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Optimization of Biodiesel Production from *Ceiba Pentandra* (Kapok Seed Oil) Using Response Surface Methodology Assisted by Ultrasonic Energy Method

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Abstract : Application of ultrasonic energy-assisted biodiesel production method from *Ceiba pentandra* (Kapok seed oil KSO) catalyzed by KOH catalyst was studied at different conditions. Response Surface Methodology (RSM) based on central composite rotatable design (CCRD) was employed to optimize the four important process parameters such as methanol/oil molar ratio (X_1), KOH catalyst concentration (X_2), reaction time (X_3) and reaction temperature (X_4) for transesterification of KSO oil using ultrasonic energy. The results of the experimental matrix were analyzed. It was found that the optimal reaction parameters were found to be methanol/oil molar ratio (10.29 mol/mol), KOH catalyst concentration (1.55wt %), reaction time (32.37 min) and reaction temperature (38.45°C) for a biodiesel yield of 99.40%. The determined properties of KSO biodiesel were within the recommended biodiesel standards limits.
Keywords: Biodiesel, Transesterification, RSM.

1. Introduction

Increasing concerns with respect to environmental impacts, the price rise of petroleum products, the exhaustion of fossil fuels and strict regulations on exhaust emissions have necessitated the replacement of fossil fuels causing less pollution and easily available renewable fuels for use in internal combustion engines[1,2] . From the point of view of protecting the global environment and the interest for long term supplies of conventional diesel fuels, triggers the researchers to develop alternative source of fuels comparable to conventional diesel fuels. Continuous efforts are being taken throughout the world to reduce the consumption of liquid petroleum fuels wherever possible. The increased use of fossil fuel resulted in depletion of its fossil reserves. This triggers researchers for initiatives to search for alternative fuel that are domestically, easily available environmentally agreeable, and technically feasible as well as can fulfill the global energy demand. It has been reported that renewable energy source will become the world's second-largest resource of power generation by 2016 and, by 2035 it can replace such fossil fuel.[3]

Biodiesel is one of the most promising alternative fuels to address these problems and is gaining more and more interest among researchers as an attractive source of fuel due to the depleting nature of fossil fuel resources[8-21]. Biodiesel is defined as a mixture of monoalkyl esters of long chain fatty acids (FAME) derived from a renewable lipid feedstock, such as vegetable oil or animal fat. Biodiesel typically comprises alkyl fatty acid (chain length C14–C22) esters of short-chain alcohols, primarily, methanol or ethanol. There are more than 350 oil-containing crops identified worldwide as potential feedstocks for biodiesel production. Biodiesel feedstocks can be divided into four main categories sucha as: (1). Edible vegetable oil: peanut, canola, soybean, sunflower, palm and coconut oil (2).Non-edible vegetable oil: *Jatropha curcas, Moringa oleifera, Calophyllum inophyllum* and *Croton megalocarpus,* (3).Waste or recycled oil, (4).Animal fats: (a) Slaughterhouse animal fat viz. beef tallow, pork lard, fleshing oil, lamb meat, mutton fat; and (b) poultry farm animal fats/poultry fats viz. chicken fat, duck tallow and feather meal.

India is diversifying its biodiesel feedstock towards non-edible plant oils, such as *Ceiba pentandra* (Kapok), *Calophyllum inophyllum* (bintangorlaut/ nyamplung/ penagalaut), *Jatropha curcas* (Jatropha), *Ricinus communis* (Castor), *Heveabra siliensis*(Rubber), and waste from palm oil processing. These non-edible plant oils have attracted researchers' attention as biodiesel feedstock due to their potential and generous supply. *Ceiba pentandr a*locally known as Kapok or Kekabu is grown in India, Malaysia and other parts of Asia. *Ceiba pentandra* pod contains 17% fiber that is mainly used in making mattresses and pillows, whereas the seeds are traditionally considered as waste Kapok seeds which make up about 25 - 30wt% of each pod with an average potential oil yield of 1280 kg/ha annually. The most common method for extracting oil from the Kapok seeds is mechanical expeller. Since oil extracted from Kapok seeds is non-edible it's well in line with the purpose of the second generation biodiesel production, i.e., utilization of non-edible feedstock to avoid direct conflict with human food.

The most common techniques used for the production of biodiesel are micro emulsion direct use and blending of diesel/oil, thermal cracking (pyrolysis), and transesterification (alcoholysis). Among them, the most popularly used method is transesterification in the presence of a catalyst[4]. However, there are numerous problems associated with this existing technique, i.e., long reaction time, non-uniform heat distribution, and large energy requirements. These drawbacks have triggered researchers to find alternative methods. Besides this, ultrasonic energy can also be used for the transesterification reaction, resulting in faster reaction kinetics and higher conversion of oil and product yield. Low frequency ultrasonic energy assisted method in biodiesel production is an efficient, time saving, economically functional and effective technique to solve the problems related with the immiscible nature of the reactants. Applying ultrasonic waves to the reaction mixture compels the fluids to generate a huge number of cavitation bubbles which grow rapidly resulting in violent collapse of the reaction mixture[5]. The vigorous collapses of these bubbles will lead to the formation of micro jets generating fine emulsion between the reactants. As a result, the mixing requirement during the process is also significantly lowered, translating into reduced energy consumption. Under ultrasonic assisted energy method, the transesterification can be carried out at a low temperature, and smaller amounts of catalyst and methanol are needed[6]. The most important criteria in Transesterification are modeling and optimization of reaction parameters to increase the % of (FAME) yield. The conventional method of optimization technique includes varying one parameter at a time and keeping the remaining reaction parameters constant. This technique is not only time-consuming but also does not describe the complete effects of the reaction parameters in the process and neglects the combined interactions between the reaction parameters. In this regard response surface methodology (RSM) has attracted great interest since it is an effective statistical tool that can be used to define the relationships between the response and the independent variables RSM also defines the effect of the independent variables, alone or in combination, in the processes. In addition to analyzing the effects of the independent variables, this technique also generates a mathematical model. It has many advantages like being more economical, reduction in the number of experimental runs required to generate sufficient information for a statistically acceptable result, giving information on interaction between parameters in response, predicting response, and checking the adequacy of the method. This process employs low-order polynomial equations in a predetermined region of the independent variables, which will be analyzed to locate the optimum values of independent variables for the best responses. The emphasis of the present work is to experimentally evaluate the possibilities of using biodiesel transesterifiedfrom *Ceiba pentandra* (Kapok). Accordingly, the process parameters in respect to the ultrasonic energy based transesterification process for biodiesel extraction were designed through the application of RSM.

2. Materials and Methods

2.1Extraction of crude *Ceiba pentandra* seed oil

The *Ceiba pentandra* seeds were collected from surrounding villages of Theni District Tamil Nadu, South India and the collected seeds were cleaned dried under sun and were ground to powder. The oil extraction was carried out by soxhlet extraction method. The extracted oil was stored under atmospheric condition so that solid materials present in the oil were allowed to settle down. The extracted crude *Ceiba pentandra* oil was filtered to remove unwanted particles in the oil. After filtration, the oil was heated to 120°C so that the water content in the crude oil was removed. The byproduct after oil extraction can be used as livestock feed and as fertilizer. The percentage of oil yield was calculated as follows:

$$\text{Yield of oil (\%)} = \frac{Weight\ of\ the\ extracted\ oil\ (g)}{Weight\ of\ kapok\ seed\ (g)} \text{ X } 100$$

The physico-chemical properties of the extracted kapok seed oil were examined by using the standard method and are reported in (Table 1).

Table 6.1 Physico chemical properties of kapok seed oil

SL.No	Properties	Units
1	Calorific Value (MJ/kg)	37.54 MJ/kg
2	Kinematic Viscosity @ 40°C in Cst	34.75
3	Density @15(ºC) (kg/m³)	923.1
4	Acid value (mg KOH/goil)	11.13
5	Saponification value	198.7
6	Flash Point (° C)	310° C
7	Fire Point (° C)	320° C
8	Molecular weight (g/mol)	897.3
9	Calculated Cetane Index	37

2.2 Acid catalyzed esterification process

The acid catalyzed esterification is a pretreatment process in which the acid value of the feedstock is reduced below 2 mg KOH/goil. To reduce the acid value of the Kapok Seed Oil, acid esterification reaction was employed by reacting KSO with methanol (Molar ratio 9:1) in the presence of sulphuric acid (1.5 wt.%) as catalyst at a temperature of 60 ºC for 30 min in a three neck flask immersed in an ultrasonic cleaning bath. After the reaction the reactant were poured into a separating funnel and was allowed to settle for 6 hours. The denser free fatty acid oil content layer moved to the bottom and the methyl ester layer settled at the top and was separated. To remove the excess methanol the separated oil was heated to 70°C and washed with distilled water in a separating funnel. Finally the acid value of the product separated at the bottom was determined by standard method and was ensured that the acid value was below 2%

2.3 Alkali catalyzed transesterification process

Ultrasonic Cleaner (UCB -15 Macro Scientific Works Pvt Ltd, Delhi, India), was used as the reactor for biodiesel production. The reaction was carried out in a 250 ml three-neck glass flask equipped with a reflux condenser which was immersed in an ultrasonic cleaning bath (total power: 500 W operating at 33 ± 3 kHz frequency). The condenser was fitted to the central neck of the flask and a thermocouple thermometer was inserted into another neck. All the experiments were conducted in the ultrasonic reactor containing the three-neck flask filled with sample mixtures (Kapok seed oil, methanol and catalyst) which was submerged in water bath, and subsequently the water bath was heated. The experimental runs were carried out in the presence of air under atmospheric conditions.

2.4. Experimental procedure

Methanol and KOH were fed into a three-neck glass flask. The vessel was placed in the ultrasonic bath. The Kapok seed oil was added to the three neck glass flask, and the reaction was timed. The denser glycerol layer moved to the bottom, the methyl ester layer settled at the top and was separated by allowing it to settle for 12 hours. Then it was washed by spraying hot water (10% by volume) gently until the layer was clear. Vigorous shaking with water was avoided as it would lead to emulsification of methyl esters. Anhydrous sodium sulphate was used to dry the product.

2.5. Design of experiments

The software, Design Expert 6.0 was used in this study to design the process parameter and the response was statistically analyzed and validated to derive objective inferences. The experimental design as a function of the selected process variables was carried out using Central Composite Rotatable Design (CCRD). A five-level-four -factor CCRD was used. The CCRD consist of 30 experimental and provided sufficient information to fit a full second-order polynomial model[7-9].The independent input process variables were primarily classified in terms of low and high levels. The factors were further distributed into versatile points called axial, center and factorial points. The axial points were coded by the CCD as -2 and +2. The low and high lever factor points were designated as -1 and +1. Whereas the centre points were coded as 0 and the repeated experimental arrays were designed on the centre points. The experimental factors selected for optimization and their respective ranges were as follows: reaction time (20 – 50 min), oil-to-methanol molar ratio (1: 3 – 1:15), quantity of KOH catalyst (1.0 – 2.0 weight %), and reaction temperature (20 -50 °C). Table 2 provides the levels used for each factor, and to avoid bias, the 30 experimental runs were performed in randomized order Table 3.

Table 2.Independent Variable and Levels used for CCRD in Methyl Ester Production.

Variables	Symbols	Levels				
		Low(-)	High(+)	0	Axial(-α)	Axial (+α)
Methanol/oil molar ratio (mol/mol)	X_1	3	6	9	12	15
Catalyst concentration (wt %)	X_2	1	1.25	1.5	1.75	2.0
Reaction time (min)	X_3	10	20	30	40	50
Reaction Temp(°C)	X_4	20	30	40	50	60

Table 3.CCRD Arrangement and Responses for Methyl Ester Production.

Std	Run	A:Molar Ratio mol/mol	B:Catalyst Concentration wt/wt	C:Reaction Time min	D:Reaction Temp °C	Biodiesel yield %
10	1	12	1.25	20	50	97.23
21	2	9	1.5	10	40	95.73
2	3	12	1.25	20	30	92.21
26	4	9	1.5	30	40	99.87
1	5	6	1.25	20	30	85.45
5	6	6	1.25	40	30	88.23
17	7	3	1.5	30	40	66.73
20	8	9	2	30	40	90.52
6	9	12	1.25	40	30	92.56
15	10	6	1.75	40	50	82.15
9	11	6	1.25	20	50	85.45
14	12	12	1.25	40	50	92.21
22	13	9	1.5	50	40	94.45
4	14	12	1.75	20	30	93.14
25	15	9	1.5	30	40	99.76
18	16	15	1.5	30	40	86.88

27	17	9	1.5	30	40	99.81
3	18	6	1.75	20	30	84.67
24	19	9	1.5	30	60	94.31
11	20	6	1.75	20	50	84.05
29	21	9	1.5	30	40	99.72
16	22	12	1.75	40	50	90.04
13	23	6	1.25	40	50	85.56
7	24	6	1.75	40	30	84.83
28	25	9	1.5	30	40	99.82
19	26	9	1	30	40	95.17
8	27	12	1.75	40	30	88.24
12	28	12	1.75	20	50	93
23	29	9	1.5	30	20	92.03
30	30	9	1.5	30	40	99.59

2.6. Statistical Analysis

The experimental design was analyzed with RSM, and the quadratic response surface model was fitted as equation (1) which was designed by Design- Expert 6 software (Stat-Ease, Inc., Minneapolis, MN, USA). The fit of the model was evaluated using the coefficients of determination and analysis of variance (ANOVA).

$$Y = \beta_{ko} + \sum_{i=1}^{3} \beta_{ki}\, x_i + \sum_{i=1}^{3} \beta_{kii}\, x_i^2 + \sum_{i=1}^{2} \sum_{j=1+j}^{3} \beta_{kij}\, x_i\, x_j \qquad (1)$$

in which Y is the response factor that is fatty acid ester , x_i is the i^{th} independent factor, β_{k0} is the intercept, β_{ki} is the first-order model coefficient, $\beta_{kii}$is the quadratic coefficient for the factor i, and β_{kij} is the linear model coefficient for the interaction between factors i and j.

3. Results and Discussions

3.1. RSM Modeling

3.1.1. Analysis of variance (Anova)

In order to optimize the reaction parameter for KSO oil biodiesel production, a CCRD with a five-level four-factor design that addressed reaction temperature, oil-to methanol molar ratio, reaction temperature and the quantity of catalyst was designed and 30 designed experiments were conducted in a randomized order and the results were analyzed with multiple regressions using Design-Expert 6 software. Regression analysis yielded three linear coefficients (X_1, X_2, X_3), three quadratic coefficients (X_1^2, X_2^2, X_3^2), and three cross-product coefficients (X_1 X_3, X_1 X_4) for the full model shown in and the ANOVA for the response surface quadratic model is shown in Table 4. The coefficients of the response surface model, as provided by the above quadratic model equation, were also evaluated. From the ANOVA of response surface quadratic model for FAME conversion, the Model F-value of 41.06 and Prob> F less than 0.0001 implied that the model was significant. For the model terms, values of Prob>F less than 0.05 indicated that the model terms were significant. In this case X_1, X_2, X_3, X_1^2, X_2^2, and $X_3^2$are significant model terms (all have Prob> F less than 0.050). This reveals that the molar ratio, catalyst concentration, reaction time, and the quadratic terms affect the yield much significantly. Values greater than 0.1000 indicate the model terms are not significant.

Table 4. Result of the analysis of variance (Anova).

Source	Sum of Squares	Degrees of Freedom	Mean Square	F Value	Probability > F
Model	1471.595	14	105.1139	67.05466	< 0.0001
X_1 (methanol /oil	404.5888	1	404.5888	258.0968	< 0.0001
X_2 (KOH Concentration)	32.8536	1	32.8536	20.95809	0.0004
X_3 (Reaction time)	8.096817	1	8.096817	5.165151	0.0382
X_4 (Reaction temperature)	1.0086	1	1.0086	0.64341	0.4350
X_1^2	945.3048	1	945.3048	603.0324	< 0.0001
X_2^2	94.95567	1	94.95567	60.57447	< 0.0001
X_3^2	46.30973	1	46.30973	29.54207	< 0.0001
X_4^2	86.84367	1	86.84367	55.39964	< 0.0001
$X_1 X_2$	0.04	1	0.04	0.025517	0.8752
$X_1 X_3$	11.6964	1	11.6964	7.461411	0.0154
$X_1 X_4$	9.455625	1	9.455625	6.031967	0.0267
$X_2 X_3$	3.822025	1	3.822025	2.43816	0.1393
$X_2 X_4$	0.8281	1	0.8281	0.528265	0.4785
$X_3 X_4$	4.1616	1	4.1616	2.654783	0.1241
Residual	23.51378	15	1.567586		
Lack of Fit	23.4651	10	2.34651	240.9973	< 0.0001
Pure Error	0.048683	5	0.009737		
Cor Total	1495.109	29			

3.1.2. Prediction of FAME yield by RSM Model

The experimental data on FAME yield were analyzed by the multiple regression model to develop second-order polynomial equations based on values of coded and uncoded levels of operational variables in terms of coded factors are as follows:

$$\text{FAME yield (\%)} = 99.76 + 4.11 X_1 - 1.17 X_2 - 0.58 X_3 + 0.20 X_4 - 5.87 X_1^2 - 1.86 X_2^2 - 1.30 X_3^2 - 1.78 X_4^2 - 0.050 X_1X_2 - 0.85 X_1X_3 + 0.77 X_1X_4 - 0.49 X_2X_3 - 0.23 X_2X_4 - 0.51 X_3X_4 \quad (2)$$

in which Y, fatty acid methyl ester content (wt.%). X_1, X_2, $X_3$and X_4 are the values of the four independent reaction parameters, the oil-to-methanol molar ratio, catalyst concentration (wt. %) the reaction time (minutes) and the reaction temperature (°C). The significance of each coefficient in Equation (2) was evaluated by the P-value as shown in Table 4. The table provides the F-values and Prob> F values that indicate the significance of each coefficient. In general, larger F-value and smaller Prob> F value indicates higher significance of the corresponding coefficient. The most influencing reaction parameter on the model response was the molar ratio, catalyst concentration, reaction time followed by reaction temperature. From the Anova table the response surface quadratic model for percentage of yield has F-value of 67.05 and the corresponding p-value of 0.0001 indicates that the model is significant with only 0.01% chance that a 'Model F-value' could have occurred due to noise. In this model, the factor methanol/kapok seed oil molar ratio is the major contributing factor for the FAME yield, where maximizing the yield is the objective. This is confirmed by the very high F-value (258.09) for that factor. The other reaction parameters such as catalyst concentration, reaction time and reaction temp, (F-value = 20.95,5.16,0.64) also has significant influence on the yield. Beside their single effects, the influences of their squared values and the two level interactions between these two reaction parameters were also found to significantly affect the process yield. For slow reactions such as the transesterification reaction, the reaction time is having an influence on the yield of FAME. However, the reaction time tested in this study was only up to 32.02 min. This range was deemed suitable for the ultrasonic-assisted process as compared to that of non-ultrasonic process that could extend to few hours. This clearly indicates that the influence of ultrasonic energy is accelerating the reaction rate. The quality of the model developed was evaluated based on the correlation coefficient value, R^2. The R^2value for Equation (7) was 0.9843. This indicates that 98.43% of the total variation in the biodiesel yield (KSOME) was attributed to the experimental process variables studied. The closer the R^2value to unity, the better the model will be as it will give predicted values which are closer to the actual

values for the response. From the ANOVA and regression analysis on (Table 4) and (Table 5) respectively, it can be seen that the linear terms of X_1, X_2,and X_3, the quadratic terms X_1^2, X_2^2, and X_3^2 were significant (because Prob> F less than 0.05), but the cross product terms X_1X_2, X_1X_3, X_2 X_3 (cross products) were insignificant. Considering the probability of error in the experiment taken into account the optimum reaction parameters for maximum FAME yield is tabulated in (Table 5).

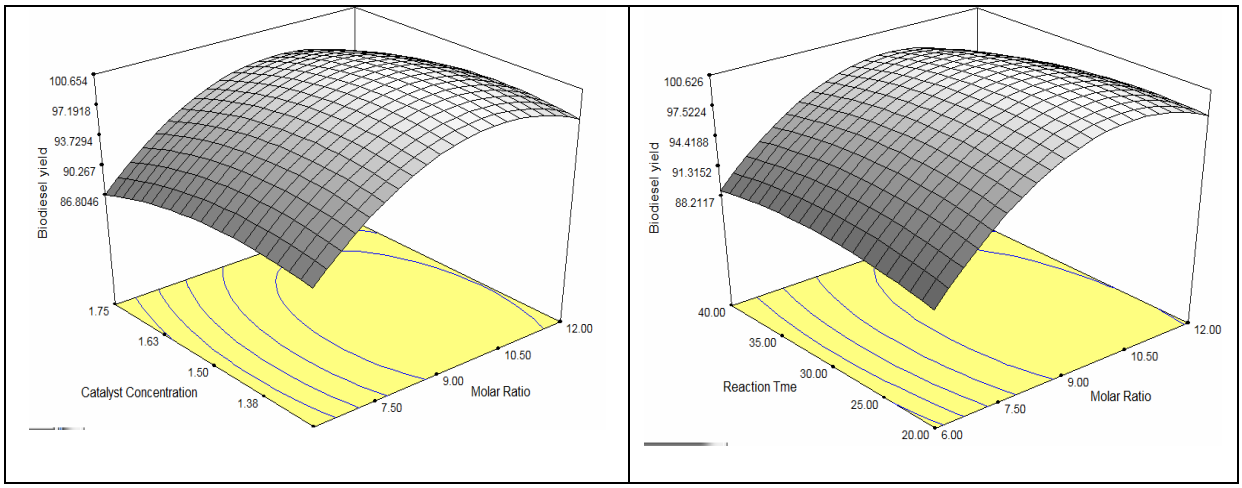
Table 5. The optimum predicted reaction parameters for maximum FAME Yield using design of experiment methodology.

Variables	Optimum Value
Methanol/oil molar ratio (mol/mol)	10.29 :1 mol/mol
Catalyst concentration (wt %)	1.55 % w/w
Reaction time (min)	32.37 minutes
Reaction temperature °C	38.45°C

3.1.4 Effect of interactive parameters on biodiesel yield

Contour plots (Figure 1) were drawn to show the relationships between dependent and independent variables of the developed model. Each contour curve presented the effect of two variables on the methyl ester yield, holding the third variable at constant level. The third variable is held at zero level. However, the interaction factor also must be considered as the individual effect plot does not give information regarding the significant interaction involved. Remarkable interaction between the independent variables could be observed if the contour plots had an elliptical profile. The relationship between independent and dependent variables of the developed model in the response surface plots at the stationary value of 9:1 methanol-to-oil molar ratio, 1.5% catalyst concentration,30 min reaction time and 30° C reaction temperature is shown in (Figure 1).

The methanol-to-oil ratio is one of the important factors that affect the conversion of triglyceride to FAME. Stoichiometrically, three moles of methanol are required for each mole of triglyceride, but in practice, a higher molar ratio is required in order to drive the reaction towards completion and produce more FAME as products. The results obtained in this study are in agreement with this.



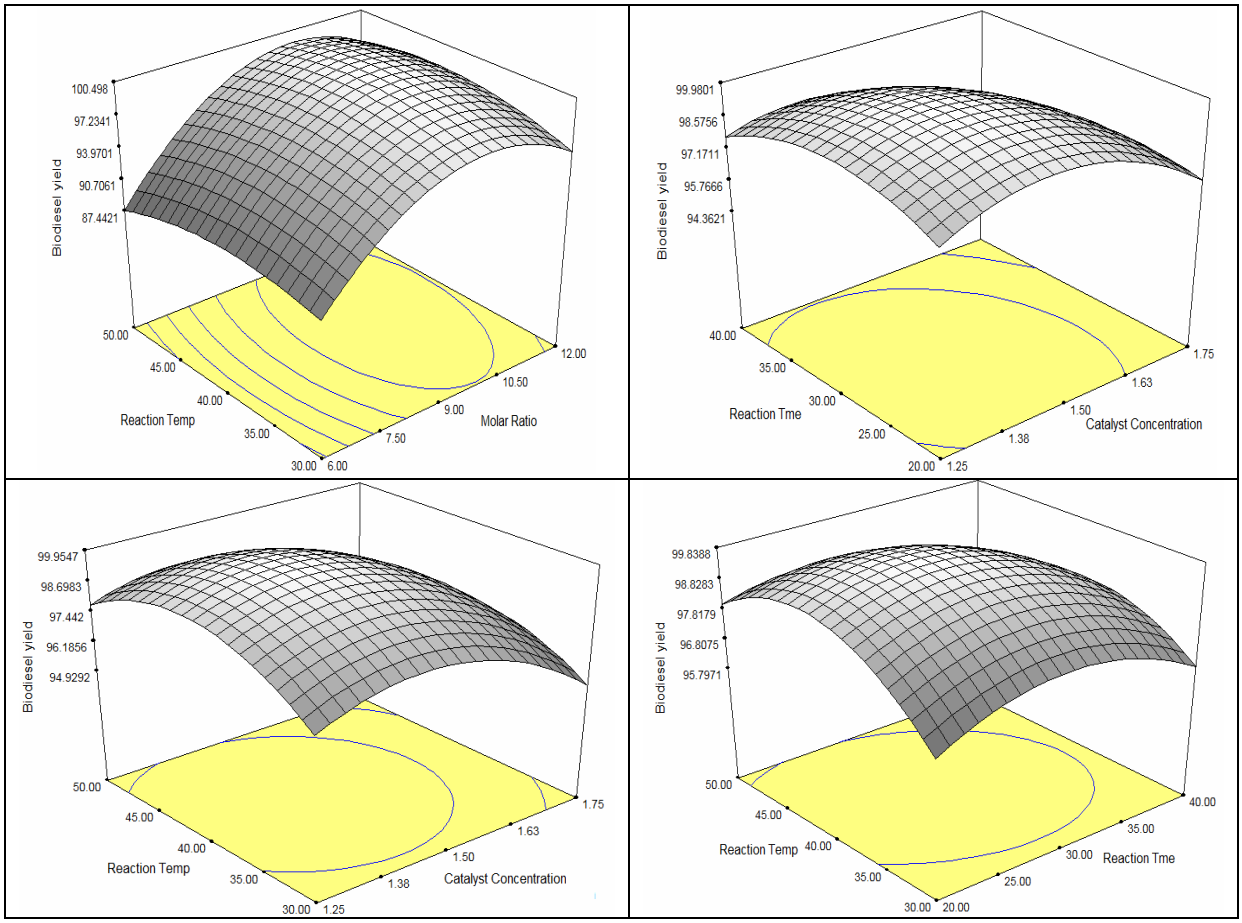


Figure 1. 3-D plots of process variables with respect to fatty acid methyl ester conversion

The optimum conditions for the three variables, methanol to oil molar ratio, reaction time and catalyst concentration were obtained using numerical optimization feature of Design Expert Software. The optimized reaction parameters found by Design of Experiments were as follows: X_1 = 10.29 mol/mol, X_2 = 1.55 (wt % of oil), X_3 = 32.37min and X_4 = 38.45°C. The theoretical fatty acid methyl ester yield predicted under the optimum conditions were Y = 99.40 %. Laboratory experiments were carried out in order to validate the equation (7) using these optimal values. It was found that the experimental value of 99.20 ± 0.3% of FAME content agreed well with the predicted value.

The properties of biodiesel produced from kapok seed oil was compared with standard Petro –diesel as per ASTM standards (Table 6). The direct usage of kapok seed oil in diesel engine as a fuel was restricted because of the much higher values of fuel properties. Transesterification of kapok seed oil into FAME resulted in reduced kinematic viscosity which was within permissible limit. The flash point, fire point and cetane number were in a limit of safe storage and handling conditions.

Table 6: Properties of Kapok seed Methyl Ester

1	Gross Calorific Value(MJ/kg)	38.96
2	Kinematic Viscosity @ 40° C	4.73 Cst
3	Density @ 15°C (kg/m^3)	889.9
4	Flash Point °C	98 °C
5	Fire Point °C	108 °C
6	Calculated Cetane Index	50

Conclusions

In this study ultrasonic energy-assisted biodiesel production process from *Ceiba pentandra* (Kapokseed oil KSO) catalyzed by KOH catalyst was studied at different conditions and was found to very efficient. The most optimum contribution of parameters is found to be Methanol/oil molar ratio 10.29:1 mol/mol, Catalyst concentration 1.55 %w/w, Reaction time 32.37 min, Reaction temperature 38.45°C. The statistical analysis predicted that effect of all parameters on the FAME yield was significant, and influence of all parameters was strongly interrelated. It could be concluded that ultrasonic energy assisted transesterification of biodiesel from Kapok seed oil is an efficient method with reduced reaction time when compared with conventional methods.

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