RICINUS CUMMUNIS AS FEEDSTOCK FOR RAW VEGETABLE OIL EXPRESSION VIA MECHANICAL EXTRACTION PROCESS: OPTIMIZATION STUDY

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ABSTRACT

Crude castor oil (CCO) extracted by Mechanical extraction process (MEP) using mechanical screw press has attracted much attention and widely recognized as one of the useful methods of expressing oil from any feedstock. Therefore, in this study, the use of Ricinus Cummunis as feedstock to produce raw vegetable oil is studied. This study was performed using design of experiment (DOE), emphasis is on response surface methodology (RSM) based on three independent variables at five levels of central composite design (CCD) with α = 2. The extraction process variables are reaction temperature (40-200 ^{o}C), force (550-750 kN) and mass (20-100 g). Interactions between the variables were found to possess' significant effect on the yield of crude castor oil. However, a coefficient of determination (R2) value of 0.99 shows the fitness of a second-order model for this study. The mathematical model developed is found to be adequate to describe the experimental parameters range and also provide a statistically accurate prediction of the optimum expression of oil yield. At 199.45 ^{o}C reaction temperature, 745.38 kN force and mass of 99.88 g, an optimum crude oil yield of 31.01% at 0.9 efficiency model prediction was obtained, which means mechanical extraction process has the ability as a cost effective process to express oil from any feedstock containing oil.

Keywords: Biodiesel; central composite design(CCD); Crude castor oil (CCO);Mechanical extraction process (MEP);Ricinuscummunis.

1.0 Introduction

Vegetable oil, animal fat and waste vegetable oil have attracted much global attention as a basic feedstock in producing biodiesel (Suranchai and Gumpon, 2011). Much research in recent years has focused on procuring cheap, available and most especially non edible oil crops as feedstock for its production because, low cost biodiesel depends mainly on the cost and nature of raw material (Cynthia *et. al.,* 2012) and the process or technology used for the production (Khalizani and Khalisanni, 2011). For more than 100years, researchers have been stressing the use of traditional ways of extraction oil most especially mechanical extraction process. Numerous experiment have established the turbidity effect of product of mechanical extraction process (Sukhdev,*et al*., 2008), nevertheless purification by boiling had been adopted to remove the foot from the oil obtained.

In an attempt to look for a cost effective process that will not make the downstream recovery and purification very difficult and expensive, an alternative approach is necessary which thiswork focuses on. The approach aim at analyzing the reaction variables involve to attain optimal conditionsat which the mechanical extraction process will give a quality yield.

2.0 Experimental and Methods

2.1 Materials

Production of crude castor oil was carried out in the Chemical and Mechanical Engineering Departments, Ladoke Akintola University of Technology, Ogbomoso, Nigeria. The raw castor seeds were obtained from Masifa village, near Ogbomoso city. It was sorted and sun dried to reduce the moisture content.

2.2 Pretreatment

The dried seeds obtained were cleaned, decorticated, and cooked at 80^{o}C under air tight conditions for twenty (20) minutes using water bath, this is done to reduce the protein content in the seeds. Then oven dry at 100^{o}C to further reduce the water content. The oven dried seeds were then grounded into powdered form using already washed and dry mortar and pestle.

2.3 Experimental Design of Mechanical Extraction Process

The factors for consideration here for the oil yield are reaction temperature, force and mass. However, these three factor parameters were found to be oflevel of medium quality standards and optimum performance. Response surface methodology (RSM) was employed to evaluate the effect of various parameters on the extraction process. The experimental design for this reaction was carried out by utilizing a central composite design (CCD). A CCD with three independent variables at five levels was employed and the total number of experiments was 20 (=2^k+2k+8) where k is the number of independent variable (Rashid *et. al.*, 2009) which included 8 factorial points, 6 axial points and 6 center points. The chosen independent parameters for the optimization in this study are reaction temperature (X_1), the force (X_2), mass (X_3). The reaction temperature selected is in the ranges 40-200°C, force 550-750kN the mass 20-100g. The response measured was the oil yield (%) obtained from mechanical extraction process of crude castor seed. The range and levels of the variables to be investigated in this study are presented in Table 1 the value of α (alpha) was fixed at 2, while combination of variables consists of one at its lowest (-2) level or highest (+2) level with other variables at zero level constituting the axial points.

Table 1: Central Composite Design Process Variables for Mechanical Extraction Process.

Independent variables	Level				
	- α (-2)	-1	0	+1	+ α (+2)
X_1: Temperature (°C)	40	80	120	160	200
X_2: Force (kN)	550	600	650	700	750
X_3: Mass (g)	20	40	60	80	100

Transformation of variable levels from coded (X) to un-coded could obtained as: X_1= 120+40X, X_2= 650+50X, X_3= 60+20X,

2.4 Experimental procedure

Weigh 20g of Castor powder into the oven tray, oven dry the powder at 150°C for one hour thirty minutes using Laboratory Oven:SM 9053A Laboratory oven Surgifield Medical England. After the desired time is attained, the dried powder was packed into a muslin cloth, tied and placed in between the expeller of the mechanical screw press machine; HFI Model**,** Ram diameter 182m, Load in kN Made in New Delhi, India. Force was applied on the powder through the lever. The oil expressed was collected, weighed and set aside for further analysis.

2.5 Statistical analysis

The CCD procedures used to obtain the experimental data (Table 2) were analyzed by the response surface methodology (RSM) using the second-order polynomial equationbelow, which was developed to describe the relationship between the independent variables of the mechanical extraction process to the predicted response variable (crude oil yield).

$$\boldsymbol{R = \varphi_o + \sum_{i=1}^{k} \varphi_i x_i + \sum_{i=1}^{k} \varphi_{ii} x_i^2 + \sum_{i>j}^{k} \sum_{j}^{k} \varphi_{ij} x_i \ x_j + \in \ldots\ldots\ldots\ldots (1)}$$

Where **R** is the response, (crude oil yield),$\boldsymbol{\varphi_o}$; $\boldsymbol{\varphi_i}$, $\boldsymbol{\varphi_{ii}}$ and $\boldsymbol{\varphi_{ij}}$ are intercept, linear, quadratic and interaction constant coefficients respectively. $\boldsymbol{x_i}$ *and* $\boldsymbol{x_j}$ aretheencoded independent variables, k is the number of factors studied and optimized in the experiment. ∈ is the random error.For regression and graphical analysis of the experimental data, statistical Design Expert Software Version 6.0.8 (STAT-EASE Inc., Minneapolis, U.S.A) was used and the statistical analysis of the model was done to evaluate the analysis of variance (ANOVA). The coefficient of determination (R^2) was used to determine the quality of the fit of the model and a response surface plot was developed using a fitted quadratic polynomial equation obtained from regression analysis.

Table 2:Experimental design matrix and results

Std	A: Temperature (°C)	B: Force (kN)	C: Mass (g)	Oil Yield (wt %)	Predicted Value (wt %)
10	280.00	650.00	60.00	23	6.06
	120.00	650.00	60.00	23	7.86
20	120.00	650.00	60.00	23	9.94
12	120.00	850.00	60.00	23	19.23
14	120.00	650.00	140.00	28	22.86
8	200.00	750.00	100.00	31	27.15
7	40.00	750.00	100.00	19	19.23
4	200.00	750.00	20.00	19	31.03
6	200.00	550.00	100.00	27	22.91
19	120.00	650.00	60.00	23	15.08
11	120.00	450.00	60.00	15	22.83
3	40.00	750.00	20.00	10	27.91
2	200.00	550.00	20.00	8	22.99
16	120.00	650.00	60.00	23	22.99
17	120.00	650.00	60.00	23	22.99
18	120.00	650.00	60.00	23	22.99
1	40.00	550.00	20.00	6	22.99
5	40.00	550.00	100.00	23	22.99

3.0 Results and Discussion

3.1 Development of Regression Model Equation

A quadratic polynomial equation (in coded form) was obtained from the design experimental data equation 2 was generated to produce the oil yield.

3.2 Final Equation in Terms of Coded Factors:

$$Y = +22.99 + 3.4 * A + 1.94 * B + 7.15 * C - 1.72 * A^2 - 1.01 * B^2 - 2.34 * C^2 + 1.88 * A * B + 0.6 * A * C - 1.87 * B * C \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots . (2)$$

Y is the response variable, that is the yield and the actual values of predictors are temperature, force and mass which were represented by **A, B** and **C** respectively. The positive sign in front of the terms indicates synergistic effect while the negative sign indicates antagonistic effect.

3.3 Model Adequacy Checking

Performance of statistical analysis was done in order to evaluate the analysis of variance (ANOVA) and to check the model adequacy. Here 95% confidence level was adopted; the coefficients of the response surface model as given in equation 1 were also evaluated. The probability of error value (p-value) was used as a tool for checking the significance of each coefficient (strength of interaction of each parameter). In relation to table 3, the p-value of the model was less than 0.0001 which shows the high significance in predicting the value of response and suitability of the obtained model.For a model term p-value < 0.05 indicates the significance at the confidence interval of 95%. The model F-value of 2978.90 implies the model is significant and there is only a 0.01% chance that a "model F" value this large could occur due to noise.

Table 3: ANOVA For Response Surface Quadratic Model

Source	**Sum of Squares**	**DF**	**Mean Square**	**F Value**	**Prob > F**	
Model	776.21	9	86.25	2978.90	< 0.0001	significant
A	*101.25*	*1*	*101.25*	*3497.29*	*< 0.0001*	
B	*60.06*	*1*	*60.06*	*2074.54*	*< 0.0001*	
C	*448.19*	*1*	*448.19*	*15480.33*	*< 0.0001*	
A^2	*34.86*	*1*	*34.86*	*1204.08*	*< 0.0001*	

B^2	*25.09*	*1*	*25.09*	*866.68*	*< 0.0001*
C^2	*64.86*	*1*	*64.86*	*2240.27*	*< 0.0001*
AB	*28.13*	*1*	*28.13*	*971.43*	*< 0.0001*
AC	*3.13*	*1*	*3.13*	*107.94*	*< 0.0001*
BC	*28.13*	*1*	*28.13*	*971.43*	*< 0.0001*
Residual	0.23	8	0.029		
Lack of Fit	*0.23*	*3*	*0.077*		
Pure Error	*0.000*	*5*	*0.000*		
Cor Total	776.44	17			

Table 4: Analysis of variance (ANOVA) for response surface model

Source	Sum of Squares	Degrees of Freedom	Mean squares	*F*-value	*P*-value
Model	776.21	9	86.25	2978.90	< 0.0001
Residual	0.23	8	0.029		
Lack of Fit	*0.23*	3	*0.000*		
Pure Error	*0.000*	5	1.5		
Cor Total	776.44	17			
C.V. = 0.83%	R^2= 0.9997	Adj. R^2= 0.9994	Pred R^2= 0.9948		

Lack of fit according to Table 4, is the weighted sum of the squared deviation between the mean response at each parameter level, and the corresponding fitted value. Here, there was no significance relative to a pure error. At the other end, the low value of the coefficient of variation (C.V= 0.83) indicatedthat the result of the fitted model are reliable.

The quality of model developed could be evaluated using correlation coefficient. The coefficient of determination (R^2) shows that 99.97% the experiment data confirm compatibility with the predicted data of the model (R^2) is always between 0 and 1 and its magnitude indicates the aptness of the model, from a good statistical model, the R^2 value should be close to 1.0). Adjusted coefficient of determination value reconstructs the expression with all the significant terms included. Here, the value of regression coefficients R^2=0.9997 and Adj. R^2 which is 0.9994 shows a high correlation between the experimentally observed and predicted values and also give details of any variability in the response(Hassan*et al;* 2013, Ittipon *et. al.;* 2011).

The significance of each parameter in the model can be accessed using p-value. From table 3, any p-value less than 0.05 indicate the significance of such term. It can be seen from the p-value obtained for each model term that there are three linear coefficient of A, B and C, three quadratic coefficients of A^2 B^2 and C^2 and three cross- product coefficient of AB, AC and BC were all significant at the 1% level i.e. p-value <0.001.

Figure 1a depicts the normal probability plot of residual and studentized residuals. It shows that there is a characteristic compression of constant variables in the data (Ittipon et. al.; 2011). Figure 1b gives a detail picture of actual values obtained from the experiment against the predicted values by applying the model equated developed. It shows that the regression model equation gave an accurate description of the experimental data, in which all the plotted points are very close to the line of perfect fit (Bello and Ahmad, 2011c). This resulted in adequate capturing of the correlation between the three mechanical extraction variables to the yield of raw oil.

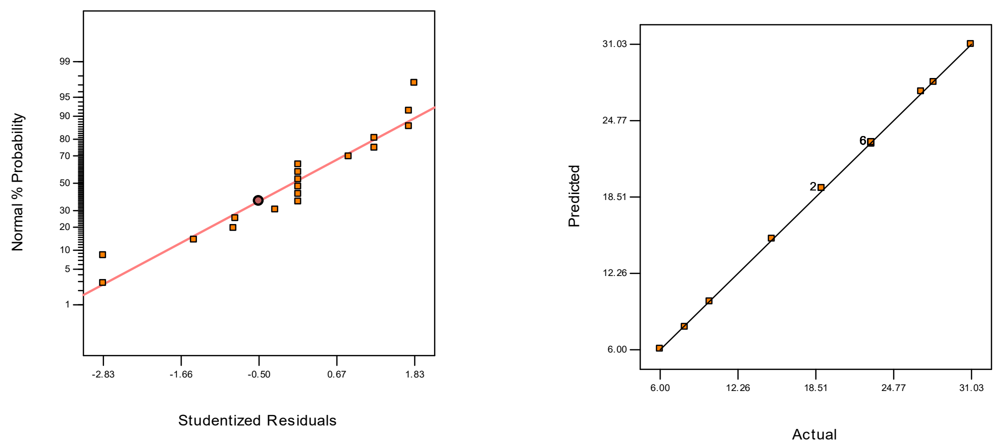

Figure 1(a and b): Normal probability plot of residual and studentized residual

3.4 Influence of the Parameters on the Yield of Crude Oil

Mechanical extraction process was significantly affected by different interacting between the process variables based on the analysis of variance most especially reaction temperature (A) and mass (C). This resulted from the application of design of experiment in capturing the interaction between the variables that affect the extraction process.

3.4.1 Effect of individual process variables

There is direct proportionality of yield on mass of lump of castor meal used at the actual factor of temperature of 120^{o}C and force of 650kN. It could be deduced from figure 2a that as the mass of castor lump increases so also the percentage of crude oil obtained increases. A yield of 13.25% was obtained at 20g of castor meal and 28.25% of oil at 200^{o}C reaction temperature. This shows that the more the quantity of castor meal, the more the oil to be obtained.

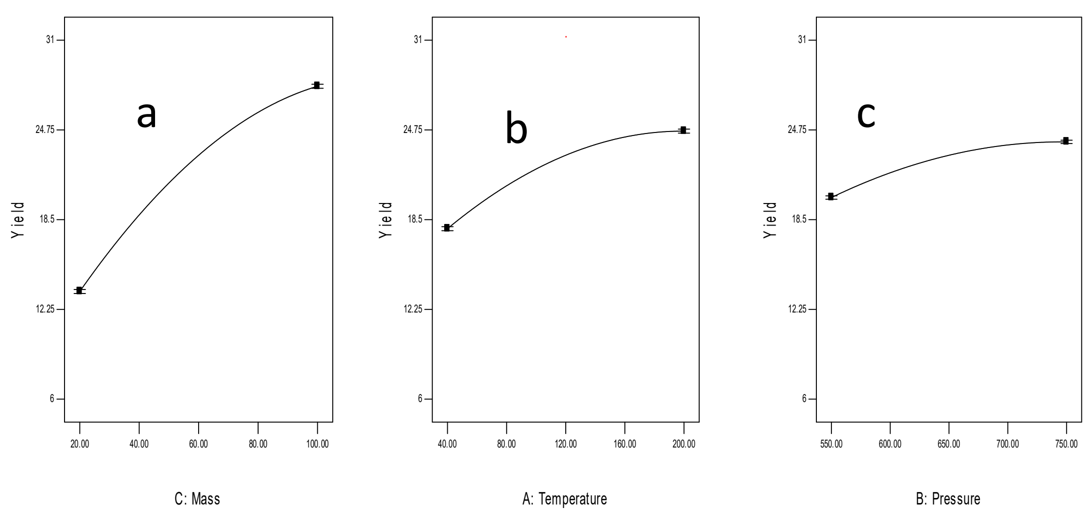

Figure 2(a-c): Effect of Individual Process Variables Yield of Crude Oil

The reaction temperature effect on the yield of crude oil is shown in figure 2b, at the actual factors of force of 650kN and mass of 60g. It can be established that as the reaction temperature increases, so also the yield of crude oil significantly increases from 18.0% at 40^{o}C to 24.70% at 200 ^{o}C.(Felix and Clement, 2011) This is due to the high diffusion of oil out of the meal at higher temper

ature. The interphasial bonds between the oil molecules and the solid are broken so that the oil will be released and more yields are obtained(Ramli *et al.,* 2012).

Figure 2c shows the effect of force (550-750kN) on the yield of extraction process at the actual factor of temperature 120 ^{o}C and mass 60g. It could be estimated that with increased in force of extraction there is increase in the yield of crude castor oil. The yield of oil increases from 20.75% at 550kN to 24% at 750kN the result reveals that, as the force was being applied, the oil was expressed,but at about 700kN most of the quality of oil therein had been totally expressed. Between a force of 700kN and 750kN, a little quantity of oil was obtained, eventually, the cake was found to be dried of crude oil.This was found to be similar to report of Alfaro *et. al.;* (2003), they established the fact that higher energy level does not bring about any benefits once enough energy has been applied to affect the rupture of material structure in order to release the chemicals therein and Li *et al.* (2012) who reported that at high level of energy the yield of anthocyanins reached a plateau.

3.4.2 Effect of Interaction between Process Variables

As it was earlier discussed, process variables of extraction have great interaction effects on the yield of crude oil, most especially temperature and mass. The phenomenon still indicated by high F-value of ANOVA ofmass and reaction temperature were 15480.33 and 3497.29 respectively. Figure 3a shows that at actual force of 650kN, lower temperature (40 ^{o}C) and little quantity of mass 20g gives low yield of 8% and at higher temperature the yield was 15%. But at higher quantity of lump of castor meal, 100g even at lower temperature the yield was 23.75%, at higher temperature and higher quantity of castor meal, the yield was 31%. This is in line with work of Yee and Lee (2008), which confirmed that reaction temperature has great effect on yield of fatty methyl ether.

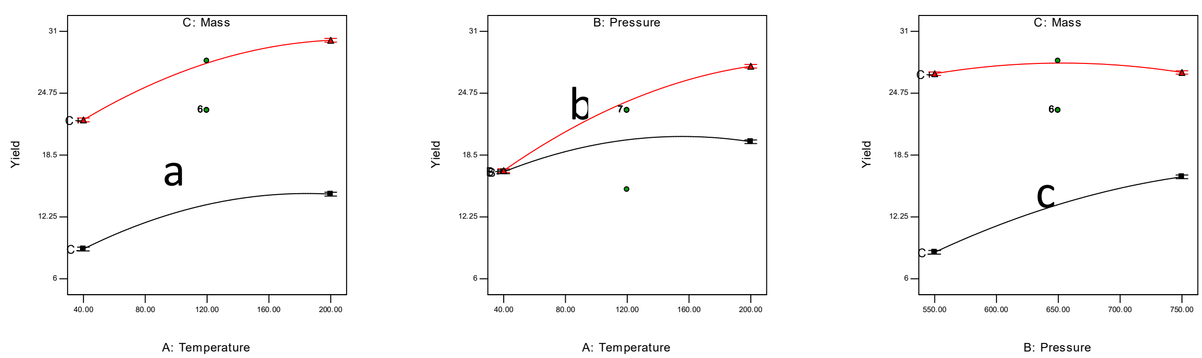

Figure 3(a-c): Interaction between Process Variables

The interaction effect of reaction temperature and force was depicted in figure 3b, at actual mass of 60 g at low reaction temperature of 40^{o}C and force of 550kN, the yield was 17.5% even when the reaction temperature increased to 200^{o}Cwith little forceeffected, a little increase in yield of 19.5 % was obtained. At higher force 750kN the yield increases to 27.75% which means, if the necessary reaction temperature is applied, there must be enough force to express the oil out of the lump of castor meal, so as to obtain a qualitative and quantitative yield of crude oil.

The interactive effect of force and mass on yield is shown on figure 3c at actual temperature of 120 ^{o}C. At 550kN of force on 20g of meal of castor seed, there is ascending progression on yield as the force increased to 750kN. Thus the yield was 8% at force of 550kN and 17.56 % at 750kN. At higher quantity of mass(100g)the yield was 25.5% at force of 550kN, it could be observed that the yield was still at 25.5% at 750kN (higher force)this is because at 650kN the quantity of oil present in the mass of castor meal had been totally expressed, so no matter the amount of force one might exerted there will not be any increase in yield, this is reflected in the curve obtained in figure 4c, which shows the significant of the variables in the model. The phenomena is still indicated by 15480.33 and 3497.29 of F-values in the ANOVA (Table 3) which are highest and higher values for both mass and reaction temperature respectively.

3.5 Optimization of Process Variables

Table 5:Verification Experimental at Optimum Conditions.

Optimum condition			Oil Yield (%)	
Temperature	Force	Mass	Experimental Predicted	Model Efficiency
199.45^{o}C	745.38kN	99.88g	31.01%	0.90

The result obtained above has shown that the two variables out of the mechanical extraction variables affected the yield of crude oil, especially the interaction among the variables. And to optimize the process variables in order to obtain the highest yield using the model regression developed. With the point prediction function of model at 0.90 efficiency (Table 5); reaction temperatureof 199.45^{o}C,force of 745.38 kN mass of99.88g, and an optimum crude

4.0 Conclusion

As a result of the experimental result obtained, it was found that all the factors shown asignificant interaction effects on the yield of crude castor oil. Application of response surface methodology (RSM) to the model to optimize condition for extraction was successful. The effect of mass and temperature were found to be most significant for yield but reaction force had a little importance. At 199.45^{o}C reaction temperature, 745.38 kN of force and 99.88g mass, an optimum crude oil yield of 31.01% was obtained using a point prediction function of 0.90. Therefore, it could be concluded that mechanical extraction process is an effective method for the expression of oil from any green feedstock containing oil such as castor seeds.

REFERENCES

Alfaro, M. J; Bélanger, J. M. R and Padilla, F. C (2003). Influence of solvent, matrix dielectric properties and applied power on the liquid-phase microwave-assisted process. (MAPTIM), extraction of ginger; Zingiber officiale. Journal of Food Research International, 36: 499-504.

Angelo C. Pinto; Lilian L. N. Guarieiro; Michelle J. C. Rezende; Núbia M. Ribeiro; Ednildo A. Torres; Wilson A. Lopes; Pedro A. de P. Pereira; Jailson B. de Andrade*, (**2005**)Biodiesel: an overview. J. Braz**.** Chem. Soc. vol.16 no.6b São Paulo Nov. /Dec.

Bello, O.S. and Ahmad, M.A. (2011c) Response Surface Modelling and Optimization of Remazol Brilliant Blue Reactive Dye Removal Using Periwinkle Shell-Based Activated Carbon, Separation Science and Technology, 46: 15, 2367-2379

Castor Bean *Ricinus Communis* An International Botanical Answer To Biodiesel Production & Renewable Energy. www.dovebiotech.com

Cynthia Ofori-Boateng, Ebenezer M. Kwofie and Moses Y. Mensah (2012) Comparative Analysis of the Effect of Different Alkaline Catalysts on Biodiesel Yield. World Applied Sciences Journal 16 (10): 1445-1449.

Felix U. Asoiro, M.Eng.* and Clement O. Akubuo, Ph.D. (2011), Effect of Temperature on Oil Extraction of *Jatropha curcas* L. Kernel. *The Pacific Journal of Science and Technology.12 (2):456-463*

Hassan, S.Z., Chopade S.A. and Vinjamur, M. (2013).Study of Parametric Effects and Kinetic Modeling ofTrans-esterification Reaction for Biodiesel Synthesis. *Research Journal of Recent Science.2:* 67-75.

Ittipon W. Prachasanti T. and Kulachate Pianthong. (2011) SWU Engineering Journal 6(1),16-30.

Khalizani Khalid and Khalisanni Khalid. (2011) Transeterification of Palm Oil for the Production of Biodiesel. American Journal of Applied Sciences 8 (8): 804-809.

Li Y.; Xu X,; Wang J.; Wang Z. and Chen F. (2012**)**.Kinetics and thermodynamics characteristics of microwave assisted extraction of anthocyanins from grape peel.Transactions of the Chinese Society of Agricultural Engineering. 28: 326-332.

Ramli, M. Rubyatul, A. S. Mahadhir, M. and Anwar, J. (2012). Solid Catalyst and Their Application in Biodiesel Production. Bulletin of Chemical Reaction Engineering & Catalysis. 7 (2): 142-149.

Rashid U, Anwar F and Arif M, "Optimization of base catalytic methanolysis of sunflower(*Helianthus annuus*) seed oil for biodiesel production by using response surface methodology", *Ind.Eng.Chem.Res,* Vol. 48, pp. 1719-1726, (2009).Rebeiro, A., Castro F., and Carvalho. J. (2011). Waste:Solution, Treatment and Opportunities. 1st International Conference. September 12th-14th.

Sukhdev Swami Handa, Suman Preet Singh Khanuja, Gennaro Longo, Dev Dutt Rakesh. (2008) Extraction Technologies for Medicinal and Aromatic Plants. United Nations Industrial Development Organization .
and the International Centre for Science and High Technology, International Centre for Science and High Technology Trieste, 2008

Surachai Jansri and Gumpon Prateepchaikul. (2011) Comparison of Biodiesel Production from High Free Fatty Acid and Crude Coconut Oil via Saponification Followed by Transesterification or A Two-Stage Process. Kasetsart Journal (Natural science). 45: 110-119.

Yee Kian Fei, and Lee Keat Teong (2008) Palm Oil Feedstocks for Biodiesel Production via Heterogeneous Transesterification: Optimization Study. International Conference on Environment Vol. 3 No 2 June, pp 382-387