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Optimization of see do il extraction process parameters from *Brucea javanica* using Design of Experiment (DoE)

K Hasni^{1,2} and Z Ilham^{1,*}

¹Biomass Energy Lab, “Institute of Biological Sciences Faculty of Science, University of Malaya 50603 Kuala Lumpur Malaysia

² Lasbella University of Agriculture, water and Marine Sciences, Uthal, Pakistan”

E-mail: ilham@um.edu.my.

Abstract. The project was done to improve Soxhlet process for extraction of oil for seeds of *Brucea javanica* and explore its various applications. The optimization was done by RSM (response surface methodology) using Box Behnken design of experiment (DoE). Properties of the *Brucea javanica* oil (BJO) was determined using Gas/Mass Spectrometric approach and Fourier Transform Infrared Spectroscopy (FT-IR) analysis. GCMS analysis indicated that important fatty acid are presence in BJO and FTIR suggested the presence of core functional groups in BJO. Quadratic model was used to analyse the results obtained from the optimised experimental design. ANOVA of developed model showed good presentation of model with R^2 value of 0.9078. Similarly, it was discovered that model were significant because their probability values (p-values) were discovered to be less than 0.05. The design of experiment showed a highest yield of 44.89 % at 75°C, 6hour extraction time and hexane 425ml.

1. Introduction

Research on recovery of valuable products from various agricultural biomass waste such as essential oils and bioactive compounds has increased recently [1-3]. Plant seed oils or commonly known as essential oils are considered as core value components for various applications in food and medicine industry [4]. A suitable extraction solvent is required to exploit the amount of oils extracted and a non-polar solvent such as hexane is commonly used [5]. The most effective method to recover oil from seeds is solvent extraction especially for the seeds with low oil yield. At lower temperature, solvent extraction method is preferred over other methods because its good quality of oil production [6]. To obtain better oil yield from less turbid oil, solvent extraction is preferred and oil produced from this method has suitable viscosity in comparison to fossil fuel [7].

The optimisation was analysed for different variables and their effect on yield was carried applying DoE(response surface methodology) using Box-Behnken approach of experiment (DoE) for extracting oil from BJO that isn't applied yet for this plant. DoE shows best result in enhancing and improving methods for different purposes including oil extraction from different plant seeds [8]. DOE (RSM) has the ability to provide suitable experiments and are statistically accepted results could be obtained.

BJO seeds have potential and a significant amount of oils which can be used in pharmacy, agro-food and bio-fuel. This seeds are from family, Simaroubaceae, an ever-green plant. It was, first, cited in the Chinese literatures cite it in *Collection of Materia-medica* [9]. A traditionally used plant for medicine



[10] and anti-diabetic treatment [11]. Metabolites (secondary one) are extracted from this said plants like flavonoid, tri-ter-penoid, alkaloid and lignin [12].

In this research, oil extraction from *Brucea javanica* seeds using Soxhlet extraction was conducted. In order to improve the oil extractions condition for the process, DoE (RSM) was used to estimate the independent parameter effect on oil extraction. Furthermore, the oil quality was determined by physio-chemical analyses.

Hence, the purpose of this study is to analyse different Soxhlet extraction parameters such as hexane volume, extraction time and temperature for *Brucea javanica* seeds using the RSM. In addition, GCMS and FTIR analyses were done to detect functional groups in the oil.

2. Experimental

2.1 Materials

The seed was obtained from Glami Lemi Biotechnology Research Center, Negeri Sembilan, Malaysia. Washing was done and oven was used to dry at 35°C for 3 days to remove water content. The seeds were grinded to powder form using grinder.

2.2 Design of experiment

In order to optimize oil extraction from *Brucea javanica* seeds oils, DoE (RSM) was employed based on Behnken approach. Selected extraction parameters were extraction time (h): X1, hexane volume (ml): X2, and temperature (°C): X3. The un-explained variability effect minimization was done by using randomization approach in the response observed.

2.3 Oil extraction from BJO

Extraction took place in a flask (500ml) and solvent for extraction was hexane. Grinded seed powder put into extracting thimbles set in a condenser. Solvent was out in the flask with tightly fixed condenser at bottom. A constant 70° C temperature as set in mantle and extracted oil obtained from the flask. An equation given below for yield.

$$\text{Oil Yield} = \frac{\text{Weight of oil produced}}{\text{Weight of oil used}} \times 100 \quad (1)$$

2.4 FTIR

Infrared-radiation is circulated over the sample to get spectrum of correspondence, several data acquisitions was arranged. FT-IR spectrum were developed in the wavenumber with different ranges (400 to 4000 cm⁻¹). Normalization of whole spectrum was baseline adjusted using FTIR software.

2.5 GC-MS analysis

“Gas chromatography mass spectrophotometry analysis (GC-MC) was done by using Trace GC-2000 gas chromatograph coupled to a Polaris Q Ion trap mass-spectrometer” (“Thermo-Finnigan, Austin, TX USA). “Zebron ZB-5ms (“Phenomenex, Torrance CA USA).

2.6 Data analysis

DoE of seventeen experiments result was evaluated using Design of Expert software 10. To relate the variable responses Oil yield and independent variable Extraction time, Solvent and Temperature, regression (multiple) was obtained in order to fit co-efficient (polynomial model) and ANOVA. Equation is given below,

$$y = b_0 + \sum_{i=1}^k b_i X_i + \sum_{i=1}^k b_{ii} X_i^2 + \sum_{i < j}^k b_{ij} X_i X_j + e \quad (2)$$

“y :yield
 bo :value of intercept
 bi :co-efficient model
 bij :interaction effects
 bii :co-efficients of quadratic Xi
 e :“error”

3. Results and Discussion

3.1 Physico chemical characterizations

35% seed oil was obtained from extraction process. Physico and chemical characterization which is a vital criteria to oil quality determination. So, these characteristics were obtained and reported displayed in Tab1. The physical appearance of oil was yellow and index (refractive) 1.58. The ultrasonic (speed of 1.42 ms⁻¹). 4.65 (mg KOH/g) acid value shows FFA in oil. Oxidation strength of 3.20 hours was (ASTM standard = 4hours) and the kinematic viscosity of the oil was 1.383mm²/s. The results of Gas GC analysis for FFA in the *Brucea javanica* oil showed composition of palmitic acid (9 %), steric acid (6 %) and linoleic acid (20 %). The results are in agree with preceding findings on *Brucea javanica* oil [13].

Table 1. Physico and chemical characterization of seed oil.

“Physico &chemical characterization”	Oil Properties
Physical appearance	Yellow
FFA	4.665 (mg KOH/g)
Viscosity (Kinematic)	1.383 (mm ² /s)
Refractive-Index	1.58
Percentage of oil	35 %
Ultrasonic-speed	1.42 ms ⁻¹
Oxidative Strength	3.2 h

3.2 Fourier transform infrared spectroscopy (FT-IR)

“In the functional group –CO–O–CH₃ of the BJME (*Brucea javanica* methyl ester), “which was generated during the ‘transesterification, 2853 cm⁻¹ and 2923 cm⁻¹ were attributed to the stretching “and asymmetric stretching vibrations of –CH₃, 1461 cm⁻¹ corresponded to the asymmetric bending vibration of “–CH₃, 1167 cm⁻¹ was due to the stretching vibration from O–CH₃”; and “1740 cm⁻¹ was assigned to the vibration of “–C=O shown in Figure 1.

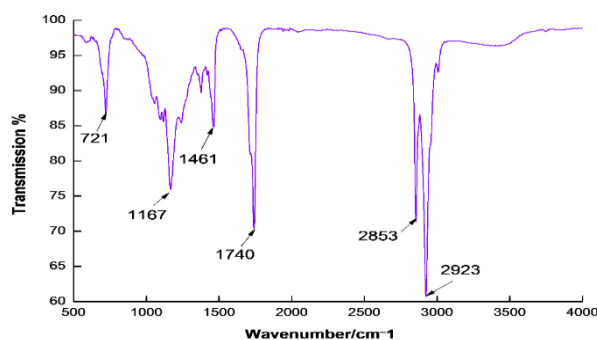


Figure 1. FT-IR analysis of *Brucea javanica* methyl ester.

These results represent the conversion of tri-acyl-glycerols” in “*Brucea javanica*oil which could be converted into methyl esters’[14].

3.3 Gas chromatography mass spectrometry (GCMS)

To detect potential compound in seeds oil, identification was carried out and analysis was done. The whole process of identifying compounds was obtained using GC-MS. Therefore, vital and important compounds were detected which play major role in anti-oxidation process such as flavonoids and phenolic compounds.

A special technique can infusion (direct) was used to determine phyto-chemical analysis of seed oil using hexane extraction from BJO seed oil. Table 2 is displaying peaks of many compounds which were checked and identified using global library of reference.

Table 2. GCMS Analysis of *Brucea javanica* seeds oil.

“Retention Time	”Area	“Chemical Compound	Reference”
10.102	0.3125	Cyclopentane	69587
10.6113	0.0564	Dodecanoic acid, methyl ester	72688
12.4022	0.5184	8-Heptadecene	92565
13.9815	0.0318	Naphthalene	16914
15.0858	6.0428	Hexadecanoic acid, methyl ester	119407
15.8068	5.2165	n-Hexadecanoic acid	107549
17.5234	22.5493	Oleic Acid	129337
17.9697	3.5004	Octadecanoic acid	131262
18.2386	8.5806	9-Eicosyne	126189
19.2171	4.9914	10-Heneicosene (c,t)	139793
24.7388	9.6426	Heneicosane	141424
26.8044	0.7648	Erucic acid	175491

3.4 Parameters optimization using RSM

Table 3 showed the results obtained from all the variables (extraction time, temperature and hexane) calculated in this exploration. The table shows DoE experiments obtained by Response Surface Methods. Oil yield, predicted oil yield and RV (residual-value). The outcomes directed that the deviation to the variable/parameter are affecting the *Brucea javanica* seeds oil (yield) are displayed in the table as well.

Once the results obtained for oil extraction, an equation is drawn labelled as equation 3. Dependent variables and output co-relation is studied using equation 3. Parameters are labelled as A, B, C (time, solvent and temp) in the equation.

$$\text{Yield} = +42.64 - 0.81 * A - 1.44 * B - 4.22 * C + 0.25 * AB + 0.92 * AC + 0.19 * BC - 0.087 * A^2 - 0.64 * B^2 - 4.92 * C^2 \quad (3)$$

ANOVA (quadratic model) is shown in Table 4, which was developed to observe the capability of obtained result and empirical way tested for BJO oil yield. P-value and R2 were used to see that model was able to fit the experimental data parameters. The probability values of the model and that of each of the factors of the model were meant to show their significances. The results achieved was indicating that p-value was value was >0.05 and was suggesting that each of factors was as well significant in the developed model.

Table 3. Prediction and seed oil yield.

Run	Extraction time	Solvent	Temperature	Experimental Yield (%)	Prediction Yield (%)	Residual Value
1	7	425	75	44	42.5	1.4
2	7	425	75	44.3	42.5	1.7
3	7	425	75	43.88	42.5	1.2
4	6	500	75	40	41.4	-1.4
5	8	350	75	44.32	42.7	2.7
6	7	350	80	33.9	34.1	-0.2
7	6	350	75	43.99	43.8	0.1
8	7	500	70	39.88	39.6	0.2
9	7	425	75	40	42.5	-2.5
10	6	425	75	44.89	43.5	1.3
11	6	425	80	33.43	33.2	0.1
12	8	500	75	39.33	39.3	-0.1
13	7	500	80	32.99	31.5	1.4
14	7	350	70	41.55	42.9	-1.3
15	7	425	75	41	42.5	-1.5
16	8	425	70	40	40.1	-0.1
17	8	425	80	32.21	33.4	-1.2

Table 4. Analysis of variance.

“Analysis of variance table” (“Partial sum of squares”)						
“Source”	“Sum of Squares”	“Degree of freedom df”	“Mean Square”	F	“p-value Prob > F”	
Model	274.24	9	30.47	7.66	0.0068	Significant
A- Extraction Time	5.20	1	5.20	1.31	0.2906	
B-Hexane	16.70	1	16.70	4.20	0.0797	
C- Temperature	142.72	1	142.72	35.86	0.0005	
AB	0.25	1	0.25	0.063	0.8093	
AC	3.37	1	3.37	0.85	0.3882	
BC	0.14	1	0.14	0.036	0.8543	
A ²	0.032	1	0.032	7.962	0.9314	
B ²	1.72	1	1.72	0.43	0.5319	
C ²	101.79	1	101.79	25.58	0.0015	
Residual	27.86	7	3.98			
Fit	12.05	3	4.02	1.02	0.4731	not significant
Error	15.80	4	3.95			
Total	302.10	16				

Furthermore, R^2 was used to see that data is represented well or not. R^2 0.9078 (model) showing closeness to unity which is 1, showing that equation was real representative of the model. Experimental design and predicted yield also display good co-relation Figure 2.

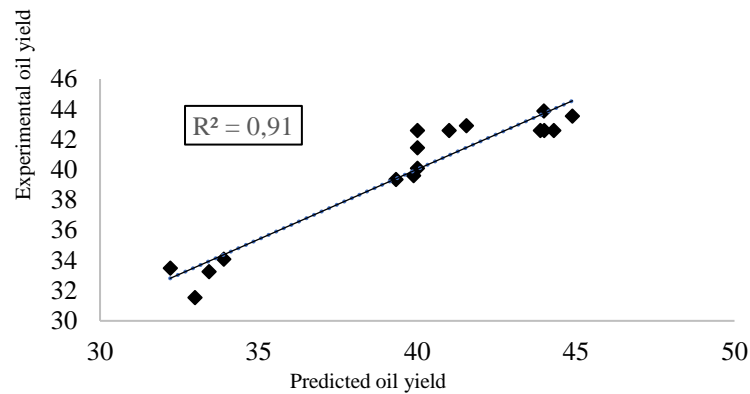


Figure 2. Predicted oil yield vs experimental oil yield (Scattered diagram).

Comparative analysis was made to study to see the performance of model designed between predicted and obtained results. The diagram (scattered) obtained from regression model analysis, which did comparison between obtained results and predicted results. Linear model shows fitness between these results verified by R square displayed in Figure 2. Outlier are displayed in Figure 3 for the oil yield. The acceptable value range is from -4.8 and $+4.8$. All the value which are outside the “interval renders is an error” in the model and data [15]. Figure points out that no value of this data is outside this interval that propose the model was reliable with experimental data with none error data recorded.

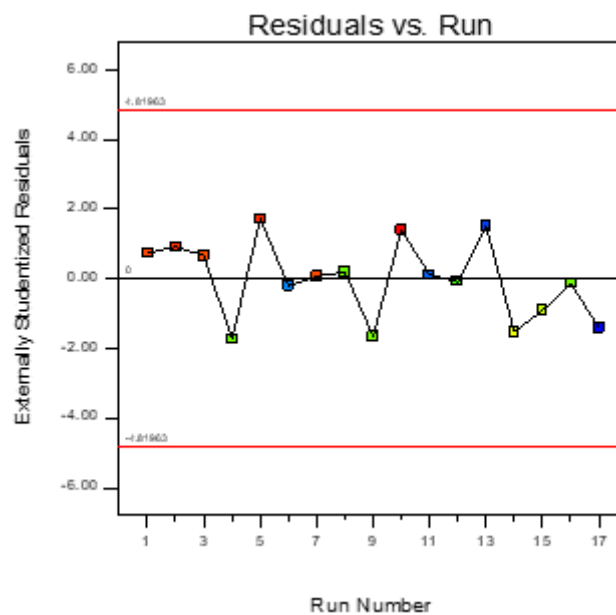


Figure 3. Experimental run vs residual t plot.

Variations effect in time, solvent and temperature of extraction on oil yield percentage for the quadratic model Box-Behnken design was studied. The 3-D surfaces plot and surface nature of *Brucea javanica* oil parameters (extraction time, hexane and temperature) optimized by RSM can be seen in “Figure 4 (a,b and c). “Figure 4 is showing a good mutual interactive effect between solvent with time and hexane with sample weight and their yield reciprocal interaction”.

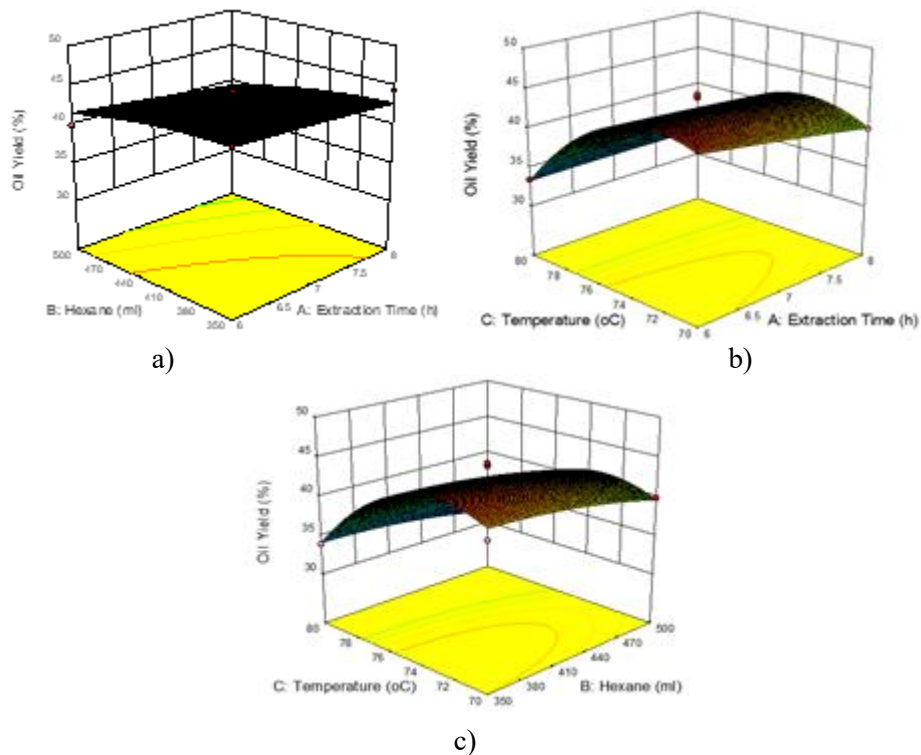


Figure 4. (a) Hexane and extraction time, (b) extraction time and temperature, and (c) temp and hexane presented in 3-D graph.

Regression equation was used to obtain optimum value for oil extraction shown in (equation (2)) using the dx10.1. The optimum condition was set at A= 6hour, B = 425ml and C= 75°C. The experimental yield was 44.89 % and predicted yield under this optimal condition was $Y = 43.5$ %. Using these optimal values for these replicates, which are independent, a mean of 30.47 % yield was obtained, within the range predicted (model used). The results suggested that DoE (RSM) be effective in optimization of oil extraction process using Soxhlet apparatus for plant seeds oil.

4. Concluding remarks

The proposed research suggested that *Brucea javanica* seeds oil is a natural rich source for oil. This work also showed that DoE (RSM) design is a useful tool in optimizing the extraction variables using design expert. The optimal condition for this model was obtained at A= 6 hours, B = 425 ml and C= 75°C. The experimental yield was 44.89% and predicted yield under this optimal condition was $Y = 43.5$ %. 30.47 mean % oil yield was attained, showing its presence in range model prediction. Physico & chemical characterisation of the seed oil recommends that it can extensively use as potential source in agro-food and bio-fuel industry.

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