# Exploring the Impact of Alcohol Type on the Yield of Biodiesel from Cottonseed Oil Using NaOH Catalyst and Response Surface Methodology Statistical Tools

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## ABSTRACT

A study on the impact of alcohol type on the yield of biodiesel from cottonseed oil oil was conducted by carrying out transesterification of cottonseed oil with three different alcohols (methanol, ethanol and butanol) in presence of sodium hydroxide (NaOH) as catalyst. Response Surface Methodology based on Central Composite Design was used to optimize the process and analysis of variance (ANOVA) used for conducting the relevant statistical analysis. Quadratic models were developed to predict biodiesel yield as a function of the transesterification process variables - alcohol/oil molar ratio, catalyst concentration, reaction temperature, reaction time, and agitation rate. The statistical tool made predictions on biodiesel yield from the processes as follows, methanol, 83.25% ethanol, 84.36% and butanol, 74.59 % at the optimal condition for methanol process as methanol/oil molar ratio 6:1, temperature 55ºC, time 45 mins, catalyst concentration 1%, and rate of mixing 300rpm. Ethanol/oil molar ratio18:1. temperature 45ºC, time 45 mins, catalyst concentration 1%, and rate of mixing speed, 300rpm. Butanol/oil molar ratio 19:1, temperature 40ºC, time 15mins, catalyst concentration 0.5%, and mixing speed 300rpm, The optimized conditions were validated with the actual biodiesel yield of 82.5% for methanol, 85% for ethanol and 75% for butanol. Experiments were conducted to validate the predicted optimal conditions presented actual biodiesel yield of 82.5% for methanol, 85% for ethanol and 75% for butanol. The error between the experimental and predicted yield was found to be is less than 0.6%, showing that the model correctly explains the influence of the process variables on the production of alkyl ester from cotton seed oi and have sufficient accurancy to predict the amount of alkyl ester yield and therefore it can be concluded that the generated models have sufficient accurancy to predict the amount of alkyl ester yield.

**Key words***:* Butanol, Biodiesel, Transesterification, Exploring.

## INTRODUCTION

Rapid increase in global energy demand besides limited fossil fuel supplies and growing environmental concerns has led to an intense search for sustainable and renewable energy sources (Yalew, et al., 2020). Biodiesel - a mono-alkyl ester of long-chain fatty acids is prominent among the other options as a strong competitor for biofuels because of its biodegradability, non-toxicity, and lower emission profiles than traditional diesel (Chang, et al.,2017, Jeswani, et al., 2020). Biodiesel is an essential part of future energy portfolios since its use greatly lowers greenhouse gas emissions and promotes energy independence.

Triglycerides undergo a chemical reaction (transesterification) with short-chain alcohols in the presence of a catalyst to produce fatty acid alkyl esters, or biodiesel, and glycerol as a by-product (Long, et al., 2021, Sales, et al., 2022). Transesterification process's efficiency and yield is influenced by several crucial factors including the type and quantity of alcohol, catalyst concentration, reaction temperature, agitation speed, and reaction duration. For the transesterification of refined vegetable oils, sodium hydroxide (NaOH), a potent alkali, is well known as a cost-effective and efficient homogeneous catalyst that provides high reaction rates and yields under ideal circumstances (Gholami, and Pourfayaz., 2024, Jamil, et al., 2022) The choice of alcohol type is important because of its obvious effect on the kinetics of the reaction, the solubility of the reactants and products, and, eventually, the yield and characteristics of the finished biodiesel. Traditionally, methanol is a preferred alcohol due to its low cost and high reactivity even though, the use of other alcohols, such as ethanol and butanol, is gaining traction due to their renewable nature (ethanol from biomass) and superior qualities of the resulting biodiesel (e.g., higher cetane number and lubricity with butanol) (Yun., 2020, Callegari, et al., 2020)

In many areas, cottonseed oil is a plentiful non-edible oil source that offers a practical and affordable feedstock for the manufacturing of biodiesel, allaying worries about the conflict between food and fuel (Khan, 2024, Patel , et al., 2025) Therefore, to optimize the production process and customize the features of biodiesel for particular uses, a thorough examination of the effects of several alcohol types—methanol, ethanol, and butanol—on the yield of biodiesel from cottonseed oil catalyzed by NaOH is essential.

A crucial statistical technique for methodically assessing and optimizing the intricate interactions between various process variables is Response Surface Methodology (RSM). When a response of interest is impacted by multiple variables, RSM is a set of statistical and mathematical methods that can be used to build, enhance, and optimize processes (Myers et al., 2016). RSM makes it possible to model the link between several independent factors and the response by using experimental designs like Central Composite Design (CCD). This allows for the identification of ideal operating conditions with fewer experiments. In order to provide vital information for effective and sustainable biodiesel production, this study intends to methodically investigate the effects of methanol, ethanol, and butanol on the yield of biodiesel made from cottonseed oil using NaOH as a catalyst. Response Surface Methodology will then be used to optimize the process parameters.

## MATERIALS AND METHODS

### Materials

Refined cotton seed oil was obtained from Roban Stores, Enugu, reagents from Ogbete Main market Enugu, Enugu State, Nigeria.

### Methods

### Characterisation of Refined Cottonseed Oil

The refined cottonseed oil was characterized to determine its vital properties.

The properties and method adopted for their determination are shown in table 1

**Table 1 - Methods employed for oil characterization**

| Property | Analytical Method (Standard |
|---|---|
| Specific gravity (S.G) | ASTM D74 (1986). |
| Melting point | ALCA H-16 |
| Flash point | ASTM D93. |
| Moisture content | ASTM D2709. |

| | |
|---|---|
| Saponificaton value | ASTM D615 |
| Iodine value | ASTM D4067-86 (1986) |
| Peroxide value | AOAC 965.33 |
| Free fatty acid (FFA) value | ASTM D7638-10 (2021) |
| Calorific value | ASTM D3286 |
| Cloud point | ASTM D2500 |
| Viscosity | ASTM D445 |

**Transesterification Reaction**

The refined cottonseed oil reacts with methanol, ethanol and butanol in the presence of NaOH to produce alkyl esters of fatty acids (biodiesel) and glycerol. The refined cottonseed oil was precisely quantitatively transferred into a flat bottom flask placed on a hot magnetic stirrer. Specific amount of catalyst (by weight of refined cottonseed oil) dissolved in the required amount of methanol, ethanol and butanol was added. The reaction flask was kept on a hot magnetic stirrer under constant temperature with defined agitation throughout the reaction. At the defined time, sample was taken out, cooled, and the biodiesel (i.e. the alkyl ester in the upper layer) was separated from the by-product (i.e. the glycerol in the lower layer) by settlement overnight under ambient condition. Percentage of the biodiesel yield was determined by comparing the volume of layer biodiesel with the volume of refined cottonseed oil used. The procedure was repeated by varying the factors affecting the transesterification reaction such as; time, catalyst concentration, temperature, alcohol/oil molar ratio and agitation speed.

**Design of Experiment for Transesterification Reaction**

Design Expert software (version 9) was used in this study to design the experiment and to optimize the reaction conditions. The experimental design employed in this work was a two-level-five factor fractional factorial design involving 32 experiments. Catalyst concentration, A, methanol/oil molar ratio, B, Reaction temperature, C, reaction time, D, and agitation speed, E were selected as independent factors for the optimization study. The response chosen was the ester yields obtained from transesterification of cottonseed oil. Eight replications of centre points were used in order to predict a good estimation of errors and experiments were performed in a randomized order. The actual and coded levels of each factor are shown in Tables 1- 3 below. The coded values were designated by −1 (minimum), 0 (centre), +1 (maximum), −α and +α. Alpha is defined as a distance from the centre point which can be either inside or outside the range, with the maximum value of 2n/4, where n is the number of factors whereby the value of alpha is set at 0.5. It is noteworthy to point out that the software uses the concept of the coded values for the investigation of the significant terms, thus equation in coded values is used to study the effect of the variables on the response. The empirical equation is represented as:

$$Y = \beta_0 + \sum_{i=1}^{5} \beta_i X_i + \sum_{i=1}^{5} \beta_{ii} X^2{}_i + \sum_{i=1}^{5} \sum_{j=i+1}^{5} \beta_{ij} X_i X_j \quad (1)$$

Selection of levels for each factor was based on the experiments performed to study the effects of process variables on the application of solid base catalysts for transesterification reaction of cottonseed oil.

**Statistical Analysis of Transesterification using Central Composite Design (CCD)**

To optimize the transesterification of the cottonseed oil using methanol, ethanol and butanol, Response Surface Methodology with Central Composite Design program was used to determine the optimum values of the process variables. The fractional factorial design was used to obtain a quadratic model, consisting of factorial trials to estimate quadratic effects. To examine the combined effect of the five respective factors (independent variables): catalyst concentration, alcohol/oil molar ratio, reaction temperature, reaction time and agitation

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speed, on biodiesel yield and derive a model, a two-level- five –factor ($2^{5-1}$ + 2*5 + 6) Central Composite Response Design = 32 experiments were performed. The factors levels are shown in tables 1, 2, and 3. The matrix for the five variables were varied at two levels (-1 and +1). The lower level of variable was designated as “-1” and higher level as “+1”. The experiments were performed in random order to avoid systematic error. Equations (1, 2,and 3) represent the mathematical model relating the transesterification reaction using methanol, ethanol and butanol respectively with the independent process variables obtained with the Design Expert 9. The design of the experimental matrix of transesterification using methanol, ethanol and butanol. The experimental and the predicted values, calculated by Equations (3), (4) and (5), is presented in table 3, 4 and 5. The response was expressed as % yield, calculated as ;

$$Y=\{(V_b)/V_o\} \times 100 \quad (2)$$

where $V_o$ is the initial volume of oil and $V_b$ is the volume of biodiesel produced.

**Table 1: Studied range of each factor in actual and coded form (Methanol).**

| Factor | Units | Low level | High level | -α | +α | 0 level |
|---|---|---|---|---|---|---|
| Catalyst conc. (A) | Wt% | 0.6(-1) | 1.2(+1) | 0.6(-2) | 1.4(+2) | 1.0 |
| Methanol, (B) | Mol/mol | 5(-1) | 8(+1) | 4(-2) | 9(+2) | 6 |
| Temperature, (C) | °C | 50(-1) | 60(+1) | 45(-2) | 65(+2) | 55 |
| Reaction time (D) | Hours | 30(-1) | 60(+1) | 15(-2) | 90(+2) | 45 |
| Agitation speed ( E) | Rpm | 250(-1) | 350(+1) | 200(-2) | 400(+2) | 300 |

**Table 2: Studied range of each factor in actual and coded form (Ethanol).**

| Factor | Units | Low level | High level | -α | +α | 0 level |
|---|---|---|---|---|---|---|
| Catalyst conc. (A) | Wt% | 0.8(-1) | 1.2(+1) | 0.6(-2) | 1.4(+2) | 1.0 |
| Ethanol, (B) | Mol/mol | 16(-1) | 20(+1) | 14(-2) | 22(+2) | 18 |
| Temperature, (C) | °C | 30(-1) | 55(+1) | 15(-2) | 65(+2) | 45 |
| Reaction time (D) | Hours | 30(-1) | 60(+1) | 15(-2) | 90(+2) | 45 |
| Agitation speed ( E) | Rpm | 250(-1) | 350(+1) | 200(-2) | 400(+2) | 300 |

**Table 3: Studied range of each factor in actual and coded form (Butanol).**

| Factor | Units | Low level | High level | -α | +α | 0 level |
|---|---|---|---|---|---|---|
| Catalyst conc. (A) | Wt% | 0.2(-1) | 0.6(+1) | 0.1(-2) | 0.8(+2) | 0.4 |
| Butanol/oil, (B) | Mol/mol | 18(-1) | 20(+1) | 16(-2) | 24(+2) | 18 |
| Temperature, (C) | °C | 35(-1) | 45(+1) | 30(-2) | 50(+2) | 40 |
| Reaction time (D) | Hours | 10(-1) | 20(+1) | 5(-2) | 30(+2) | 15 |
| Agitation speed ( E) | Rpm | 250(-1) | 350(+1) | 200(-2) | 400(+2) | 300 |

## RESULTS AND DISCUSSIONS

**Table 4: Characterization of refined cotton seed oil, biodiesel from cotton seed oil and ASTM standard.**

| S/N | Properties | Units | Refined cottonseed oil | Biodiesel from cottonseed oil | ASTM D6751 standard |
|---|---|---|---|---|---|
| 1 | Moisture content | % wt | 0.020 | 0.020 | 0.050 max |
| 2 | Acid value | Mg/KOHg | 0.24 | 0.22 | – |
| 3 | FFA | % | 0.15 | 0.11 | – |
| 4 | Saponification value | Mg/g | 187.95 | 165.47 | – |
| 5 | Ester value | Mg/g | 187.72 | 165.19 | – |
| 6 | Iodine value | $mgI_2/100$ g | 68.90 | 125.20 | – |
| 7 | Peroxide value | Meq/kg | 80.00 | 26.01 | – |
| 8 | Specific gravity | | 0.906 | 0.87 | 0.88 |
| 9 | Kinematic viscosity | $mm^2/s$ | 29.22 | 6.81 | 1.9–6.0 |
| 10 | Odour | | Agreeable | Agreeable | – |
| 11 | Colour | | Brown | Light brown | – |
| 12 | Refractive index | (28 °C) | 1.4233 | 1.344 | – |
| 13 | Flash point | (°C) | 255 | 173 | 100–170 |
| 14 | Cloud point | (°C) | −3.0 | 7.0 | −3–12 |
| 15 | Pour point | (°C) | −2.3 | 5.0 | −15–10 |
| 16 | Fire point | (°C) | – | 193 | _ |
| 17 | Cetane number | | | 56.06 | 48–65 |
| 18 | High heating value | MJ/kJ | 41.25 | 39.54 | – |

### Phsico-chemical Characteristics of Cottonseed Oil

Characteristics of the refined cotton seed oil, biodiesel and ASTMD6751 standards are summarized in table 4. The results show that the free fatty acid (FFA) value of 0.15% for the refined cottonseed oil is less than 1% while the moisture content of 0.02% is very close to zero. These are acceptable levels for transesterification reaction, since higher amount of free fatty acids (FFA) (>1% w/w) in the feedstock can directly react with the alkaline catalyst to form soaps. The soaps are subject to form stable emulsions and thus prevent separation of the biodiesel from the glycerol fraction and decrease the yield of biodiesel (Demirbas, 2003). Base-catalyzed transesterification reaction requires water free and low acid value (< 1) raw materials for biodiesel production (Amit, 2012). The presence of water and FFA greater than 1% in raw materials resulted in soap formation and decrease in yield of alkyl ester, consume much catalyst and reduce the effectiveness of the catalyst (Demirbas, 2006). The results of the physiochemical characteristics of the cottonseed oil and biodiesel, along with the standard (ASTM D6751-02), as presented, show that the major characteristics (kinematic viscosity, acid value, free fatty acid) are in good agreement with the standard.

It was observed that the specific gravity of refined cottonseed oil was reduced from 0.906 to 0.87 after transesterification and it is within the acceptable limit. Saponification value of cottonseed oil is 187.95 mg/g while that of biodiesel is 165.47 mg/g. This implies that the triglycerides of cottonseed oil have higher molecular weight fatty acids (saturated and unsaturated). This result obtained compares favorably with the saponification value of palm oil (187–205), olive oil (185–187), and soy oil (187–193) (Mohammed et al, 2012). Saponification value is most important in that it is a good indicator of the extent of transesterification. The iodine value for cottonseed oil, 68.90 mg$I_2$ justifies the fact that the oil is edible. Iodine value for edible oil is less than 100 mg$I_2$. In general, the greater the iodine value, the higher the degree of unsaturation and the higher the tendency of the oil to undergo oxidative rancidity. It also indicates that cottonseed oil is a non-drying oil and would produce a non-drying alkyd (Panda, 2010). Even though the biodiesel has the iodine value of 125.20 mg$I_2$/g, which is relatively high according to Europe's EN 14214 specifications of iodine value, it indicates that cottonseed oil is a good source of raw material for biodiesel production because the higher the iodine value the more the number of unsaturated double bonds present in molecular structure and less the viscosity of the oil (Mohammed et al, 2012).

Peroxide value useful in monitoring oxidation is not specified in the biodiesel standards (Mohammed et al, 2012) but it influences cetane number, a parameter that is specified in the fuel standard. An increase in peroxide value indicates an increase in cetane number and therefore may reduce ignition delay time (Mohammed et al, 2012).

**Evaluation of regression model for transesterification efficiency**

The correlation between the experimental process variables and the transesterification efficiency was evaluated using the CCD modelling technique. Second order polynomial regression equation was fitted between the response (Transesterification efficiency, ($Y$)) and the process variables for the respective alcohols ; (methanol, ethanol, butanol): alcohol – oil molar ratio, A, catalyst weight %, B reaction temperature, C and reaction time, D. and agitation speed, E. From Tables 6, 8 and 10, the ANOVA results showed that the quadratic model is suitable to analyse the experimental data. The model in terms of the coded values of the process parameters is given by eqns 3, 4, and 5 for methanol, ethanol and butanol respectively.

$$Y=83.25+0.32A-1.01B-1.56C+1.92D+2.09E-4.23AB-2.58AC+4.13AD-2.52AE+0.92BC+4.51BD+0.73BE+1.51CD-2.42CE-2.43DE-1.02A^2-6.39B^2-6.52C^2-5.13D^2-3.20E^2 \quad (3)$$

$$Y = 84.36+2.58A+0.75B+0058C-1.58D-0.17E+0.38AB+0.13AC-1.37AD-1.25AE-3.50BC+4BD-0.88BE-1.75CD-0.13CE-2.13DE-4.86A^2-2.49B^2-2.74C^2-4.74D^2-7.24E^2 \quad (4)$$

$$Y = 74.59+0.58A+0.33B+0.83C-0.83D+1.42E-1.37AB+0.50AC+0.25AD-0.23AE+1.50BC-0.50BD+1.75BE+0.88CD+0.63CE-1.38DE-2.47A^2-3.09B^2-2.22C^2-2.47D^2-4.84E^2 \quad (5)$$

To develop a statistically significant regression model, the significance of the regression coefficients was evaluated based on the $p$-values. The coefficient terms with $p$-values more than 0.05 were insignificant and were removed from the regression model. The analysis in Tables (6, 8, 10) show that the linear terms A, B, and C; the quadratic terms, $A^2$, $B^2$, and $C^2$ and the interaction terms of AB, AC and CD; are significant model terms but D was included in the model because of its importance. The models were reduced to Eqns. (6, 7, 8) respectively, after eliminating the insignificant coefficients.

$$Y = 83.25+1.92D+2.09E-4.23AB-2.58AC+4.13AD-2.52AE+4.51BD-2.42CE-2.43DE-6.39B^2-6.52C^2-5.13D^2-3.2 \quad (6)$$

$$Y=84.36+2.58A-3.50BC+4BD-4.86A^2-2.49B^2-2.74C^2-4.74D^2-7.24E^2 \quad ( \quad (7)$$

$$Y = 74.59+1.42E-1.37AB+1.50BC+1.75BE-1.38DE-2.47A^2-3.09B^2-2.22C^2-2.47D^2-4.84E^2 \quad (8)$$

Where Y is the response variable (percentage yield of biodiesel) and A-E are the coded values of the independent variables. The above equations represent the quantitative effect of the factors (A, B, C, D, and E) upon the response (Y). Coefficients with one factor represent the effect of that particular factor while the

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coefficients with more than one factor represent the interaction between those factors. Positive sign in front of the terms indicates synergistic effect while negative sign indicates antagonistic effect of the factor. The adequacy of the above proposed model was tested using the Design Expert sequential model sum of squares and the model test statistics.

**Table 3: Experimental design matrix for the factorial design of iodiesel production from cotton seed oil using methanol.**

| **Run** | Catalyst concentration **(A)** | Alcool/oil molar ratio **(B)** | Reaction temperature **(C)** | Reaction temperature **(D)** | Agitation speed **(E)** | Experimental Yield **(Y)** | Pred. Yield (Y) |
|---|---|---|---|---|---|---|---|
| | wt% | Minutes | Degree Celsius | mol/mol | rpm | (%) | (%) |
| 1 | 0.8 | 30 | 50 | 5 | 350 | 72 | 74.3 |
| 2 | 1.2 | 30 | 50 | 5 | 250 | 69 | 67.9 |
| 3 | 0.8 | 60 | 50 | 5 | 250 | 52 | 50.98 |
| 4 | 1.2 | 60 | 50 | 5 | 350 | 53 | 55.4 |
| 5 | 0.8 | 30 | 60 | 5 | 250 | 60 | 58.88 |
| 6 | 1.2 | 30 | 60 | 5 | 350 | 55 | 57.3 |
| 7 | 0.8 | 60 | 60 | 5 | 350 | 65 | 67.38 |
| 8 | 1.2 | 60 | 60 | 5 | 250 | 41.5 | 40.48 |
| 9 | 0.8 | 30 | 50 | 8 | 250 | 46.3 | 45.27 |
| 10 | 1.2 | 30 | 50 | 8 | 350 | 68.1 | 70.49 |
| 11 | 0.8 | 60 | 50 | 8 | 350 | 65.6 | 68.07 |
| 12 | 1.2 | 60 | 50 | 8 | 250 | 69 | 68.07 |
| 13 | 0.8 | 30 | 60 | 8 | 350 | 49 | 51.37 |
| 14 | 1.2 | 30 | 60 | 8 | 250 | 71.6 | 70.57 |
| 15 | 0.8 | 60 | 60 | 8 | 250 | 70.1 | 69.15 |
| 16 | 1.2 | 60 | 60 | 8 | 350 | 57.89 | 60.36 |
| 17 | 0.6 | 45 | 55 | 6.5 | 300 | 79.88 | 78.54 |
| 18 | 1.4 | 45 | 55 | 6.5 | 300 | 81.21 | 79.83 |
| 19 | 1 | 15 | 55 | 6.5 | 300 | 60.88 | 59.7 |
| 20 | 1 | 75 | 55 | 6.5 | 300 | 57.2 | 55.66 |
| 21 | 1 | 45 | 45 | 6.5 | 300 | 61.7 | 60.32 |
| 22 | 1 | 45 | 65 | 6.5 | 300 | 55.4 | 54.07 |
| 23 | 1 | 45 | 55 | 3.5 | 300 | 60.1 | 58.9 |
| 24 | 1 | 45 | 55 | 9.5 | 300 | 68.1 | 66.58 |
| 25 | 1 | 45 | 55 | 6.5 | 200 | 60.8 | 66.27 |
| 26 | 1 | 45 | 55 | 6.5 | 400 | 82.8 | 74.62 |
| 27 | 1 | 45 | 55 | 6.5 | 300 | 82.8 | 83.25 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 28 | 1 | 45 | 55 | 6.5 | 300 | 82.8 | 83.25 |
| 29 | 1 | 45 | 55 | 6.5 | 300 | 82.8 | 83.25 |
| 30 | 1 | 45 | 55 | 6.5 | 300 | 82.8 | 83.25 |
| 31 | 1 | 45 | 55 | 6.5 | 300 | 82.8 | 83.25 |
| 32 | 1 | 45 | 55 | 6.5 | 300 | 82.8 | 83.25 |

**Table 4: Experimental Design Matrix For the Factorial Design of Biodiesel Production from Cotton Seed Oil using ethanol.**

| **Run** | Catalyst concentration **(A)** | Alcool/oil molar ratio **(B)** | Reaction temperature **(C)** | Reaction temperature **(D)** | Agitation speed **(E)** | Experimental Yield **(Y)** | Pred. Yield (Y) |
|---|---|---|---|---|---|---|---|
| | **wt%** | **Minutes** | **Degree celcius** | **mol/mol** | **rpm** | **(%)** | **(%)** |
| 1 | 0.8 | 30 | 30 | 16 | 350 | 60 | 62.05 |
| 2 | 1.2 | 30 | 30 | 16 | 250 | 65 | 63.05 |
| 3 | 0.8 | 60 | 30 | 16 | 250 | 56 | 55.13 |
| 4 | 1.2 | 60 | 30 | 16 | 350 | 67 | 65.96 |
| 5 | 0.8 | 30 | 55 | 16 | 250 | 65 | 65.3 |
| 6 | 1.2 | 30 | 55 | 16 | 350 | 78 | 78.13 |
| 7 | 0.8 | 60 | 55 | 16 | 350 | 56 | 57.21 |
| 8 | 1.2 | 60 | 55 | 16 | 250 | 67 | 64.21 |
| 9 | 0.8 | 30 | 30 | 20 | 250 | 54 | 52.96 |
| 10 | 1.2 | 30 | 30 | 20 | 350 | 53 | 51.8 |
| 11 | 0.8 | 60 | 30 | 20 | 350 | 67 | 66.88 |
| 12 | 1.2 | 60 | 30 | 20 | 250 | 80 | 75.88 |
| 13 | 0.8 | 30 | 55 | 20 | 350 | 56 | 57.05 |
| 14 | 1.2 | 30 | 55 | 20 | 250 | 65 | 62.05 |
| 15 | 0.8 | 60 | 55 | 20 | 250 | 63 | 61.13 |
| 16 | 1.2 | 60 | 55 | 20 | 350 | 60 | 57.96 |
| 17 | 0.6 | 45 | 42.5 | 18 | 300 | 62 | 59.74 |
| 18 | 1.4 | 45 | 42.5 | 18 | 300 | 64 | 70.08 |
| 19 | 1 | 15 | 42.5 | 18 | 300 | 73 | 72.91 |
| 20 | 1 | 75 | 42.5 | 18 | 300 | 72 | 75.91 |
| 21 | 1 | 45 | 17.5 | 18 | 300 | 70 | 72.24 |
| 22 | 1 | 45 | 67.5 | 18 | 300 | 73 | 74.58 |
| 23 | 1 | 45 | 42.5 | 14 | 300 | 69 | 68.58 |
| 24 | 1 | 45 | 42.5 | 22 | 300 | 58 | 62.24 |

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| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 25 | 1 | 45 | 42.5 | 18 | 200 | 50 | 55.74 |
| 26 | 1 | 45 | 42.5 | 18 | 400 | 57 | 55.08 |
| 27 | 1 | 45 | 42.5 | 18 | 300 | 85 | 84.36 |
| 28 | 1 | 45 | 42.5 | 18 | 300 | 85 | 84.36 |
| 29 | 1 | 45 | 42.5 | 18 | 300 | 85 | 84.36 |
| 30 | 1 | 45 | 42.5 | 18 | 300 | 85 | 84.36 |
| 31 | 1 | 45 | 42.5 | 18 | 300 | 85 | 84.36 |
| 32 | 1 | 45 | 42.5 | 18 | 300 | 85 | 84.36 |

**Table 5: Experimental Design Matrix For the Factorial Design of Biodiesel Production from Cotton Seed Oil using butanol.**

| Run | Catalyst concentration (A) | Alcool/oil molar ratio (B) | Reaction temperature (C) | Reaction temperature (D) | Agitation speed, (E) | Experimental Yield (Y) | Pred. Yield (Y) |
|---|---|---|---|---|---|---|---|
| | wt% | Minutes | Degree celcius | mol/mol | rpm | (%) | (%) |
| 1 | 0.3 | 10 | 35 | 18 | 350 | 60 | 60.51 |
| 2 | 0.6 | 10 | 35 | 18 | 250 | 63 | 62.09 |
| 3 | 0.3 | 20 | 35 | 18 | 250 | 57 | 57.09 |
| 4 | 0.6 | 20 | 35 | 18 | 350 | 62 | 61.84 |
| 5 | 0.3 | 10 | 45 | 18 | 250 | 54 | 53.84 |
| 6 | 0.6 | 10 | 45 | 18 | 350 | 62 | 61.59 |
| 7 | 0.3 | 20 | 45 | 18 | 350 | 68 | 68.59 |
| 8 | 0.6 | 20 | 45 | 18 | 250 | 58 | 57.18 |
| 9 | 0.3 | 10 | 35 | 20 | 250 | 60 | 59.01 |
| 10 | 0.6 | 10 | 35 | 20 | 350 | 59 | 57.76 |
| 11 | 0.3 | 20 | 35 | 20 | 350 | 58 | 57.76 |
| 12 | 0.6 | 20 | 35 | 20 | 250 | 55 | 53.34 |
| 13 | 0.3 | 10 | 45 | 20 | 350 | 56 | 55.51 |
| 14 | 0.6 | 10 | 45 | 20 | 250 | 65 | 63.09 |
| 15 | 0.3 | 20 | 45 | 20 | 250 | 60 | 59.09 |
| 16 | 0.6 | 20 | 45 | 20 | 350 | 65 | 63.84 |
| 17 | 0.15 | 15 | 40 | 19 | 300 | 64 | 63.56 |
| 18 | 0.75 | 15 | 40 | 19 | 300 | 63 | 65.89 |
| 19 | 0.45 | 5 | 40 | 19 | 300 | 60 | 61.56 |
| 20 | 0.45 | 25 | 40 | 19 | 300 | 62 | 62.89 |
| 21 | 0.45 | 15 | 30 | 19 | 300 | 63 | 64.06 |
| 22 | 0.45 | 15 | 50 | 19 | 300 | 66 | 67.39 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 23 | 0.45 | 15 | 40 | 17 | 300 | 67 | 66.39 |
| 24 | 0.45 | 15 | 40 | 21 | 300 | 60 | 63.06 |
| 25 | 0.45 | 15 | 40 | 19 | 200 | 50 | 52.39 |
| 26 | 0.45 | 15 | 40 | 19 | 400 | 58 | 58.06 |
| 27 | 0.45 | 15 | 40 | 19 | 300 | 75 | 74.59 |
| 28 | 0.45 | 15 | 40 | 19 | 300 | 75 | 74.59 |
| 29 | 0.45 | 15 | 40 | 19 | 300 | 75 | 74.59 |
| 30 | 0.45 | 15 | 40 | 19 | 300 | 75 | 74.59 |
| 31 | 0.45 | 15 | 40 | 19 | 300 | 75 | 74.59 |
| 32 | 0.45 | 15 | 40 | 19 | 300 | 75 | 74.59 |

Table 3 Characterization of refined cotton seed oil, biodiesel from cotton seed oil and ASTM standard.

**Analysis of Variance (ANOVA)**

The analysis of variance (ANOVA) indicated that the quadratic polynomial model was significant and adequate to represent the actual relationship between transesterification efficiency and the significant model variables as depicted by very small *p*- values (<0.0001). The significance and adequacy of the established models were further elaborated by a high value of coefficient of determination ($R^2$) value of 0.9642 for methanolysis, 0.9516 ethanolysis, 0.9691butanolysis and adj. $R^2$ value of 0.8982 for methanolysis, 0.8562 ethanolysis, 0.9130 butanolysis. This means that the model explains 96.42%, 95.16%, 96.91% of the variation in the experimental data for methanolysi, ethanolysis and butanolysis respectively. The adequate correlation between the experimental values of the independent variable and predicted values further showed the adequacy of the models.

From the statistics test for methanolysis as shown in Table 6, the regression coefficient ($R^2 = 0.9642$) is high, and the adjusted $R^2$(0.8991) is in close agreement with the predicted $R^2$ (0.8982) value. Also, the sequential test for ethanolysis shown in table.8 show that the model F-value (10.81) of the quadratic model is large compared to the values for the other models for the equation. And from the statistics test, the regression coefficient ($R^2 = 0.9516$) is high, and the adjusted $R^2$ (0.8635) is in close agreement with the predicted $R^2$ (0.8562) value.

**Table 6:Analysis of variance (ANOVA) for the fitted quadratic polynomial model for Methanol (Methanolysis)**

| Source | Coefficient estimate | Degree of freedom | Sum of square | F-value | P-value (Prob >F) |
|---|---|---|---|---|---|
| Model | 83.25 | 20 | 4496.19 | 14.81 | < 0.0001 |
| A | 0.32 | 1 | 2.50 | 0.16 | 0.6925 |
| B | -1.01 | 1 | 24.54 | 1.62 | 0.2298 |
| C | -1.56 | 1 | 58.63 | 3.86 | <0.0752 |
| D | 1.92 | 1 | 88.51 | 5.83 | 0.0343 |
| E | 2.09 | 1 | 104.54 | 6.89 | <0.0237 |
| AB | -4.23 | 1 | 286.54 | 18.87 | 0.0012 |
| AC | -2.58 | 1 | 106.66 | 7.02 | 0.0226 |
| AD | 4.13 | 1 | 272.99 | 17.98 | 0.0014 |

| | | | | | |
|---|---|---|---|---|---|
| AE | -2.52 | 1 | 101.56 | 6.69 | 0.0253 |
| BC | 0.92 | 1 | 13.49 | 0.89 | 0.3662 |
| BD | 4.51 | 1 | 324.81 | 21.39 | 0.0007 |
| BE | 0.73 | 1 | 8.54 | 0.56 | 0.4690 |
| CD | 1.51 | 1 | 36.27 | 2.39 | 0.1505 |
| CE | -2.42 | 1 | 93.65 | 6.17 | 0.0304 |
| DE | -2.43 | 1 | 94.62 | 6.23 | 0.0297 |
| $A^2$ | -1.02 | 1 | 30.31 | 2.00 | 0.1853 |
| $B^2$ | -6.39 | 1 | 1198.81 | 78.95 | < 0.0001 |
| $C^2$ | -6.52 | 1 | 1245.19 | 82.01 | < 0.0001 |
| $D^2$ | -5.13 | 1 | 771.31 | 50.80 | < 0.0001 |
| $E^2$ | -3.20 | 1 | 300.91 | 19.82 | 0.0010 |
| **Residual** | | | **15.18** | | |
| **Cor. Total** | | | **4663.21** | | |

Std. Dev. = 3.90; Mean = 66.56; C.V.% = 5.85; PRESS = 4372.66; $R^2$ = 0.9642; Adj. $R^2$ = 0.8991; Pred. $R^2$ = 0.8982; Adeq. Precision = 13.551

**Table 8: Analysis of variance (ANOVA) for the fitted quadratic polynomial model for Methanol (Ethanolysis)**

| **Source** | **Coefficient estimate** | **Degree of freedom** | **Sum of square** | **F-value** | **P-value (Prob >F)** |
|---|---|---|---|---|---|
| Model | 84.36 | 20 | 3502.59 | 10.81 | < 0.0001 |
| A | 2.58 | 1 | 160.17 | 9.88 | 0.0093 |
| B | 0.75 | 1 | 13.50 | 0.83 | 0.3810 |
| C | 0.58 | 1 | 8.17 | 0.50 | 0.4926 |
| D | -1.58 | 1 | 60.17 | 3.71 | 0.0802 |
| E | -0.17 | 1 | 0.67 | 0.041 | 0.8430 |
| AB | 0.38 | 1 | 2.25 | 0.14 | 0.7165 |
| AC | 0.13 | 1 | 0.25 | 0.015 | 0.9034 |
| AD | -1.37 | 1 | 30.25 | 1.87 | 0.1992 |
| AE | -1.25 | 1 | 25.00 | 1.54 | 0.2401 |
| BC | -3.50 | 1 | 196.00 | 12.09 | 0.0052 |
| BD | 4.00 | 1 | 256.00 | 15.79 | 0.0022 |
| BE | -0.88 | 1 | 12.25 | 076 | 0.4032 |
| CD | -1.75 | 1 | 49.00 | 3.02 | 0.1100 |
| CE | -0.13 | 1 | 0.25 | 0.015 | 0.9034 |
| DE | -2.13 | 1 | 72.25 | 4.46 | 0.0584 |

| | | | | | |
|---|---|---|---|---|---|
| $A^2$ | -4.86 | 1 | 693.88 | 42.81 | <0.0001 |
| $B^2$ | -2.49 | 1 | 181.67 | 11.21 | 0.0065 |
| $C^2$ | -2.74 | 1 | 220.00 | 13.57 | 0.0036 |
| Residual | | | 178.29 | | |
| Cor. Total | | | 3680.88 | | |

Std. Dev. = 4.03; Mean = 67.81; C.V.% = 5.94; PRESS = 4570.14; $R^2$ = 0.9516; Adj. $R^2$ = 0.8635; Pred. $R^2$ = 0.8562; Adeq. Precision = 9.986

Similarly, the sequential test as in table 10 show that the model F-value (17.27) of the quadratic model is large compared to the values for the other models for the equation. And from the statistics test, the regression coefficient ($R^2$ = 0.9691) is high, and the adjusted $R^2$ (0.9130) is in close agreement with the predicted $R^2$ (0.9011) value.

The experimental in tables 3 – 5 were also analyzed to check the correlation between the experimental and predicted biodiesel yield using methanol, and the normal probability and residual plot, and actual and predicted plot are shown in Figures 1 and 2 respectively.

**Table 10: Analysis of variance (ANOVA) for the fitted quadratic polynomial model for Methanol (Butanolysis)**

| Source | Coefficient estimate | Degree of freedom | Sum of square | F-value | P-value (Prob >F) |
|---|---|---|---|---|---|
| Model | 74.59 | 20 | 1413.46 | 17.27 | < 0.0001 |
| A | 0.58 | 1 | 8.17 | 2.00 | 0.1854 |
| B | 0.33 | 1 | 2.67 | 0.65 | 0.4366 |
| C | 0.83 | 1 | 16.67 | 4.07 | 0.0686 |
| D | -0.83 | 1 | 16.67 | 4.07 | 0.0686 |
| E | 1.42 | 1 | 48.17 | 11.77 | 0.0056 |
| AB | -1.37 | 1 | 30.25 | 7.39 | 0.0200 |
| AC | 0.50 | 1 | 4.00 | 0.98 | 0.3440 |
| AD | 0.25 | 1 | 1.00 | 0.24 | 0.6308 |
| AE | -0.23 | 1 | 1.00 | 0.24 | 0.6308 |
| BC | 1.50 | 1 | 36.00 | 8.80 | 0.0128 |
| BD | -0.50 | 1 | 4.00 | 0.98 | 0.3440 |
| BE | 1.75 | 1 | 49.00 | 11.98 | 0.0053 |
| CD | 0.88 | 1 | 12.25 | 2.99 | 0.1115 |
| CE | 0.63 | 1 | 6.25 | 1.53 | 0.2422 |
| DE | -1.38 | 1 | 30.25 | 7.39 | 0.0200 |
| $A^2$ | -2.47 | 1 | 178.37 | 43.59 | <0.0001 |
| $B^2$ | -3.09 | 1 | 280.24 | 68.49 | <0.0001 |
| $C^2$ | -2.22 | 1 | 144.03 | 35.20 | <0.0001 |

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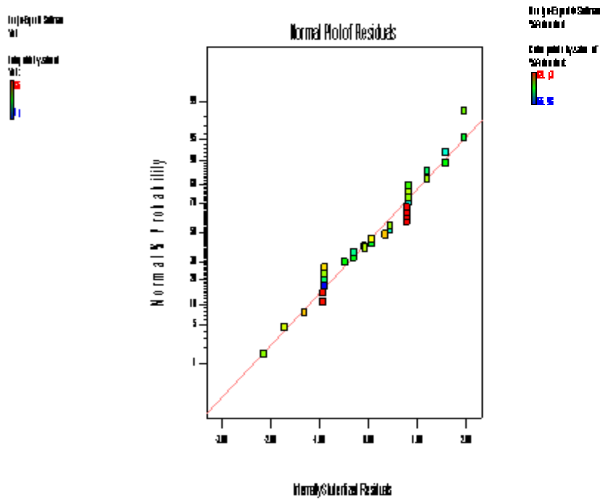
| | | | | | |
|---|---|---|---|---|---|
| $D^2$ | -2.47 | 1 | 178.37 | 43.59 | < 0.0001 |
| $E^2$ | -4.84 | 1 | 687.41 | 168.01 | <0.0001 |
| **Residual** | | | **45.01** | | |
| **Cor. Total** | | | **1458.47** | | |

Std. Dev. = 2.02; Mean = 63.28; C.V.% = 3.20; PRESS = 1119.60; $R^2$ = 0.9691; Adj. $R^2$ = 0.9130; Pred. $R^2$ = 0.9011; Adeq. Precision = 13.546

It can be seen from the Figures that the data points on the plot were reasonably distributed near to the straight line, indicating a good relationship between the experimental and predicted values of the response, and that the underlying assumptions of the above analysis were appropriate. The result also suggests that the selected quadratic model was adequate in predicting the response variables for the experimental data.

The experimental data in Table 4 were also analyzed to check the correlation between the experimental and predicted biodiesel yield using ethanol, and the normal probability and residual plot, and actual and predicted plot are shown in Figures 3 and 4. respectively.



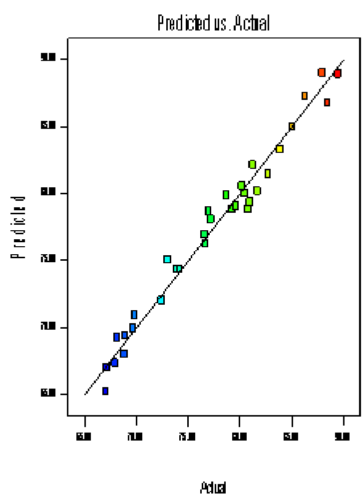




**Figure 1: Plot of predicted versus the actual experimental values for biodiesel yield using methanol.**

**Figure 2: Plot of normal probability versus residuals values for biodiesel yield using methanol.**

It can be seen from the Figures that the data points on the plot were reasonably distributed near to the straight line, indicating a good relationship between the experimental and predicted values of the response, and that the underlying assumptions of the above analysis were appropriate. The result also suggests that the selected quadratic model was adequate in predicting the response variables for the experimental data.

Analysis of experimental data in Table 5 was also performed to check the correlation between the experimental and predicted biodiesel yield using butaanol, and the normal probability versus residual plot, and actual versus predicted plot are shown in Figures 5 and 6 respectively. It can be seen from the figures that the data points on the plot were reasonably distributed near to the straight line, indicating a good relationship between the experimental and predicted values of the response, and that the underlying assumptions of the above analysis were appropriate. The result also suggests that the selected quadratic model was adequate in predicting the response variables for the experimental data.

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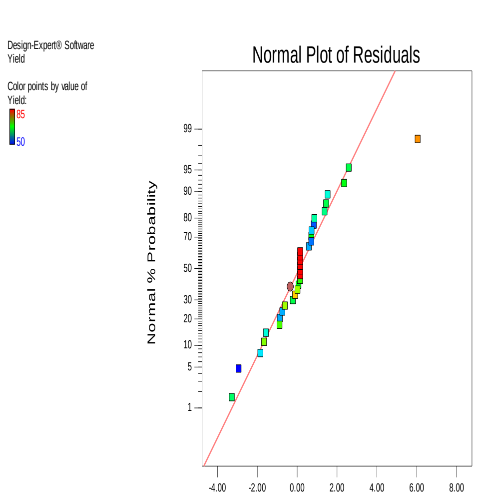


Figure 3: Plot of normal probability versus residuals for biodiesel yield using ethanol.

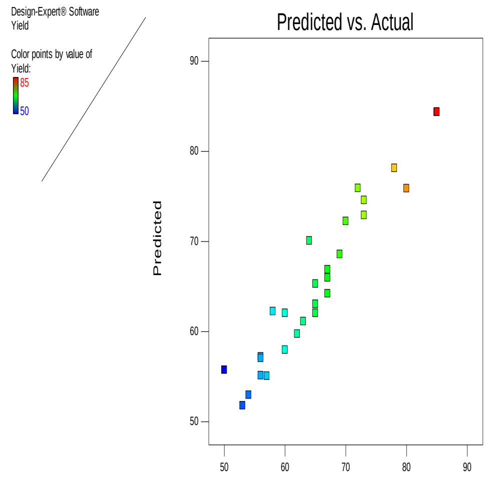


Figure 4.: Plot of predicted values versus the actual experimental values for biodiesel yield using ethanol.

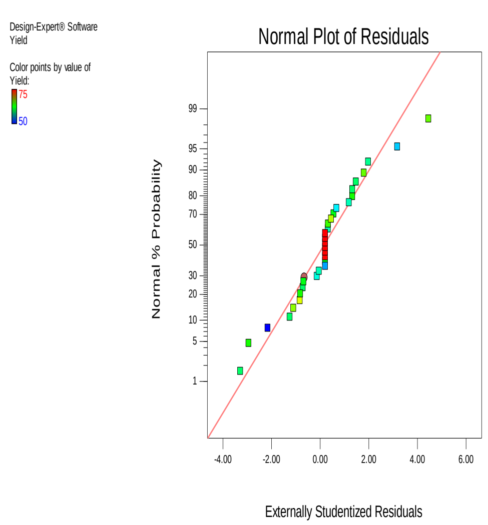


Figure 5: Plot of normal probability versus residualsvalues for biodiesel yield using butanol.

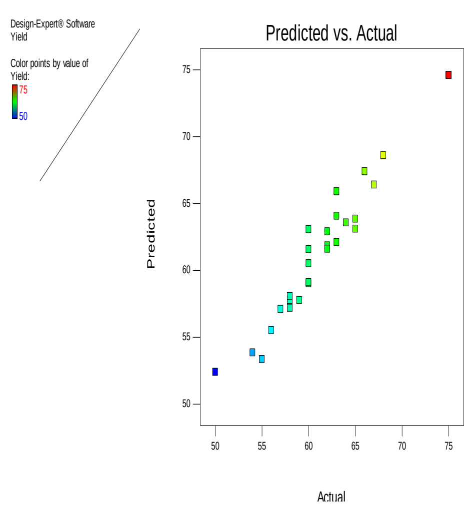


**Figure 6: Plot of predicted versus the actual values for biodiesel yield using butanol.**

**Response surface estimation**

Interactive effects of the process variables on the transesterification efficiency of the respective models were studied by plotting three dimensional surface curves against any two independent variables, while the other variables were kept at their central (0) level. The plots aid the understanding of the interaction of the variables and to determine the optimum level of each variable for maximum response. Figures (7 – 10) show the 3D curves of the response (transesterification efficiency) from the interactions between the variables in the methanol process while figures (11 – 12) show for ethanol and figures 13 - 16 are for butanol. On the curves, the elliptical shape of the curves indicates a good interaction of the two variables and circular shape indicates no interaction between the variables. The curves obtained in this study showed that there is a relative significant interaction between all the variables and for all the alcohols. Optimum conditions were also obtained from the response surface plots. For all the alcohols, the stationary point or central point is the point at which the slope of the contour is zero in all directions.

The coordinates of the central point within the highest contour levels in each of the plots will correspond to the optimum values of the respective variables. The maximum

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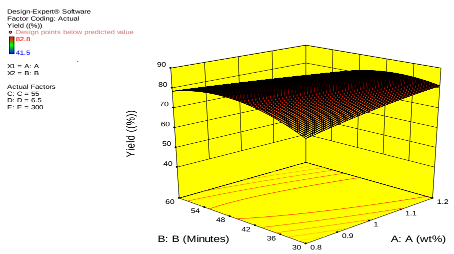


**Figure 7 : 3D Plot showing the interaction effect of time and catalyst concentration on the biodiesel yield**

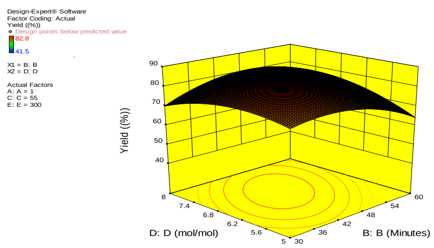


**Figure 8; 3D Plot showing the interaction effect of time and methanol/oil ratio on the biodiesel yield**

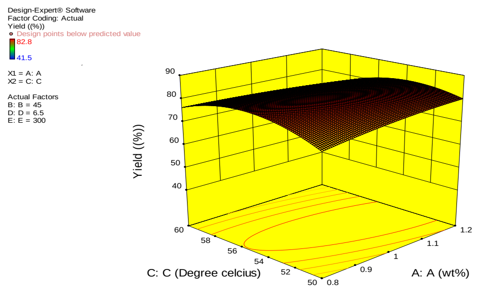


**Figure 9: 3D Plot showing the effect of temperature and catalyst concentration on the biodiesel yield**

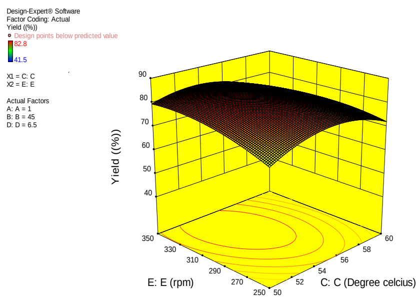


**Figure 10:3D Plot showing the interaction effect of Agitation speed and temperatureon the biodiesel yield**

predicted yield is indicated by the surface confined in the smallest curve of the contour diagram.

In the methanolysis process (Fig. 4 – 7), the optimum values of the variables were: reaction temperature, 55 °C; reaction time, 45min; catalyst weight 1.0%, methanol oil molar ratio 6:1 and agitation speed 300rpm. The predicted response value at these optimum values was 83.25%.

To confirm this optimum values, experiments were performed at these values and the experimental response value was 82 80%. This showed that the model correctly explains the influence of the process variables on the production of FAME from cotton seed oil.

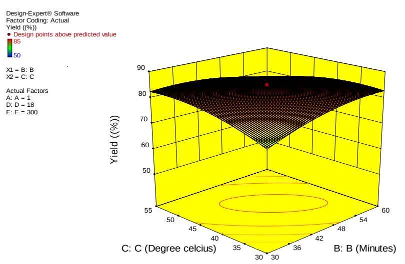


**Figure 11: 3D Plot showing the interaction effect of temperature and time on the biodiesel yield**

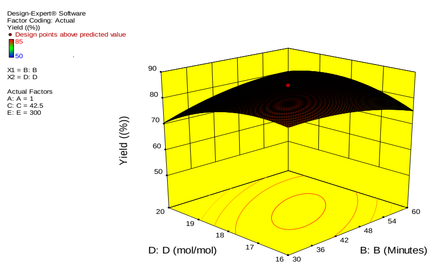


**Figure 12: 3D Plot showing the interaction effect of ethanol oil molar ratio and time on the biodiesel yield .**

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In the ethanolysis process (Fig. 11– 12), the optimum optimum values of the variables were: reaction temperature, 40 °C; reaction time, 45 min; catalyst weight 1.0 % agitation speed,and ethanol oil molar ratio 18:1. The predicted response value at these optimum values was 84.36%.

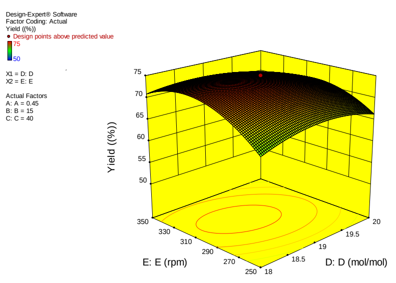


**Figure 13: 3D Plot showing the interaction effect of butanol/oil molar ratio and agitation speed on the biodiesel yield**

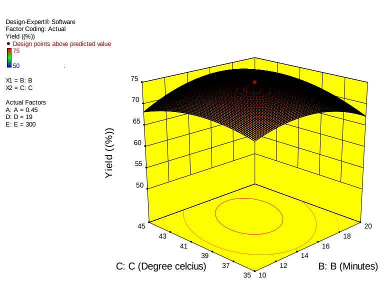


**Figure 14: 3D Plot showing the interaction effect of temperature and time on the biodiesel yield**

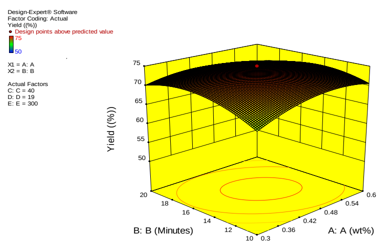


**Figure 15: 3D Plot showing the interaction effect time and catalyst concentration on the biodiesel yield**

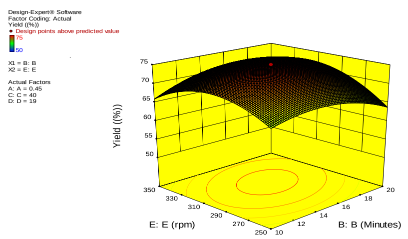


**Figure 16: 3D Plot showing the interaction effect of agitation speed and time on the biodiesel yield.**

To confirm this optimum values, experiments were performed at these values and the experimental response value was 85%. This showed that the model correctly explains the influence of the process variables on the production of FAEE from cotton seed oil.

In the butanolysis process (Fig. 13– 16), the optimum values of the variables were: reaction temperature, 40 °C; reaction time, 15 min; catalyst weight 0.5%, agitation speed,300rpm and methanol oil molar ratio 19:1. The predicted response value at these optimum values was 74.9%. To confirm this optimum values, experiments were performed at these values and the experimental response value was 75%. This showed that the model correctly explains the influence of the process variables on the production of fatty acid butyl ester (FABE) from cotton seed oil.

## Model Validation

Transesterification reaction under the obtained optimum operating conditions methanolysis, ethanolysis and butanolysis were carried out in order to evaluate the precision of the quadratic model; the experimental value and predicted values are shown in table 3, 4. 5. Comparing the experimental and predicted results, it can be seen that for each of the alcohols, the error between the experimental and predicted is less than 0.6%, therefore it can be concluded that the generated models have sufficient accurancy to predict the amount of alkyl ester yield.

**Table 7: Results of the model validation ( experiment 1 indicates the optimum reaction conditions and yield)**

| Experi-ment | Catalyst conc. (%wt oil) A | Methanol/oil molar ratio B | Temp .C | Time (Mins) D | Agitation speed (rpm) E | Experimental Yield (%) | Predicted yield (%) |
|---|---|---|---|---|---|---|---|
| 1 | 1 | 6 | 55 | 45 | 300 | 82.80 | 83.25 |

**Table 9: Results of the model validation ( experiment 1 indicates the optimum reaction conditions and yield)**

| Experiment | Catalyst conc.(%wt oil) A | Ethanol/oil molar ratio B | Temperature (ºC) C | Time (Minutes) D | Agitation speed (rpm) E | Experimental Yield (%) | Predicted yield (%) |
|---|---|---|---|---|---|---|---|
| 1 | 1 | 18 | 40 | 45 | 300 | 85 | 84.36 |

**Table10: Results of the model validation ( experiment 1 indicates the optimum reaction conditions and yield) for butanolysis.**

| Experiment | Catalyst conc.(%wt oil) A | Butanol/oil molar ratio B | Temperature (ºC) C | Time (Minutes) D | Agitation speed (rpm) E | Experimental Yield (%) | Predicted yield (%) |
|---|---|---|---|---|---|---|---|
| 1 | 0.5 | 19 | 40 | 15 | 300 | 75 | 74.59 |

**Effect of alcohol types on biodiesel yield.**

The result obtained in this study revealed that the amount of biodiesel fuel produced by using different types of alcohol decreased in the following order: ethanol > methanol > butanol. This result obtained was different from the findings of Hossain et al, (2010). They reported that methanol yields higher than ethanol and that both alcohols yield greater quantity of biodiesel than butanol. The result also contradicts the findings of Nye et al. (1983), which reported that methanol was the alcohol that can give the highest biodesel yield followed by butanol and then ethanol. Meher et al (2006) also reported that the production of biodiesel using ethanol in alkali-catalyzed transesterification is more difficult than that by using methanol. This is due to the formation of stable emulsion during ethanolysis. In methanolysis the less stable emulsion formed would breakdown easily to form lower glycerol rich layer and upper methyly ester rich layer while in ethanolysis, the emulsions formed are more stable due to the presence of larger non polar group in ethanol, thus making the separation and purification of biodiesel more difficulty (Zhou et al 2003). On the other hand, Mittelbach et al (2001) reported that the ethanol and butanol catalyzed transesterification gave much higher yields than methanol catalysed transesterification. The same result was also reported by Abigor et al (2000). Obviously from this review, the yield of biodiesel using different alcohols does not depend only on the type of alcohol but also on the catalyst as well as the triglyceride oil used.

## CONCLUSION

The production of biodiesel from cottonseed oil using methanol, ethanol and butanol and were carried out. The low acid value, iodine value and saponification value of the oil enable it to undergo direct transesterification without treatment. The methyl ester was produced by transesterification of cottonseed oil. Increase in process parameters such as reaction time, catalyst concentration, methanol/oil ratio, reaction temperature and agitation speed increase the yield of methyl ester to a reasonable point before it decreased. Optimization of the reaction parameters for biodiesel production from cottonseed oil was carried out using response surface methodology and central composite design. The effects of the reaction time, reaction temperature, catalyst concentration, methanol/oil molar ratio and agitation speed on the amount of methyl ester yields were significant parameters to predict the response values. The optimum values of the parameters were reaction time of 45minutes for both methanol and ethanol and 15minutes for butanol, reaction temperature of 55ºC (methanol), 40ºC (ethanol and butanol), catalyst concentration of 1% (methanol and ethanol) and 0.5% (butanol), methanol/oil molar ratio 7:1 (methanol), 18:1 (ethanol) and 19:1 (butanol) and agitation speed of 300rpm; under these conditions the amount of methyl ester yields achieved were 82.8% (methanol), 85% (ethanol) and 75% (butanol). The density, viscosity, cetane index and higher heating values of biodiesel produced under optimized protocol in the present work meet the ASTM standard and were within the acceptable limits.

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