

Process Parameter of Biodiesel from Kapok Seed Oil by Roto Matric Magnetic Stirrer Assisted Method

Prabu M.¹, Karthikeyan N²

^{1,2}*Department of Mechanical Engineering, Karpaga Vinayaga College of Engineering and Technology, Kancheepuram, Tamilnadu, India*

Abstract - Energy consumption is not expected to decrease in this century, because the world population is increasing and the economics of developing centuries are expanding rapidly. In contrast, the source and supply of primary energy sources like coal, oil and natural gas seems to decrease to a critical point. Alternate fuels will fulfill such demands in future. Biodiesel is quickly becoming one of the fastest growing alternative fuels in the world. Biodiesel from kapok seed oil by transesterification process is a mixture of calcium oxide(Cao) and ethanol. The high viscosity component, glycerol, is removed and hence the product has low viscosity like the fossil fuels. The transesterification can be done in a number of ways such as using an alkalai catalyst, acid catalyst, bio catalyst, heterogeneous catalyst in their supercritical state. The transesterification of kapok seed oil was carried out by using rotary magnetic stirrer method in the presence of ethanol as a reagent and calcium oxide(Cao) as the catalyst. Application of rotary magnetic stirrer method provides easy way to get a biofuel with advantages of enhancing reaction rate and improving the separation process. Creating biodiesel in a sustainable manner will allow this clean, renewable, and cost effective fuel to help ease the world through increasing shortages of petroleum, while providing economic and environmental benefits well into the future.

Keywords: kapok seed oil, esterification, magnetic stirrer, transesterification.

I. INTRODUCTION

Biodiesel is an alternate fuel for diesel engine, where it is produced by chemically reacting a vegetable oil or animal fat with an alcohol such as ethanol [1]. The early diesel engines had complex injection systems and they are designed to run on many different fuels, from kerosene to coal dust. It was only a matter of time before someone recognized that, because of their higher energy content, vegetable oils would make excellent fuel.

1.1. The Future of Biodiesel

Due to its clean emissions profile, ease of use, and many other benefits, biodiesel is quickly becoming one of the fastest growing alternative fuels in the world. With animal subsidy biodiesel is cost competitive with petroleum diesel, and millions of users have found and enjoyed the benefits of the fuel. The future of biodiesel lies in the world's ability to produce renewable feedstock such as vegetable oils and fats to keep the cost of biodiesel competitive with petroleum.

1.2. Methods for the Production of Biodiesel

Many methods are used to produce the biodiesel such as

1. Pyrolysis
2. Micro emulsion
3. Dilution
4. Trans-esterification

1.2.1 Pyrolysis

Pyrolysis refers to chemical change caused by application of heat to get simpler compound from a complex compound. This process is also known as cracking. Kapok seed oil has been cracked to reduce the viscosity and to increase the cetane number. According to the operating conditions the pyrolysis process can be subdivided into three types such as

1. Conventional pyrolysis
2. Fast pyrolysis
3. Flash pyrolysis

1.2.2 Micro-Emulsification

Micro-emulsification is the formation of micro-emulsions and it is the potential solution for solving the problem with the oil has the high viscosity. Micro-emulsification is another technique that has been reported to produce bio-diesel and components of bio-diesel micro-emulsion includes diesel fuel, vegetable oil and alcohol with suitable proportions. Alcohol such as ethanol used as viscosity lowering additives. Viscosity reduction, increase in cetane number encourages the usage of micro-emulsions

1.2.3 Dilution

The vegetable oil is diluted with petroleum diesel to run the engine. Caterpillar, Brazil, in 1980, used pre combustion chamber engines with the mixture of 10% vegetable oil to maintain total power without any alteration or adjustment in engine. The low viscosity is good for better performance of an engine, which decreases with increasing the percentage of diesel.

1.2.4 Transesterification

The most popular method for extracting biodiesel is the trans-esterification process. Biodiesel is obtained by transesterification process is a mixture of mono-alkyl esters of higher fatty acids. It consists of three consecutive reversible reactions where diglycerides are converted into monoglycerides followed by the conversion of monoglycerides into glycerol. The transesterification process requires a catalyst to split the oil molecules such as ethanol to combine with the separated esters. A catalyst is used to increase the reaction rate and yield. [1]

II. MATERIALS AND METHODS

The materials used in this investigation is

1. Kapok oil
2. Ethanol
3. Calcium oxide catalyst
4. 250 ml conical flask
5. Magnetic stirrer
6. Thermometer
7. Sulphuric acid

The kapok oil was esterified from 1.6 to 0.4 acid value by heating 300 g of oil with 0.5% (w/w of oil) and 0.1 g of concentrated sulphuric acid on a magnetic stirrer for 3 hours at 55°C.

2.1 Methods

2.1.1 Water Heating

Suitable amount of water was taken into a container flask and heated till temperature rises to 70°C.

2.1.2 Purification of Kapok Seed Oil

Suitable amount of kapok seed oil was taken into the container flask containing hot water, and wait for 30 minutes still impure particles settled down.

2.1.3 Separation of Kapok Seed Oil

Hot water containing kapok seed oil was collected into the separating funnel and shaken it vigorously for 5-10 minutes. Because of low density of kapok seed oil settles on the top of the funnel and high density water and impurities settled at bottom of the funnel. Then disperse water and impure particles by opening the funnel valve, after that the purified kapok seed oil was collected into the jar, Removal of moisture from purified kapok seed oil. Collected purified kapok seed oil was heated up to 65°C to remove moisture contained in oil.



Fig 2.1 Water and oil separation process

III. EXPERIMENTAL SETUP

Transesterification was carried out in a batch reactor consists of the following components



Fig 3.1 Batch reactor

1. Magnetic stirrer with heater arrangement
2. Single necked round bottomed glass beaker
3. Temperature controller
4. Stirrer controller

Single necked round bottomed glass beaker is used to collect sample of mixture (oil+ethanol+catalyst) magnetic stirrer and heater provides the stirring and heating effect simultaneously. Temperature controller is used to control the desired heating effect. Stirrer controller is used to control the stirring effect.

IV. STEPS FOLLOWED FOR BIODIESEL PRODUCTION

4.1. Transesterification Reaction Assays & Analysis

The transesterification reaction was carried out with methanol/oil ratio (i.e., 3:1, 6:1, 9:1, 12:1 and 15:1 M), using 0.3 wt%, 0.5wt%, 0.7wt% and 1 wt% of CaO, as a base catalyst. The reactor shown above was preheated, and then the oil was added. When the oil reached the temperature established for the reaction, the methanol and catalyst was added, in amounts established for each experiment. The reaction was carried out at 50, 55, 60, and 65°C and the reaction times were 2, 2.5, 3, and 3.5h. The choice of the said variables and their range were selected based on several outside sources and on preliminary studies in our laboratory.

The combination of these variables (temperature, catalyst concentration, reaction time, and molar ratio of alcohol to oil) was used in factorial experimental design for biodiesel production optimization by transesterification of Kapok seed oil with methanol.

4.2 Separation Process

The ester/glycerol separation is typically the first step of product recovery in most biodiesel processes. The separation process is based on the facts that fatty acid alcohol esters and glycerol are sparingly mutually soluble, and that there is a significant difference in density between the ester and glycerol phases. Glycerol is heavier than biodiesel. The settling will begin immediately, but the mixture is left a minimum of four to five hours. The difference in viscosity and colour between the two liquids can be seen. The objective is to remove only the Glycerol and stop when the biodiesel is reached. Two phases (having different density) are formed as a result of trans-esterification. Separation is done using a separating funnel. Upper layer consists of bio-diesel, alcohol, and some soap (formed as a result of side reaction saponification - free fatty acids get converted to soap). Lower layer consists of glycerin, excess alcohol, catalyst, impurities, and traces of unreacted oil. Purification of upper layer (to obtain biodiesel) is done in two steps.

- Removal of alcohol – by keeping mixture at elevated temperature ~80 °C.
- Removal of saponified products – by washing with warm water. Water is immiscible with bio-diesel, hence can be easily separated from bio-diesel.

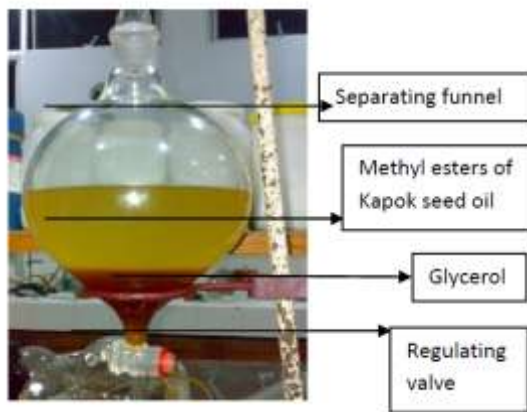


Fig 4.1 Separation Process

4.3. Washing Process

The primary purpose of the ester washing step is the removal of any soaps formed during the transesterification reaction. In addition, the water provides a medium for addition of acid to neutralize the remaining catalyst and a means to remove the product salts. The use of warm water prevents precipitation of saturated fatty acid

esters and retards the formation of emulsions with the use of a gentle washing action.

Gentle washing prevents the formation of emulsions and results a rapid and complete phase separation. The phase separation between esters and water is typically very clean and complete. However, the equilibrium solubility of water in esters is higher than the specified water content. Therefore, after the washing step there will be more than the equilibrium amount of water present. Water was added with the Biodiesel and it was mixed. Water was drained out and Indicator Phenolphthalein was added. This washing was repeated until the drained water maintains the pink color.

4.4. Drying

The final product of purified ester was heated to 110°C under vacuum to remove residual moisture around three hours to heat by using oven. Then the purified biodiesel is obtained. Many of the parts in the diesel fuel injection system are made of high carbon steels, thus they are prone to corrosion when in contact with water. Hence it is necessary to dry the water washed biodiesel product.

4.5. Factorial Experimental Design and Optimization of Parameters

Temperature, methanol-to-oil ratio, catalyst concentration and reaction time were chosen as independent variables and the production of methyl esters as dependent variables. The experimental range and levels of independent variables for bio diesel production are given in table 4.1. A 2⁴ full- factorial BBD for four independent variables at five levels was employed and the total number of experiments was 29 (= 2^k + 2k + 5). Twenty – four experiments were augmented with five replicants to evaluate the pure error. Usually a lower- order polynomial in some range of independent variables is employed for modeling. If the response is well modelled by a linear function of independent variables, then the approximating function is the first-order model. If there is a curvature in the system, then a polynomial of higher degree must be used, such as second order model. In this study there was a curvature.

Variables	Symbol coded	Units	Range	
			Low	High
Molar Ratio	X ₁	Moles	3	15
Catalyst concentration	X ₂	Wt%	0.3	1
Temperature	X ₃	°C	55	65
Time	X ₄	H	2.5	3.5

Table 4.1 Experimental range and levels of the independent variables

Experiments were then employed to fit the second order polynomial, which indicated that 29 experiments were required for this procedure.

The Design Expert 8.0 software was used for regression and graphical analyses of the data obtained. The maximum values of the yield were taken as the responses of the design experiment. Statistical analysis of the model was performed to evaluate the analysis of variance (ANOVA).

RUN	Independent variables				RESPOND
	X1	X2	X3	X4	YIELD %
1	3	0.3	60	3	62
2	3	0.65	60	3.5	65
3	9	0.65	65	2.5	86
4	9	0.3	55	3	82
5	3	0.65	60	2.5	63
6	9	1	55	3	80
7	15	1	60	3	76
8	3	0.65	65	3	69
9	9	0.65	60	3	94
10	15	0.65	65	3	78
11	9	0.65	60	3	95
12	9	0.65	60	3	94
13	15	0.65	60	3.5	74
14	3	1	60	3	67
15	9	0.65	60	3	95
16	9	0.65	60	3	94
17	15	0.3	60	3	72
18	15	0.65	60	2.5	73
19	9	0.65	65	3.5	91
20	9	1	65	3	88
21	9	0.3	60	3.5	84
22	9	0.65	55	3.5	80
23	9	0.65	55	3	64
24	9	1	60	3.5	79
25	9	0.65	55	2.5	81
26	15	0.65	55	3	79
27	9	0.3	65	3	85
28	9	0.3	60	2.5	83
29	9	1	60	2.5	88

Table 4.2 Full Factorial Box Behnken Design Matrix for Biodiesel Production

V. RESULTS AND DISCUSSION

5.1. Regression Analysis

If there is a functional relationship between the two variables X and Y the points in the scatter diagram will cluster around some curve called the curve of regression. If the curve

is a straight line it is called line of regression between two variables. If we fit a straight line by the principle of least squares to the points of the scatter diagram in such a way that the sum of squares of distance is parallel to the Y-axis, from the points to the line is minimized. We obtain a line to best fit for the data and are called regression line of Y on X. Regressions are of two types a) Simple regression b) Multiple regression

Simple regressions: The relationship between one independent variable and one dependent variable.

Multiple regressions: The relationship between two or more independent variables and one dependent variable.

The response surface contours, which are the graphical results of interactive effects, are shown in below Figs. Fig 5.1 shows that methyl ester conversion increases when high catalyst concentration is applied. However, it was observed that temperatures greater than 62°C and catalyst concentrations greater than 0.7 wt % led to the production of large amount of soap in this study. Furthermore, the addition of an excessive amount of catalyst increases emulsion formation.

When cottonseed oil and CaO dissolved in methanol, there are two reaction pathways: transesterification to produce biodiesel and saponification to produce soap; forecasting the temperature effect is not straightforward. There are two equilibrium reactions. When saponification is favoured, CaO is lost and overall process rate decreases. CaO is a catalyst on transesterification reaction and a reagent on saponification reaction. On the other hand, the transesterification reaction can be favoured when adequate temperatures are used. Low temperatures decreases the saponification rate, thus transesterification reaction is favoured. When temperature increases, the reaction rates are obviously higher because molecules have more energy, but the saponification reaction rate speeds up,

Therefore the transesterification reaction yield decreases.

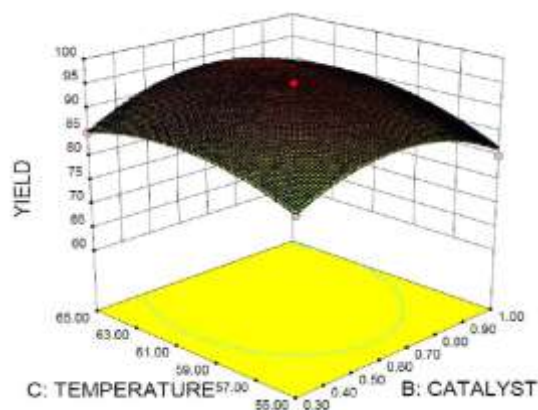


Fig 5.1 Response surface contour interaction on methyl esters production between temperature and catalyst concentration

Fig 5.1 Shows the response for the interactive factors of temperature and molar ratio. The 3D response surface plots indicate that the methyl esters production increases when methanol concentration increases (molar ratio: methanol/oil). Therefore, the maximum biodiesel conversions are obtained for high molar ratio. This is caused by the stoichiometry of transesterification, which requires a 3:1M ratio of alcohol to triglyceride, since this reaction involves the conversion of one ester and an alcohol towards another ester and another alcohol, as an excess of alcohol is used to drive the reaction near completion. On the other hand, an excessive amount of alcohol makes the recovery of glycerol difficult. Therefore, the ideal alcohol/oil ratio has to be established empirically, considering each individual process. It was found a molar ratio between 9:1 and 12:1 to be best for methanol.

Therefore temperature was tested as an important variable to enhance the reaction in biodiesel production. To summarize, defining the best temperature is clearly an optimization problem.

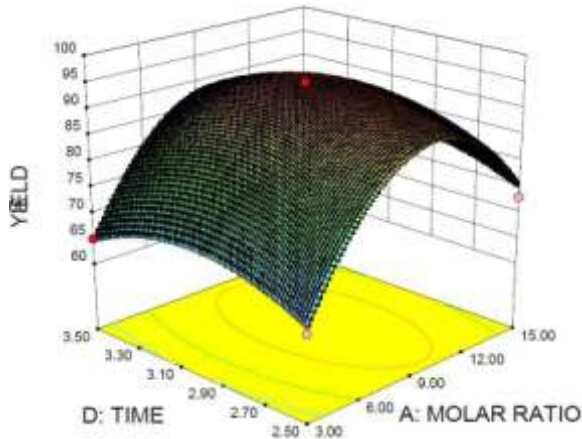


Fig 5.2. Response surface contour interaction on methyl esters production between molar ratio and temperature.

Fig 5.3. Shows higher production of methyl esters is strongly favoured when higher molar ratio is employed for a certain time of reaction. The molar ratio is a fundamental variable in the transesterification of the biodiesel production. The said molar ratio affects the separation and recovery of glycerol. A molar ratio of 6:1 is generally considered the most appropriate for ethanol, although in this work, as been indicated, it was found to be molar ratio 9:1 to be best for methanol.

Some researchers have hypothesized that the quality of esters depends on the large excess of alcohol. However, in this study, the high molar ratio of alcohol to vegetable oil interfered in the glycerol separation, since there was increase in solubility. When glycerol remains in solution it helps driving the equilibrium back to the left, thereby lowering yield of esters. The transesterification of cottonseed oil with methanol was observed at molar ratio between 3:1 and 15:1

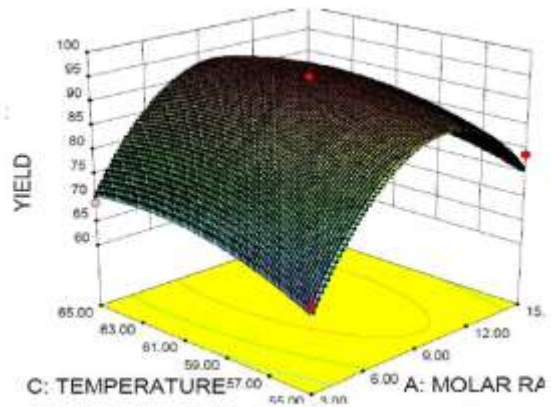


Fig 5.3. Response surface contour interaction on methyl esters production between molar ratio and time

For molar ratio less than 6:1, the reaction incomplete. For a molar ratio of 15:1 the glycerol separation was difficult and the apparent yield of esters decreased, since some of the glycerol remained in biodiesel phase. Therefore, molar ratio 9:1 seems to be the most appropriate. Several researchers have found similar results for biodiesel production.

Fig 5.4. Shows that the methyl ester yield is a function of the molar ratio and catalyst concentration, under the experimental conditions is defined by factorial design. It is possible to observe that higher methyl ester yield occur at high molar ratio greater than 12:1 and at high catalyst concentration greater than 0.7 wt%.

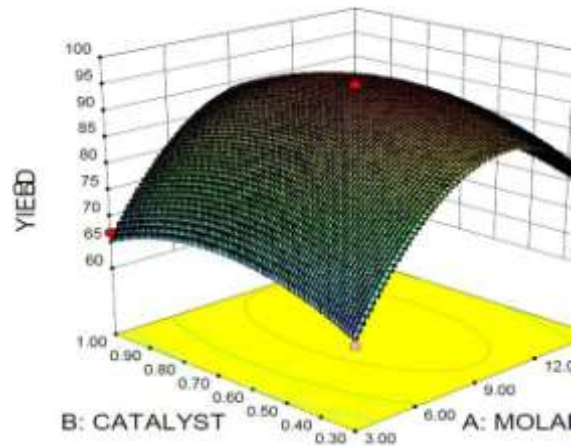


Fig 5.4 Response surface contour interaction on methyl esters production between molar ratio and catalyst concentration

When molar ratio values were very low less than 6:1 yields increased with higher catalyst concentration. However, when molar ratio was kept in its higher level (15:1) a higher methyl ester was always achieved. Thus, catalyst concentration is the most important factor in improving methyl ester yield. But an excess catalyst can produce emulsions and the biodiesel that is produced has difficulty in the separation phase.

It has been reported that low catalyst concentration increases conversions with ethanol-oil-ratio. In this present study applying response surface design, we observed that for methanol, methyl esters production increases when catalyst concentration increases for low molar ratio.

The optimum values for selected variables were obtained by solving the regression equation. The optimum values of the process variables for maximum methyl esters production are shown in table 5.3 when a yield of 95% was achieved. The ester yield increased as the molar ratio also increased up to a value 12:1. The best results were for molar ratios between 9:1 and 12:1. The reaction was incomplete for molar ratios less than 6:1. For a molar ratio of 15:1, the glycerol separation was difficult and the apparent yield of esters decreased, because of a portion of the glycerol remaining in the biodiesel phase. Therefore, the phase separation becomes more difficult when molar ratio of methanol increases due to its miscibility increasing for both phases (glycerol and methyl ester).

Parameters	Optimum values
Ethyl esters (%)	95
Molar ratio(moles)	9:1
Catalyst concentration (wt %)	0.7
Temperature ($^{\circ}$ C)	60
Time (h)	3

Table 5.1. Optimum values of the process parameter for maximum efficiency

The optimum value temperature was 600C. This temperature is below the boiling point of alcohol. Therefore, molar ratio is used between 6:1 and 12:1, since it did not evaporate. The transesterification rate increases as the temperature increases. However, the maximum operating temperature should not exceed the boiling point of the reactants.

The highest methyl ester yields were achieved when the reaction time was 3h. Methanolysis is quicker than

ethanolysis. The optimum value of catalyst concentration was at 0.7wt%.

A typical catalyst concentration for transesterification reactions (0.3 to 1wt %), the results that were obtained agreed with those obtained from the response surface analysis, confirming that RSM was effectively used to optimize biodiesel production.

VI. CONCLUSION

The search for feedstock for the production of biodiesel was extended to kapok oil. The transesterification of kapok oil was carried out with heterogeneous catalyst Cao. Though the FFA of the feedstock was reduced to 0.8 this result indicates that the FFA need to be reduced for higher methyl ester yield[3].the physical and chemical properties of kapok oil biodiesel was verified and these values are equivalent to ASTM standards[2].

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