Solvent Extraction of Peanut Oil: Optimization Using Response Surface Methodology

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Abstract— **This study focused on seed oil extraction by conventional method and response surface methodology. The study was performed by using different parameters as to find the optimum condition for the extraction process. The parameters that being studied were type of solvent, extraction times, temperature, stirring power intensities and solvent to feed (S/F ml of solvent to gram of feed) ratios. The process parameters were further optimized by statistical approach using historical data design of response surface method (RSM) to find the optimum parameters for the highest oil yield. Results show that maximum oil yield (61%) was obtained by using ethyl acetate as solvent at 18 ml/g of S/F ratio for 25 minutes, 50 °C and at 700 rotational per minute (rpm). The process was further optimized by surface response method (RSM), the maximum oil yield (72%) occur at 29.07 ml/g of S/F ratio for 29.39 minutes, 45.24 °C and at 600 rpm.**

***Keywords* — Peanut (Arachis Hypogaea), conventional extraction, one factor at a time (OFAT), response surface methodology (RSM), peanut oil**

I. INTRODUCTION

Vegetable oils are edible oils that extracted from plants. Vegetable oils can be extracted by various techniques. Conventional extraction method such as mechanical extraction is a continuous treatment of oleaginous seeds without solvent mostly used to extract traditional oil such as coconut oil and olive oil [1]. Sufficient forces were exerting on confined seed and the pressure will be high enough to rupture the cells and the oil will be forced out from the seed [2]. Other conventional techniques that can be used are hydrodostillation and steam distillation that were widely used for extraction of the oil. Seed or leaves will be submitted to hydrodistillation or steam distillation by using a Clevenger-type apparatus and oil will be extracted [3]. Next, the vegetable oils can be extracted by using soxhlet method by aids by solvent such as hexane, ethanol and methanol [4]. Normal solvent extraction which is so simple involve only magnetic stirrer to extract the vegetable oils [5]. Modern techniques that were assisted equipment such as microwave, ultrasonic and supercritical carbon dioxide are introduce as it reduce the time extraction and the oil yield is higher than the conventional method. Modern techniques may be assisted by solvent. Solvent mixed with the oil extracted is separate by rotary evaporator as to gain pure oil [6].

Demand of vegetables oils start increasing worldwide as the consumer tends to become aware of its benefits to human health [7]. Due to this high demand in the market, an increase in the production of vegetables oils needs to be made in order to fulfill it. This action is applied to production of peanut oil as it is one of the vegetable oils[8]. Soup made from peanut root often consumed as to help kids grow taller and give them strong muscles. Researchers at National Chiayi University, Taiwan, have identified potent antioxidant, in matured peanut roots known as resveratrol. Resveratrol, a phytoalexin, was found to be potential antimutagen and antioxidant[9]. It inhibits the development of preneoplastic lesions in cultures of carcinogen-treated mouse mammary glands [10]. Tremendous tonnages of peanut hulls annually were produced due to approximately 20% weight of the dried pod and nut weight was contributed by peanut hulls[11]. Peanut hull frequently used as roughage in commercial feeds and cattle diets, soil conditioners, act as fuel for manufacturing processes to run the boilers, bedding in broiler houses, mulch for horticultural plants, and carriers for other chemicals and fertilizers[12]. If peanut hulls contain very low of aflatoxin content, it can be efficiently utilized as bedding in dairy cattle loafing barns [13]. The vines with leaves are excellent high protein hay for horses and ruminant livestock [14]. Peanut skins which are high in antioxidants and dietary fiber are consumed as they are beneficial for health [15]. Problems concerning blood and bleeding can be treated by using seed coat of peanut and peanut shells are involved in prescriptions for treating cough.. Nonfood products such as soaps, cosmetics, lubricants and medicines can be yield from peanuts[16].

This experiment was conducted to evaluate the yield of peanut oil using solvent extraction to satisfy the demand of peanut oil in the market. The use of this conventional method is preferred for this production compared to Soxhlet method as the latter method is more time consuming and involve heat which may rupture the quality of oil produce[17]. Factors that returns the best possible response or yield of oil can be determine by screening. There are two different approach for screening that is one factor at a time (OFAT) and response surface methodology (RSM)[18]. One factor at a time (OFAT) is a method of observing one parameter while holding all other parameter constant while in response surface methodology (RSM), Design of Experiment (DOE) is employ[19]. This research focused on production of maximum oil yield based on four parameters from peanut that bought from local store.

The extraction was done under conventional extraction. The extraction were done and being optimized by one factor at a time (OFAT) and research surface methodology (RSM). On the scope of OFAT, the parameters that was investigated was the types of solvent, stirring speed, ratio of solvent to the peanut (S/F), extraction temperature and the duration of the process while in the study scope of RSM, parameters that involved are ratio of solvent to the peanut, stirring speed, extraction temperature and the duration of the process.

II. METHODOLOGY

A. *Materials*

The peanut was purchased from local market in Shah Alam, Selangor and manually cleaned by hand-picking to remove foreign objects. The peanut were grind by using blender and was separated by a vibrator sieve into a uniform nominal particle size of 850 microns. To finish, the uniform size of peanut were stored in air tight container to avoid contamination for further progress.

B. Extraction of Oil by OFAT

Sieved peanut (5g) was placed in a flask covered by aluminium foil, with different type of solvent (ethanol, methanol, acetone, ethyl acetone), occupied different volume levels (6-36 mL/g) of extraction solvent for duration of time (5-30 min) at a certain temperature (35-60 °C) with variable intensity stirring speed. 5 g of sieved peanut were mixed with solvent and was put on hot plate magnetic stirrer to extract the peanut oil. Mixture was centrifuged at 5000 rpm for 30 minutes as to separate the solid from the solution. Solution was placed in rotary evaporator to separate the oil sample and solvent. Samples were placed in desiccator until the weight is constant. Each experiment was performed in duplicate.

C. Optimization by Using Surface Response Method

Historical obtained data was selected as experimental design of response surface method (RSM, Design Expert 6.0.4 software). The oil yield was the response and a quadratic regression polynomial model was assumed to foresee the response. Central composite design (CCD) was used to study the influence of the stirring speed (X1), time involved (X2), the solvent to feed ratio (X3) and the temperature (X4) during the oil extraction process. The results of this design were used to generate a predicted model by fitting a second order polynomial, which is shown in equation. Analysis of variance (ANOVA) and analysis of factors (F test) were used to determine the suitability and statistical significance of the model.

$$y = b_o + \sum_{i=1}^{k} b_i\, x_i + \sum_{i=1}^{k} b_{ii}\, x_i^2 + \sum_{i=1}^{k=1} \sum_{j=i+1}^{k} b_{ij}\, x_i x_j + \varepsilon \tag{1}$$

where the Y is the predicted response (yield of the oil extracted), β_0 is a constant coefficient, βi is the coefficient of linear parameter, βii and βij are the interaction and second order coefficients, respectively. Xi and Xj are coded independent variables and ε is the residual error as shown in Equation 1 [20].

D. Determination of Oil Yield from Peanut Seeds

Oil yield of the peanut oil was determined by using Equation 2 [21]:

$$oil\ yield = \frac{mass\ of\ extracted\ oil}{mass\ of\ dried\ material} x100\% \tag{2}$$

III. RESULTS AND DISCUSSION

A. One Factor At a Time Method (OFAT)

The extraction result by using different solvent was shown in Figure 1. Extraction process by using ethyl acetate as solvent gave the highest oil yield (42%) followed by acetone (31%), methanol (26%) and ethanol (24%). According to Ibrahim, Omilakin, & Betiku (2019), oil extraction that was done by using sandbox seed, oil yield showed that ethyl acetate was the most efficient (72.20 ± 0.35 wt.%) followed by acetone (57.90 ± 1.27 wt.%) and *n*-hexane (56.25 ± 1.77 wt.%). Based on G. Catalkaya and D. Kahveci, extraction of lycopene from industrial tomato waste that was carried out using acetone, ethyl acetate, ethanol shown that ethyl acetate was the most suitable solvent as it gave the highest yield of lycopene [23]. Ethyl acetate is excellent for extraction process due to its low value of polarity and the process involve of polar and non-polar interaction [24]. Ethyl acetate has two chemical and biological characteristics including medium polarity and minimum toxicity which help to extract many polar and non-polar compounds [22]. Ethyl acetate is excellent solvent with low toxicity and safer to handle compared to n-hexane. From this information, ethyl acetate was selected as permanent solvent to be used during the study.

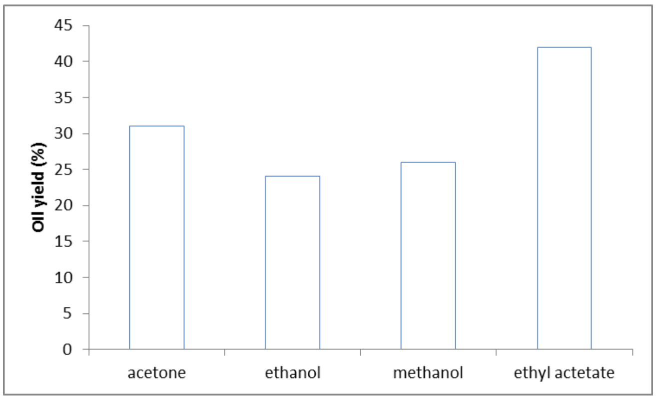


Figure 1: Effect of type solvents on the oil yield of the peanut seeds (parameter : S/F ratio 18 mL/g, extraction time 15 minutes, extraction temperature 45°C, extraction stirring speed 600 rpm)

As shown in Figure 2, the oil yield was not greatly increased with increasing S/F ratio from 6 ml/g to 37 ml/g. Significant increase in the oil yield was achieved from 6 ml/g to 18 ml/g, due to the concentration gradient between the solvent and the solid phase becomes greater which improved mass transfer, leading to higher oil yield [12]. The oil yield significantly increased from 46% to 56% by increasing the volume of solvent from 6 ml/g to 18 ml/g. As the volume of solvent increased from 24 ml/g to 36 ml/g, the oil yield begins to constants. This condition may due to maximum oil content in the seed have been extracted starting from 90 ml of ethyl acetate. According to Chokchai Muenmas et al (2018), efficiency of the extraction process was increased as the volume of solvent was increased and this occurrence may due to the concept of diffusion. Concentration gradient between solvent and the solute are the driving force of the mass transfer. As the volume of solvent increases, the diffusion rate will increase due to the increasing concentration gradient. This will result to higher extraction by the solvent but once it reached equilibrium, increasing the solvent volume will not increase the extraction process. The study was done by manipulating the volume of solvent (30-110 ml) and the efficiency of extraction process does not increase after the volume of solvent was higher than 90 ml [25]. As the solvent and solute reached equilibrium, the performance will be constant. Therefore, liquid/solid ratio of 18:1 or 90 ml of solvent would be sufficient for the extraction process.

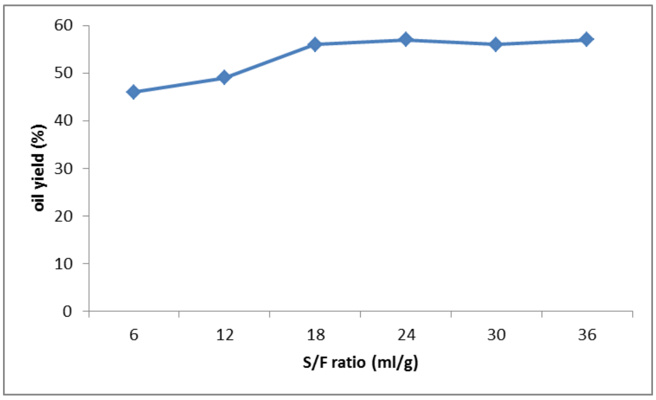


Figure 2: Effect of solvent to feed ratio on the oil yield of the peanut seeds (parameter : extraction solvent ethyl acetate, extraction time 15 minutes, extraction temperature 45°C, extraction stirring speed 600 rpm)

Extraction time is a crucial parameter in solvent extraction as a lower extraction time reduces both energy and the cost of the extraction process. Figure 3 shows the oil yield obtained at various stirring time. The oil yield increased from 52% to 58% with increasing stirring time from 5 minutes to 25 minutes. It was observed that further increase in extraction time to 30 minutes resulted in a minor drop in the oil yield. At the initial stage, none to low concentration of oil in the solvent cause the extraction rate to increase and the oil will diffuse more into the solvent. As the absorption of oil in the solvent increase and more than the concentration in the peanut seed, the extraction rate will decrease due to lower mass transfer and it started to reached equilibrium [26]. Thus, time of 25 minutes stirring was the optimum time for the experiment. Based on a study done in 2018, increased extraction time from 1 to 5 min increased the extracted oil from 11.73 ± 0.3% to 13.67 ± 0.2% due to increased contact time between the solvent and solute which increased the opportunity to extract the oil but the quantity of extracted oil changed insignificantly when the time was greater than 5 min. This show that extraction process has achieved equilibrium and additional time will not increase the oil yield [25].

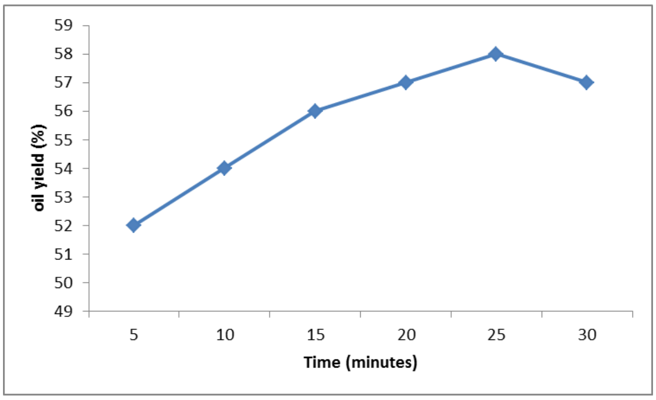


Figure 3: Effect of time on the oil yield of the peanut seeds (parameter : extraction solvent ethyl acetate, S/F ratio 18 mL/g, extraction temperature 45°C, extraction stirring speed 600 rpm)

As shown in Figure 4, as temperature increased from 35°C to 50°C, the oil yield was also increased due to an increase in oil solubility and diffusivity which normally improve the mass transfer. However, the oil yield begins to constant as temperature was further increased to 60 °C. The possible reason for this trend may be explained due to the volatilization of some ingredients existed in the oil and the solvent during extraction [2]. According to Jinfeng Zhong et al (2018), the oil yield increased as the extraction temperature was increased from 30 to 40 °C but the yield was significantly decrease as the temperature was further increase until 50°C [27]. In this study, it was observed that no significant difference in oil yield was observed from 50 to 60 °C. Therefore, temperature of 50 °C was chosen for the subsequent experiment studying the effect of stirring power on oil yield.

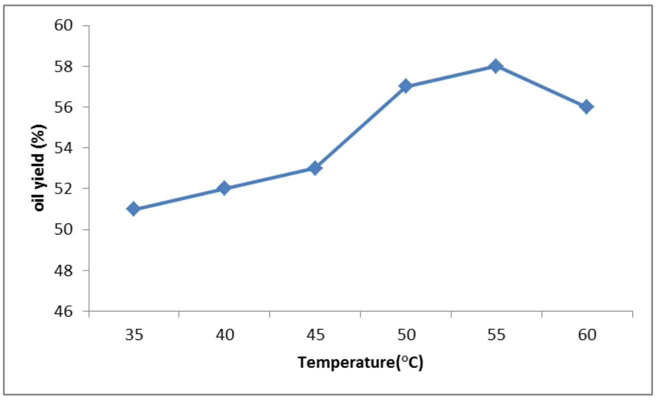


Figure 4: Effect of temperature on the oil yield of the peanut seeds (parameter : extraction solvent ethyl acetate, S/F ratio 18 mL/g, extraction time 25 minutes, extraction stirring power 600 rpm)

Based on Figure 5, the oil yield increased from 55% to 61% by using stirring power of 400 rpm to 700 rpm. As the strring power was increased to 800 rpm and 900 rpm, the production of oil extracted start to fall to 56% and 52%. This may due to rupture of bond in the solvent cause by excessive stirring. The oil yield increase only 1% from 600 rpm to 700 rpm, thus the optimum stirring power chosen would be 600 rpm as the value increased is not significant.

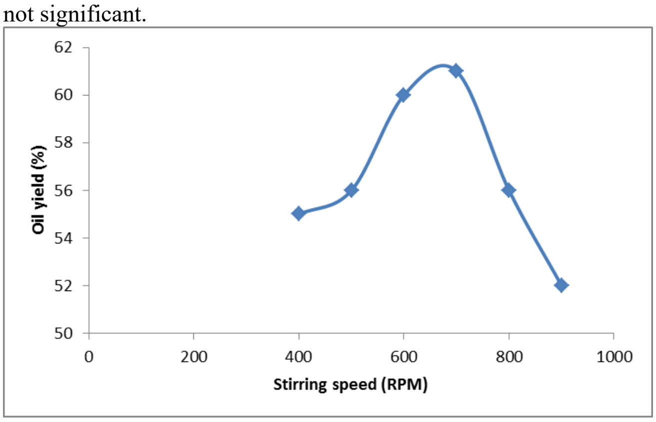


Figure 5: Effect of stirring speed on the oil yield of the peanut seeds (parameter : S/F ratio 18 mL/g, extraction time 25 minutes, extraction temperature 50°C, extraction stirring speed 600 rpm)

B). Fitting of The Response Surface Model (RSM)

The yield of peanut oil by using conventional method at different operating conditions optimized by RSM was run. Response surface method (RSM) was employed to determine the interactions between the independent variables (stirring speed, time, volume, temperature) and response (peanut oil yield) in order to optimize the extraction conditions. The design of experiments (DOE) was based on historical data design. The experimental data were used to calculate the coefficients of the second order polynomial equation. A three-factor, three-level Central Composite Design (CCD) was used to manage relationships between the variables [20].

Based on table 1, the "Pred R-Squared" of 0.8451 is in reasonable agreement with the "Adj R-Squared" of 0.9117. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable and as ratio in the experiment was 17.835, it indicates an adequate signal and the model can be used to navigate design space.

Table 1
Model summary statistics

Standard dev.	2.97	R-squared	0.9543
Mean	59.52	Adj R-squared	0.9117
C.V. %	4.99	Pred R-squared	0.8451
PRESS	448.38	Adeq Precision	17.835

Table 1

Based on table 2, the model F-value of 22.39 implies the model is significant. There is only a 0.01% 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 X_1, X_2, X_3, X_4, X_{13}, X_{14}, X_{23}, X_{11}, X_{22}, X_{33}, X_{44} are significant model terms. Within the model X_1 (stirring speed), X_2 (extraction time), X_3 (S/F ratio), X_4 (extraction temperature), X_{13} (stirring speed x S/F ratio), X_{14} (stirring speed x extraction temperature), X_{23} (extraction time x S/F ratio), X_{11} (stirring speed^2), X_{22} (extraction time^2), X_{33} (S/F ratio^2), X_{44} (extraction temperature^2) are the significant ($p<0.0001$) model parameter.

Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The "Lack of Fit F-value" of 0.26 implies the Lack of Fit is not significant relative to the pure error. There is a

96.71% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good as the the model was supposed to fit.

Table 2
ANOVA analysis of experimental data

Source	Sum of squares	df	Mean Square	F Value	p-value Prob>F	
Model	2762.87	14	197.35	22.39	<0.0001	significant
X_1	*110.36*	*1*	*110.36*	*15.52*	*0.0030*	
X_2	*332.05*	*1*	*332.05*	*37.68*	*<0.0001*	
X_3	*690.80*	*1*	*690.80*	*78.39*	*<0.0001*	
X_4	*223.80*	*1*	*223.80*	*25.40*	*0.0001*	
X_{12}	*4.22*	*1*	*4.22*	*0.48*	*0.4994*	
X_{13}	*60.76*	*1*	*60.76*	*6.90*	*0.0191*	
X_{14}	*316.48*	*1*	*316.48*	*35.91*	*<0.0001*	
X_{23}	*293.61*	*1*	*293.61*	*33.32*	*<0.0001*	
X_{24}	*23.33*	*1*	*23.33*	*2.65*	*0.1245*	
X_{34}	*0.15*	*1*	*0.15*	*0.017*	*0.8972*	
X_{11}	*66.44*	*1*	*66.44*	*7.54*	*0.0150*	
X_{22}	*23710*	*1*	*23710*	*26.91*	*0.0001*	
X_{33}	*156.98*	*1*	*156.98*	*17.81*	*0.0007*	
X_{44}	*51.63*	*1*	*51.63*	*5.86*	*0.0287*	
Residual	132.18	15	8.81			
Lack of fit	*45.11*	*10*	*4.51*	*0.26*	*0.9671*	*Not significant*
Pure error	*87.07*	*5*	*17.41*			
Total	2895.05	29				

Table 2: ANOVA analysis experimental data

Analysis of variance (ANOVA) showed that the behavior of the system was best approximated by a second order polynomial model. In equation 3, Y represents the predicted peanut oil yield, a_0 represent the model intercept terms, xi is the independent variables and ai is the model coefficient parameters [28].

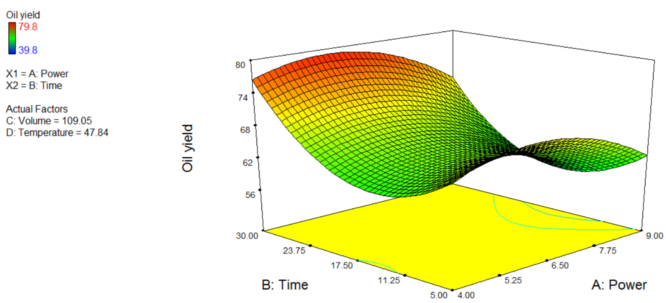
$$Y = a_0+a_1x_1+a_2x_2+ a_3x_3+a_{11}x_{12}+a_{22}x_2^2+a_{33}x_3^3+ a_{12}x_1x_2 a_{13}x_1x_3+ a_{23}x_2x_3 \quad (3)$$

The second order polynomial model in this study showed the relationship between the independent variables of stirring speed (X_1), time involved (X_2), the solvent to feed ratio (X_3) and the temperature (X_4) during the oil extraction process and the dependent variable which is peanut oil yield (*Y*) as,

$$Y= 64.17 - 2.48X_1 + 4.29X_2 + 6.19X_3 + 3.53X_4 - 0.51X_{12} - 1.95X_{13} + 4.45X_{14} + 4.28X_{23} - 1.21X_{24} - 0.097X_{34} - 5.06X_{11} + 9.57X_{22} - 7.78X_{33} - 4.46X_{44} \quad (4)$$

The figures below show the interaction between the parameters and oil yield. Based on figure 6(a)(1) and 6(a)(2), the oil yield increases as the stirring speed/power and extraction time increases. The highest yield was at 650 rpm and 30 minutes. This may due to enhanced cavitation which has improved the mass transfer and better oil extraction by the solvent. After 650 rpm of stirring speed, the oil yield starts to decrease. At high stirring speed, cavitational bubble collapse is extra violent [29]. If temperature and pressure are high inside bubble, the collapse of bubbles happened over a very diminutive time. This could develop solvent penetration into the feed and enhance the oil extraction by disrupting the cell walls [7]. As the stirring power was further increase, the oil yield start to decrease. This condition may due to plant material degradation [30]. Based on a study done by Somayeh Rahimi and Mohaddeseh Mikani, the greatest lycopene amount was reached at higher ultrasonic power and extended ultrasonic times, from 1.59 to 10 min, while reduced from 10 min and longer extracting time [23] which similar to this study.

(a)(1)



(a)(2)

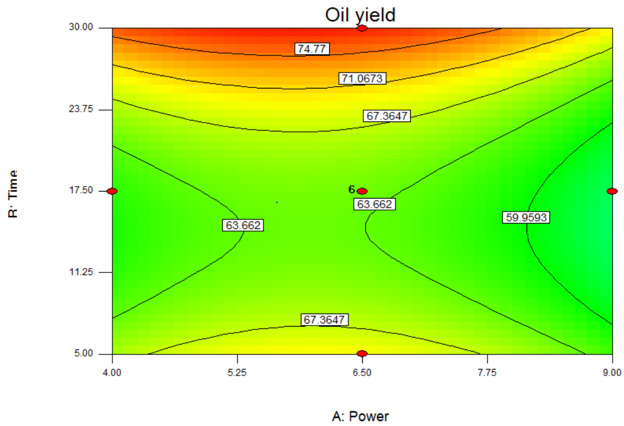
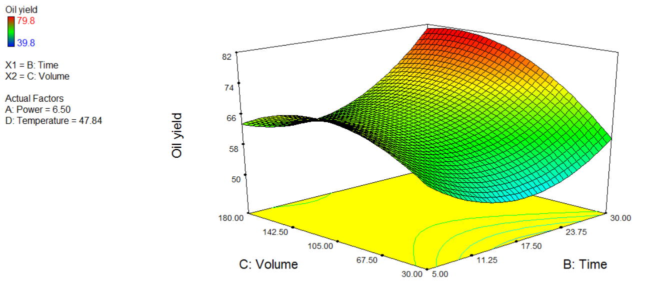


Figure 6 (a)(1) and 6(a)(2): Response surfaces plot: Interaction plot of stirring speed/power and extraction time

Based on figure 6(b)(1) and 6(b)(2), the oil yield was achieved the highest as the extraction time and volume of solvent (S/F ratio) increase. The highest oil yield achieved at 27°C and 142.50 ml of solvent/28.5 ml/g of S/F ratio. This may due to present of large concentration gradient between the solvent and the solid phase becomes greater which improved mass transfer, leading to higher oil yield [12]. The yield start to constant as the volume of solvent (S/F ratio) and extraction time increased and this may due to the oil gradually more saturated so that the solubility of peanut oil decreased [29].

(b)(1)



(b)(2)

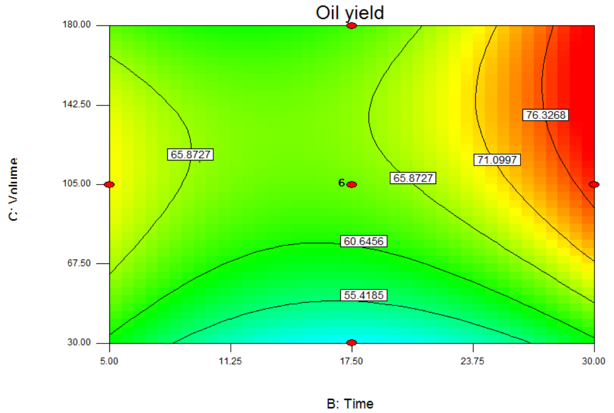
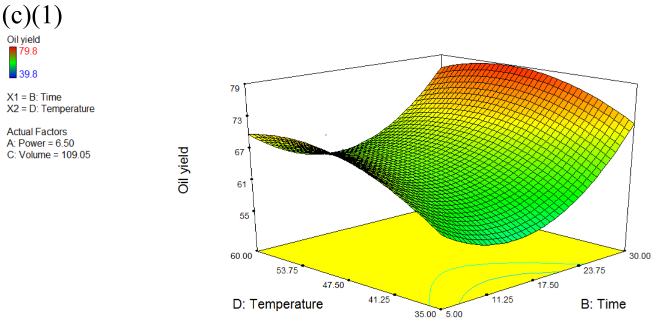


Figure 6 (b)(1) and 6(b)(2): Response surfaces plot: Interaction plot of volume of solvent(S/F ratio) and extraction time

Based on figure 6(c)(1) and 6(c)(2), the oil yield increased as the extraction time and extraction temperature increased. As the temperature increased, the solubility and diffusivity was improved. However, the oil yield begins to constant as temperature was further increased and this may due to the volatilization of some ingredients existed in the oil and the solvent during extraction [2]. Present of impurities such as insoluble substances that suspended in the extract will also lower the permeability into cell structures[31].

(c)(1)



(c)(2)

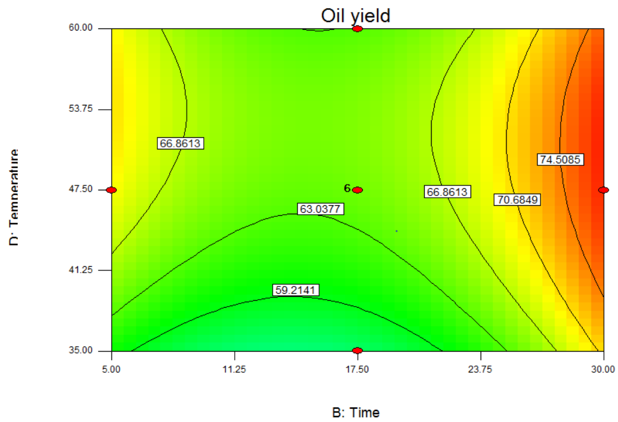


Figure 6 (c)(1) and 6(c)(2): Response surfaces plot: Interaction plot of extraction temperature and extraction time

In order to maximize the oil yield, numerical optimization was being done. The parameters that were chosen were 705 rpm, 30 minutes, 174.92 ml of ethyl acetate and operate at 56.20 ^{O}C. The oil yield that should be extracted by this condition was 79.80% but due to limitation of the equipment used, the yield achieved was only 77.23%. There was error as much as 0.7%.

IV. Conclusion

In conclusion, the extraction yield increases proportionally with increasing the parameters up to a limit before the rise becomes insignificant or decline. In OFAT, the optimum parameters that used in this study were ethyl acetate as the solvent, S/F at 18 ml/g, conducted at 50 ^{O}C for 25 minutes and by using 600 rotational per minute (rpm) of power and after the process were further optimized by surface response method (RSM), the maximum oil yield occur at 705 rpm, 30 minutes, 174.92 ml of ethyl acetate and operate at 56.20 ^{O}C.

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