THE RELATIONSHIP OF EXTRACTION PARAMETERS OPTIMIZATION WITH MASS BALANCE IN MAXIMIZING PRODUCTION RATE OF SPENT BLEACHING EARTH OIL

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ABSTRACT: *Spent bleaching earth (SBE) is classified as an industrial waste generated by almost all crude palm oil refinery around the globe. It contains oil content in the range of 20-40% by weight, which is being extracted out by a solvent extraction process plant to cater for biodiesel production. Previous studies were merely focusing on the optimization of various plant and seeds extraction parameters based on a laboratory scale. Hence, a study on the optimization of the solvent extraction parameters is the key element for an enhanced SBE oil extraction process plant management. This study shall pioneer in establishing the relationship of mass balance with the optimized extraction parameters to enable plant managers to monitor the inputs and outputs of each plant equipment to operate the plant more efficiently. In this study, the extraction parameters, namely, settling rate, n-hexane temperature, and slurry concentration were optimized by response surface method (RSM) using Box-Behnken Design (BBD) to produce the maximum extraction oil rate. The independent variables, settling rate (13ml, 11ml, and 8ml), n-hexane temperature (45°C, 50°C, and 55°C), and slurry concentration (30%, 35%, and 40%) were selected for optimization by coding at three-factorial levels and their values were selected based on the extraction plant current operating condition and limitation. The BBD consisted of 17 experimental actual production plant runs with 3 hours of continuous process control with a steady-state operation for each run. A second-order polynomial model was used for predicting the response outcomes. ANOVA of the conducted experimental runs concluded that 96.9% of the variation was explained by the models. The optimized extraction parameters were 13ml, 55°C, and 30%, for settling rate, n-hexane temperature, and slurry concentration, respectively. Under the optimized extraction parameters, the values of the corresponding response, extraction oil rate were 2.08 tonne per hour. Mass balance computation was established subsequently based on the studied extraction process plant's process flow diagram (PFD) of various equipment's input and output. The computation which was performed via SuperPro Designer software was able to calculate the extracted oil rate within the 95% statistical confidence level generated by the RSM.*

Keywords: Spent bleaching earth, Optimization, RSM, Box-Behnken Design, ANOVA, mass balance, SuperPro Designer

1. INTRODUCTION

Today's globalized and competitive market demands not only to force a manufacturing plant management to react reactively on accommodating the challenges but also to formulate a concrete result-driven action plan to achieve excellence in their business[1].

The need to reduce the cost of operations and the increase in global competition amid tough economic climates, particularly during and post the COVID-19 pandemic's adverse effects, are the ultimate drivers for most of the manufacturing business owners to eliminate non-value adding operations, reinvent the process control and increase production volume and yield. Hence, business owners who enduring themselves in fierce and stiff competition are zooming into operational excellence to enable them to stand firm on their profit ground with broader market capitalization. By definition, operational excellence is the state of any business organization that the organization achieves through the development of technology and innovation in the service and product development as well as their distributions [2].

Operational excellence becomes a major concern for the manufacturing sector around the world to improve product yield, increasing production rate, and reducing production costs. While operational excellence predominantly deals with production process optimization, market and customer orientation, and productivity efficiency, process plant respective manufacturing plants can sustain and compete efficiently in their respective business field [3].

Malaysia, being one of the main hubs for various manufacturing plants, is not excluded from the radar of the synergy of operational excellence. In particular, the solvent extraction plant of spent bleaching earth (SBE) oil, which serves as the environmental friendly waste disposer for hundreds of palm oil refineries in Malaysia, is in great desire to improve productivity while efficiently control their production cost to ensure business continuity is sustained for years to come. SBE is an industrial waste generated during the refining process of crude palm oil (CPO). CPO refineries, which have a bleaching process as part of its mandatory process flow, utilize bleaching earth to adsorb heavy metals, impurities, phosphatides, and chlorophyll [4, 5].

The bleaching earth is dosed in the range of 9 – 15kg per metric CPO and contains 18-40% of oil by weight [6]. The bleaching post bleaching process is known as SBE which contained residual oil which a valuable feedstock material for HVO production [7]. Solvent extraction found to be the most efficient process to recover the oil residue in SBE. Higher yield and better quality of oil are the main justification for niche industry players to build a solvent extraction plant to extract thousands of tons of SBE in Malaysia and Indonesia [8].

Almost all the solvent extraction plants are consuming *n*-hexane as the solvent to extract out the adsorbed oil content in the SBE [9]. *n*-hexane is relatively expensive; hence, it requires an optimized process control for maximum recovery of extracted oil to ensure the production cost of the solvent extraction plant is well-controlled based on the budgetary figures. However, *n*-hexane consumption is not the only extraction parameter that contributes to the maximization of the extracted oil production.

Various independent variables that significantly contribute to the production rate needs to be studied to operate the plant most efficiently [10].

Optimization of extraction parameters in the SBE solvent extraction plant's process management is one of the most challenging tasks for engineers and management. The reason behind, multiple independent factors such as raw material quality, solvent recovery volume, and solvent temperature shall contribute significantly to the efficiency of the plant's performance. The efficiency of an extraction plant measured by dependent factors such as extracted oil production throughput and n-hexane consumptions.

Generally, process engineers who need to optimize the plant's operations on daily basis found it very complicated to operate the plant efficiently since the very least amount of attention is given to study variables present on the extraction parameters which determine the production and efficiency of the extraction plant. It has been a norm whereby engineers operate the plant only based on their feeling and non-data orientated assumptions, which was less optimized.

Most of the studies were conducted mainly on extraction optimization of various parameters of plants and seeds. Those studies were done on a lab-scale approach rather than on industrial plants [11]. Besides, none of study done on the optimization of SBE extraction parameters on an industrial scale to maximize the extracted oil production rate.

Referring to the optimization of the extraction parameter, a study has conducted an optimization study on the extraction parameter of spent bleaching clay (SBC). However, this study was focusing on SBC which used as an adsorbent in used lubrication oil (ULO) refining. Moreover, the study is conducted on a lab-scale with a different solvent than *n-hexane*, which is methyl ethyl ketone [12].

On the other hand, an industrial scale optimization study was conducted on the optimization extraction process of sesame seed's oil and centralized on two extraction parameters, namely heating temperature and moisture contents to minimize remained oil content in the pressed cake [13]. However, despite the study was conducted with *n*-hexane as the solvent, the type of raw material and optimization extraction parameters are irrelated to the SBE extraction process.

In 1937, an article was published on the general process flow of an SBE extraction plant which gives an overview of how oil is extracted out from SBE with a systematic way to recover the n-hexane consumed during extraction [14]. While this study focused mainly on a very basic process flow, very least information could be gathered on the optimization of various extraction parameters involved.

Undoubtedly, several studies were carried out mainly on experimenting with different types of solvents to maximize oil extraction from SBE. Various alcohols and hydrocarbons as solvents to extract residual oil in SBC were conducted to determine the best solvent for the highest oil yield [15]. In that study, isopropanol (IPA) was concluded as the best solvent based on lab-scale testing. However, the feasibility of IPA as a solvent for the industrial scale is yet to be studied and proven in any study to date. Besides studying the optimization parameters, the establishment of mass balance for the whole extraction process is considered the desired step to ensure quality control done on various parameters is reflected in the actual operations of the plant [16]. Besides, the mass balance shall help engineers to monitor closely the efficiency of each piece of equipment involved in the extraction plant [17]. On the other hand, a check and balance on the process control shall be well-established with the aid of mass balance. Subsequently, any losses on extracted oil production at any process flow stages shall be promptly detected [18] To date, no study was done on mass balance for an SBE extraction plant.

Therefore, a detailed and comprehensive study on optimization of SBE extraction parameters to maximize the extracted oil production shall be a novelty study to assist extraction plant management to manage and operate their respective plant in a most efficient manner.

The present study is aimed to identify the critical extraction process control parameters which contribute to the maximum production rate of SBE Oil. Those parameters shall be grouped as independent variables for optimization using various laboratory testing methods. Optimization of the process control parameters (independent variables) to maximize the yield of the extracted oil production was conducted using Box-Behnken design (BBD). Furthermore, the development of a comprehensive mass balance for the entire process flow of the SBE Oil extraction plant was established using SuperPro Designer software.

2. EXPERIMENTAL DETAILS

The raw material used for this study is spent bleaching earth (SBE) which is sourced from various refineries from West Malaysia. SBE, which presents in solid similar to typical cement physical appearance, is transported by trucks from the refinery into the extraction plant. A shovel is used to unload the SBE from trucks into a shed, whereby segregation, stacking, and storing activities are done. As for this study, segregation of 36mt of SBE according to the pre-assigned settling rate is done before feeding into the SBE feeding conveyor. The 36mt is based on the feeding rate of 12mt per hour which multiplied into 3 hours for the experiment runs. A tolerance of 0.5mt is given in anticipation of SBE losses during the feeding such as spillages, silo overflows, and impurities separations by the mesh and magnets.

Only one kind of solvent is used for the extraction process studied, which is the *n*-hexane. Based on the material safety data sheet (MSDS) obtained from the bulk supplier, the n-hexane consumed for the whole extraction process has 100% concentrations with combination isomers (concentrations) of cyclohexane (1.0-3.0%, hexane of mixed isomers (30-55%) and n-hexane (44-70%). It has a density of 680kg/m^3 at a temperature of 15°C with a boiling point of 65°C at atmospheric pressure [19]. This solvent is stored in a storage tank with a capacity of 25,000 liters and equipped with safety equipment such as a flame arrester and electrostatic earthing.

Laboratory analysis is being carried out on the quantification of the three optimization parameters studied. Firstly, to quantify the settling rate (SR) parameter, a 100 ml measuring cylinder was used with the *n*-hexane similar to the production's consumptions. The level of the clear miscellany layer was quantified based on the level reading of the measuring cylinder in milliliter (ml). Secondly, as for *n*-hexane temperature, reading is taken directly from a bimetal temperature gauge which is mounted on the solvent pipeline outlet from the plate heat exchanger and inlet to the extractor. The temperature gauge which has a nominal dial size of 80mm with scale spacing of 1°C and an error limit of ±1°C, is manufactured by WIKA Alexander Wiegand SE & Co. KG, Germany in accordance with EN 13190 standard with Ingress Protection (IP) 43 as per IEC/EN 60529 standard. As the temperature gauge is newly purchased and installed for this study, pre-factory calibration was done by the manufacturer itself. Thirdly, for the determination of slurry concentration, laboratory apparatus such as Buchner filter funnel, 500ml vacuum filter flask, and a vacuum pump is being used. Besides Whatman filter paper of No.1 was being used as the filtration aid.

The design of the experiment (DOE) for this study was to be initiated with the selection of the most suitable design model of response surface method (RSM), which is to be either central composite designs (CCD) or Box Behnken Design (BBD) [20, 21]. By taking into considerations that all the three extraction parameters to be optimized were to be studied based on three different quantitative values, hence, the design model must be a three-level design to enable Design-Expert software to compute the optimized value of those parameters studied statistically for the maximized output [22]. Thus, Box Behnken Design (BBD) was selected as the design model.

The Box-Behnken experimental design model was used to study the relationship between the response functions which are the extracted oil rate and extraction yield and extraction parameter variables which are settling rate, *n*-hexane temperature, and slurry concentration. For this study, a design of five center points per block was constructed which resulting in 17 runs. The number of BBD runs that are required for each factor can be calculated based on the formula of N=2k(k-1) + cp, where k is the number of factors, and (cp) is the number of central points [23], [24].

With 3 factors, k studied for this study, hence the number of BBD runs shall be 17 with 5 central points. For the three-level three factorial Box-Behnken experimental design, a polynomial equation was referred to the equation (1) as below: -

$$y = \beta_o + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_{23} \quad (1)$$

Where, y is the predicted response, β_o, model constant, x_1, x_2 and, x_3 independent variables, β_1, β_2, and β_3 are linear coefficient, β_{11}, β_{22}, and β_{33} are cross-product coefficients and β_{12}, β_{13}, and β_{23} are quadratic coefficients [25, 26].

With the assistance of Design-Expert software, analysis of variance (ANOVA) shall be utilized to analyze further the collected response data from the experiment runs to identify if the studied extraction parameters contribute significantly to the whole optimization process [27].

The RSM experiment setup was carried out according to the design of an experiment developed by the Design Expert (V 12.0.8.0). The coding scheme was used to represent the level of each factor [28]. The extraction parameters were assigned as X_1 for SR, X_2 for n-hexane temperature, and X_3 for slurry concentration. Binary numbers were assigned accordingly for the level range, whereby -1 assigned for low level, 0 for medium level, and +1 for high level [29]. Design Expert (V 12.0.8.0) assigned 17 sets of runs for the BBD model based on 5 center points and response, extracted oil (tonne per hour) were recorded as the mean of three hours of a continuous process in a steady-state condition. The range of the independent variables with coded level range experimental design was shown in Tables 1 and 2, respectively.

Table 1: Extraction parameters and coded level for SBE oil extraction process

Independent Variable	**Symbol**	**Coded Variable Level**		
		-1	**0**	**+1**
Settling rate (ml)	X_1	8	11	13
n-hexane temperature (°C)	X_2	45	50	55
Slurry concentration (%)	X_3	30	35	40

Table 2: Box-Behnken Design for SBE oil extraction process optimization

Runs Number	**Independent Variable Coded**		
	X_1	**X_2**	**X_3**
1	+1	+1	0
2	+1	0	-1
3	-1	0	0
4	0	0	0
5	0	0	0
6	0	0	0
7	-1	0	0
8	+1	-1	0
9	+1	0	+1
10	0	+1	-1
11	0	0	0
12	0	-1	-1
13	-1	0	+1
14	0	0	0
15	0	+1	+1
16	-1	+1	0
17	+1	-1	+1

One of the ultimate objectives of this study is the volume of the extracted oil. Extracted oil rate was directly measured by a mass-flow meter which was installed on the outlet pipeline which transports the oil from drier to a storage tank. The mass flow meter was manufactured by Rota Yokogawa, Germany with model RCCT36-AN0M02A1SL/KF5/BG/P3/P6/HP and serial number D1R901172. It was being calibrated within a working range of 0 kg/hr to 3,000 kg/hr on 31st October 2019 by UCAL TECHS, Johor Bahru with certification number UJB/IS-MM/181001783, which is valid at the time of writing. The calibration report revealed that the flowmeter was within working condition with ±5kg/hr of error.

ANOVA for this study was obtained from the Design Expert (V 12.0.8.0) software with being displayed under the response analysis tab.

had its p-value of more than 0.05 were classified as not significant. Model F-value implied if the model is significant with a tolerance of less than 0.05% of change that an F-value

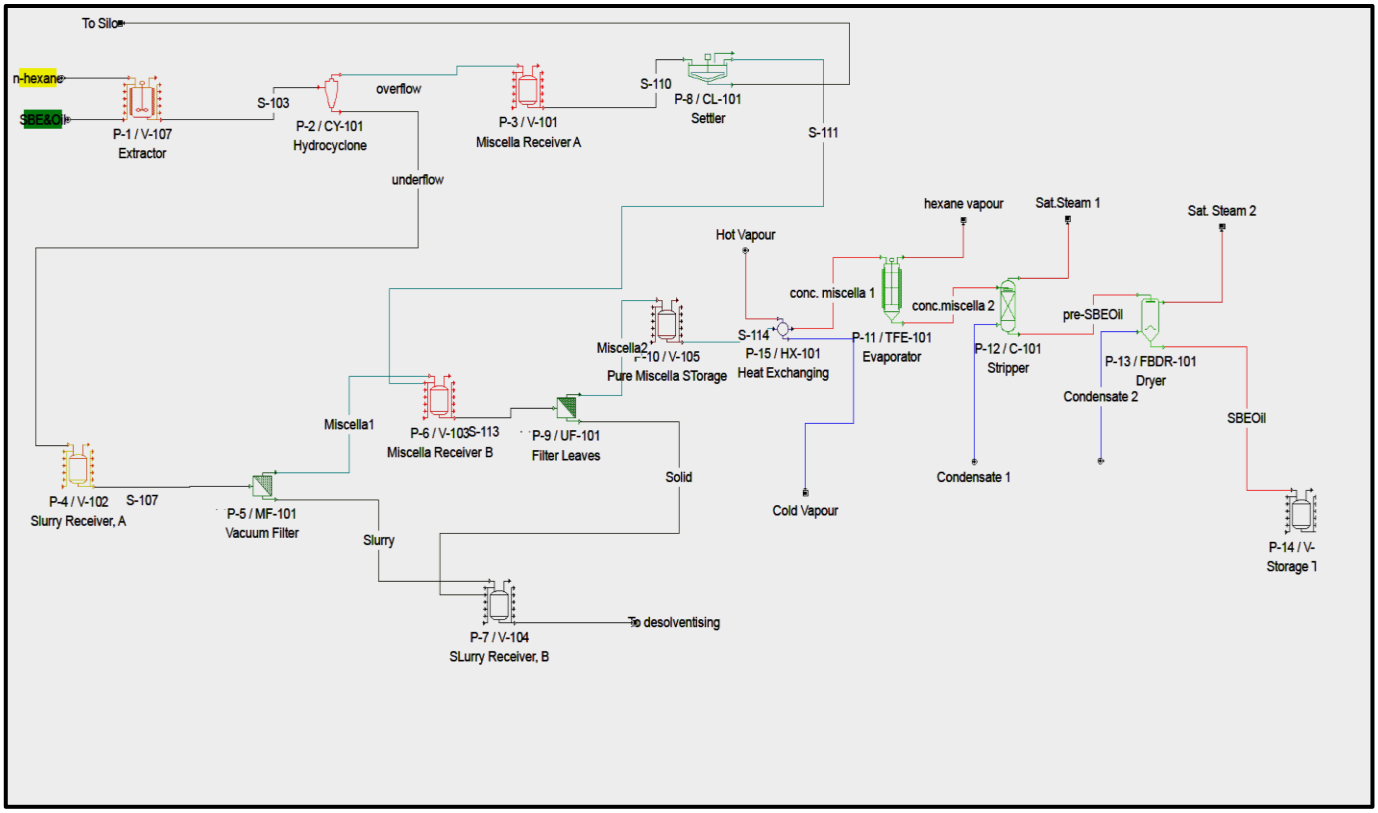

Figure (1) Process Flow Diagram (PFD) of SBE oil extraction process plant

Firstly, transform was regarded as inapplicable for this study, and the transformation of none was selected. Secondly, a fit summary for each response was analyzed. On the fit summary analysis, the most suitable model for each response was being suggested based on the highest values of adjusted R^2 and predicted R^2 and the smallest value of predicted residue of sum, PRESS. For this study, the quadratic model was predicted as the suggested model [30].

Besides, referring to the sequential model sum of squares, a design model was further evaluated by referring to the p-value and F-value, whereby comparison between available model was done and model with the lowest p-value was compared and suggested [31].

Thirdly, a lack of fit tests was analyzed to select a model that has the lowest value of the sum of squares, mean square, and F-value while restricting the p-value to less than 0.05.

Finally, the model summary statistic was referred to as the final confirmation of the model suggested based on the maximization of adjusted R^2 and predicted R^2 [32]. Following up on the fit summary and model selection, ANOVA for the suggested model was conducted in detail. Each response was analyzed statistically by ANOVA based on coefficients, the sum of the square, degree of freedom, mean square, F-value, and p-value.

For the selected quadratic polynomial model, each variable was evaluated individually and with the combination of variables generated by the software. Based on the p-value of less than 0.05, any variables or combination of variables that could occur due to noise or error. The lack of fit was also concluded in this analysis section based on the p-value of less than 0.05 [33].

Before a final equation for the studied response's correlation with the experimented variables being established, a fit statistic was evaluated to determine if the predicted R^2 is close to the adjusted R^2. A model was concluded as the best model if the Adeq Precision, which measures the signal to noise ratio indicated an adequate signal [34].

Finally, an equation to predict the response for given levels for each factor was established by the software, which shall be utilized for the optimization process. The software generated the optimized value for all three variables which produce the maximum mass of extracted oil and extraction yield.

Mass balance was computed using Microsoft Office Excel of the Microsoft 365 version. The earlier established process flow was transformed into a spreadsheet whereby each input and output of the material's mass for each piece of equipment were calculated. Further enhancement of mass balance was carried

out with the aid of SuperPro Designer® Version 10.3 whereby the detailed process flow diagram was established based on the studied process plant. Operation data for each piece of equipment was keyed in based on the historical and current process control parameters. The complete process flow diagram is as per figure (1).

In general, the mass balance of process equipment is as per equation 3, as below: -

$$\frac{dm}{dt} = \sum_{i=1}^{n} q_{ini}(t) - \sum_{j=1}^{k} q_{outj}(t) \quad (3)$$

Where m is the mass of accumulated material, t is the time, $q_{in\,i}(t)$ is the i^{th} input mass flow, and $q_{outj}(t)$ is the j^{th} output mass flow [35].

As to ensure mass balance computation be able to produce a precise and consistent extraction rate and yield, the calculation was conducted in a steady-state condition with no mass accumulation takes place and hence, dm/dt= 0 [36].

The mass balance framework and calculation were focused on extraction and distillation process flow only. The other process flows namely desolventizing and absorption and desorption sections are merely functional to recover the n-hexane with the limitation of equipment capacity.

For each piece of equipment, the mass balance was calculated based on individual flowrate of SBE, oil, and hexane in ton per hour measurement unit.

Prior to computing mass balance with the spreadsheet formulated, specific data such as SBE oil content, SBE feed rate, extractor slurry concentration, hydrocyclone flowrate, slurry concentration, oil content, and n-hexane content for the various vessel and filters were obtained either through lab analysis result, plant's flowmeter or plant's historical data.

3. RESULTS AND DISCUSSION

The outcome of the experiment runs, which was the measured extracted oil rate was tabulated according to the set of runs generated by the Design Expert V12.0.8.0 software, as shown in Table 3. response number one which is the extraction oil rate was fitted into a linear model source. The linear model was selected based on the highest value for adjusted R^2 and predicted R^2, which were 0.9618 and 0.9458, respectively. While both values were more than 90% of confidence, the linear source had a p-value of less than 0.05 and the highest lack of fit p-value, 0.6435, as shown in Table 4. Hence, the linear was suggested and shall be applied for this study purpose.

The selected model for the responding variables, extracted oil rate, was further analyzed based on the sum of squares. Linear vs Mean was suggested given the highest polynomial where the additional terms were significant, and the model was not aliased. The suggested source model's sum of squares, mean square, F-value, and the p-value, which were 0.1399, 0.0466. 135.43 and less than 0.0001 respectively, were the highest compared to others. The summary of the statistical analysis of all available models is tabulated in Table 5.

The selected model source for the response of the extracted oil rate underwent a lack of fit tests to ensure the selected model shall have an insignificant lack-of-fit. The linear model, which was selected earlier was proven to be insignificant lack-of-fit based on the highest value of the sum of squares, 0.0029. The outcomes of the analysis are shown in Table 6.

Before ANOVA, the selected model source was examined for a final round statistically by considering standard deviation, R2, adjusted R2, predicted R2, and PRESS [37]. The linear source was affirmed further given the highest value for both adjusted and predicted R2, which were 0.618 and 0.9458, respectively. Furthermore, a high coefficient of determination was achieved for the selected source, R2 = 0.9690, which was close to 1 and concluded that 96.90% of the response variation could be explained as a function of the three independents

Table 3: Responding variable outcomes based on Box-Behnken Design runs

Runs Number	Independent Variable Coded			Responded Variables
	X_1	X_2	X_3	R_1
	Settling Rate	*n*-hexane temperature	Slurry concentration	Extraction oil rate
1	13	55	35	2.04
2	13	50	30	2.00
3	8	50	30	1.80
4	11	50	35	1.86
5	11	50	35	1.86
6	11	50	35	1.85
7	8	45	35	1.70
8	13	45	35	1.84
9	13	50	40	1.92
10	11	55	35	2.02
11	11	50	35	1.90
12	11	45	30	1.80
13	8	50	40	1.76
14	11	50	35	1.88
15	11	55	40	1.96
16	8	55	35	1.86
17	13	45	40	1.76

Table 4: Fit summary for the responding variable of extraction oil rate

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2	Decision
Linear	< 0.0001	0.6435	0.9618	0.9458	Suggested
2FI	0.5030	0.6047	0.9604	0.9173	
Quadratic	0.3489	0.6569	0.9636	0.9051	
Cubic	0.6569		0.9557		Aliased

Table 5: The sequential model sum of squares for the responding variable of the extraction oil rate

Source	Sum of Squares	df	Mean Square	F-value	p-value	Decision
Mean vs Total	59.52	1	59.52			
Linear vs Mean	0.1399	3	0.0466	135.43	< 0.0001	Suggested
2FI vs Linear	0.0009	3	0.0003	0.8388	0.5030	
Quadratic vs 2FI	0.0013	3	0.0004	1.29	0.3489	
Cubic vs Quadratic	0.0007	3	0.0002	0.5833	0.6569	Aliased
Residual	0.0016	4	0.0004			

Total	59.67	17	3.51			

Table 6: Lack of fit test for the responding variable of the extraction oil rate

Source	Sum of Squares	df	Mean Square	F-value	p-value	Decision
Linear	0.0029	9	0.0003	0.7990	0.6435	Suggested
2FI	0.0020	6	0.0003	0.8235	0.6047	
Quadratic	0.0007	3	0.0002	0.5833	0.6569	
Cubic	0.0000	0				Aliased
Pure Error	0.0016	4	0.0004			

variables studied and only 3.1% of the total variation was not explained by the selected source. The summary of the analysis is as shown in Table 7. Analysis of Variance (ANOVA) for the experimental runs conducted with the linear source was performed for the responding variables of the extraction oil rate**.** The ANOVA performed was as shown in Table 8.

Based on the ANOVA performed, the model F-value of 135.43 implied the model is significant. Furthermore, there is only a 0.01% chance that an F-value this large could occur due to noise.

Table 7: Model summary statistic for the responding variable of the extraction oil rate

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	Decision
Linear	0.0186	0.9690	0.9618	0.9458	0.0078	Suggested
2FI	0.0189	0.9752	0.9604	0.9173	0.0119	
Quadratic	0.0181	0.9841	0.9636	0.9051	0.0137	
Cubic	0.0200	0.9889	0.9557			Aliased

Table 8: ANOVA for responding variable of the extraction oil rate

Source	Sum of Squares	df	Mean Square	F-value	p-value	Decision
Model	0.1399	3	0.0466	135.43	< 0.0001	significant
A-Settling Rate	0.0578	1	0.0578	167.86	< 0.0001	
B-n-hexane Temperature	0.0761	1	0.0761	220.85	< 0.0001	
C-Slurry Concentration	0.0060	1	0.0060	17.57	0.0011	
Residual	0.0045	13	0.0003			
Lack of Fit	0.0029	9	0.0003	0.7990	0.6435	not significant
Pure Error	0.0016	4	0.0004			
Cor Total	0.1444	16				

Besides, the F-value of 135.43 for this model was found to be significantly larger than the tabulated F value of 3.41, corresponding to the degree of freedom of model and residual, 3 and 13 respectively at the p-value of 0.05 [22]. Hence, the null hypothesis (H_o) was rejected for the responding variables, and optimization of the extraction parameters must be conducted to optimize the parameters for the maximization of the extracted oil rate.

p-values of less than 0.0500 indicated that model terms are significant. In this study, settling rate, *n-hexane* temperature, and slurry concentrations were significant model terms. P-values of greater than 0.1000 indicate the model terms are not significant.

The Lack of Fit F-value of 0.7990 implied it is not significant relative to the pure error. There was a 64.35% chance that a Lack of Fit F-value this large could occur due to noise. The ultimate objective was to ensure the model selected has an insignificant lack of fit, which was achieved for the selected model for this study.

The fit statistic was conducted to analyze the selected model from any influence of signal to noise ratio. Based on the analysis performed, the predicted R^2 of 0.9458 is in reasonable agreement with the adjusted R^2 of 0.9618 whereby the difference is less than 0.2. Adeq Precision measures the signal-to-noise ratio [38]. A ratio greater than 4 is desirable, as recommended by the analysis outcomes. The analysis performed showed a ratio of 40.550, which indicates an adequate signal. Hence, the selected model can be used to navigate the design space. The outcomes of the analysis are as shown in Table 9.

Table 9: Fit statistic for the selected liner model

Std. Dev.	0.0186	R^2	0.9690
Mean	1.87	Adjusted R^2	0.9618
C.V. %	0.9917	Predicted R^2	0.9458
		Adeq Precision	40.5500

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant [39], The analysis performed for the responding variables of the extracted oil rate revealed that the independent variables of settling rate and *n*-hexane temperature were directly proportional to the extraction oil rate by having the positive coefficient estimates. On the other hand, slurry concentration was analyzed to be inversely proportional to the extraction oil rate by having a negative coefficient. The summary of coefficients for the analyzed independent variables is as shown in Table 10.

Table 10: Coefficient of independent variables studied

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	1.87	1	0.0045	1.86	1.88	
A-Settling Rate	0.0850	1	0.0066	0.0708	0.0992	1.0000
B-n-hexane Temperature	0.0975	1	0.0066	0.0833	0.1117	1.0000
C-Slurry Concentration	-0.0275	1	0.0066	-0.0417	-0.0133	1.0000

Based on the coefficient table produced by the analysis, a coded equation was generated. The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. Equation 4 shows the predicted extracted oil rate model.

$$\text{Extracted oil rate (tph)} = 1.87 + 0.0850A + 0.0975B - 0.0275C \quad (4)$$

It is crucial to diagnose the selected model and ANOVA carried out to ensure are not affected by abnormalities such as outliners, trends, or isolated influential runs [40]. Firstly, the prediction of the studentized residuals was done by referring to the best-fit normal distribution. A plot of the graph was done against the studentized residuals obtained from the experiments. The plotted graph is as shown in Figure (2).

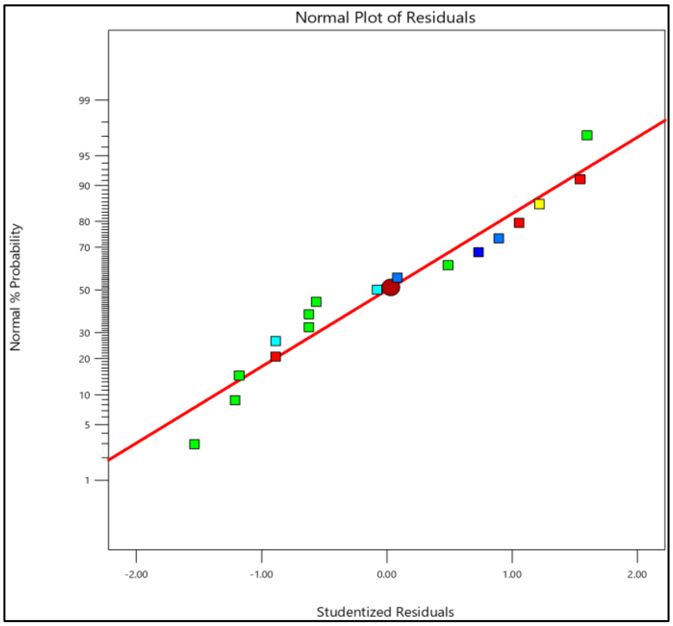

Figure (2) Residual plots of studentized residuals versus normal probability

The plotted residuals shall form a roughly straight line with the exact normal distribution that does not need to be followed by the data. Rather, a well-behaved distribution, with the tails end rapidly and roughly symmetric is expected [41]. Based on Figure (2), the studentized residuals follow a normal distribution, and no abnormalities were observed, which indicates that the selected model and experiments run were fitted to the response surface.

Secondly, the studentized residuals were plotted against the predicted extracted oil rate, as shown in Figure (3). A crystal-clear observation can be done that the experiment data points were scattered randomly in the plot, which is ideal and no data transformation is further needed [41]. Furthermore, the plot indicated that the values of the extracted oil rate were irrelated to the original observations. Hence, it can be concluded that the quadratic regression model produced an adequate description of the extracted oil rate outcomes.

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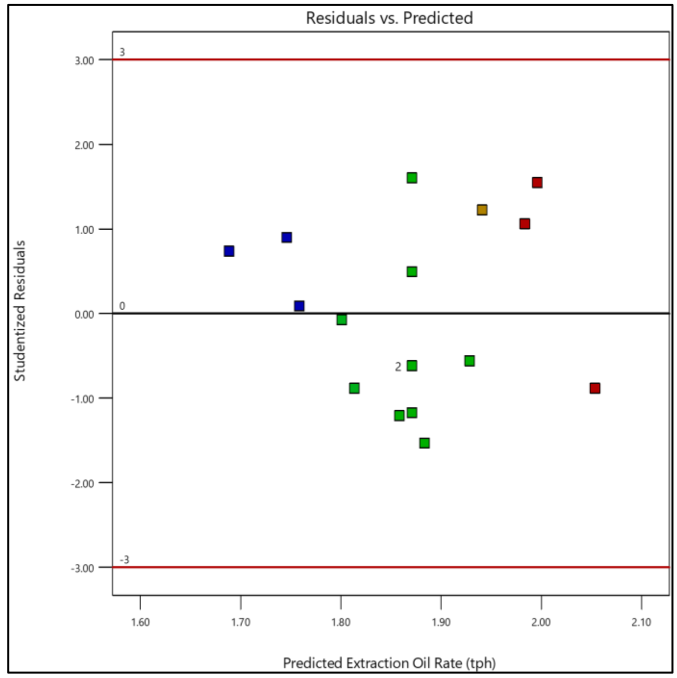

Figure (3) Plot of the predicted extraction oil rate versus the studentized residuals

Thirdly, the outliner t plot, which indicates the value of residual for all experimental runs for the responding variable of extracted oil rate was plotted as in Figure (4). Based on this plot, the experimental run which has a large residual can be determined. The conducted experiment runs contained all the studentized residuals to be well within the ±3.00 interval. Hence, a good approximation of the fitted model to the response surface was well indicated.

Finally, the difference between the experimental and predicted value of the extracted oil rate was analyzed. Based on the analysis performed as shown in Figure (5), the differences between the experimental and predicted values were less than 0.1, indicating that there was a good agreement between the experimental data and the model. These findings further conform to the R^2 and adjusted R^2 obtained previously which had a value close to the unity. Hence, the regression model gave a good estimate of the response of the system, which is the extraction oil rate, with the changes in the value of settling rate, *n*-hexane temperature, and slurry concentrations.

Based on the ANOVA conducted for extraction oil rate, we understood that settling rate and *n-hexane* temperature both were directly proportionate to the extraction oil rate outputs while slurry concentration was inversely proportionate to the extraction oil rate outputs. Hence, the three-dimensional (3D) the model graph was plotted with the x-axis of *n-hexane* temperature and z-axis of settling rate against the y-axis of the extraction oil rate. Slurry concentrations were manipulated between the experimented range of 30-40% to produce a perfect combination of all the three independent variables to maximize the extraction oil rate output, as shown

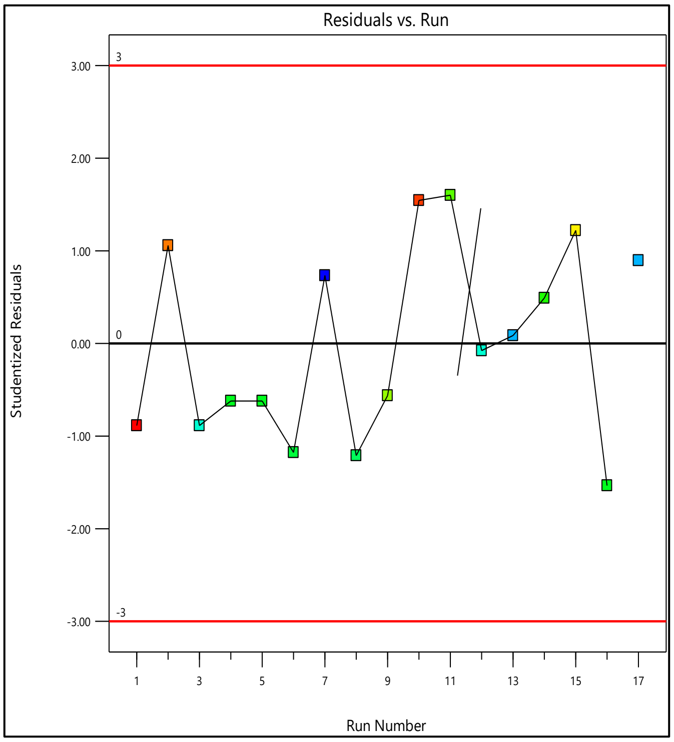

Figure (4) Outlier t plot for responding variables of extracted oil rate

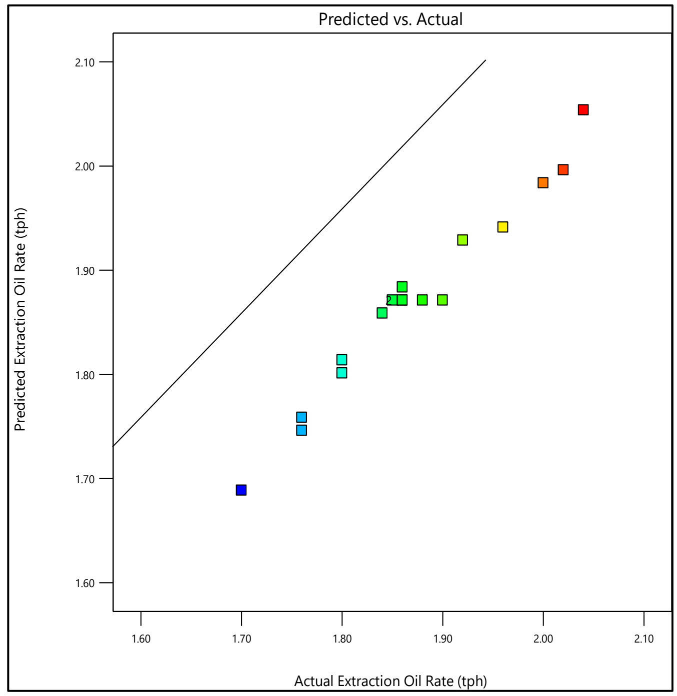

Figure (5) Plot of the actual extraction oil rate versus the predicted value

in Figure (6), (7), and (8). Based on the analysis conducted, the extraction oil rate output was maximized when the settling rate was 13ml, n-hexane temperature was 55°C and the settling rate was 30%. Figure (6) clearly illustrated the maximized extraction oil rate output within the red-colored region and with the minimum blue-colored region. On the other hand, despite the settling rate and n-hexane temperature were maintained at the same value of 13ml and 55°C, both higher slurry concentrations of 35% and 40% respectively produced less extraction oil rate. Figure (7) and (8) 3D surface plots further confirmed the analysis with the less red-colored region and a more blue-colored region compared to Figure (6).

Hence, the maximum extraction oil rate of 2.08 tons per hour was achieved at the settling rate of 13ml, n-hexane temperature of 55°C and slurry concentrations of 30%.

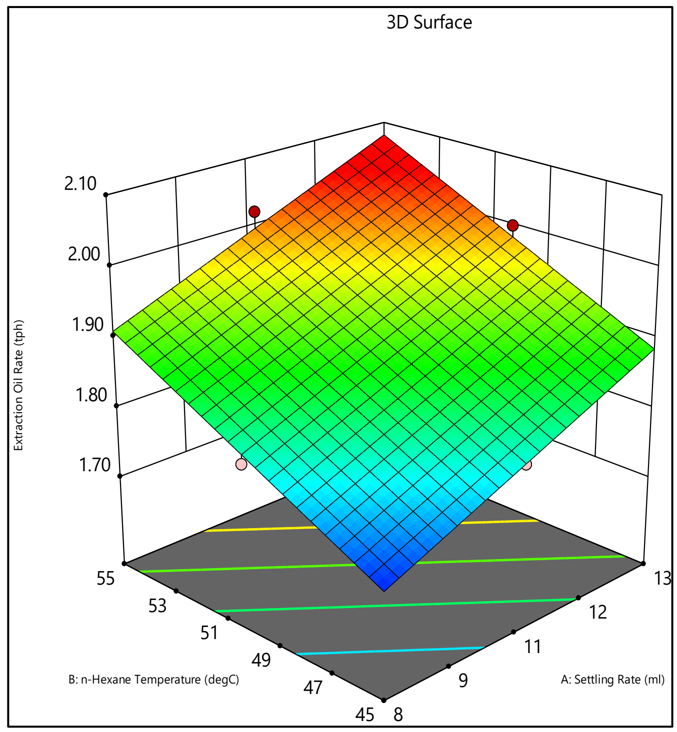

Figure (6) Optimized extraction oil rate output with the slurry concentration of 30%

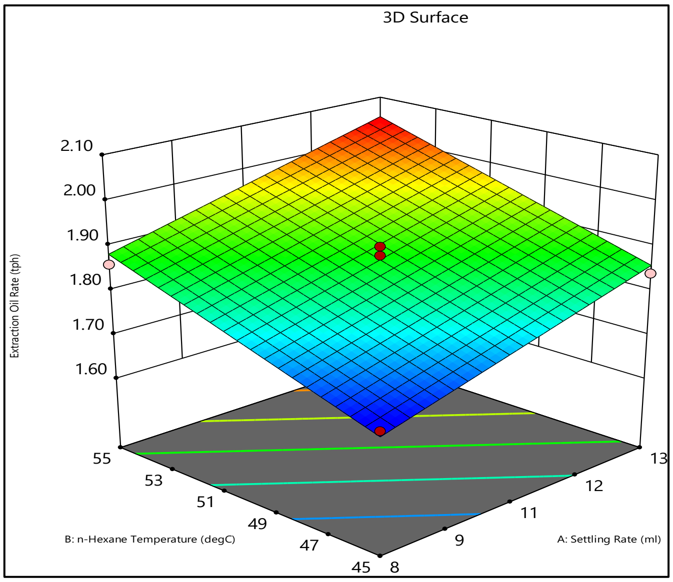

Figure (7) Optimized extraction oil rate output with the slurry concentration of 35%

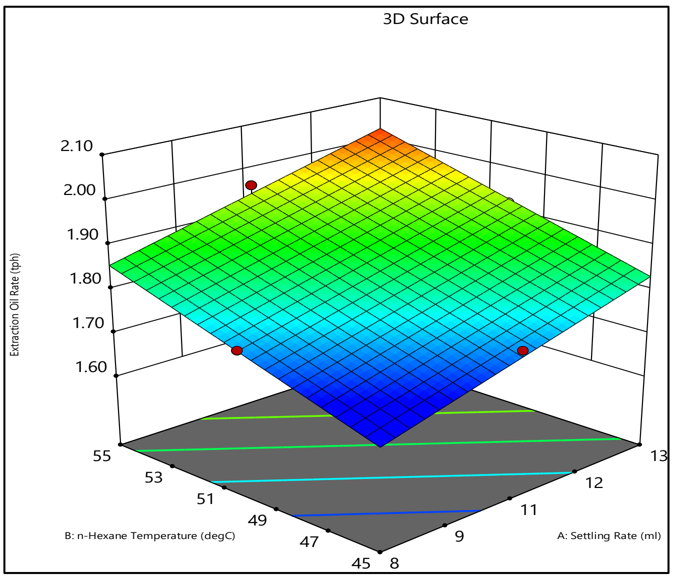

Figure (8) Optimized extraction oil rate output with the slurry concentration of 40%

The novelty of this study besides being the pioneer on optimization of the extraction parameters for n-hexane solvent extraction of SBE is the mass balance computation to quantify the input and output of each of the equipment involved in the extraction plant, based on the input of the optimized extraction parameters. In reference to the optimized extraction parameters discussed in this chapter, the extraction oil rate was calculated automatically by SuperPro Designer software based on the input of the optimized extraction parameters' value. The mass balance computation was conducted with the slurry concentration of 30% and 35%. The reason behind, optimization analysis resulted in that slurry concentration of 40% was proven to be the least optimized parameter which yielding the lowest extracted oil rate and yield. Hence, the other two lower slurry concentration was focused to improve the extraction plant's efficiency. On the other hand, the n-hexane temperature was fixed at 55°C based on the most optimized temperature as a result of the optimization analysis done earlier. Besides, the top two most optimized settling rates, 13ml and 11ml were chosen for the mass balance computation given the objective of this study which is to maximize the extraction rate and yield output. The outcomes of mass balance computation using SuperPro Designer software were as tabulated in Table 9.

Table 9: Mass balance computation outcomes using SuperPro Designer software

SR (ml)	Slurry Concentration (%)	n-hexane Temperature (°C)	Flowrate (tonne per hour)			
			SBE Feed	SBE Oil	*n*-hexane	Extracted Oil
13	30	55	9.78	2.22	18.00	2.09
	35		9.84	2.16	13.71	2.05
11	30		9.89	2.11	19.20	2.00
	35		9.91	2.09	14.74	1.98

Based on the mass balance computation conducted, the responses' outcomes of mass balance computations were well within the 95% confidence level generated by the Design-Expert software statistic analysis, which indicated that the values obtained were statistically fit and accurate. Hence, the mass balance computation formulated in this study shall be utilized fully to calculate precisely the output values for each of the responses studied, extraction oil rate, and yield based on the optimization of the respective extraction parameters conducted in detail in this study. The mass balance outcomes were tabulated as per Table 10.

Table 10: Statistical analysis of SuperPro Designer mass balance computation outcomes with Design-Expert predicted outcomes

Extraction Parameters		Response	SuperPro Designer Mass Balance	Design-Expert		
SR (ml)	Slurry Concentration (%)			Predicted Median	95% PI low	95% PI high
13	30	Extraction Oil Rate (tph)	2.09	2.08	2.05	2.12
13	35	Extraction Oil Rate (tph)	2.05	2.05	2.02	2.09
11	30	Extraction Oil Rate (tph)	2.00	2.01	1.98	2.05
11	35	Extraction Oil Rate (tph)	1.98	1.99	1.96	2.01

4. CONCLUSIONS

As a result of this research, a few conclusions were derived as follows: -

1. Response Surface Method (RSM) was applied for the optimization of extraction parameters to maximize the extraction oil rate. The Box-Behnken Design (BBD) was chosen to be the ideal design of the experiment for this study based on the three-level factorial and seventeen experiment runs.
2. The experiment runs, and statistical analysis conducted concluded that the optimized extraction parameters of

settling rate, n-hexane temperature, and slurry concentration to maximize the extraction oil rate were 13ml, 55°C, and 30% respectively. The combination of these optimized extraction parameters produced a 2.09tph of extraction oil rate.

3. The study on the relationship of optimized extraction parameters with the mass balance in maximization of extraction oil rate concluded that a complete mass balance computation was able to be established to calculate the extraction oil rate accurately with a 95% statistical confidence level based on the combination of settling rate and slurry concentration of 13 ml and 11ml as well as 30% and 35% respectively.
4. Settling rates of 13ml and 11ml along with the slurry concentration of 30% and 35% were found to be the practical combination of managing the optimization of the extraction parameters' input concerning the availability of the SBE based on supply and demand market scenario.
5. This study established a novelty in the SBE solvent extraction research field whereby the optimized extraction parameters were being identified and a comprehensive mass balance was formulated as a future reference for all the plant managers and engineers to operate their respective plants more efficiently. The extraction process plant management shall be greatly improvised with reference to this study's outcomes with crystal clear data and information on the inputs and outputs.

5. REFERENCES

1. Rusev, S.J. and K. Salonitis, *Operational Excellence Assessment Framework for Manufacturing Companies.* Procedia CIRP, 2016. **55**: p. 272-277.
2. Ojha, S.K., *Operational Excellence for Sustainability of Nepalese Industries.* Procedia - Social and Behavioral Sciences, 2015. **189**: p. 458-464.
3. Jaeger, A., K. Matyas, and W. Sihn, *Development of an Assessment Framework for Operations Excellence (OsE), based on the Paradigm Change in Operational Excellence (OE).* Procedia CIRP, 2014. **17**: p. 487-492.
4. ONG, J.T.L., *Oil Recovery from Spent Bleaching Earth and Disposal of the Extracted Material.* Journal of the American Oil Chemists' Society, 1983. **60**(2): p. 266A - 277A.
5. Beshara, A. and C.R. Cheeseman, *Reuse of spent bleaching earth by polymerization of residual organics.* Waste Manag, 2014. **34**(10): p. 1770-4.
6. Kalam, A. and J.B. Joshl, *Regeneration of Spent Earth in Aqueous Medium.* Journal of the American Oil Chemists' Society, 1988. **65**(12): p. 1917-1921.
7. Bohl, T., et al., *Particulate number and NO trade-off comparisons between HVO and mineral diesel in HD applications.* Fuel, 2018. **215**: p. 90-101.
8. Al-Zahrani, A.A. and M.A. Daous, *Recycling of Spent Bleaching Clay and Oil Recovery.* Process Safety and Environmental Protection, 2000. **78**(3): p. 224-228.
9. C.G. Lee, C.E. Seng, and K.Y. Liew, *Solvent Efficiency for Oil Extraction from Spent Bleaching Clay.* JAOCS, 2000. **77**(11).
10. Jan Rydberg, C. Musikas, and G.R. Choppin, *Principles and Practices of Solvent Extraction*. 1992, New York: Marcel Dekker Inc.
11. Jassim, N.J., *Solvent Extraction Optimization of Chlorophyll Dye from Conocarpus Lancifolius Leaves.* Journal of Engineering and Technological Sciences, 2020. **52**(1): p. 14-27.
12. Hatami;, A.M., M.R. Sabour;, and A. Amiri, *Recycling process of spent bleaching clay: Optimization by response surface methodology.* J. Environ. Sci. , 2018. **4(1)**: p. 9-18.
13. Rostami, M., et al., *Optimizing the extraction process of sesame seed's oil using response surface method on the industrial scale.* Industrial Crops and Products, 2014. **58**: p. 160-165.
14. Sieck, H., *Extraction of Spent Bleaching Material.* J. of the American Oil Chemists' Society, 1937: p. 314-315.
15. C.G. Lee, C.E. Seng, and K.Y. Liew, *Solvent Efficiency for Oil Extraction from Spent Bleaching Clay.* J. of the American Oil Chemists' Society, 2000. **77**(11).
16. Dahlen, J.H. and L.A. Lindh, *Mass Balance of Hexane Losses in an Extraction Plant.* JAOCS, 1983. **60**(12): p. 2009-2010.
17. Warn, A.E. and J.S. Brew, *Mass Balance.* Water Research, 1980. **4**: p. 1427-1434.
18. Ochsenbein, D.R., et al., *Industrial application of heat- and mass balance model for fluid-bed granulation for technology transfer and design space exploration.* Int J Pharm X, 2019. **1**: p. 100028.
19. *Safety Data Sheet Exxsol Hexane Fluid*. 2019, Exxon Mobil: Kuala Lumpur, Malaysia.
20. Varala, S., et al., *Process optimization using response surface methodology for the removal of thorium from aqueous solutions using rice-husk.* Chemosphere, 2019. **237**: p. 124488.
21. Antony, J., *Design of Experiments for Engineers and Scientists*. 2nd ed. 2014, London: Elsevier. 1-17.
22. Anderson, M.J., *DOE Simplified*. 2nd ed. 2007, New York: Productivity Press. 147-157.
23. Anderson, M.J., *RSM Simplified*. 2005, New York: Producitivity Press. 57-122.
24. Elkelawy, M., et al., *Maximization of biodiesel production from sunflower and soybean oils and prediction of diesel engine performance and emission characteristics through response surface methodology.* Fuel, 2020. **266**.
25. Montgomery, D.C., *Design and Analysis of Experiments*. 6th ed. 2005, United States of America: John Wiley & Sons, Inc. 8-22.
26. Ferreira, S.L., et al., *Box-Behnken design: an alternative for the optimization of analytical methods.* Anal Chim Acta, 2007. **597**(2): p. 179-86.
27. Bezerra, M.A., et al., *Response surface methodology (RSM) as a tool for optimization in analytical chemistry.* Talanta, 2008. **76**(5): p. 965-77.
28. Gullón, B., et al., *Optimization of solvent extraction of antioxidants from Eucalyptus globulus leaves by response surface methodology: Characterization and assessment of their bioactive properties.* Industrial Crops and Products, 2017. **108**: p. 649-659.

29. Muppaneni, T., et al., *Optimization of biodiesel production from palm oil under supercritical ethanol conditions using hexane as co-solvent: A response surface methodology approach.* Fuel, 2013. **107**: p. 633-640.
30. Nissy, S.M., et al., *Optimization for the extraction of Bisphenol A; Response surface Methodology*. Materials Today: Proceedings, 2018. **5**(9): p. 17914-17923.
31. Latchubugata, C.S., et al., *Kinetics and optimization studies using Response Surface Methodology in biodiesel production using heterogeneous catalyst.* Chemical Engineering Research and Design, 2018. **135**: p. 129-139.
32. Wang, J., et al., *Bayesian modeling and optimization for multi-response surfaces.* Computers & Industrial Engineering, 2020. **142**.
33. Mohapatra, T., S.S. Sahoo, and B.N. Padhi, *Analysis, prediction and multi-response optimization of heat transfer characteristics of a three fluid heat exchanger using response surface methodology and desirability function approach.* Applied Thermal Engineering, 2019. **151**: p. 536-555.
34. Li, F., et al., *Optimization of enzymatic pretreatment for n-hexane extraction of oil from Silybum marianum seeds using response surface methodology*. Food and Bioproducts Processing, 2012. **90**(2): p. 87-94.
35. Itävuo, P., et al., *Mass balance control of crushing circuits.* Minerals Engineering, 2019. **135**: p. 37-47.
36. Hocaoglu, S.M., et al., *Assessment of technology modification for olive oil sector through mass balance: A case study for Turkey*. Journal of Cleaner Production, 2018. **188**: p. 786-795.
37. A.W. Nursulihatimarsyila, et al., *Deoiling and Regeneration Efficiencies of Spent Bleaching Clay*. American Journal of Applied Sciences, 2010. **7**(3): p. 434-437.
38. Aslan, N. and Y. Cebeci, *Application of Box–Behnken design and response surface methodology for modeling of some Turkish coals.* Fuel, 2007. **86**(1-2): p. 90-97.
39. Jung, K.A., et al., *Response surface method for optimization of phenolic compounds production by lignin pyrolysis.* Journal of Analytical and Applied Pyrolysis, 2016. **120**: p. 409-415.
40. Zhang, P., et al., *Biodiesel production from palm oil and methanol via zeolite derived catalyst as a phase boundary catalyst: An optimization study by using response surface methodology*. Fuel, 2020. **272**.
41. Vining, G., *Technical Advice: Residual Plots to Check Assumptions.* Quality Engineering, 2011. **23**: p. 105-110.

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