D-Optimal Experimental Design of Biodiesel Production from Waste Cooking Oil of ABUAD Cafeterias

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Abstract - Biodiesel is a renewable, biodegradable, alternative, environmental friendly, and clean fuel used in engines. It can be produced from renewable sources like vegetable oils and animal fat. However, it is better if it can be obtained from a non-edible source or a waste. As a result of this, waste cooking oil, which had low free fatty acid content, obtained from two cafeterias of Afe Babalola University, Ado-Ekiti (ABUAD), Ekiti State, Nigeria has been utilized in this work to produce biodiesel via a transesterification reaction between the oil and methanol in the presence of sodium hydroxide catalyst by applying the D-optimal technique with the aid of Design-Expert to design the experiments that were carried out. The input parameters of the process were reaction temperature, catalyst concentration, and reaction time. After the production, the biodiesel samples were analysed and some of their properties were compared and found to fall within the ranges of the standard values, which was an indication that the material produced was actually biodiesel. In addition, a model of the system was formulated and optimized using the D-optimal design technique, and it was discovered to represent the system very well because it was found to be significant as its probability value was estimated to be less than 0.05 that was chosen based on the confidence level of 95%, and its high R-squared value was also found to be very close to unity (1). Therefore, D-optimal experimental design technique has been successfully applied to biodiesel production from waste cooking oil of the cafeterias of ABUAD.

Keywords: Biodiesel, waste cooking oil, D-optimal, Design-Expert, ABUAD.

1. INTRODUCTION

The increase in energy demand around the globe, the depletion of fossil fuels, and the fluctuation of crude oil price in the international market as well as the greater recognition of the unfavourable environmental consequences of fossil fuels have made renewable bio-fuels an attractive alternative to conventional fuels. Today, it is very essential to use alternative fuels because of energy security, environmental concerns and socio-economic reasons. Escalating oil prices and depletion of oil reserves necessitate better alternatives of energy from fossil fuels. With the rise in concern for pollution caused by fossil fuels such as petroleum, coal and natural gas, alternative fuels and renewable sources of energy such as biodiesel are coming in vogue (Garlapati *et al.,* 2013). Besides, the side effect of petroleum based fuels is that over the years there has been a steady increase in the amount of pollution produced by these fuels. The use of these energy sources over many years has resulted to the rise in global temperature levels, otherwise known as "global warming" (Ogunwole, 2012). As a result of this, over the last few years, biodiesel, which can be produced from used cooking oil, has gained importance as an alternative fuel for diesel engines because it could be used to prevent global warming. Moreover, biodiesel is gaining ground because it is a biodegradable, environmental friendly, and readily available fuel (Adepoju and Olamide, 2014).

Manufacturing biodiesel from used vegetable oil is relatively easy and possesses many environmental

benefits. The use of vegetable oils as frying oils produces significant amounts of used oils that may present a disposal problem. Their use for biodiesel production has the advantage of their low price. Used vegetable oil is described as a 'renewable fuel' as it does not add any extra carbon dioxide gas to the atmosphere, as opposed to fossil fuels, which cause changes in the atmospheric conditions.

Generally, vegetable oil from plant sources is the best starting material to produce biodiesel because the conversion of pure triglyceride to fatty acid methyl ester is high and the reaction time is relatively short. The most common way to produce biodiesel is by transesterification, which refers to a catalysed chemical reaction involving vegetable oil and an alcohol to yield fatty acid alkyl esters and glycerol (Thirumarimurugan *et al*., 2012). In the reaction for the production of biodiesel, there is a displacement of alcohol from an ester under alkaline catalytic conditions producing glycerol and fatty acid esters of the respective alcohol (Knothe *et al.*, 2007).

So far from the literature, Minodora *et al.* (2010) produced biodiesel by lipase transesterification of vegetable oils which could become a viable alternative to conventional techniques due to its advantages. Response surface methodology was used and found to be very convenient because it allowed the determination of optimum set of experimental conditions. Jahirul *et al.* (2014) produced biodiesel from beauty leaf oil with high content of free fatty acids and due to this, a two-step biodiesel conversion method consisting of acid catalyzed pre-esterification and alkali catalysed transesterification was used. A response surface method based on Box-Behnken design was employed to determine a feasible experimental plan to optimize the beauty leaf oil to biodiesel conversion procedure. Hossain and Boyce (2009) studied the comparison of optimum conditions of alkaline catalysed transesterification process for biodiesel from pure sunflower cooking oil (PSCO) and waste sunflower cooking oil (WSCO). This study provided the evidence that waste cooking sunflower oil might be employed as a substantial source of biodiesel as fuel in diesel engines because the biodiesel produced in the work was of good quality within the array of standard method specifications and the production yield was approximately 99% under optimum conditions. Junaid *et al.* (2013) produced biodiesel using non-edible rubber seed oil with high free fatty acid content of 45% in which a two stage process of acid esterification to reduce free fatty acid (FFA) content and base transesterification was employed. Response surface methodology was adopted for the study on the reduction of FFA and the production of biodiesel from rubber seed oil. Four different types of variables were considered. Catalyst concentration and alcohol to oil ratio were found to be the most influencing variables in FFA reduction and for base transesterification, the most influencing variable was found to be alcohol to oil ratio. Ofoefule *et al.* (2013) assessed the fuel quality of biodiesel produced from tiger nut (*Cyprus esculentus*) oil and its blends with petro-diesel. The oil was transesterified using potassium methoxide at the temperature of 60 ^{o}C for 60 min using a catalyst concentration of 0.65% and a constant stirring speed. The biodiesel was blended with petro-diesel to obtain various blends of B10, B20, B30 and B40. The study showed that tiger nut was a good supplementary feedstock for biodiesel production. Ruengwit *et al.* (2011) used a plug flow reactor for the continuous production of biodiesel from refined palm kernel oil with supercritical methanol and carried out optimization using response surface methodology. The modified quadratic regression model developed in the work demonstrated that the biodiesel content of the product obtained was more sensitive to changes in pressure. Uddin *et al.* (2013) produced biodiesel from waste cooking oil (WCO) through a three-step method (saponification, acidification, and esterification) in which aqueous sodium hydroxide was used for the saponification. The experimental results obtained supported that biodiesel from WCO by this method could be successfully used.

As can be noticed from the literature review carried out, most of the researches have centred on the use of response surface methodology for the investigation and the optimization of biodiesel production processes from waste cooking oil because it is a very good tool in carrying out the design and optimization of a process. Actually, there are different types of techniques available in response surface methodology, viz: central composite, Box-Behnken, D-optimal, and so on (Giwa *et al*., 2015). This research work is aimed at applying the D-optimal design technique of the response surface methodology to a process producing biodiesel using waste cooking oil obtained from two cafeterias of Afe Babalola University, Ado-Ekiti (ABUAD), Ekiti State, Nigeria in order to establish the effect

of process parameters (such as reaction temperature, catalyst concentration, and reaction time) on biodiesel yield, investigate the fuel properties of the produced biodiesel, develop a mathematical model for the relationship between the percentage yield of biodiesel produced and the parameters considered, and optimize the process conditions with the aid of Design-Expert. In this work, D-optimal has been chosen to be used because its design matrices are usually not orthogonal and effect estimates are correlated. These types of designs are always an option regardless of the type of model the experimenter wishes to fit (for example, first order, first order plus some interactions, full quadratic, cubic, etc.) or the objective specified for the experiment (for example, screening, response surface, etc.). D-optimal designs are straight optimizations based on a chosen optimality criterion and the model that will be fit (NIST/SEMATECH, 2015).

2. METHODOLOGY

The step-by-step approaches used to accomplish this research work on the production of biodiesel from a reaction between waste cooking oil and methanol using D-optimal experimental design technique are as outlined below.

2.1 Determination of Free fatty Acid Content of the Oil

In order to know whether the waste cooking oil would be suitable for either esterification or transesterification reaction, its free fatty acid (FFA) content was determined using the procedures described below:

1) 5.0 g of the waste cooking oil (shown in Figure 1) was weighed and poured into a conical flask (F1),
2) 50 ml of ethanol was weighed and poured into another flask (F2),
3) the ethanol was heated to boiling point and neutralized with 0.1 N of KOH using 0.5 ml phenolphthalein indicator,
4) the neutralized ethanol was poured into the flask (F1) and heated to boiling point,
5) as hot as possible, the solution in step 4 above was titrated against 0.1 N of KOH until the addition of a single drop produced a slight but definite colour change (pink) persisting for at least 15 sec.

Figure 1: Waste cooking oil collected from ABUAD cafeterias

After the free fatty acid content of the oil was determined, it was used via an alkaline-catalysed process for biodiesel production through the series of experiments designed with the aid of Design-Expert using D-optimal experimental design technique.

2.2 Experimental Design and Statistical Analysis

In carrying out the experimental design, the lower and upper limits of the variables (reaction temperature, catalyst concentration and reaction time) to be varied were inputted into the Design-Expert (Stat-Ease, 2005) based on D-optimal design. The temperature range used was 40 to 63 ^{o}C, catalyst concentration range was 0.5 to 2.0 wt%, and the range used for the reaction time was 1-3 hr. Application of D-optimal experimental design gave 12 runs given in Table 1 that were performed.

Table 1: Generated D-optimal experimental design parameters

Run	Reaction temperature (^{o}C)	Catalyst concentration (wt%)	Reaction time (hr)
1	63	2	2.29
2	40	0.5	1
3	40	1.39	1.81
4	53.74	0.5	1.80
5	40	2	3
6	63	1.04	1
7	53.71	1.40	3
8	62.64	2	1
9	40	0.5	3
10	63	0.5	3
11	40	2	3
12	47.89	2	1

Using the values given in Table 1 for each of the parameters considered, and the waste cooking oil that was collected from Afe Babalola University Cafeteria One and Two, twelve (12) samples of biodiesel were produced using the procedures outlined thus:

1) the water bath/oven was first calibrated to obtain the desired reaction temperature,
2) 10 ml of oil and 60 ml of alcohol were measured and poured into a beaker (Figure 2) and weighed,
3) measured catalyst was poured into the beaker containing the oil and alcohol mixture,
4) the beaker was placed in the water bath (Figure 3)/oven (Figure 4) and the reaction was carried out depending on the time to be used,
5) after the reaction, the produced mixture was poured into a separating funnel and left to stay for 24 hours, and two layers containing biodiesel and glycerol were formed,
6) the biodiesel was separated from the glycerol and the separated biodiesel (Figure 5) was kept in a sample bottle while the glycerol was also collected in another bottle (Figure 6),
7) at the end of all the experiments, the samples of biodiesel produced were analysed in the Chemical Engineering Laboratory of Afe Babalola University, Ado-Ekiti (ABUAD), Ekiti State, Nigeria.

Figure 2: Sample of reactants in conical flasks

Figure 3: Water bath used for the experiments being calibrated

Figure 4: The oven used for the experiment

Figure 5: Samples of biodiesel produced

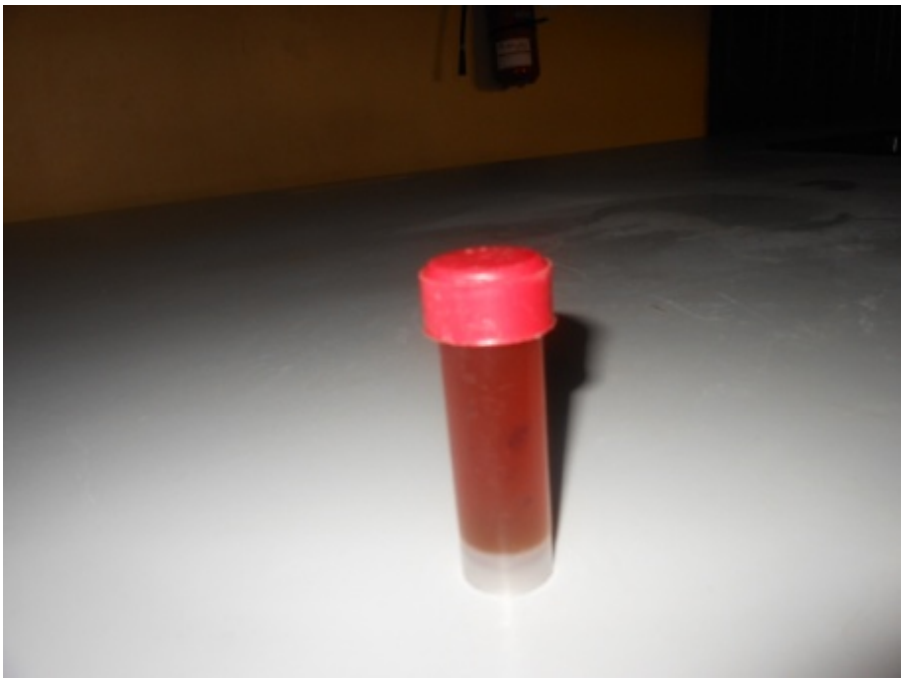

Figure 6: Sample of glycerol obtained

After the production of the twelve (12) samples of the biodiesel, the percent yield obtained from each of the runs was calculated, using Equation 1, and inputted into the Design-Expert, and the entire results were analysed using quadratic process model with manual selection, which was later modified using backward selection.

$$\text{Percentage yield of biodiesel} = \frac{weight\ of\ biodiesel\ obtained}{weight\ of\ reactants(oil\&alcohol)} \times 100 \quad (1)$$

Apart from the statistical analysis that was carried out on the produced biodiesel, some properties of the produced samples like the density, the flash point, and the cloud point were as well determined and compared to the literature values so as to be sure that the product produced was actually biodiesel.

2.3 Determination of Density of the Biodiesel

To determine the densities of the samples of biodiesel produced,

1) dried empty density bottle (Figure 7) was weighed,
2) it was filled with distilled water and weighed again,
3) it was emptied and filled with a biodiesel sample,
4) the weight of the sample was estimated and recorded,
5) and the density of the sample was thereby calculated using the relationships (Equations 2 and 3) given as:

$$\text{Specific gravity} = \frac{weight\ of\ sample}{weight\ of\ equal\ volume\ of\ water} \quad (2)$$

$$\text{Density of sample in } \frac{g}{cm^3} = specific\ gravity \times 1\ \frac{g}{cm^3} \quad (3)$$

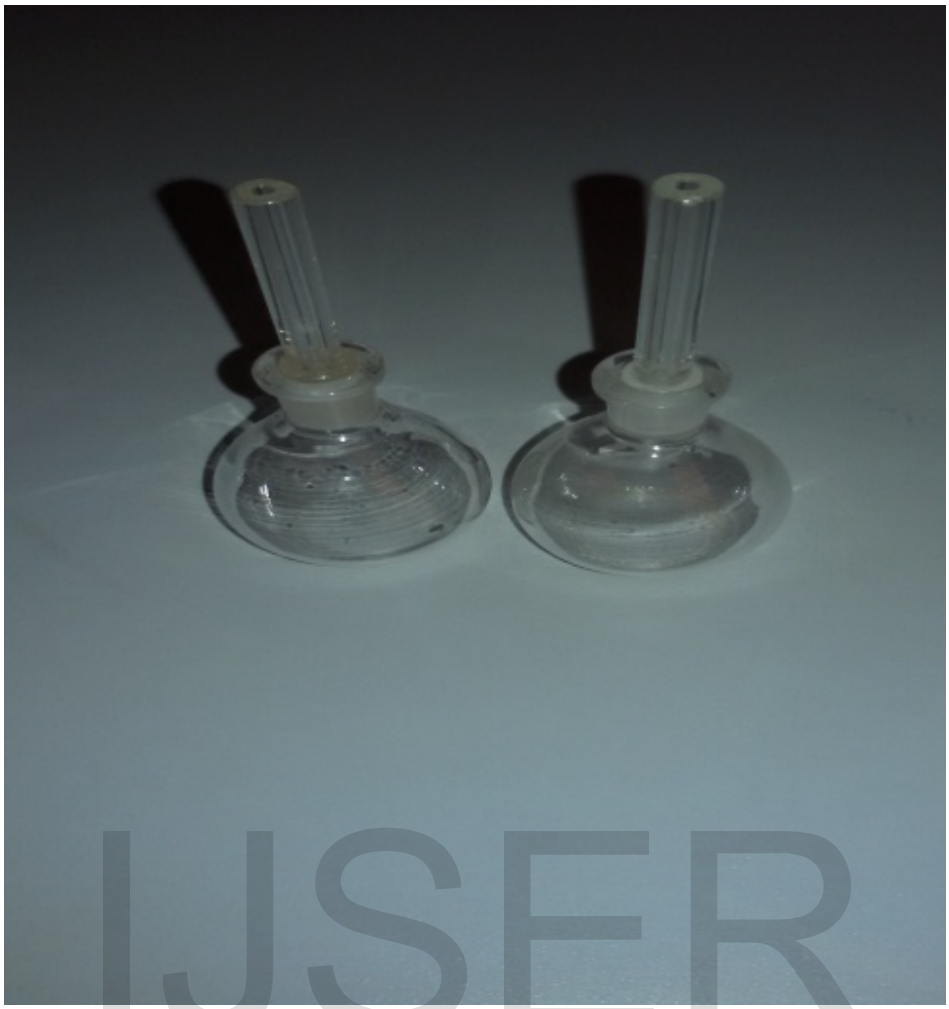

Figure 7: Density bottles

2.4 Determination of Flash Point of the Biodiesel

The flash point of each of the produced biodiesel sample was determined through the following procedures:

1) a liquid biodiesel sample was poured into a beaker and allowed to be heated until vapour started forming,
2) flame was then applied to the vapour,
3) the lowest temperature at which application of test flame caused the vapour to ignite above the oil was then recorded as the flash point.

2.5 Determination of Cloud Point of the Biodiesel

The step-by-step approach used to determine the cloud point of the biodiesel produced from the waste cooking oil obtained from the two cafeterias of Afe Babalola University are thus:

1) a biodiesel sample was heated and poured into a test bottle into which a thermometer was inserted,
2) the heated sample was then kept in a refrigerator and allowed to stay for some time until cloud started to form,
3) the point at which cloud started to form was noted as the cloud point of the produced biodiesel.

3. RESULTS AND DISCUSSION

3.1 Waste Cooking Oil Analyses

Before proceeding to producing the biodiesel of this work, the waste cooking oil used was first analysed, especially to know the free fatty acid content, so as to determine the process to be used between esterification and transesterification for the production, and the results of the analysis were as given in Table 2.

Table 2: Properties of the waste cooking oil

S/N	Parameter	Value
1	Density (g/cm^3)	0.9112
4	pH	5.16
5	Free fatty acid content (%)	0.2

It could be observed from the results given in Table 2 that the free fatty acid content of the biodiesel was too low for esterification reaction of biodiesel production. Hence, the transesterification reaction involving an alkaline catalyst was employed in this work.

3.2 Yield of Biodiesel Produced

The percentage yields obtained from the analysis of the produced biodiesel samples are given in Table 3. From the table, it was observed that the experiment carried out using the input parameters given in runs 5 and 11 of the experiments generated with the D-optimal design technique gave biodiesels that had very high yields in the range of approximately 96 and 93%, respectively. Moreover, each of the parameters considered were discovered to affect the yield of the biodiesel produced in different manners.

Table 3: Yields of the produced biodiesel samples at various conditions

Run	Reaction temperature (°C)	Catalyst concentration (wt%)	Time (hr)	%Yield
1.	63	2	2.29	64.29
2.	40	0.5	1	72.86
3.	40	1.39	1.81	57.14
4.	53.74	0.5	1.8	71.43
5.	40	2	3	95.71
6.	63	1.04	1	75.71
7.	53.71	1.4	3	68.57
8.	62.64	2	1	80.00
9.	40	0.5	3	85.71
10.	63	0.5	3	54.29
11.	40	2	3	92.86
12.	47.89	2	1	78.57

3.2.1 Effect of temperature on biodiesel yield

Knowing that the boiling temperature of methanol is 64.65 °C (Nwambuonwo and Giwa, 2015), the high limit of reaction temperature considered was thus made to be less than that value (64.65 °C) in order to avoid evaporation of methanol (one of the reactants of the process) before the reaction occurred so that the reaction conversion could be high. Now, considering the results (Table 3) obtained from the analysis of the different biodiesel samples produced, the highest yield of all was found to occur at low temperature of 40 °C (run 5), of course, with the combinations of the other parameters. Also noticed from the results given in Table 3 was that the biodiesel with the lowest yield was given at the highest reaction temperature (63 °C) (see run 10) considered. This was found to be an indication that low temperature favoured the biodiesel production from the reaction between the waste cooking oil and methanol used.

3.2.2 Effect of Catalyst Concentration on biodiesel yield

Based on the information obtained from the literature, an insufficient amount of catalyst could result in an incomplete conversion of triglycerides into fatty acid methyl esters. As such, the amount of catalyst used had a significant role on the production of biodiesel. In this work, the catalyst used was sodium hydroxide, and from the results (Table 3) obtained, 2 wt% of it was discovered to give the biodiesel with the highest yield. This observation has shown that catalyst amount as low as 2 wt% could be able to bring about high conversion of the reactants for biodiesel production.

3.2.3 Effect of reaction time on biodiesel yield

The results given in Table 3 revealed that the biodiesel produced at the high limit of the reaction time (3 hr) was the one that had the highest yield among all the samples obtained from the experiments carried out. This was an indication that high reaction time was able to favour the conversion of the reactants to the desired product (biodiesel).

3.3 Density

Generally, it has been discovered that density is one of the important properties of biodiesel. In order to have knowledge about the actual product given by the experiments carried out, the densities of most of the samples produced were estimated, and it was discovered that the density values obtained, given in Table 4, which varied between 0.84 and 0.88 were found to fall within the standard density range of biodiesel found in the literature, which was 0.85 – 0.88. This was a preliminary hint that the products produced were, actually, biodiesel.

Table 4: Densities of the produced biodiesel samples at various conditions

Run	Reaction temperature (^oC)	Catalyst concentration (wt%)	Time (hr)	Density (g/cm^3)
1.	63	2	2.29	0.87
2.	40	0.5	1	0.86
3.	40	1.39	1.81	0.86
4.	53.74	0.5	1.8	0.86
5.	40	2	3	0.85
6.	53.71	1.4	3	0.86
7.	40	0.5	3	0.84
8.	63	0.5	3	0.88
9.	40	2	3	0.85
10.	47.89	2	1	0.85

3.4 Cloud Point

The result of the cloud point analysis carried out on the sample that gave the highest yield of biodiesel, which was the 5th run in Table 3, gave a value of -3 ^oC. This value was found to fall within the standard range of cloud point of biodiesel, which is -3 to 12 ^oC. This was discovered to be another indication supporting the conclusion reached from the analysis of the density of the samples that the product obtained was biodiesel.

3.5 Flash Point

Given in Table 5 are the flash points of the produced biodiesel samples. According to the results shown in the table, the flash point of the product was found to vary as the conditions used to carry out the experiments were also varied. However, all the values of the flash points obtained from the samples of the produced biodiesel were found to be within the literature range of 130 to 170 ^oC. This was found to be another evidence showing that the produced material was biodiesel.

Table 5: Flash point of the produced biodiesel samples at various conditions

Run	Temperature (°C)	Catalyst conc. (wt %)	Time (hr)	Flash point (°C)
1.	63	2	2.29	145
2.	40	0.5	1	135
3.	40	1.39	1.81	135
4.	53.74	0.5	1.8	130
5.	40	2	3	157
6.	63	1.04	1	132
7.	53.71	1.4	3	136
8.	62.64	2	1	142
9.	40	0.5	3	146
10.	63	0.5	3	148
11.	40	2	3	148
12.	47.89	2	1	148

3.6 Statistical Analysis

Having ascertained that the samples produced were biodiesel, and having obtained the response of the experimental design carried out, which was the percentage yield of the biodiesel produced, the values of the parameters together with the responses (percentage yield) were analysed and the results of the statistical analysis carried out are given in Table 6. From the results shown in Table 6, the model was found to be significant because the probability value was less than 0.05 that was chosen using a confidence level of 95% (0.95), but its predicted R-squared value was observed to be negative. For that reason, it (the model) was modified.

Table 6: Full D-optimal analysis results

Source	Sum of squares	df	Mean square	F value	p-value (Prob> F)
Model	1807.306	9	200.8118	28.43296	0.0344
A-temperature	97.37788	1	97.37788	13.78774	0.0655
B-catalyst concentration	12.52267	1	12.52267	1.773085	0.3145
C-reaction time	4.937957	1	4.937957	0.699166	0.4910
AB	2.22726	1	2.22726	0.315358	0.6309
AC	606.0168	1	606.0168	85.80596	0.0115
BC	93.32679	1	93.32679	13.21415	0.0680
A^2	69.81894	1	69.81894	9.885669	0.0880
B^2	220.3401	1	220.3401	31.19797	0.0306
C^2	168.5531	1	168.5531	23.86545	0.0394
Residual	14.12528	2	7.062642		
Lack of Fit	10.06403	1	10.06403	2.478063	0.3603
Pure Error	4.06125	1	4.06125		
Cor Total	1821.432	11			
R-squared: 0.9922;	Adj R-squared: 0.9573;		Pred R-squared: -0.8575		

To modify the model, the selection was changed from manual to backward on the Model page of the Analysis section of Design-Expert, and the results obtained are given in Table 7.

Table 7: Modified D-optimal analysis results

Source	Sum of squares	df	Mean square	F value	p-value (Prob> F)
Model	1805.079	8	225.6349	41.39445	0.0055
A-temperature	97.9226	1	97.9226	17.96465	0.0240
B-catalyst concentration	11.3102	1	11.3102	2.074943	0.2454
C-reaction time	9.082011	1	9.082011	1.666165	0.2872
AC	609.5365	1	609.5365	111.8241	0.0018
BC	91.17055	1	91.17055	16.72594	0.0264
A^2	67.59292	1	67.59292	12.40044	0.0389
B^2	230.6577	1	230.6577	42.31593	0.0074
C^2	167.2813	1	167.2813	30.68904	0.0116
Residual	16.35255	3	5.450848		
Lack of Fit	12.2913	2	6.145648	1.51324	0.4984
Pure Error	4.06125	1	4.06125		
Cor Total	1821.432	11			
R-squared: 0.9910;	Adj R-squared: 0.9671;		Pred R-squared: 0.7245		

The results, given in Table 7, obtained from the analysis of variance (ANOVA) of the modified model (Equation 4) revealed that it was significant and, also, its R-squared value was very high and its predicted R-squared value was found to be positive indicating that the model was very good in predicting the percentage yield of biodiesel, given the values of the input parameters.

$$
\begin{aligned}
\%\text{Yield} = {} & -60.45500 \\
& +6.38363 \times \text{temperature} \\
& -58.29530 \times \text{catalyst concentration} \\
& +2.32007 \times \text{reaction time} \\
& -0.94175 \times \text{temperature} * \text{reaction time} \\
& +5.35561 \times \text{catalyst concentration} * \text{reaction time} \\
& -0.046799 \times \text{temperature}^2 \\
& +19.68556 \times \text{catalyst concentration}^2 \\
& +9.61239 \times \text{reaction time}^2
\end{aligned} \qquad (4)
$$

After the model was modified and found to be significant with favourable R-squared and predicted R-squared values, it was simulated using the coefficients of the actual factors of the model obtained, with the aid of Spreadsheet, and the results of the simulation were compared with the experimental ones as given in Figure 8. According to the figure, good correlation was found to exist between the experimental and the simulated percentage yield of the biodiesel samples produced. The good agreements between the values (experimental and simulated) was also discovered to support the fact that the model was able to represent the process of biodiesel production from the transesterification reaction between the waste cooking oil of ABUAD Cafeterias and methanol in the presence of sodium hydroxide catalyst very well, apart from the fact that the R-squared value of the developed model was high and approximately equal to 0.991 in addition to a favourable predicted R-squared value (0.7245) obtained from the model analysis.

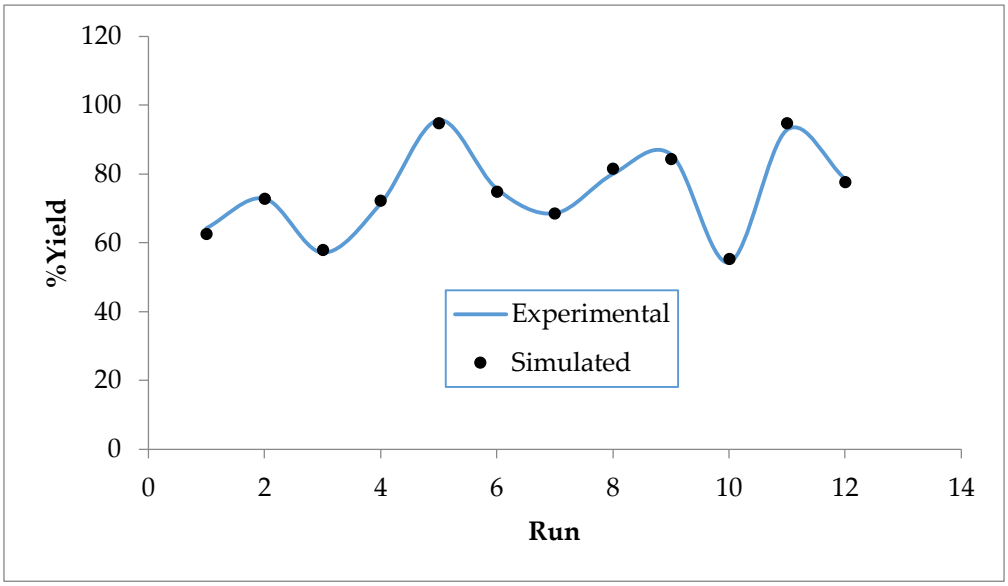

Figure 8: Experimental and simulated percentage yield of biodiesel

Having obtained a model for the system that related the percentage yield of biodiesel produced to the parameters (reaction temperature, catalyst concentration and reaction time), an attempt was made to obtain the values of the parameters that could give higher yield of biodiesel than the one already obtained by carrying out optimization on the developed model, and the optimum values obtained were found to be reaction temperature of 60 ^{o}C, catalyst concentration of 2.54 wt% and reaction time of 3 hr. The objective function of the optimization was also estimated to be 98% yield of biodiesel.

4. CONCLUSION

The density, the cloud point and the flash point values obtained for the produced material of this work were found to fall within the range of the standard values for biodiesel, and they were used as the criteria to say that the material produced from the experiments designed using D-optimal experimental design technique was biodiesel. In addition, the modified model of the system obtained from the D-optimal design analysis was discovered to represent the system very well because its high R-squared value (0.991) was found to be very close to unity (1). Apart from that, the model developed was found to be significant as its probability value was less than 0.05 that was chosen based on the confidence level of 95%. It was also discovered in this work that very high yield of biodiesel could be obtained using the optimum values of the input parameters estimated to run the experimental system.

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NOMENCLATURE

A	Temperature
Adj	Adjusted
B	Catalyst concentration

C Reaction time
df Degree of freedom
Pred Predicted
Prob Probability

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