International Journal of Engineering Sciences & Research Technology

(A Peer Reviewed Online Journal)
Impact Factor: 5.164

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IJESRT
INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY

IMPROVING JATROPHA BIODIESEL YIELD THROUGH THE BOX-BEHNKEN PROCESS VARIABLES OPTIMIZATION METHOD

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DOI: 10.5281/zenodo.2540680

ABSTRACT

Evolving challenges due to global warming, emissions and depleting natural reserves have prompted the search for alternatives to fossil fuels. Biodiesel is one such promising alternative. However, the significantly low yield from biodiesel feedstock sources has been a deterring factor preventing its large scale production in Nigeria. This research work is therefore aimed at optimizing process variables affecting the yield of biodiesel from jatropha curcas crude. Esterification of the crude was carried out using 1% (w/w) of sulphuric acid as catalyst in order to reduce its free fatty acid content. Upon esterification, three process variables; catalyst concentration, reaction temperature and the reaction time were varied simultaneously at different ratios for 15 run of the Box-Behnken Design while transeterifying the crude maintaining a constant methanol to oil ratio of 6:1. The Box-Behnken method was used to optimize the investigated variables. The result show an optimal yield of 99 % biodiesel for a 60°C reaction temperature, 90 minutes reaction time and 1% (w/w) catalyst concentration. A model equation was also developed that could be used to determine the optimum operating ratios of the process variables for best yields.

KEYWORDS: Biodiesel, Transesterification, Optimization, Yield, Box-Behnken.

1. INTRODUCTION

The importance of energy in life generally cannot be over emphasized. It is essential to the socio economic and technological development of any nation, such that the increase in its demand around the world is linked to the prosperity and material well-being of the people. It is the predominant and most universal measure of work by human beings and nature [1]. From the beginning of the industrial revolution till date, a large percentage of the world's energy needs are supplied by fossil fuels (coal, petroleum and its derivatives and natural gas), and countries draws their energy needs from these variety of resources locally or import them. In Nigeria, diesel fuel (petro diesel), one of the petroleum resource derivatives there is, plays a vital role in in the transportation of goods and services, power generation and in the operation of heavy duty machineries in the construction and agricultural industries thus contributing a lot to advancing the economy of the country. However, regardless of the enormous contribution of the fuel, its use has been associated with exhaust pollutant emissions that are hazardous to the environment. In line with best global practices therefore, there is the dire need to search for suitable environmentally friendly fuels to replace it. In addition to the aforesaid, depleting natural reserves, irregular supply, frequent rise in pump prices etc. are some of the factors that have also added pressure to the search for newer alternative fuels. The eventual use of alternative fuels will reduce the country's overdependence on petroleum. One such alternative is biodiesel. Biodiesel consists of fatty acid methyl esters produced by transesterifying long chain fatty acids obtained from vegetable oil or animal fat with methanol [2]. Not only is biodiesel a renewable fuel, it possesses some properties that are superior to those of petro diesel [3]. For instance the flash point of biodiesel is higher than that of petro diesel thus making it a safer fuel to handle during transportation and handling when compared to petro-diesel. In addition to the flash point, it has better lubricity benefits [3, 4]. Furthermore, it can be used in diesel engines, sometimes only with little modifications [2]. It is in this regard that the Federal Government of Nigeria (FGN) through the National Biofuel Policy and Incentives recommended the addition of biodiesel in the country's energy mix [5].

The transesterification of biodiesel involves either the use of a base or an acid catalyst. The common base catalysts used in base-catalyzed transesterification are potassium hydroxide (KOH) and sodium hydroxide (NaOH) while hydrochloric acid (HCl) and Sulphuric (H_2SO_4) are utilized for the acid-catalyzed transesterification. Today biodiesels are mostly produced using a base-catalyst together with methanol. It results in a relatively short reaction time and more yield compared to the use of acid catalyst, but, the vegetable oil and alcohol must be substantially anhydrous and have low free fatty acid (FFA) content [6]. The presence of water or high FFA content or both promotes soap formation. Soap formed lowers the yield of esters and renders the downstream separation of the products difficult, requiring additional processing. Other setbacks arising from the use of homogeneous base catalysts are difficulty in purification of glycerol byproducts and the need for wastewater treatment. [7]. In spite of these however, base-catalyzed transesterification is still the preferred method for the production of biodiesel. Biodiesel can be produced from vegetable oils such as soya oil, neem oil, palm oil, castor oil, algae oil and jatropha oil. However, the use of Jatropha for producing biodiesel in Nigeria has been the priority because it is considered non edible. Therefore utilizing it for biodiesel production will not add to the pressure on soya, palm oil used for food production in the country. Besides, Jatropha plant can be cultivated in different parts of the country particularly in the northern part. Jatropha curcas referred to as “lapa-lapa” in Yoruba, “oluluidu” in Igbo and “bini da zuugu” in hausa is a perennial, non edible oil bearing plant belonging to *Euphorbiaceae* family. A substantial amount of oil could be obtained from its seeds. It is majorly found in arid, semi-arid and tropical regions of the world. Jatropha is a drought resistant tree that grows in marginal lands that is known to have the capacity to live over 50 years [8]. Some other uses of the jatropha tree includes its stem which is used as a natural tooth paste and brush, latex from stem is being used as natural pesticides and wound healing, its leaf as feed for silkworms among other uses [8]. It grows rapidly and can be easily propagated. One hectare of Jatropha can produce up to 2000 litres of biodiesel as against 5950 and 2638 litres/hectare for palm oil and avocado respectively [9]. Thus considering the challenges of food security that has afflicted the country, it is essential to promote the conservation and use of under-utilized and neglected crops, such as jatropha for biodiesel fuel production. It has a great potential of taking the place of petro-diesel in the energy market but for its low yield per hectare compared to conventional biodiesel feedstock such as soya oil, rape seed oil, etc. There is therefore the need to optimize its yield particularly for the purpose of large scale commercialization. Optimization of biodiesel yield is usually dependent on certain process variables. These process variables as identified by Barnwal and Sharma [10] are reaction temperature, ratio of alcohol to vegetable oil, catalyst concentration, mixing intensity and purity of reactants. Either one or two or three of these variables are varied separately or simultaneously depending on the experimental design setup. Dixit and Dixit [11] optimized the production of biodiesel from water degummed linseed by varying the concentration of the Sodium hydroxide catalyst used, reaction time and reaction temperature. A 90% biodiesel yield was obtained at optimum operating conditions of 0.8% w/w catalyst concentration, I hour and 60ºC. Arnold [12] carried out a study on the optimization of the transesterification stage of canola oil biodiesel using a statistical method. Six process variables affecting yield were examined using twelve (12) run Plackett-Burman Design, with potassium carbonate as the catalyst in order to study the variation of this variables on the FFA content of the oil. Linoleic acid was used to stimulate oleic content of the investigated oil. Apart from varying the amount of catalyst, temperature, stirring rate, alcohol to oil ratios, the time of reaction was also varied. The results obtained from the design were imputed into the Minitab software to determine the active factors that will affect yield and an experiment conducted showed that the methanol to oil ratio affects the biodiesel the least while the FFA affects it the most. Arnold [12] again applied the Box Behnken method in optimizing the FFA content, catalyst amount and the stirring speed on the canola oil. The Analysis of variance method (ANOVA) was further used in the determination of the relationship between the yield of the Biodiesel and the three (3) process parameters mentioned above. A yield of greater than 98% for optimum conditions of 0.5 % w/w FFA, 400 rpm, 4 % w/w catalyst at 60ºC, 6:1 methanol-oil ratio for 100g of the canola oil. David and Julius [13], conducted a study on the optimization of factors affecting the yield of palm kernel oil biodiesel from its crude and ethanol. The study focused on the effect of varying ethanol to oil ratio, reaction temperature, catalyst concentration and reaction time on the yield using the completely randomized 2^4 factorial design. The result revealed the optimal conditions to be 1% w/w of KOH catalyst, 1hr 30min reaction time, 1:5 mean ratio of ethanol to oil and a reaction temperature of 30^0C for an 83.9% yield. Refaat, et al [14], investigated certain process variables: methanol-oil ratio, catalyst concentration, reaction temperature and time affecting the yield waste vegetable oil biodiesel. The result revealed that at optimum operating conditions of 6:1methanol to oil molar ratio, 1 % (w/w) of the catalyst, 65ºC and 60 minutes, a maximum yield of 96.15% was obtained. However no known statistical methods were employed in the investigation. Many other researchers too have investigated the effect of optimizing process variables involved in the production of biodiesels using the Box Behnken method and the relationship between the yield and parameters varied with the aid of the ANOVA. The results deduced from the investigation indicates an improvement of well above 97% yield [15, 16]. Jatropha is one of the promising energy crop in Nigeria for the actualization of commercial scale production of biodiesel. However the yield and quality of the biodiesel produced from the crude also depends on the geography of the location of cultivation. Several researchers have produced biodiesel from Nigerian Jatropha [17 18], but there is the need to further research into effective methods of boosting its yield for the sake of commercialization. Therefore, this research work is aimed at determining the optimum operating condition that will give maximum yield by optimizing the process variables affecting the production of biodiesels from the Jatropha crude investigated.

2. MATERIALS AND METHODS

2.1 Materials

The Jatropha crude used for the investigation was obtained from the National Research Institute for Chemical Technology (NARICT) Zaria, Kaduna State. The crude was characterized for the kinematic viscosity, lower heating value (LHV), density, acid and saponification values, ash and moisture content at the Chemical Engineering Department Laboratory of the Kaduna Polytechnic. The transesterification of the crude to biodiesel was carried out in accordance to the American Society for Testing and Materials (ASTM) D 6751-15 CƐ1 in the same institution and so also, the optimization process. A 250ml Erlenmeyer conical flask, a measuring beaker, a Gallernkang magnetic stirrer hot plate heater, a temperature probe device, potassium hydroxide pellets, analytical grade methanol and the Minitab software were some of the equipment utilized for the investigation.

2.2 Methods

2.2.1 Experimental Design

The Box-Behnken method was used in optimizing the process variables examined: catalyst concentration, reaction temperature and time in the design of the experiment. It is a spherical design with three levels having all points lying on the sphere of radius $\sqrt{2}$. The design does not allow for any point in vertices of the cubic region created by the limit (upper and lower) of any considered variable. The Box-Behnken design (BBD) as obtained in the Minitab 16 software is utilized in place of the central composite design because the axial and corner points are extremely high and such high levels are not common. This feature significantly reduces the number of experimental runs for the BBD. A model was also developed based on the BBD to describe the effect of yield as an output response factor. It records an illustration of how the three process variables being studied can be used to obtain the maximum possible biodiesel yield from the Jatropha crude. The table 2.1 below shows the possible combinations of the variables as obtained from the BBD that is expected to produce the maximum yield.

Table 2.1 Experimental design of possible combinations of the process variables

Number of Runs	Temperature (^{0}C)	Time (mins)	Catalyst concentration (%wt.vol)
1	50	120	1.0
2	50	90	1.5
3	60	90	1.0
4	70	120	1.0
5	60	60	1.5
6	70	90	0.5
7	60	120	0.5
8	60	90	1.0
9	60	120	1.5
10	50	90	0.5
11	60	60	0.5
`12	70	90	1.5

13	50	60	1.0
14	60	90	1.0
15	70	60	1.0

2.2.2 Experimental Set-up

The Jatropha crude was pretreated with 1% w/w H_2SO_4, and 20% methanol in an esterification reaction contained in a glass reactor with a stirrer inside. The preheating was to reduce the FFA content to a value below 3% required for optimal production. After stirring, the crude was allowed to settle for 24 hrs before proceeding with transesterification. The reactor is a 250ml Erlenmeyer conical flask. The Biodiesel was produced in batches using the conditions of process variables specified by the Box Behnken experimental design. Also100ml of the pretreated Jatropha crude was poured into the reactor and different the different concentration of KOH pellet specified from the experimental design was dissolved in approximately 21 ml methanol which is 21% of Jatropha crude. The 21% used is for maintaining the 6:1 methanol-oil ratio in order to have an excellent yield. The dissolution of the catalyst in methanol formed a methoxide mix. The methoxide mix was then poured into the crude which was already being pre heated on a Gallenkang heater magnetic stirrer regular hotplate. The mix was then for the BBD design time and allowed to settle for about 24hrs. Glycerin settled at the bottom; a separating funnel was used to collect the glycerin leaving the raw biodiesel. The raw biodiesel was washed using de-ionized water in order to remove impurities. The pure biodiesel was afterward collected, its volume measured and yield calculated as a percentage of the Jatropha crude used in the production process. The same process was repeated for fifteen times using different values of time, temperature and catalyst concentrations as specified in each batch of the experimental design. The results of yields for the fifteen (15) experimental runs are presented in the next section.

2.2.3 Optimization

The yields obtained from each experimental run were imputed into the mini tab Design Expert 9.0 software and the response surface Design (RSD) analyzed. The analysis of variance (ANOVA) was statistically use to analyze the response model. The interactions between the three process variable considered were also ANOVA analyzed and their levels of significance tabulated. The conditions for optimal yield of bio-diesel from the Jatropha crude were obtained with the relevant contour and surface plots displaying regions of optimization. These conditions for optimal yield were then taken back to the Laboratory for experimental validation.

3. RESULTS AND DISCUSSION

The results of the physicochemical properties of the Jatropha crude, Jatropha biodiesel and ASTM cƐ1 are presented in Table 3.1.

Table 3.1Physicochemical properties of the Jatropha crude, Jaropha biodiesel produced and ASTM c Ɛ1 Standard

S/No	Physicochemical property	Jatropha crude	Jatropha biodiesel	ASTM cƐ1 [19]
1	Acid value (mgKOH/g)	1.86	0.76	0.50
2	Ash content (%)	0.02	0.02	0.02
3	Density (kg/m^3)	828	868	-
4	Iodine value (mg/100g)	53.6	98	130max
5	Moisture content (%)	0.07	0.06	0.05
6	Kinematic Viscosity (mm^2/s) @ 40^{o}C	6.629	6.054	1.9-60
7	Lower heating value (MJ/kg)	38.26	41.29	-

Table 3.1 compares the physicochemical properties: acid value, ash content, density, iodine value, moisture content, kinematic viscosity and the lower heating value of the Jatropha crude and the resulting biodiesel with that of the ASTM cƐ1. As can be seen from the table transesterification had a significant impact on the lower heating value. The lower heating value increased from 38.26 to 41.29 MJ/kg. It is a boost that is good and desirable as it pertains to the heat liberated when the fuel is combusted in an engine under standard conditions. On the other hand, the kinematic viscosity was not significantly influenced by transesterifying the Jatropha crude. As revealed on the Table, it only reduced from 6.629 to 6.054 mm^2/s and marginally exceeded the ASTM

maximum by 0.054 mm^2/s. If not further reduced, on the long run, the continual use of the fuel might present operability problems in an engine particularly the issue of poor atomization. A marginal drop in the value of the moisture content from 0.07 for the Jatropha crude to 0.06 % for the biodiesel was also observed. The value exceeds the 0.05 stipulated by the ASTM standard by 0.01%. The acid value is one of the most important properties for biodiesel quality check. It quantifies the amount of corrosive acid as well as oxidation products present in the fuel should be lower than 0.50 mgKOH/g specified by ASTM standard. The acid value deduced herein is found to be higher (0.76 mgKOH/g) that of the ASTM standard even though transesterification reduced it from 1.86 mgKOH/g for the crude. Therefore the Jatropha biodiesel needs to be treated to reduce the acid value to the acceptable limit. The ash content did not change. It is the same for both the crude and the biodiesel, and meets the ASTM requirement. The transesterification conducted boosted the density from 828 to 868 kg/m^3. The Iodine value was also enhanced from 53.6 to 98 mg/100g and falls within the ASTM limit. The result in percentage yield of Bio-diesel from the BBD is presented in Table 3.2. It consists of the average values of yield for each 15-runs undertaken. The corresponding estimated regression coefficient of yield, standards deviation and confidence intervals deduced from the ANOVA analysis are presented in Table 3.3 while Table 3.4 presents the results of the ANOVA analysis.

Table 3.2 Percentage yield of the BBD

Runs	Temperature (°C)	Time (mins)	Catalyst concentration (% w/w)	Yield(% w/w)
1	50	120	1.0	82.0
2	50	90	1.5	65.0
3	60	90	1.0	99.0
4	70	120	1.0	83.5
5	60	60	1.5	67.0
6	70	90	0.5	70.5
7	60	120	0.5	69.5
8	60	90	1.0	98.7
9	60	120	1.5	62.5
10	50	90	0.5	60.0
11	60	60	0.5	61.0
12	70	90	1.5	70.0
13	50	60	1.0	77.0
14	60	90	1.0	98.5
15	70	60	1.0	85.0

Table 3.3 Estimated Regressions Co-efficient For Yield

Term	Coef	SE Coef	T	P	Significance
Constant	-448.171	29.4655	-15.210	0.000	Significant
Temp (x)	10.365	0.8457	12.256	0.000	Significant
Time (y)	2.396	0.1931	12.412	0.000	Significant
catalyst concentration (z)	233.808	10.2982	22.704	0.000	Significant
temp*temp (x^2)	-0.077	0.0068	-11.450	0.000	Significant
time*time (y^2)	-0.010	0.0008	-13.484	0.000	Significant
catalyst concentration*catalyst concentration (z^2)	-98.467	2.7044	-36.409	0.000	Significant
temp*time (xy)	-0.005	0.0022	-2.502	0.054	Not significant
Temp*catalyst concentration (xz)	-0.275	0.1299	-2.117	0.088	Not significant
time*catalyst concentration (yz)	-0.217	0.0433	-5.003	0.004	Significant

Table 3.4 Analysis of variance (ANOVA)

Source of variation	Degrees of Freedom	Sum of squares	Adjusted Squares	Adjusted mean square	F	P
Regression	9	2684.46	2684.46	298.273	176.72	0.000
Linear	3	86.69	1119.91	373.302	221.17	0.000
Square	3	2537.40	2537.40	845.799	501.11	0.000
Interaction	3	60.37	60.37	20.125	11.92	0.010
Residual Error	5	8.44	8.44	1.688	-	-
Pure Error	2	0.13	0.13	0.063	-	-
Total Error	14	2692.90	-	-	-	-

Table 3.3 shows the estimated regression coefficient of the biodiesel yield as a result of interaction between the process variables temperature, time and catalyst concentration represented by the terms x, y and z respectively. An interaction of a variable with itself yielded a square or quadratic terms while an interaction between any two different variables yields a product as is the case with the interaction between time (y) and catalyst concentration (z). The P- value for all the linear and quadratic terms are seen to be 0.000 but for temperature-time (x y) and temperature-catalyst concentration (x z) with 0.054 and 0.088 respectively. The interaction of time-catalyst concentration (y z) was observed to be 0.004 just a little than 0.000. All the interactions with P-values less than 0.05 [B] have significant impact on the biodiesel yield. The regression model, equation 1, was generated from the Table 3.3. The regression model could be used to predict the optimum ratios of the investigated variables that will give a maximum biodiesel Yield (Y) under the same operating condition as applied to Jatropha crude. It is further observed that from the source of variation on table 3.4, that the sum of the squares as a result residual error is higher than that of pure error but less than the total error. The sum of squares due to pure error is much smaller when compared to that of the total error. This observation is comparable that in the literature [12]. It is further observed that the values of the sum of squares and adjusted squares are the same except for the total error with no adjusted squares value generated. However a significant reduction in the values of the adjusted mean squares observed. These observations show that the model is well adjusted and the obtained P-values at 0.000 but for the scenario when one of the process variable interacts with another at 0.010. The analysis on Table 3.3 was used to generate the following regression model for estimation of yield (Y). It indicates the correlation between the yield and the independent parameter deduced from the response surface method by the BBD.

$$Y = -10.365x + 2.396y + 233.808z - 0.077x^2 - 0.010y^{2+} - 98.467z^2 - 0.005xy - 0.275yz - 0.217xz \quad (1)$$

The Fig 1 is the normality graph of the residual plots for the biodiesel yield. It shows the normal probability plot of the residuals, residual versus the fitted values, residuals versus the order of data and a histogram of frequency versus residuals.

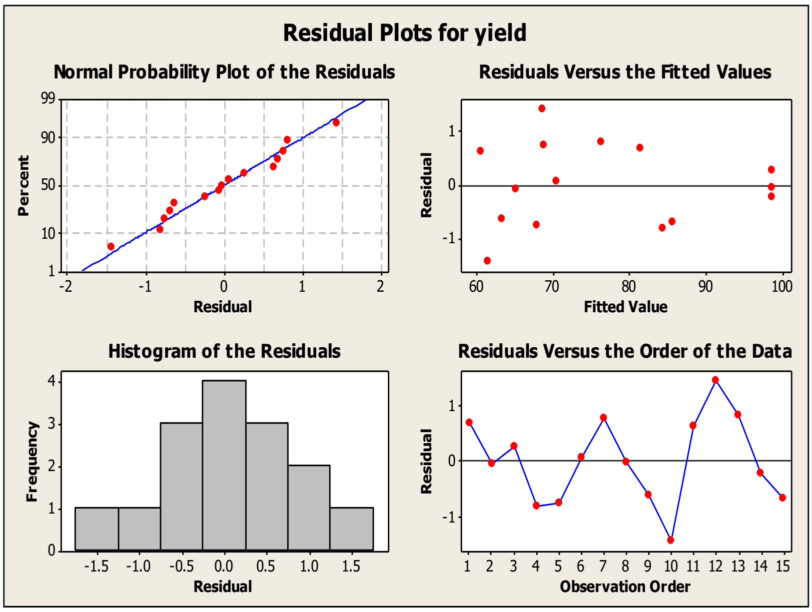

Figure 1 Normality Graphs

The residual probability plot for the investigation is shown in Fig 1 as the normal probability plot of the residuals. Residuals are the differences between the observed response values and the values predicted by the model at each combination of the values of the process being examined and helps determine the validity of the obtained model. As expected almost all the points (number of runs) fall on the line, an indication that the model fits the data well and thus follow a normal distribution. The residual versus the fitted values plot reveals a scatter of the points about zero as anticipated. The observation is a further substantiation of how good the model fits the data. Similarly, the residual histogram shows a normal distribution of the equally spaced residual from -1.5 to +1.5 for 0.5 interval. Likewise, the plot of residuals against the order of data of the 15 runs used in the BBD show their random distribution about 0 and fluctuating with peaks at -1.5 and +1.5 on opposite sides further confirming a good match between the model and the data. Also as displayed in the residual probability plot, at 95% level of significance, the linear and quadratic terms were found to be significant as well as the interaction term for time and catalyst concentration as shown in Table 3.3 and Table 3.4. None the less, the interaction terms for temperature and time was insignificant compared to the interactions term for temperature and catalyst concentration. The R^2 being the co-efficient of determination was found `to be 99.7% while R^2adjusted being 99.1%. The observation shows that the model accounted for 99.1% of the entire variation in the process variation and only 0.9% was not explained by the model. This means that the reliability of the model is high. The high R^2 and the repeated less than the 0.05 P-values obtained in Table 3.3 and Table 3.4 indicates that the model was appropriate and applicable for the investigation.

The contour and surface plots shown in Fig 3.2 (a-c) shows the yield in percentage terms of the Jatropha biodiesel while varying the process variables based on the design in Table 2.1. The yield for each set of run is plotted against the number of runs (Fig 2). It is observed in Fig 2 that run 3 produced the maximum yield of 99.0% while holding the temperature at 60ºC, catalyst concentration at 1.0% and time 90 minutes. Interestingly, this maximum yield is followed by run 8, 98.7% and run 14, 98.5% maintaining the same proportion as that of 3. This difference though minute, could be attributed to the effect other variables like the stirring rate. The stirring was manually carried out while the reaction chamber top was open. The least yield 60.0 % was obtained with run 10 at 50ºC, 90 minutes and 0.5 catalyst concentration. In all the 15 runs conducted, the obtained yield was found to between a minimum of 60% and maximum 99% per 100 ml of Jatropha crude used while the maximum reaction time was 120 minutes. The least catalyst concentration that was used is 0.5%. However the variables juxtaposition with 1.0 % of catalyst gave higher yields. It shows that the best catalyst concentration for the generated model is 1%. The contour plots presented in Fig 3.2 is a further revelation of the interplay of the variables for each of the run. The dark green region of the contours is a region of optimum mix of the process variables for best yield. From the reaction time and temperature plot, the contour region in between 75 and 115 minutes and 55 and 67°C will yield the optimum mix ratios, while for catalyst concentration it falls somewhere in-between 0.80 and 1.2 % and 55and 68°C. Similarly it is 0.80 and 1.20% and 75 and 105 minute for the catalyst concentration and reaction time. Holding a Specified condition for any of the process variable could be clearly marked out for a corresponding yield value in the contour plots. For instance holding reaction temperature at 60°C, reaction time at 90 minutes and 1 % catalyst concentration produce the maximum yield of 99% while maintaining a 6:1 methanol to oil ratio used throughout the investigation. It is therefore expected that using this same model under the same operating conditions for Jatropha crude, will produce a maximum yield of 99% Jatropha Biodiesel (JB100).

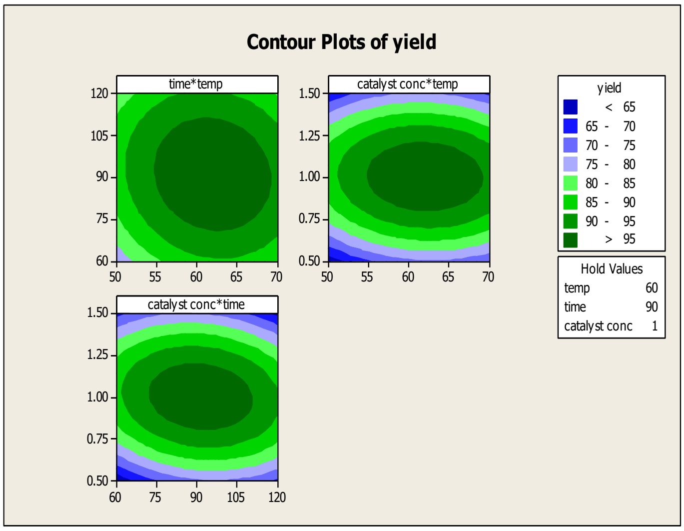

Figure 2 contour plots of the yields

4. CONCLUSION

In many fields, where optimal conditions for production are required, various designs have been proposed and have been implemented successfully. In the field of biofuels and especially biodiesel production, these methods are rarely used. The common methods applied are best guess and the one factor- at-a-time techniques. However, owing to the dependency that exists between certain process variables, the best guess and one factor at-a-time approaches could be misleading and somewhat inaccurate and therefore limited. Therefore factorial designs are used to address the limitations of the best guess and one factor at-a-time methods. The Box-Behnken method was used effectively in this investigation to determine and optimize the active process variables at the transesterification stage of biodiesel from locally sourced Jatropha crude. It was used to optimize the reaction temperature, time and the concentration of catalyst required for the biodiesel production. The contour plots were effectively used in determining the optimum process variable combinations for the maximum yield. Using the design of experiments, the numbers of runs were reduced to 15. The 15 runs is a few run compared to the more cumbersome guess and factor at a time methods. Consequently, the cost of running the experiments is less; the time to run the experiments and the time taken to obtain the optimum conditions for production of biodiesel is also less. In all the obtained results from this investigation could be used to model similar future experiments involving the optimization of Jatropha biodiesel yield from its crude. While statistical methods have been used to determine the optimum factor combinations for biodiesel production, the model equation obtained can be used as a base to predict the effects of the process variables on Jatropha crude and several other different feedstock available in the country.

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CITE AN ARTICLE

Solomon, W. C., Kolade, O. A., & A Sa’ad. (2019). IMPROVING JATROPHA BIODIESEL YIELD THROUGH THE BOX-BEHNKEN PROCESS VARIABLES OPTIMIZATION METHOD. *INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY*, *8*(1), 118-126.