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PRODUCTION AND OPTIMIZATION OF BIODIESEL FROM JATROPHA OIL USING RESPONSE SURFACE METHODOLOGY

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Abstract: This paper presents an optimization of preparation variable for biodiesel production from locally sourced Jatropha oil. The optimization of alkali-catalyzed transesterification was aimed at converting fatty acid methyl ester (FAME). The optimization variables for the renewable biodiesel preparation were reaction temperature (25-65^{O}C), catalyst concentration (0.5-1.5%) and methanol to oil ratio (4.5-7.5). The data was statistically analyzed using response surface methodology of the Design-Expert software to find the suitable model of percent fatty acid methyl ester purity (% FAME) and yield) as a function of the factors. A full quadratic model was recommended by the program with an R^2 and adjusted R^2 of 51.53 and 81.53%, respectively. The optimum FAME content of 99.28% was obtained at 65^{O}C for temperature, 0.5% for catalyst concentration, 7.5 for methanol-to-oil molar ratio. The conversion is satisfactory for biodiesel requirements.

Keywords: Optimization, response surface methodology, biodiesel, Jatropha oil

INTRODUCTION

The environmental challenges caused by fossil fuel consumption as well as the dramatic impact of oil imports on global economy demands sourcing for alternative of which biodiesel fits in. Biodiesel minimal adverse environmental impact and it production will decrease the over dependence on the crude oil [1]. However, as it is produced from vegetable oils and animal fats, biodiesel feedstock may affect food supplies in the long-term. The recent focus has been to seek a source of non-edible oils, as a feedstock for biodiesel production. *Jatropha curcas* L. (Jatropha) has been chosen as an optimal supply source. *Jatropha curcas* L. has several benefits which include use of its stem for cleaning, as source of latex that serves as natural pesticide and for healing of wounds, and its leaves are used as fodder for silkworms [2, 4]. The use of chemically altered or transesterified vegetable oil, called biodiesel, does not require any modification in the

engine or injection system or fuel lines and can be used in any diesel engine. The production of biodiesel would be inexpensive because it could be extracted from the non-edible oil sources and from certain species that are commonly grown in many parts of the world.

Transesterification is a process for the conversion of triglyceride molecules to esters using an alcohol [3-4]. Methanolysis is the process where methanol is used in biodiesel production [5-6]. The stoichiometric equation requires one mole of triglyceride and three moles of alcohol to form three moles of methyl ester and one mole of glycerol in the presence of a strong base or acid [3].

Response surface methodology (RSM) is a useful statistical technique, which has been applied in the research of complex variable processes [7]. Multiple regression and correlation analysis are used as tools to assess the effects of two or more independent factors on the dependent variables. Furthermore, the central composite design (CCD) of RSM has been applied in the optimization of several biotechnological and chemical processes with the flowchart presented in figure 1. Its main advantage is the reduction in the number of experimental runs required to generate sufficient information for a statistically acceptable result. It has been applied successfully for optimization of biodiesel production in fat and oil feedstock's, including mahua oil (*Madhuca indica*) [8], Jatropha oil [9] and animal fat [10]. The current study is concentrated on developing a technique for biodiesel production from Jatropha oil, however the study also considered RSM as a tool applied to optimize the alkali-catalyzed transesterification to produce fatty acid methyl ester (FAME) as a function of three factors: the methanol-to-oil molar ratio, sodium hydroxide and the reaction time. The fuel properties of Jatropha biodiesel for vehicle use were determined.

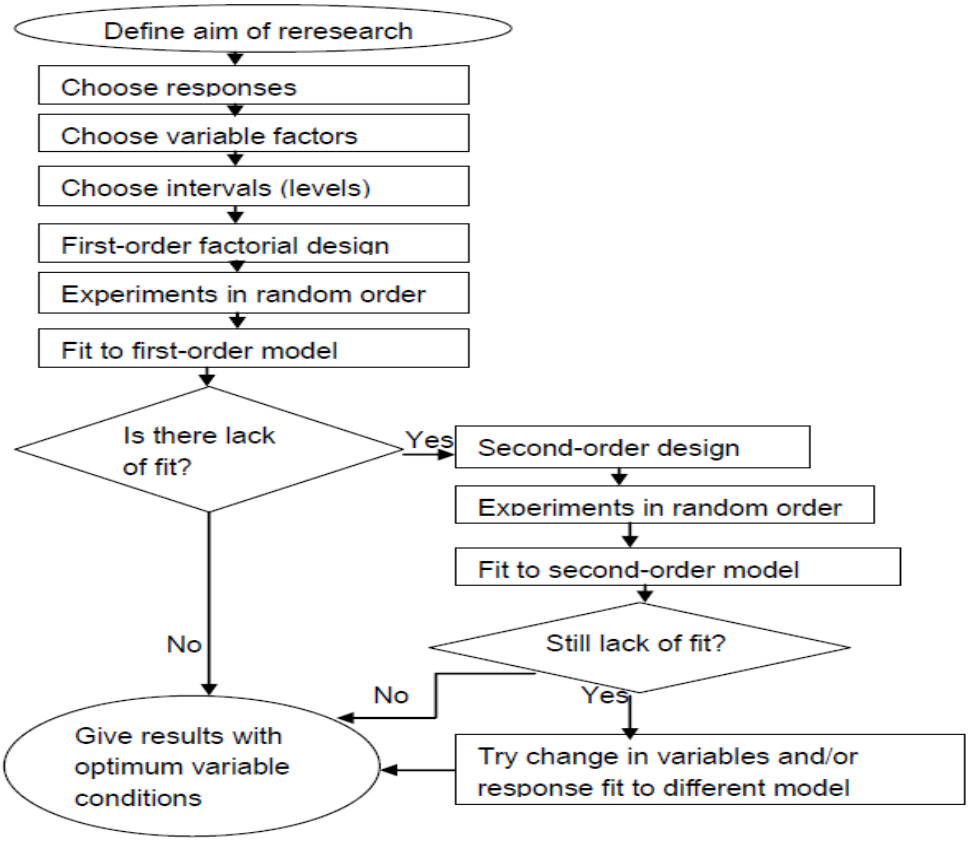

Fig 1: Flowchart of the Central Composite Design.

MATERIALS AND METHODS

Materials and Equipments

A locally source vegetable oil (Jatropha oil) from northern Nigeria was characterized for free fatty acid (FFA) content (0.15 mg KOH/g), saponification value (193.7 mg KOH/g), iodine value (130.2 mg I_2/g) and peroxide value (17.1 meq/kg). An analytical grade methanol 99.8 % and potassium hydroxide (catalyst) were used. A gas chromatograph (Hewlett Packard 5890 Series II), an integrator (Hewlett Packard 3396SA), a fused silica capillary column (Hewlett Packard OV-1), a flame-ionization detector (FID), 250 cm^3 three-necked batch reactor, reflux condenser, mechanical stirrer and a stopper and a constant-temperature oil bath were used for the experiment.

Transesterification reaction

The reactor was initially filled with the desired amount of oil, then placed in the isothermal bath with its associated equipment and heated to a predetermined temperature. The KOH catalyst was dissolved in the methanol and the resulting solution was added to the agitated reactor. The reaction was timed as soon as the catalyst/methanol solution was added to the reactor and allowed for 1 h. The mixture was transferred to a separating funnel, allowing glycerol to separate by gravity for 2 h. After removing the glycerol layer, the methyl ester layer was washed with distilled water to remove methanol, the catalyst and glycerol residuals. The methyl ester phase was then analyzed to determine the biodiesel purity and yield.

Analytical Methods

The biodiesel purity which is the methyl ester concentration (% wt) in the biodiesel was obtained by capillary gas chromatography. This method also allows for the quantification of the monoglyceride, diglyceride and triglyceride contents in the biodiesel. The analyses were performed on a gas chromatograph (Hewlett Packard 5890 Series II) connected to an integrator (Hewlett Packard 3396SA), using a fused silica capillary column (Hewlett Packard OV-1) and a flame-ionization detector (FID). The biodiesel yield (% wt) after the post-treatment stage relative to the amount of vegetable oil poured into the reactor was calculated from the methyl ester and vegetable oil weights.

Statistical analysis

The synthesis of biodiesel by Jatropha oil methanolysis using potassium hydroxide as the catalyst was developed and optimized following the Factorial Design and Response Surface Methodology. A factorial design was performed to study the effect of the variables on the process and the interaction among variables. The response surface methodology was applied to optimize the process. The experimental design applied to this study was a full 2^3 factorial design (three factors each at two levels). Application of this method requires the adequate selection of responses, factors and levels. The responses selected were the previously defined biodiesel purity, *P*, and biodiesel weigh yield, *Y*. Selection of the factors was based on the operating conditions that have a significant influence on the biodiesel process [8-10]. The factors chosen were temperature, *T*, initial catalyst concentration, *C*, and the molar ratio of methanol to vegetable oil, MR. Selection of the levels was carried out based on results obtained in preliminary studies [8], considering the experimental installation limits, and the working conditions limit for every reactant and product. The upper temperature level, 65 °C, was determined by the boiling point of methanol and the lower level was 25 °C. Catalyst concentration levels were 0.5% and 1.5% by weight of vegetable oil. The stoichiometry of reaction required 3 mol of methanol per mol of

triglyceride. The reaction was reversible and hence an excess of methanol was necessary to drive the equilibrium towards methyl ester formation. The levels of the methanol: vegetable oil molar ratios were 4.5:1 and 7.5:1 and the impeller speed was set at 600 rpm to avoid mass transfer limitations on the process [9-10].

RESULTS AND DISCUSSION

The factorial design was created and the response data collected, a model was fitted to the results and some graphs were generated to evaluate the effects. The output from fitting a mathematical model was used, and the two graphical methods were used to help predict, which factors were important for improving the yield of biodiesel in biodiesel production from Jatropha Oil. The design of experiment and reduced model are shown in table 1 and 2.

Table 1: Experiment matrix results: Factorial and Centre Points

Std Order	Run Order	Center Point	Blocks	T(^{O}C)	C(% wt)	MR	Y
12	1	1	1	65	1.5	7.5	98.42
10	2	1	1	65	1.5	4.5	90.43
4	3	1	1	65	0.5	7.5	99.28
3	4	1	1	25	1.5	4.5	97.17
12	5	1	1	65	0.5	7.5	98.32
6	6	1	1	25	1.5	7.5	96.88
11	7	1	1	65	0.5	4.5	98.7
1	8	1	1	25	0.5	4.5	96.96
6	9	1	1	45	1	6	98.4
8	10	1	1	45	1	6	97.94
5	11	1	1	45	1	6	98.63
9	12	1	1	45	1	6	97.48

The full model was fitted, which includes the three main effects, three two-way interactions, and one three-way interaction. The standard order was randomized in order to determine which of the effects are significant. Using Alpha = 0.05, the main effects for Catalyst concentration (C), the Methanol to Oil Ratio (MR) and Temperature-Catalyst Concentration-Methanol Ratio (TCMR) interaction were more significant.

After fitting the model, several plots were generated to visualize the effects, evaluate the fit of the reduced model, and do a residual analysis. A good standard by which to evaluate the model is to look at p-values. If all terms have p-values less than α level appropriate for the experiment, it can be confident that a good model was obtained. Here, α = 0.05 was chosen. The p-value for each term in the model is less than 0.05, indicating that the model is good and can be explore and validated. This model is considerably simpler and fits the data almost as well as the model with all terms. The residual error only increased by a small amount. The model is further checked by using the residual plots. The fitted values are the results predicted by the model. The residuals are the actual biodiesel yield minus the predicted biodiesel yield as presented in figure 2 and 3.

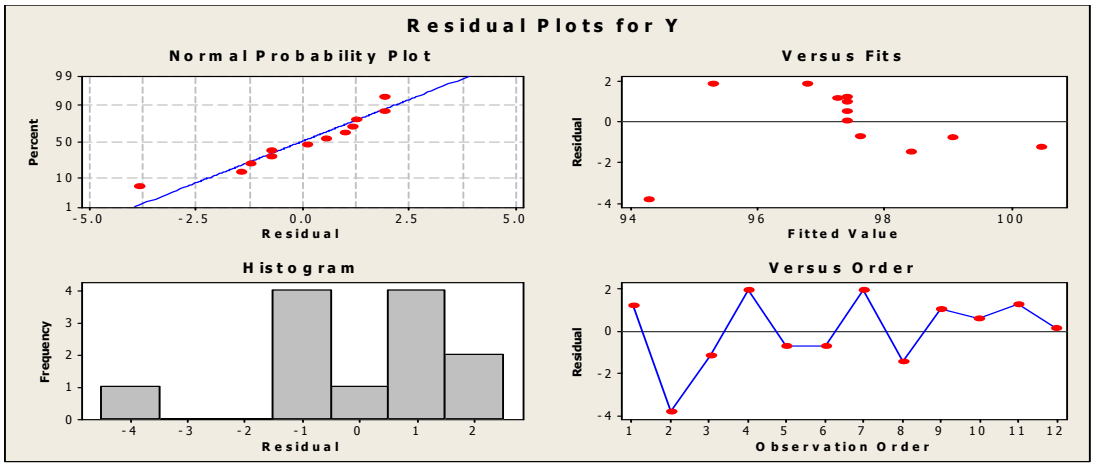

Fig 2: Residual plots for yield (Y)

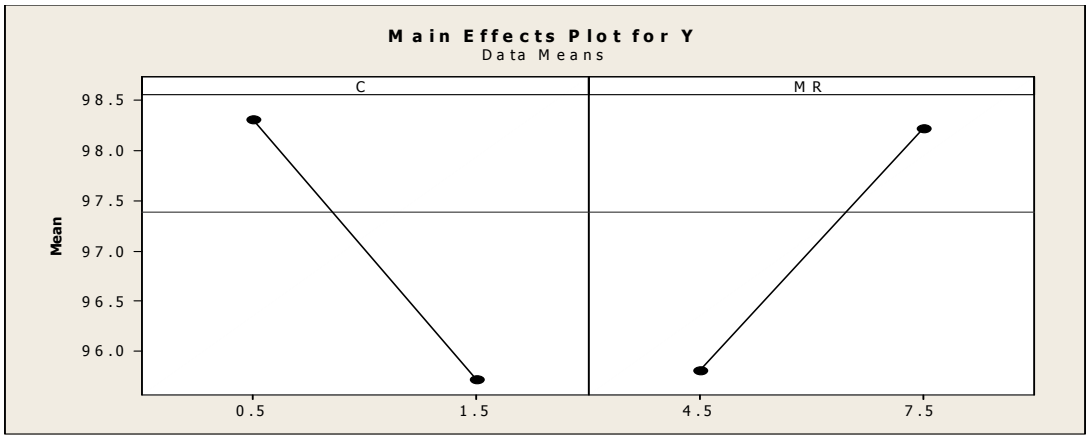

Fig. 3: Main effect plot for yield

Table 2: Estimated Statistical Parameters for Reduced Model

Statistical Parameters	Reduced Model	Full Model
S	0.995376	0.55376
PRESS	614.555	24614.555
R-Sq	93.28%	53.28%
R-Sq(adj)	81.53%	51.53%

The Biodiesel yield for methanol ratio of 7.5 is greater than Biodiesel yield for methanol ratio of 4.5 at both 0.5 and 1.5 of catalyst concentration. As observed from interaction plot (data means) for Biodiesel yield in figure 3, the difference in Biodiesel yield between runs using Methanol Ratio of 7.5 and runs using Methanol Ratio of 4.5 at catalyst concentration of 1.5 is much greater than the difference in waste removed between runs using Methanol Ratio of 7.5 and runs using Methanol Ratio of 4.5 at 1.5 catalyst concentration. In order to get the highest biodiesel yield for this experiment, the results suggest that the catalyst concentration should be set to 1.5 and use Methanol Ratio of 1.5. As it can be observed from Table 2, the adjusted root mean square values for the full model equation and reduced equation are 51.53% and 81.53% respectively.

CONCLUSION

Optimization of production of Biodiesel from Jatropha Oil by chemical reaction has been carried out. The effects of factors such as temperature, catalyst concentration and methanol to oil ratio on Biodiesel yield have been investigated. Optimization using factorial design experiment showed that catalyst concentration and methanol ratio factors affect the yield of biodiesel produced from chemical reaction. Optimization using factorial design experiment showed that the highest yield of Biodiesel is obtained by using a catalyst concentration of 1.5 methanol ratio of 7.5.

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