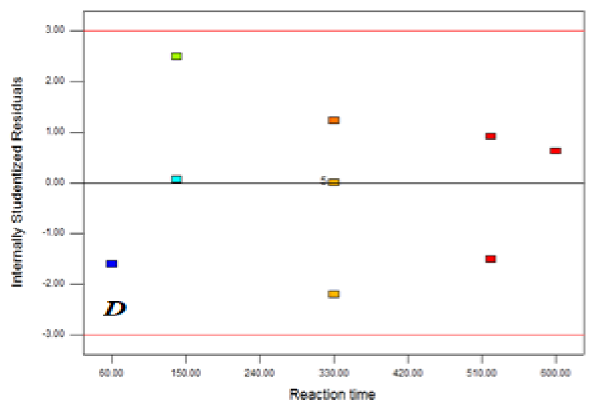


Fig. 3(c): Residuals based on predicted order for big castor seed oil yield **Fig. 3(d):** Residuals based on experimental order for big castor eed oil yield

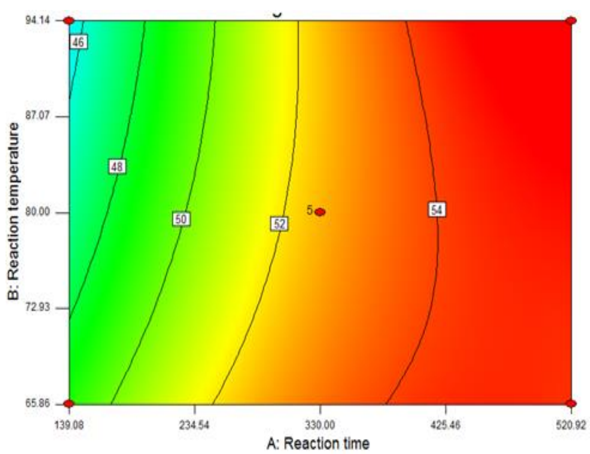


Figure 4: Isoresponse contour plots showing the effect of reaction time and reaction temperature and their interactive effect on the % yield of big castor seed oil.

The response surface plots shown in Figure 5 for the chosen model equation $Y_{Big,seed}$, illustrates the three-dimensional relationship for the effects of reaction time and reaction temperature on the % yield of castor seed oil. These constant levels are at the central levels of that variable in their respective ranges. The response surface plots indicate the respective variable ranges. The response surfaces show that % castor oil yield increases as the reaction time and reaction temperature increase to the optimum conditions. Looking at the surface response for castor oil extraction yield in Figure 5, the optimization point to get the highest oil yield performance of 55.76% was observed at a reaction time of 520.92mins (8.68hr) and a reaction temperature of 94.14℃. The findings from this study showed that the determined coefficient of determination R^2 for big castor seed oil extraction value was obtained as 0.9530 indicating that the model developed in this study explained 95.30% of variation in the original data. The respective adjusted R^2 value was 0.9195. The R^2_{pre} value determined by cross-validation suggested that the model could only explain 67% of variation in predicting novel observations. The result showed that the optimization point to get the highest oil yield performance was observed at a reaction time of 520.92mins (8.68hr) and a reaction temperature of 94.14℃. These values of the optimum process conditions were then fitted into the quadratic polynomial model to predict the optimum value of big seeded varieties of castor seed oil yield to be 55.76% compared to 48.75% reported by Shridhar *et al.* (2010), 48% oil extraction yield obtained by Abitogun *et al.* (2009) as well as 50.16% reported by Muzenda *et al.* (2012).

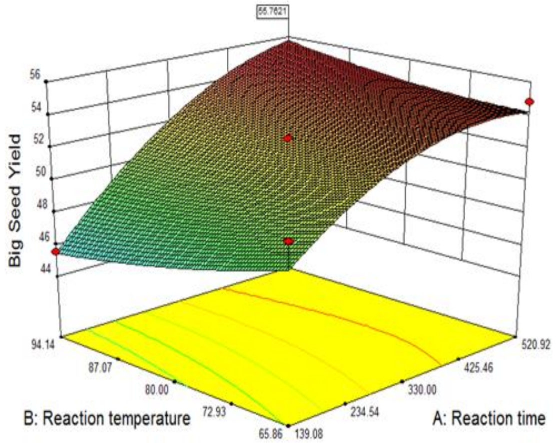


Fig 5: Response surface plots showing effect of reaction time and reaction temperature and their interactive effect on the % yield of big castor seed oil.

This value falls favourably within the standard range 30-55% specified by the American Society for Testing and Materials (ASTM). The implication of this study is that the response surface methodology adopted could effectively be employed to any process where an analysis of the optimum, effects and interaction of many experimental factors are studied.

Conclusion: This study has been able to optimized reaction time, reaction temperature and their interactive effects on the oil yield of big seeded varieties of castor-oil plant, (Ricinus communis) seed using response surface methodology (RSM). The study also discovered that the optimal conditions to get the highest oil yield performance of 55.76% in the big seeded varieties of castor-oil plant was observed at a reaction time of 520.92mins (8.68hr) and a reaction temperature of 94.14℃.

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