

Optimization of Process Variables in the production of Biodiesel from Jatropha-Neem Hybrid Feed stock Mixture

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ABSTRACT

In the search for suitable clean and renewable fuels to replace conventional diesel, alternatives such as biodiesels are being researched upon daily. Challenges of low biodiesel yield per hectare of vegetable oils have prompted the need for optimization. This research is aimed at optimizing process variables in the production of biodiesel from the mixture of Jatropha and Neem seed oils. A 31-run Central Composite Design was employed in a response surface optimization process; four independent process variables, namely temperature, time, catalyst concentration and mixing speed were optimized in an alkali based transesterification process of biodiesel synthesis. The result was a 97.05% biodiesel yield from Jatropha –Neem hybrid mixture.

Keywords: Biodiesel, Optimization, Process variables, Yield.

1. INTRODUCTION

Biodiesel is made up of fatty acid methyl esters synthesized by the transesterification of plant/animal oil with methanol [1]. Apart from being renewable, it has excellent fuel properties that can be compared to those of petrodiesel [2]. In addition, it has better lubrication advantages than diesel. [2, 3] It is also environmentally friendly [4]. The use of edible oil feed stocks for the production of biodiesel leads to problems of food security especially in developing countries. Besides, world population growth has increased the demand for food, giving way to the increase in price of edible oils. This will no doubt affect the potential for commercialization in the biodiesel industry with edible oil feedstock and cause inflation in food prices [5]. Biodiesel production from single edible oil feedstock is not sustainable in the long term because of the cultivation gap and non-availability of specific types of comestible oil plants for its adaptability to propagate in diverse regions with diverse eco-friendly conditions [6]. Accordingly, non-edible oil feed stocks have commercial potentials for biodiesel production. Some of the non-edible oils that are mostly used in the synthesis of biodiesel are Jatropha Curcas, Pongamia pinnata, Croton megalocarpus, Moringa oleifera, Aleurites, Azadirachta indica, Hevea brasiliensis, Nicotiana tabacum, Crambe abyssinica etc. [7, 8, 9, 10, 11, and 12]. Certain challenges like competing for land, high processing bills and emerging technologies have been pinned as factors that impeded some biodiesel sources from commercial applications. To properly rank biodiesel sources, a number of factors such as development of modern technologies, economic factors and the yield obtainable in every known weight of fuel are put into consideration [13].

Despite being a sustainable low carbon fuel, biodiesel can be used to address the issues of tailpipe emission of diesel engines; however, a number of studies have shown that there is the challenge of low yield per hectare from most plant based feedstock in use. Presently, most studies focus on the use of biodiesels developed from single-inedible plant feedstock like Jatropha and Neem. To optimize their properties, there is a need for a combination of feed stocks. This study aims to optimize the process variables in the production of biodiesel from the hybridized mixture of Jatropha and Neem oils.

2. LITERATURE REVIEW

Biodiesel feed stock can be classified edible and non-edible oils in plant and animal sources. Edible oil is made from plant sources; it is mostly consumed by humans as food. It contains a number of dietary constituents that makes it healthy and fit for human and animal consumption [14]. The extraction of edible oil from sources does not require any special chemical handling. Edible oils are more expensive than non-edible ones because of their demand as food oils hence limiting supplies. Certain difficulties like competition for land including high processing bills and emerging technologies have been identified as factors that slowed some biodiesel sources from commercial viability. Commonly, soybean, palm, rapeseed, sunflower and peanut oils are edible oils that can be used for biodiesel production. [15, 16]. Non-edible oils are oils that are not suitable for consumption by human beings. They are categorized as unhealthy and unhygienic, they are majorly employed in industrial and allied applications, some of which includes biofuels, soap making, in painting industries and in the manufacture of detergents. A number of chemical processes are necessary to make non-edible oils fit for different applications; it is therefore cheaper when applied in industrial applications. Common non-edible oil feed stocks employed for biodiesel productions are *Jatropha*, algae, including waste cooking oils.

Sankalp et al., (2016) [17] optimized the synthesis of biodiesel from degummed linseed, the catalyst employed was sodium hydroxide and the optimum yield obtained was 90% at a reaction temperature of 60°C. The entire reaction occurred in 60 minutes; a catalyst volume of 0.8% wt by volume was employed, the effects of varying process variables affecting biodiesel yield was not known.

Arnold [18] worked on the optimization of the transesterification of biodiesel production using numerical method; six process variables affecting biodiesel yield were optimized. They were optimized and analyzed using twelve (12) run Plackett-Burman experimental design. Canola oil was the feedstock used, using potassium carbonate as the catalyst, variation in temperature, stirring rate, alcohol to oil ratios and time of reaction were examined, the result obtained were imputed into the Minitab experimental design software; the experimental result shows a significant effect with the amount of Free Fatty Acid (FFA); methanol to oil ratio however shows the latest significance. The significance of catalyst concentration and reaction time is however not known.

Again, Arnold applied the Box Behnken method in optimizing the Free Fatty Acid content, catalyst amount and the stirring speed in a transesterification reaction. Analysis of Variance method (ANOVA) was used in the determination of the relationship between the yield of the Biodiesel and the three (3) process parameters mentioned. The yield was found to be greater than 98% in a 60 minutes reaction process. 4wt% of catalyst concentration was used at a stirring speed of 400rpm. Canola oil was used here. The result for *Jatropha curcas* and Neem oils are however not known if same methods were applied; the effect of temperature and time were also not specified.

David et al [19], attempted the optimization of factors affecting the synthesis of biodiesel from crude palm kernel oil and ethanol; he studied the effect of ethanol to oil ratio, temperature of reaction, catalyst concentration and reaction time using the completely randomized 2⁴ factorial design. Optimal conditions were determined to be 1% wt. vol of catalyst (KOH), 1hr 30min reaction time, 1:5 ratio of ethanol to oil and a reaction temperature of 30°C. The effect of the variation of the same parameters using *Jatropha curcas* oil is underdetermined. This work intends to vary the process variables for the optimization of yield and synthesis of biodiesel from a mixture of *Jatropha* and Neem oils.

Refaat, et al., [20], worked on the production and optimization of biodiesel from waste vegetable oil, the results of two (2) catalysts where NaOH and KOH were employed with varying degree of concentrations. Methanol to oil molar ratio was also kept at 6:1. A maximum yield of 96.15% was obtained under optimum condition of 1% wt vol catalyst, 65°C and 60 minutes reaction time. The experimental results were however not optimized using any known statistical methods or software. The result is also unknown for *Jatropha curcas*.

3. EXPERIMENTAL WORK / METHODOLOGY

This chapter discusses the equipment, materials and methods used during the research. It involves experimental design process using the MINITAB 21 software; Analysis of Variance (ANOVA), Variance Inflation Factors (VIF), yield model equation and optimization graphs all characterize the optimization method employed in obtaining optimum values for independent process variables (time, temperature, catalyst concentration and mixing speed) in this work.

The central composite design was employed as the process variable optimization method. High and low values of time, temperature, catalyst concentration were imputed and the ensuing result was a 31 run experimental design table.

3.1 Design of Experiment

The table below shows the generated experimental data, which was used to develop the yield model empirical equation.

Table 3.1 showing high and low values for process (independent) variables

Independent Variable	High	Low
Temperature °C	70	50
Time (Mins)	90	60
Catalyst concentration (wt%)	0.5	1.5
Mixing speed (RPM)	200	800

Table 3.2 Central Composite Experimental Design

Temperature	Time	Catalyst concentration	Mixing speed
60	75	1	-100
60	75	1	1100
50	60	1.5	800
70	90	1.5	200
50	90	0.5	200
60	75	1	500
70	60	0.5	200
50	60	1.5	200
70	60	0.5	800
60	75	1	500
70	60	1.5	200
60	45	1	500
70	90	0.5	200
60	75	2	500
60	75	1	500
50	60	0.5	200
50	90	1.5	800
70	60	1.5	800
50	90	1.5	200
60	75	0	500
60	105	1	500
60	75	1	500
70	90	0.5	800
60	75	1	500
50	90	0.5	800
60	75	1	500
80	75	1	500
60	75	1	500
40	75	1	500
70	90	1.5	800
50	60	0.5	800

The above table represents the central composite design experimental design table.

Sample Preparation

100ml of Jatropha-Neem (J-N) oil mixture, containing 60% of Jatropha and 40% Neem was employed. An Erlenmeyer conical flask was used as the reactor and different wt% KOH pellet (as specified in the experimental design table) was dissolved in approximately 21ml of methanol to formulate 21% of the J-N 60-40 hybrid oil. The dissolved catalyst in methanol formed a good mix; the mix was then poured into a pre heated J-N oil mixture. Pre heating was done on the Gallenkang heater magnetic stirrer hot plate. Temperature and mixing speed were monitored and regulated based on values assigned in the central composite experimental design table. Time of reaction was also and regulated based on values specified by the Minitab software. The same procedure was repeated 31 times with different values as arranged in the experimental design table. For each run, biodiesel yield values were measured and recorded; recorded yield values in percentages were fed back into the Minitab 21 software, regression analysis was done and optimal values for temperature, time, catalyst concentration and mixing speed were then obtained. The values were then taken back to the laboratory for production of the J-N biodiesel. The empirical model equation, surface and contour graphs depicting interactions between process variables and other analysis of the regression design are all shown in the next section.

The yield of biodiesel produced using predicted optimal values was then compared with what was obtained experimentally.

1. RESULTS

Result of Experimental Design for Optimization

Table 4.1: Percentage Yield of Biodiesel Using the Central Composite Design

RunOrder	PtType	Blocks	Temp (°C)	Time (Mins)	catalyst concentration (%wt)	mix speed (RPM)	Yield (%)
1	-1	1	60	75	1	-100	83.8
2	-1	1	60	75	1	1100	89.4
3	1	1	50	60	1.5	800	74.9
4	1	1	70	90	1.5	200	94.6
5	1	1	50	90	0.5	200	75.8
6	0	1	60	75	1	500	91.3
7	1	1	70	60	0.5	200	78.8
8	1	1	50	60	1.5	200	77.6
9	1	1	70	60	0.5	800	88.1
10	0	1	60	75	1	500	94.1
11	1	1	70	60	1.5	200	91.1
12	-1	1	60	45	1	500	84.7
13	1	1	70	90	0.5	200	81.3
14	-1	1	60	75	2	500	88.1
15	0	1	60	75	1	500	90.2
16	1	1	50	60	0.5	200	72.2
17	1	1	50	90	1.5	800	80.2
18	1	1	70	60	1.5	800	95.1
19	1	1	50	90	1.5	200	87.6
20	-1	1	60	75	0	500	63.5
21	-1	1	60	105	1	500	92.7
22	0	1	60	75	1	500	94.1
23	1	1	70	90	0.5	800	84.4
24	0	1	60	75	1	500	93.5

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4.1

25	1	1	50	90	0.5	800	76.6
26	0	1	60	75	1	500	94.6
27	-1	1	80	75	1	500	81.2
28	0	1	60	75	1	500	94
29	-1	1	40	75	1	500	69.3
30	1	1	70	90	1.5	800	94.7
31	1	1	50	60	0.5	800	75.6

table

above represents the yield as measured in the 31 runs experimental design predicted by the Central Composite Design (CCD) using the Minitab 21 experimental design software. Employing four factors at a time, the measured yield gave an intrinsic model equation that defined the interaction between the four process variables which were employed for the optimization process. Observed interactions gave a workable model equation that can predicting the yield of biodiesel when combined values of time, temperature, catalyst concentration and mixing speed are to be considered. The results of the regression analysis were presented in coded and uncoded units.

Table 4.2: Regression table in coded coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	93.114	0.941	98.99	0.000	
Temp	4.642	0.508	9.14	0.000	1.00
Time	1.575	0.508	3.10	0.007	1.00
cat conc	4.675	0.508	9.20	0.000	1.00
mix speed	0.908	0.508	1.79	0.093	1.00
temp*temp	-4.224	0.465	-9.08	0.000	1.03
time*time	-0.862	0.465	-1.85	0.083	1.03
cat conc*cat conc	-4.087	0.465	-8.78	0.000	1.03
mix speed*mix speed	-1.387	0.465	-2.98	0.009	1.03
temp*time	-1.125	0.622	-1.81	0.089	1.00
temp*cat conc	1.425	0.622	2.29	0.036	1.00
temp*mix speed	1.400	0.622	2.25	0.039	1.00
time*cat conc	0.937	0.622	1.51	0.151	1.00
time*mix speed	-1.087	0.622	-1.75	0.100	1.00
cat conc*mix speed	-1.412	0.622	-2.27	0.037	1.00

The table 4.2 above shows the results of the regression analysis in coded terms. The table shows how significant the independent process variables are in the optimization process, it also reveals how significant the quadratic interactions between the process variables are. For a Probability value (P value) of 5%, i.e 5% significance level, time, temperature and catalyst concentration as lone independent variables are significantly important. As for mixing speed, a P value of 0.093 shows that it is less significant. The interactions between the process variables also reveals that the quadratic interaction of time is less significant to the output of the design, the interaction between temperature and time is also less important, the interaction between time and catalyst concentration is also less significant. All other interactions with P value less than 0.05 are important and have the capacity to significantly affect the yield of biodiesel. The Analysis of Variance (ANOVA) table is presented in Appendix 3.

Variance Inflation Factor (VIF) depicts the level of multi collinearity. VIF value greater than 4 requires investigation, while VIF exceeding 10 requires serious correction. Multi collinearity makes it difficult to interpret coefficients and reduces the power of the model to identify independent variables that are statistically significant.

The generated model equation in un-coded units is as shown below:

$$\begin{aligned}
 \text{Yield} = & -154.3 + 5.578 \text{ temp} + 1.125 \text{ time} + 20.3 \text{ cat conc} + 0.0180 \text{ mix speed} - 0.04224 \text{ temp*temp} \\
 & - 0.00383 \text{ time*time} - 16.35 \text{ cat conc*cat conc} - 0.000015 \text{ mix speed*mix speed} - \\
 & 0.00750 \text{ temp*time} + 0.285 \text{ temp*cat conc} \\
 & + 0.000467 \text{ temp*mix speed} + 0.1250 \text{ time*cat conc} - 0.000242 \text{ time*mix speed} - \\
 & 0.00942 \text{ cat conc*mix speed}.
 \end{aligned}$$

Below are contour and surface curves depicting the yields in different regions of optimization of process variables.

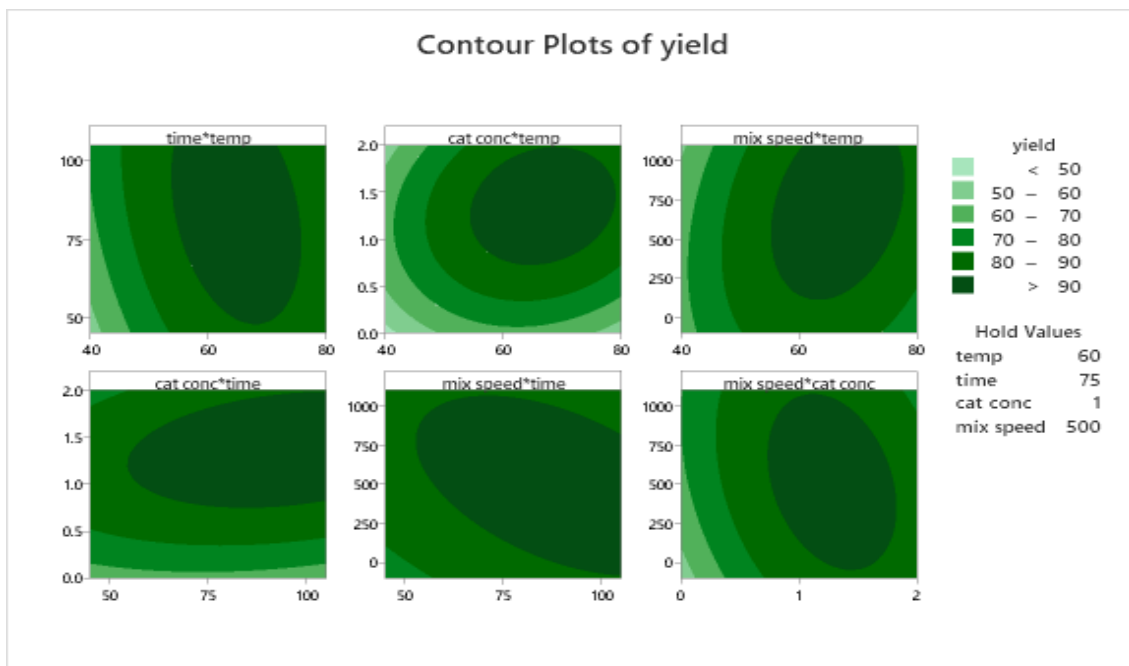


Figure 4.1 : Contour graphs Showing interaction between the process variables and yield

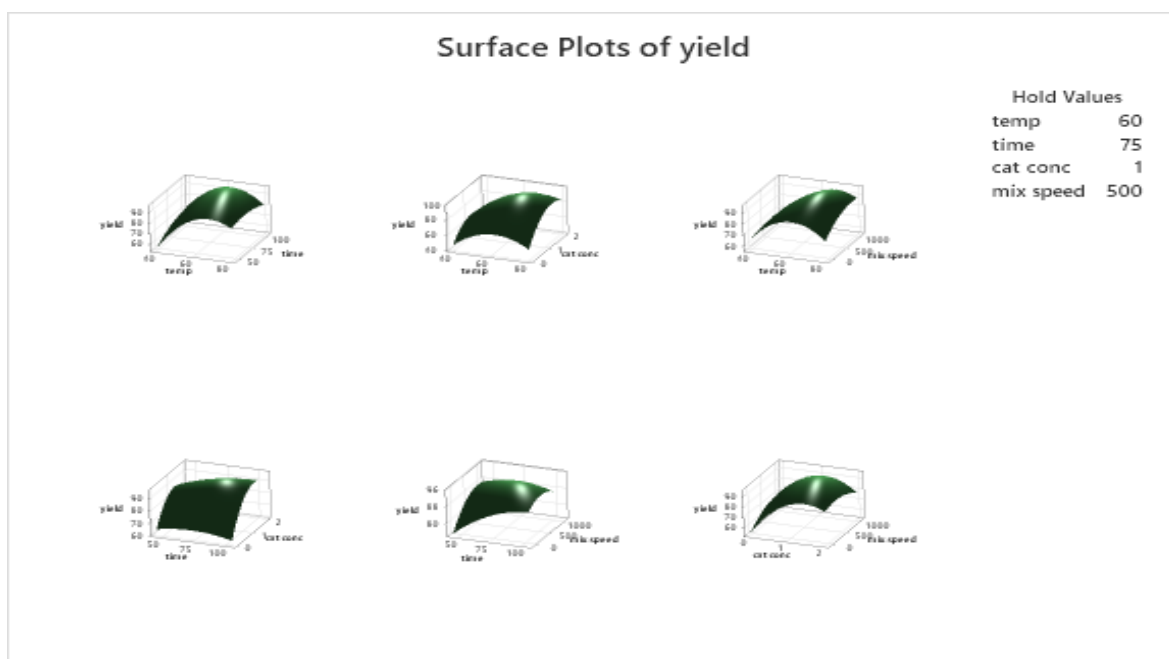


Figure 4.2: Surface curves showing the interaction between process variables and yield

The figures 4.1 and 4.2 depict the contour and surface curves showing interactions between process variables and the possible yields. The darker the region, the better the yield. The darkest region in the contour curves depicts the region with the highest yield values of biodiesel. The solution to the central composite design optimization process is depicted in the graph below:

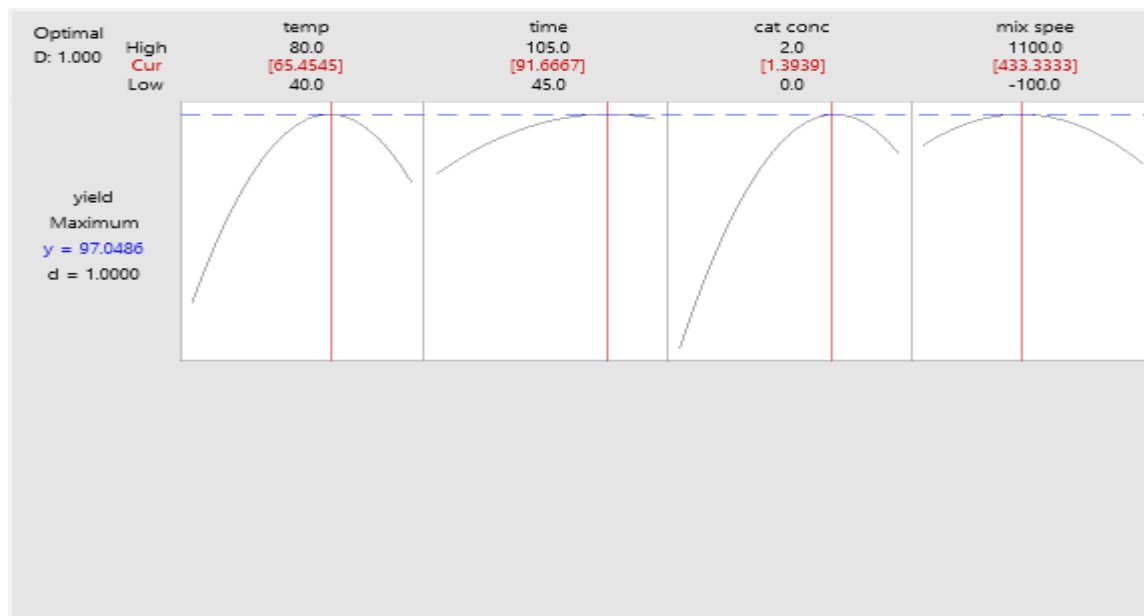


Figure 4.6: Optimization Graph

Validation

Using the solution above, Jatropha-Neem Biodiesel (JN-B) produced gave a yield of 97.9%. The percentage error is then calculated as thus:

$$\% \text{ error} = \frac{\text{observed value} - \text{theoretical value}}{\text{theoretical value}}$$

$$= \frac{97.9 - 97.0486}{97.0486} = 0.88\%$$

A low percentage error is an indication of the accuracy of the process and instrument used to measure and estimate values experimentally.

5. CONCLUSIONS

The solution predicts that if JN 60:40 is used to produce biodiesel at a temperature of 65.45°C, time of 91.67 minutes, catalyst concentration of 1.394wt% and a mixing speed of 433.33 RPM, the resulting yield is expected to be 97.0486% biodiesel.

The accuracy of the MINITAB 21 experimental design software is established, hence the percentage error was very low.

Acknowledgement:

This study is an extract from a PhD research work.

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