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STUDY OF PARAMETRIC EFFECTS OF HOT BATCH EXTRACTION PROCESS OF CASTOR BEAN OIL TOWARD SUSTAINABLE BIODIESEL PRODUCTION

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ABSTRACT

This work investigates the efficacy of hot batch extraction process on castor seeds using Soxhlet extraction apparatus with normal hexane as solvent. The seeds obtained were pretreated by decoction process whereby, both water and protein content therein were drastically reduced. The effect of the yield on duration of extraction, solvent volume and mass were carried out by adopting a central composite design of response surface methodology and based on the statistical analysis and optimization of various operating variables using a statistical Design Expert software version 6.0.8 (STAT-EASE Inc., Minneapolis, USA) A second order quadratic model was developed in order to correlate the three variables to the response. The optimum conditions for hot batch extraction process of crude castor oil were found to be 150 minutes period of time, 160 ml of solvent volume and 99.9% of mass. At the calculated optimum conditions, the yield was 80.7% at the point desirability of 99.2%. Acid value of 0.45mgKOH/g and calculated 0.23% value of free fatty acid were found to be within the standard range for biodiesel production. The mathematical model developed was found to adequately describe the compatibility with the data predicted and accurate statistical prediction of the optimum yield conditions and the quality of crude castor oil were established.

Key Words: Biodiesel, castor seeds, central composite design and hot batch separation

1.Introduction

Due to alarming socio-economic implication (feedstock competition with food) and environmental concern in some cases, biodiesel production from animal fats or vegetable oil as an alternative to fossil fuel still attracts special global attention and undeniable justification when considering the existing agricultural and industrial production capacities, social and economic growth and the interest in the exploitation of future generation feedstock such as non-edible, waste and algae oil [illegible] economically competitive, environmentally acceptable and readily available (Encina*et al.,* 2007). A critical understanding is needed for procurement of a very reliable bio-fuel such as biodiesel in terms of production technology (Khalizani and Khalisanni, 2001). The role of technology cannot be underestimated, in fact specific technologies are required to treat multiple feedstock even including low cost ones. (Aramugam, *et al.,* 2009 and Helwani*et al.,* 2009).

Demirbas, (2008), Arumugan*et al.,* (2009) and Ittipon*et al.,* (2011), considered varieties of feedstocks that can produce biodiesel, such as rape seed,soybean, canola, mustard, oil palm, sunflower, jatropha, rubber seed. Others are coconut, andiroba, algae, tobacco seed,fish, rice bran, palm kernel, groundnut,karanja, cumaru, camelina and others (Mittebach and Remschmidt, 2006).Castor oil stands out being found abundant and cheaper in order to reduce the overhead cost of biodiesel production

Any vegetable oil or animal fat can be used to [illegible] t a careful choice of feedstock should be made.Presently, biodiesel is produced mostly from both edible and non-edible vegetable oil, however, non-edible oil type of feedstock are being advocated for,(which will not interfere with food sources and production) in order to forestall food storage, hunger and famine. Any non-edible source adopted must fulfill two requirements: price (low feedstock and production cost; more than 80% of production cost correspond to the feedstock cost) and local availability (large and constant production volume) (Vito *et al.,* 2004and

Helwani*et al.,* 2009). More importantly, it is necessary to take cognizance of the oil content of the seed and the yield of the oil per hectare of land.This study is motivated by the high potential alternative fuel value of castor bean oil which also has many other uses (Akpan*et al.,* 2006).It has been discovered that castor bean can meet up with desired requirements stated above. It contain about 46-50% of oil per seed, readily and abundantly available and non-edible. Apart fromthese, it has great affinity for metal surface and higher polar (Manase, 2013).This paper is considering proper adequate and cost effective process to be adopted for biodiesel production using castor bean as feed stock from onset.

Thisis because the most detrimental properties of the feedstock adopted,that is, high viscosity, low volatility, poor atomization and auto-oxidation (*Paul et al.,* 2009) must be overcome in order to produce biodieselof higher quality and quantity. So this paper is aim at studying the optimized parameters of hot batch extraction process using normal hexane in terms of yield and quality.

The extraction of oil and other products from any seed have being a subject of interest and several new techniques and methods are being used to isolate these chemicals (Sohail, 2009) and some of these methods are mechanical expression which is no more widely used due to its low oil recovery and its turbidity effect on oil produced. However, the use of screw press is preferred by small scale processors because of its low capital cost (Liauw, 2008). Cold press oil extraction is another method which does not use organic solvent or heat to extract oil, but found to be more expensive than any other methods (John, 2006).Another type of extraction is supercritical fluid extraction, but its capital and operating costs are intensive(Johnson and Morgan,1997). Steam and high pressure method, this method makes use of high pressure extraction means to squeeze out oil from seeds but most of the active ingredients and compounds are destroyed by high temperature.Hot batch extraction processrefers to preferential dissolution of oil by contacting oil seeds with solvent, which isfound to be the most efficient techniques to recover oil from oil seeds. Extraction using solvent has several advantages such as: higher yield and less turbid oil than mechanical extraction, relative low operating cost compared to supercritical fluid extraction (Liauw, 2008; Ofoegbu*et al.,* 2006). Factors influencing the yield and quality during extraction process are the particle size, particle to solvent ratio, temperature of extraction, extraction duration and the type of solvent use (Liaw*et. al.,* 2008). However, reaction time, solvent volume and mass of castor meal were considered in this study.

2. Experimental Materials and Methods

2.1 Materials

Castor seeds were obtained from Masifa village, near Ogbomosocity; they were carefully sorted and sun dried to reduce the moisture content. Methanol (CH_3OH),analytical grade, manufactured by manufactured by BDH Laboratory Supplies, Poole BH 15 TD England. Laboratory Oven**:** SM 9053A Laboratory oven Surgifield Medical England**.** Soxhlet apparatus with accessories, available in the department of Chemical engineering, Lautech, Ogbomoso, Nigeria.

2.2 Pretreatment and extraction

In order to reduce the protein content in the seeds, the sun dried seeds obtained were cleaned, decorticated, and cooked at 80^oC under air tight conditions for twenty (20) minutes using water bath, then oven dry at 100^oC to further reduce the water content. The processed seeds were then grinded into meal form using dry mortar and pestle and extracted using hot batch extraction process.

2.3 Acid value and free fatty acid determination

The determination of acid value was carried out by titrimetric technique (AOACS, 2001) method using a relation:

$$\text{Acid value} = \frac{56.11 \times V \times N}{M} \qquad (1)$$

Where: V= Volume of KOH used in ml, N is concentration in normality and M is mass of oil sample in grams.

The constant 56.1 was derived from the molar mass of Potassium Hydroxide to obtain the respective acid in % by weight.

A standard solution of 0.1MKOH was prepared according to the method of Anya *et al.,* (2012). Then % free fatty acid was determined by:

$$\% \text{ FFA} = 0.50 * \text{acid value} \qquad (2)$$

2.4 Model development

The response (oil yield) of the extraction was used to develop a mathematical model that correlates the yield of crude castor oil to the extraction process variables studied through the first, second order and interaction term according to the following second order polynomial equation:

$$\sqrt{Y} = a_0 + \sum_{j=1}^{3} a_j x_j + \sum_{ij}^{3} a_{ij=} \; x_i x_j + \sum_{j=1}^{3} b_{jj} x_j^2 \quad (3)$$

Where Y is the square root yield of crude castor oil, x_i and x_j are the independent variables, a_o is the offset term, a_j is the linear effect, $a_{ij}$is the first order interaction effect and $b_{jj}$is the square effect.

2.4.1 Experimental design

The selected experiment design for this study is Central Composite Design (CCD) with five level three factor design (Bello and Ahmad, 2011), which addressed the reaction time, solvent volume and mass of castor meal used, while the measured response is the yield of the crude castor oil (CCO) the experiment was performance under atmospheric pressure condition.The range and levels of the three independent factors studied is shown in the table 1 while the levels of factors investigated are considered due to the operating units of the experimental apparatus.

2.4.2 Experimental design of Hot Batch Extraction Process

The optimization of reaction parameters of hot batch extraction process was done by selecting central composite design (CCD) with five-level three–factor design (table 1) that addressed reaction time, (A) solvent volume (B) and mass (C).

Table 1: Central Composite Design Process Variables for hot batch extraction process.

	Level				
Independent variables	-η (-2)	-1	0	+1	+η (+2)
Time (mins.)	30	60	90	120	150
Solvent volume (ml)	160	170	180	190	200
Mass (g)	20	40	60	80	100

Transformation of variable levels from coded (X) to un-coded could obtained as: X_1= 90+30X,X_2= 180+10X, X_3= 60+20X,

3. Results and Discussion

3.1 Chemical Analysis

By applying different formulae as indicated in the experimental procedure, acid value and the free fatty acid content were determined to be0.45mgKOH/g and 0.23% respectively, in whichacid value obtained was found to be within the range of 0.4-4.0 mgKOH/g range of ASTM specification for quality castor oil reported by Akpan*et al.,* (2006) and Manase, (2013). Table 2 gives detailed experimental parameters and the results of both experimental and predicted values on the basis of the CCD experimental design. The analysis of all the designed experiments was done through multiple regressions. The oil yield from (experimental value) ranged from 1.50 to 15.5% with the design points number 16 and 6 giving the minimum and the maximum conversion to crude castor oil respectively. The minimum conversion was obtained at 30minutes time of reaction, 160ml of solvent volume and 20g mass, whereas, the maximum yield was obtained at 2hrs 30mins time, 160 solvent volume and 100g mass. 0.23% of free fatty acid was also found to be less than free fatty acid of 2% achieved by Anya *et al.,* (2012)

Table 2: Experimental design matrix and results of hot batch extraction

Standard	Time (min.)	Solvent vol. (ml)	Mass (g)	Predicted value	Experimental value
7	30.0	200.0	100.0	3.8	1.9
8	150.0	200.0	100.0	12.9	3.6
1	90.0	180.0	60.0	6.0	1.2
10	210.0	180.0	60.0	12.6	3.6
19	90.0	180.0	60.0	5.7	2.4
5	30.0	160.0	100.0	3.8	1.9
12	90.0	220.0	60.0	6.3	2.5
2	150.0	160.0	20.0	3.1	1.8
18	90.0	180.0	60.0	5.7	2.5
4	150.0	200.0	20.0	3.0	1.8
11	90.0	140.0	60.0	5.8	2.4
14	90.0	180.0	140.0	8.0	2.8
15	90.0	180.0	60.0	5.7	2.4
16	30.0	160.0	20.0	1.5	2.4
20	90.0	180.0	60.0	5.7	2.4
17	90.0	180.0	60.0	5.7	2.4
3	30.0	200.0	20.0	2.3	1.5
6	150.0	160.0	100.0	15.5	3.9

A quadratic polynomial equation which correlates the response (oil yield) to the hot batch extraction insignificant term was obtained, that is

3.2 Final Equation in Terms of Coded Factors:

$$\sqrt{Y} = +2.40 + 0.55 * A + 7.573E\text{-}003 * B + 0.65 * C + 0.014 * A^2 + 0.015 * B^2 - 0.22 * C^2 - 0.083 * A * B + 0.36 * A * C - 0.076 * B * C \quad (4)$$

Note that square root transformation of the yield was selected by the software as it was found to give a better fitting. A positive sign in front of the terms indicates synergistic effect, while a negative sign shows antagonistic effects on final yield.

3.3 Analysis and Adequacy of the Empirical Model

Table 3: Results of regression coefficients analysis and significant of response surface quadratic model

Factor	Coefficient Estimate	Degree of Freedom	Standard Error	95% CI Low	95% CI High	*P*-value
Intercept	2.4	1	1.0×10^{-2}	2.3	2.4	< 0.0001
A	0.6	1	1.0×10^{-2}	0.5	0.6	< 0.0001
B	7.6×10^{-3}	1	8.1×10^{-3}	-0.01	0.03	0.3771
C	0.7	1	1.0×10^{-2}	0.6	0.7	*< 0.0001*
A^2	1.0×10^{-2}	1	9.4×10^{-3}	-8.2×10^{-3}	0.04	0.1897
B^2	0.02	1	6.5×10^{-3}	1.6×10^{-4}	0.03	0.0482
C^2	-0.22	1	9.4×10^{-3}	-0.24	-0.20	*< 0.0001*
AB	-0.08	1	0.01	-0.11	-0.06	*< 0.0001*
AC		1	0.01	0.33	0.39	*< 0.0001*
BC	-0.08	1	0.01	-0.10	-0.05	0.0002

Statistical analysis of empirical model was done to determine the analysis of variance (ANOVA) and to check the adequacy of the model. In the study, 95% level of confidence was desired (Table 3). The coefficients of the response surface empirical model were also evaluated as given in equation one. The significance of each of the coefficients of the model equation was check using probability of error value (p-value) which indicate the interaction strength of each parameter. The results of regression coefficients analysis and significant of response surface quadratic model were also evaluated. The significance of each parameter can be evaluated from its p-value. A p-value less than 0.05 shows that each of the model term is significant in nature. These results correlates to the work of Ittipon*et al.,* (2011) while studying the ultrasonic irradiation assisted synthesis of biodiesel from crude palm oil using response surface methodology. It could be observed from the p-value calculate for each model term that there are two linear coefficients of A and C, two quadratic coefficients of B^2 and C^2, and three cross – product coefficient of AB, AC and BC were significant at the $p < 0.0001$, whereas other coefficients of the model which did not indicate significant effects for the extraction of crude castor oil, were not considered in the final model equations.

Table 4: Analysis of Variance for response surfase quadratic model

Source	Sum of Squares	Degrees of Freedom	Mean squares	*F*-value	*P*-value
Model	8.7	9.0	9.7×10^{-1}	922.8	< 0.0001
Residual	8.4×10^{-3}	8.0	1.1×10^{-3}		
Lack of Fit	5.2×10^{-3}	3.0	1.8×10^{-3}	2.7	1.6×10^{-1}
Pure Error	3.2×10^{-3}	5.0	6.4×10^{-3}		
Cor Total	8.7	17.0			
C.V. = 1.35%	R^2= 0.999	Adj. R^2=0.998	Pred R^2= 0.991		

With reference to table 4, the p-value of the model was less than 0.000l, which demonstrates high significance in predicting the response value and the suitability of the deduced model, that is, at the 95% confidence interval and p-value < 0.05, the model term is significant. The model F-value of 922.75 shows that the model is significant, this shows that only a 0.01% chance that a model "F-value" this large would occur due to noise.

A lack of fit which is the weighted sum of the squared deviations between the mean response at each parameter level and the corresponding fitted value. The p-value of the lack of fit was 0.1562 (p>0.01) which indicated that there was no significance relative to a pure error. F-value of 2.70 implies 15.62% chance that a lack of fit this large could occur due to noise when the model in fitted to the observed experimental data. For the reliability of the model, low value of the coefficient of variation (CV = 1.35%) show that the model is reliable.

Coefficient of correlation could be used to determine the quality of the empirical model developed. The coefficient of determination (R^2) being 0.999 for the response implies that 99.9% of the experimental data confirmed the compatibility with the data predicted by the model for a good statistical model, it must be very close to 1. The adjusted coefficient of determination (Adj. R^2) of 0.998 which reconstructs the expression with all the significant terms included, confirmed the high significance of the model, because $R^2 = 0.999$ and Adj. R^2 of 0.998 indicates a high correlation between the experimentally observed and predicted values and explains any variability in the response, this corresponds to R^2 of 0.989 reported by Yee and Lee, (2008)and Ittipon*et al.,* (2011) which was found to be very close to unity.

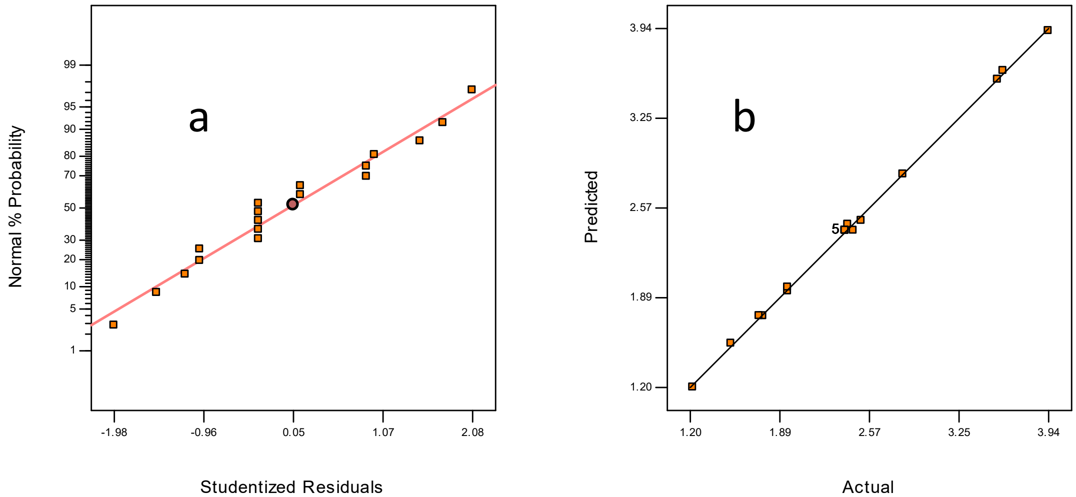

Figure 1: Normal Plot of Residuals

Figure 1a shows the normal probability plot of residuals and studentized residual, this is in line with the work of Ittipon*et al.,* (2011) which shows a dispersion character of constant variables in the data. Figure 1b shows the actual values obtained from the experiment against the predicted values using the developed model equation. The figure shows the line perfect fit with points corresponding to zero error between predicted values and actual values, it also visualizes the performance of the correlation in a very clear manner and demonstrated that the regression of the experimental data, at which all plotted points are very close to the line of best fit which correlate to the work of Yee & Lee, (2008), when checking the model adequacy of palm oil as feedstocks for biodiesel production via heterogeneous transesterification while carrying out the optimization studies. Summarily, it could be said that, the result gives a successful capturing of the correlation between the three variables of hot batch extraction process to the yield of crude castor oil.

3.4 Effect of hot batch extraction variables

Base on analysis of variance, the extraction process was significantly affected by various interactions between the process variables. Those individual variables that significantly affect the reaction are process duration (time) and the mass. The result gave the advantages of using design of experiments in capturing the interaction between the variables that affect the extraction reaction (Bello & Ahmad, 2011).

3.4.1 Effect of Individual Process Variables

Figure 2a shows the effect of time (30-150mins) on the yield of crude oil at actual solvent volume of 180ml and mass of 60g. It was found that with increasing in time of reaction, the square root yield increased. The yield of crude castor oil increased significantly from 1.90% at 30mins to 2.98% at 150mins. The increase in the yield of crude castor oil is due to enough time of contact of castor meal with solvent which in turns allow higher yield. This is also affirmed from 0.52 and 0.57% confidence interval of the time and mass respectively (Table 2) and p-value <0.001.

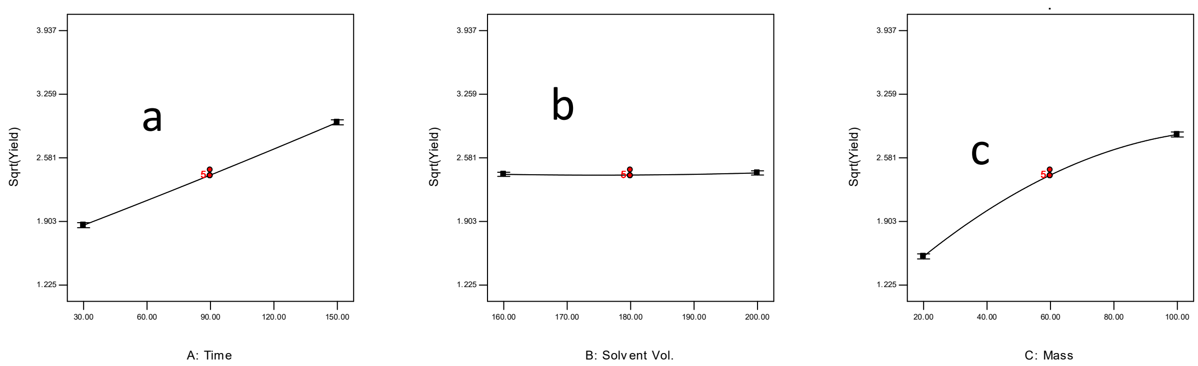

Figure 2(a-c) Individual Process Variables

The effect of solvent volume on square root of yield is shown in figure 2b at actual time of 90mins and mass of 90g. The effect was not felt much because enough quantity of solvent was used, with enough time of reaction. This shows that solvent quantity does not have effect on the yield of crude castor oil, because, it is the vapourised liquid that extractsLiauw*et al.,* (2008). It was also confirmed from table 3 whereby p-value of 0.3771 shows the insignificance of solvent volume variable.

At actual time of 90 minutes and solvent volume of 180ml (figure 3c), effect of mass (20-100g) on the square root of yield of crude castor oil has a great effect. It was shown that with increasing in quantity of mass of the meal, the square root yield increased. The yield of oil increases significantly from 1.51% at 20g meal to 2.86% at 100g. This shows that mass quantity has a great effect on the yield, which is also confirmed from table p-value<0.0001 and 67% at 95% high confidence interval. It could be deduced from the figure that there are increase in yield to certain level, and then constant position was about to set in, this is because the solvent had extracted all the oil quantity it had contact with.

3.4.2 Effect of Interaction between Process Variables

Figure 3a shows the interaction between reaction time and solvent volume on the yield. It shows that, in as much as there is enough time for the reaction and solvent quantity, there is steady increase in the yield of crude castor oil from 1.78% to 3.12% for 160ml of solvent at 30minutes and 150minutes respectively, and for 200ml of solvent, there is increase from 1.96 to 3.00% at 30mins and 150mins respectively.Figure 3b shows the interaction between reaction time with mass on oil yield of castor seed. Generally increase in contact time was found to increase the oil yield at actual factor of solvent volume 180ml.

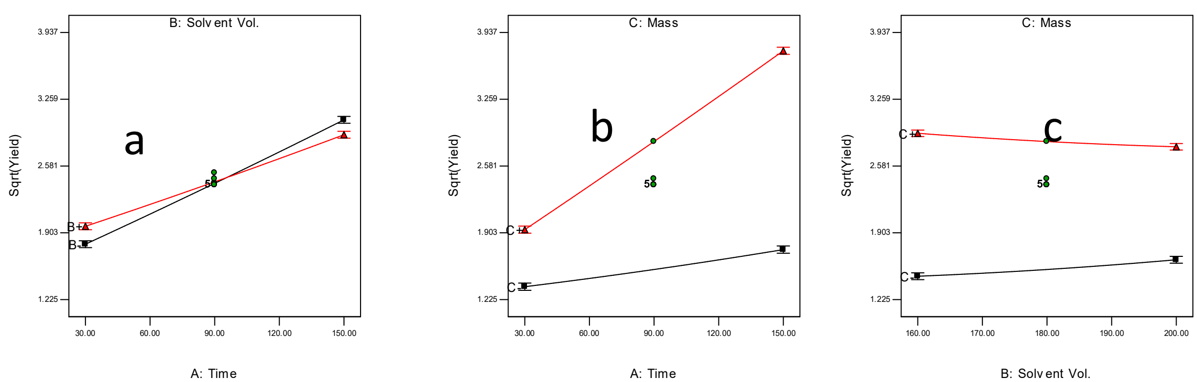

Figure 3(a-c): Interaction between Process Variables

The increase could be seen on both 20g and 100g of mass but the effect was felt so much on 100g, because the meal of castor of 100g contain more oil than that of 20g, therefore more oil was being extracted from 100g mass.Figure 3c shows that there is no reasonable interaction between the mass and solvent volume at 90minutes of actual factor. For 20g of mass; at 160ml and at 200ml of solvent the yield is 1.48% and 1.68% respectively. This shows that in as much as there are enough contact time and sufficient solvent, there is steady increase in square root of yield. But the reverse in the case of 100g mass, this is because all the oil therein had being totally expressed.

3.5 Optimization of Process Variables

The optimal values of the selected variables were obtained by solving the regression equation (equation 2) using the software design expert 6.0.8 version, having confirmed the effect of the three variables involved in the batch extraction process using the point prediction function in design expert, it was predicted that at the following conditions; 150mins of reaction period, 160ml of solvent and mass of 99.92g, an optimum oil yield can be obtained (Table 5).

Table 5:Verification experimental data at optimum conditions

	Optimum condition		Oil Yield (%)	
Time	Solvent Volume	Mass	Experimental	Model Predicted
150.0	160.0	99.9	3.9	0.9

3.5.1Validation experimental data at optimum conditions

The optimum parameter values for oil extract; time of extraction, solvent volume and mass of particles were calculated. The optimization was done to obtain maximum oil extract as much as possible. A solution with maximum desirability was selected **(**table 5**)**.Time of 150min, solvent volume of 160 ml and mass of 99.92g were selected and substituted into equation 2 as optimum parameters to obtain optimum oil yield of 80.73%

Final Equation in Terms of Coded Factors:

$$\sqrt{Y} = +2.40 + 0.55 * A + 7.573E\text{-}003 * B + 0.65 * C + 0.014 * A^2 + 0.015 * B^2 - 0.22 * C^2 - 0.083 * A * B + 0.36 * A * C - 0.076 * B * C = 0 \quad (2a)$$

Final Equation in Terms of Actual Factors:

$$\sqrt{Y} = +2.40 + 0.55 * 150 + 7.573E\text{-}003 * 160 + 0.65 * 99.92 + 0.014 * 150^2 + 0.015 * 160^2 - 0.22 * 99.92^2 - 0.083 * 150 * 160 + 0.36 * 150 * 99.92 - 0.076 * 160 * 99.92 = 0$$

$$= \sqrt{6516.6064} = 80.73\% \quad (2b)$$

4.0 Conclusions

The central composite design of response surface methodology was succesfully applied to the model developed which optimized the conditions for the hot batch exraction process reaction variables to obtain crude castor oil. The results obtained indicated that duration of extraction process had significant effects on the yield of crude oil obtained by increasing the oil yield at increase in contact time. The determination coefficient (R^2) value of 0.999 confirmed the fitness of a second order model and low value of coefficient of variation of 1.35% affirmed the reliability of the model to hot batch extraction process.

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