

Chapter 5

**RSM BASED OPTIMIZATION
OF BIODIESEL PRODUCTION
FROM TOBACCO SEED OIL**

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ABSTRACT

Biodiesel is a monoalkyl ester of fatty acid of long chain derived from vegetable oil. Response surface methodology based on Central Composite Design is used to analyze the interaction effect and to optimize the transesterification reaction variables such as reaction duration, catalyst amount and oil to methanol ratio on biodiesel yield. A quadratic model developed based on the CCD, correlating the yield of biodiesel with reaction variables. The predicted yield by optimization is found very close to the experimental values. The optimum conditions for biodiesel production is found to be at the reaction duration of 78 min, oil to methanol ratio of 1:6.09 and catalyst amount of 1.13%. The fuel properties of tobacco methyl ester at the optimized parameters are determined as per ASTM standard and the results of the TSO biodiesel are within biodiesel specifications.

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1. INTRODUCTION

Transesterification is the mostly commonly used method of reducing the viscosity of vegetable oil and it is the reaction of a lipid with alcohol to form esters and a by-product, glycerol. Transesterification seems to be the best choice as the physical characteristics of fatty acid (m) ethyl esters (biodiesel) are very close to those of diesel fuel and the process is relatively simple. Different methods to produce biodiesel abound but transesterification is the most commonly used method of reducing the viscosity of vegetable oil. Transesterification is the reaction of a lipid with alcohol to form esters and a byproduct, glycerol. By transesterifying vegetable oils, alkyl monoesters of the fatty acids present in the vegetable oil are obtained. These esters are commonly referred to as biodiesel. A catalyst is usually required to speed up the reaction; this may be basic, acidic or enzymatic in nature. The reaction is affected by several parameters such as the concentration of catalyst, oil to methanol ratio, reaction temperature, moisture, presence of free fatty acids (FFA) and agitation intensity [1]. This process involves many parameters that effect the reaction and optimizing so many reaction factors require large number of experiments, which is laborious, time consuming, and economically non-viable. Response surface methodology (RSM) is a useful statistical technique for the optimization of complex processes, as it reduces the number of experiments required to achieve ample data for a statistically pertinent result [2]. Optimization of process parameters is the key step in response surface methods to achieve high quality without cost inflation [3].

Biodiesel a renewable fuel with lesser exhaust emissions is one of the alternative sources of energy that is presently of interest worldwide. Hence, researchers have focused on the development and the optimization of the processes of biodiesel production to meet the standards and specifications needed for the fuel to be used commercially without compromising on the durability of engine parts. However, the global inevitability of fossil fuels, ever increasing demands for diesel and uncertainty in their availability, depletion rate of energy source, emission of very dangerous pollutants, rapid industrialization and increasing price that make petroleum no longer economically sustainable have necessitated the search for cheap raw material. A sustainable and economical supply of raw material is the key factor for biodiesel to be competitive commercially [4-8]. Tobacco seed oil is one of the promising alternative as the seeds are not commercial products, they are collected from fields and they are a by-product of tobacco leaf product. Veljkovic et al. [9] remarked that tobacco seed, as agricultural wastes and with quite high oil content, might be a cheap and valuable renewable raw material for biodiesel production and the authors produced tobacco biodiesel with maximum yield of 91%. They reported that tobacco biodiesel obtained had fuel properties within the limits prescribed by international standard. Tobacco (*Nicotiana tabacum* L.) seed oil (TSO), a by-product of tobacco leaf production, has been shown to be an appropriate substitute for a diesel fuel in a raw [10] or chemically modified [11] form.

Mukhtar and Mukhtar [12] highlighted that tobacco seed oil is comparable to the other commonly used vegetable oil. Usta [13] reported that properties and quality verification of biodiesel produced from tobacco seed oil. Murthy and Babu [14] concluded that B5 tobacco biodiesel is the most suitable blend in view of the engine performance and emission. Parlak et al. [15] investigated parameters that affect the yield of tobacco biodiesel and their interactions on emissions of a diesel engine. They concluded that choosing right catalyst and the tobacco

methyl ester blend rate are important two factors in view of minimization of pollutant emissions. Mohanrao et al. [16] reported that tobacco seed oil can be used as an alternate fuel in cases of emergency and short term use with proper precautions and tuning of the engine for best performance level.

The Response Surface Methodology (RSM) is a combination of statistical and optimization methods that can be used to model and optimize designs [17-19]. It has many applications in design improvement of products and process operation.

So far, few or no work has been documented on literature on optimization of experimental conditions for biodiesel production from methanolysis of esterified tobacco seed oil; therefore this chapter was aimed at optimizing the condition of transesterification of esterified tobacco seed oil into biodiesel through reaction variable such as alcohol to oil molar ratio, catalyst amount and reaction time. This paper is prepared with following objectives:

1. To study the effect of process factors such as oil-methanol ratio, catalyst amount and reaction time at various levels on yield of tobacco seed oil methyl ester using central composite experimental design
2. To determine the optimum process factors conditions for methanolysis of tobacco methyl ester
3. To characterize the tobacco methyl ester produced at the optimum process factor levels according to ASTM standard.

The world is faced with challenges of minimizing emission of green house gases and other pollutants as well as overcoming the shortage of power supply. Hence, there is a need to develop alternative cost-effective biodiesel feedstock in form of tobacco seed oil that are capable of powering everyday life without bringing harmful environmental changes. Production of biodiesel from tobacco seed oil would not compete with food uses of edible oils and thus there would be value addition as well as curtailing smoking related challenges. This chapter is restricted to optimization of methanolysis/alkaline transesterification parameters of pre-treated tobacco seed oil. The tobacco seed oil methyl ester produced at the optimum process factor levels is characterized and compared with ASTM standard. Also, effort will be made to develop model for biodiesel produced from Nigerian tobacco seed oil.

2. MATERIALS AND METHODS

Tobacco seed was obtained from Agricultural farm, Olabisi Onabanjo University, Ibogun campus, Ogun State, Nigeria. Methanol, sulphuric acid, potassium hydroxide, phenolphthalein, ethanol, acetic acid and other chemicals/reagents were obtained from Uche Scientific Company Limited, Lagos, Nigeria. All chemical/reagents were of analytical grade with purity > 99%.

2.1. Seed Preparation and Tobacco Seed Oil Extraction

Tobacco seeds were separated from foreign matters, weighed and grinded using blender in order to expose large surface area of the cells containing oil for the extraction of the oil. Due to unavailability of tobacco seed oil in markets, the oil was initially extracted from the collected seeds in laboratory conditions. The extraction procedures entailed drying tobacco seed until a constant weight was obtained and the seeds were grounded to a fine powder. A soxhlet extraction apparatus was used to extract the oil from seed and hexane was used as solvent in extraction process and the oil was separated from the hexane using a Buchi R114 rotary vacuum evaporator (Figure 1). The percentage of oil yield is given by expression in Equation 1.

$$\text{Oil yield (\%)} = \frac{\text{Mass of oil obtained (kg)}}{\text{mass of seed used (kg)}} \times 100\% \quad (1)$$



Figure 1. Soxhlet extraction set-up.

2.2. Acid-base Catalyzed Transesterification of High FFA TSO

Raw TSO was filtered to remove all the insoluble impurities followed by heating at 100°C for 15 min to remove all the moisture. TSO had high FFA (15.98%), which is far above the 1% limit suitable for alkaline catalyzed transesterification reaction. FFAs were, therefore, first converted to esters in a pretreatment process for the production of esterified tobacco seed oil. The reaction was carried out at a temperature of 60°C for 60 min. using conc. H₂SO₄ (2.5% w/w) as acid catalyst with methanol/oil ratio of 7.5:1 in author's laboratory. The

mixing solution of methanol with catalyst (sulphuric acid) is also heated prior to be added into the reactor. After the reaction completing, the products are allowed for 2 h settling down in a separation funnel and then, the methanol-water fraction at the bottom layer is removed. The high FFAs were reduced from 15.98% to 0.98% after second pre-treatment and the oily phase of reaction mixture (FAME) was subjected to alkaline catalyzed transesterification process for biodiesel production using KOH as base catalyst (Figure 2).

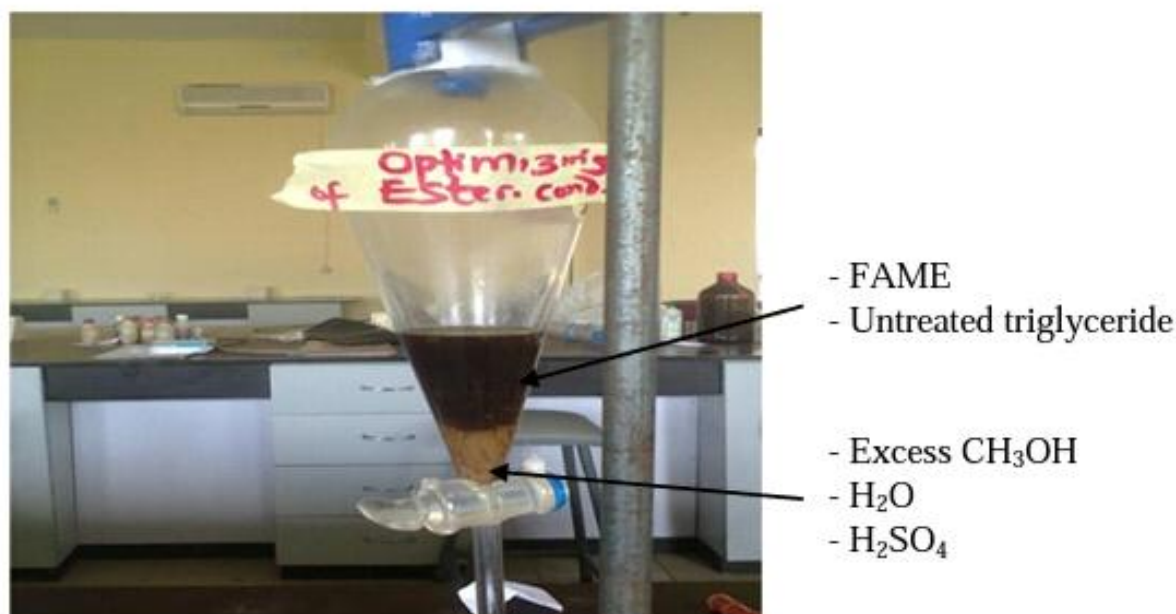


Figure 2. Product from acid esterification during the separation process.

2.3. Alkaline Transesterification Procedures

Esterified tobacco seed oil was subjected to base catalyzed transesterification for biodiesel production at specified reaction conditions according to the Central Composite Response Surface Design (CCRD) using conical flask equipped with a condenser, thermometer, chiller, heating plate and stirrer. The pre-heated oil was trans-esterified with methanol and potassium hydroxide to yield TSO biodiesel and glycerol. The resulting product was poured into a separating funnel mounted on a clamp stand and was allowed to settle down overnight. It was observed that the resulting mixture from the reaction had settled into yellow biodiesel on the top with the black glycerol at the bottom of the separating funnel (Figure 3). The ester was washed with water three times. Small amount of acetic acid (2.5ml/l of the oil) was used in the first washing. At the end of the process, the ester was heated to 90°C at vacuum to remove any water left from the oil. The final ester became clear straw yellow. The procedure was replicated three times and the transesterification of TSO has been optimized using RSM for the maximization of TSB yield which was calculated using the following Equation (2):

$$\text{Yield of TSOB (\%)} = \frac{\text{Total weight of methyl ester}}{\text{Total weight of oil in the sample}} \times 100 \quad (2)$$

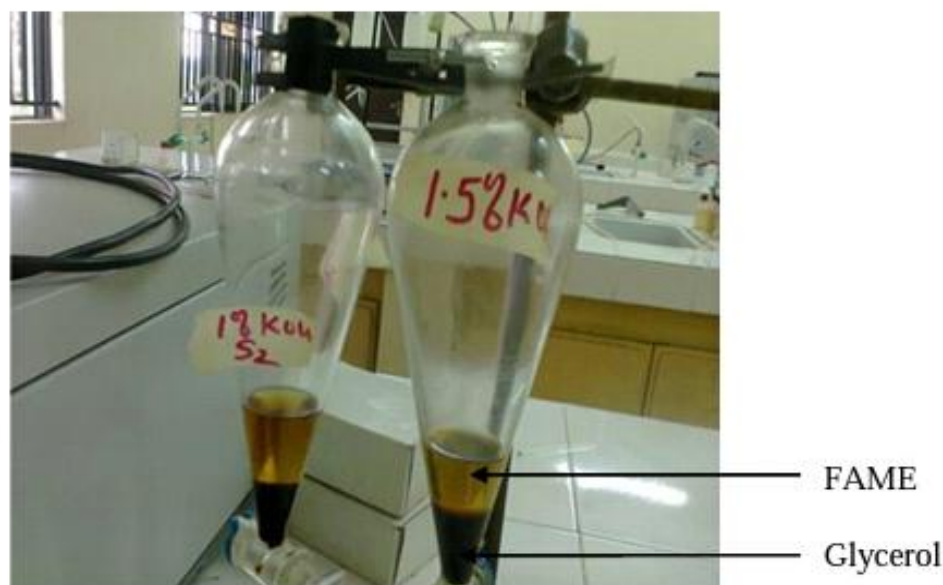


Figure 3. Two-layers separation after alkali catalyzed transesterification.

2.4. Fuel Properties Tests

The fuel properties of TSOME produced from the optimum reaction variables of TSO transesterification were determined in accordance to biodiesel standard test methods. The standards employed were by the American ASTM D 6751 and European Union EN 14214 biodiesel standards [20]. Fuel properties such as kinematic viscosity (ASTM D 445), density (ASTM D 4052), flash point (ASTM D 93) and acid value (EN 14104) were determined. Values obtained were compared with the ASTM D 6751 and EN 14214 standards.

2.5. Design of Experiment

The alkaline transesterification of tobacco seed oil and optimization of the methyl ester was studied using Design Expert 6.0.6 (Stat-Ease Inc., USA). Response Surface Methodology (RSM) and Central Composite Design (CCD) were used to find the interactions between the three variables and to predict the optimum condition for biodiesel yield. Selection of these parameters was based on the literature studies reported on the alkaline solutions. These parameters were catalyst (KOH) amount, methanol to oil molar ratio and time. Methyl ester yield was used as the response. The reported data in this work were average of triplicate measurements to ensure accuracy. Table 1 shows the levels of the variables used and the ranges. Selection of the levels was based on the preliminary study and literature research. The reaction time, methanol to oil molar ratio catalyst (KOH) amount, were varied from 40 to 80 minutes, 4 to 8 mol/ mol and 0.5 to 1.5 wt. %, respectively. The reactant mixture temperature was held constant below the boiling point of methanol (65°C) in order to prevent mass transfer limitation and bubble formation of methanol. A three-level-three-factors CCD (20 experiments) was adopted, consisting of 8 factorial points, 6 axial points and 6 replicates at the center points design. The yield of methyl ester was analyzed and optimized using Equation (3) as follows:

$$y = \beta_0 + \sum_{i=1}^3 \beta_i X_i + \sum_{i=1}^3 \beta_{ii} X_i^2 + \sum_{j=1}^3 \sum_{j=1}^3 \beta_{ij} X_i X_j \quad (3)$$

where β_0 , β_i and β_{ii} are regression coefficients for intercept, linear, quadratic and interaction coefficients, respectively are X_i and X_{ij} are coded independent variables.

3. RESULTS AND DISCUSSION

3.1. Percentage Oil Content of Tobacco Seed Oil

The oil content of the collected seeds was determined as approximately 35.6% (in weight basis) using hexane as extraction solvent.

3.2. Development of Regression Model

In this chapter, the correlation between response (yield) and three parameters, reaction time, methanol to oil molar ratio and amount of catalyst, KOH, were analyzed using alkaline transesterification. The experimental data of the three parameters and the response yield were shown in Table 1. The Response Surface Methodology was conducted to find the relationship between the time, methanol to oil molar ratio and catalyst dosage. This investigation was employed to obtain high yield of biodiesel without adding co-solvent. A model for the experimental design was fitted using Design Expert 6.0.6 (Stat-Ease Inc., USA). Final equation in terms of (coded factors) experimental data was:

$$\begin{aligned} \text{Yield} = & +86.40 + 3.70A + 0.73B + 7.45C - 0.81A^2 - \\ & 5.71B^2 - 18.17C^2 - 1.83AB + 2.01AC - 1.39BC \end{aligned} \quad (4)$$

where, A, B and C denote reaction time (min), methanol to oil molar ratio (M), and catalyst (KOH) amount (wt.%), respectively, and Zabeti et al. [21] reported that positive sign in front of each term denotes synergetic effect and negative sign shows the antagonistic effect. Figure 4 shows the predicted value and experimental value using the model equations in Equation (1) and the graph shows that the predicted values are quite close to the experimental value and confirmed by the R^2 value, 0.9828. Analysis of variance (ANOVA) was employed to investigate the significance and fitness of the model. ANOVA also represents the interaction of variables on the respond and the effect of individual parameters. Table 3 indicates that the model was very significance because the p value < 0.0001 . There was no lack of fit since the value was not significant. Lack of fit implies that the model was fitted to the data Adequate precision measures the signal to noise ratio. This model can be utilized to navigate the design region as ratio greater than 4. Therefore it is desirable and in this case, the ratio was 23.801.

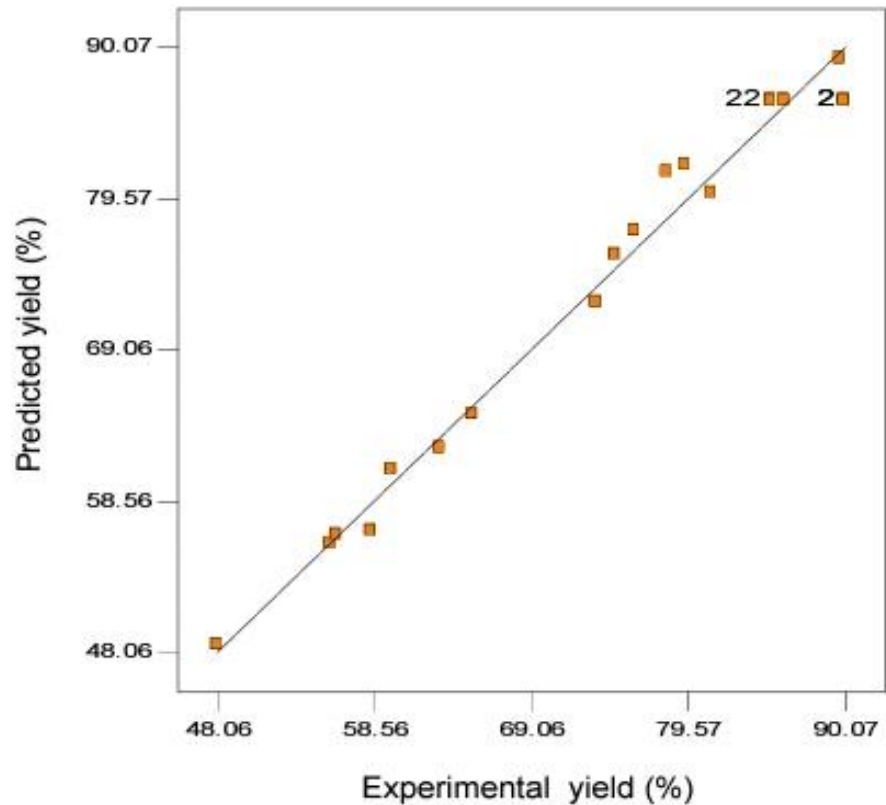
DESIGN-EXPERT Plot
Yield

Figure 4. A parity plot between actual experimental values and predicted values of TSO biodiesel.

Table 1. Levels for Biodiesel Production of Tobacco seed Oil

| Variables | Coding | Unit | Lower level | High Level |
|-----------------------------|--------|-------|-------------|------------|
| Time | A | Min | 40 (-1) | 80 (+1) |
| Methanol to oil Molar ratio | B | M | 4(-1) | 8 (+1) |
| Catalyst amount | C | wt. % | 0.5 (-1) | 1.5 (+1) |

Table 2. Central composite design for optimizing NaOH-catalyzed transesterification

| Run | Coded factors | | | Actual factors | | | Biodiesel Yield (%) | | |
|-----|---------------|----|----|----------------------|-----------------------------|-------------------|---------------------|-----------|----------|
| | A | B | C | Reaction time (min.) | Methanol to Oil molar ratio | Catalyst (w/w. %) | Experimental | Predicted | Residual |
| 1 | -1 | -1 | -1 | 40 | 4 | 0.5 | 48.06 | 48.62093 | -0.56093 |
| 2 | 1 | -1 | -1 | 80 | 4 | 0.5 | 55.63 | 55.64193 | -0.01193 |
| 3 | -1 | 1 | -1 | 40 | 8 | 0.5 | 58.36 | 56.51893 | 1.841068 |
| 4 | 1 | 1 | -1 | 80 | 8 | 0.5 | 56.04 | 56.23493 | -0.19493 |
| 5 | -1 | -1 | 1 | 40 | 4 | 1.5 | 62.97 | 62.26493 | 0.705068 |
| 6 | 1 | -1 | 1 | 80 | 4 | 1.5 | 76.01 | 77.34093 | -1.33093 |
| 7 | -1 | 1 | 1 | 40 | 8 | 1.5 | 65.13 | 64.60793 | 0.522068 |
| 8 | 1 | 1 | 1 | 80 | 8 | 1.5 | 73.45 | 72.37893 | 1.071068 |

| Run | Coded factors | | | Actual factors | | | Biodiesel Yield (%) | | |
|-----|---------------|----|----|----------------------|-----------------------------|-------------------|---------------------|-----------|----------|
| | A | B | C | Reaction time (min.) | Methanol to Oil molar ratio | Catalyst (w/w. %) | Experimental | Predicted | Residual |
| 9 | -1 | 0 | 0 | 40 | 6 | 1 | 79.38 | 81.88727 | -2.50727 |
| 10 | 1 | 0 | 0 | 80 | 6 | 1 | 89.75 | 89.28327 | 0.466727 |
| 11 | 0 | -1 | 0 | 60 | 4 | 1 | 81.15 | 79.95127 | 1.198727 |
| 12 | 0 | 1 | 0 | 60 | 8 | 1 | 78.18 | 81.41927 | -3.23927 |
| 13 | 0 | 0 | -1 | 60 | 6 | 0.5 | 59.71 | 60.78327 | -1.07327 |
| 14 | 0 | 0 | 1 | 60 | 6 | 1.5 | 74.71 | 75.67727 | -0.96727 |
| 15 | 0 | 0 | 0 | 60 | 6 | 1 | 90.07 | 86.39982 | 3.670182 |
| 16 | 0 | 0 | 0 | 60 | 6 | 1 | 86.04 | 86.39982 | -0.35982 |
| 17 | 0 | 0 | 0 | 60 | 6 | 1 | 86.04 | 86.39982 | -0.35982 |
| 18 | 0 | 0 | 0 | 60 | 6 | 1 | 90.07 | 86.39982 | 3.670182 |
| 19 | 0 | 0 | 0 | 60 | 6 | 1 | 85.13 | 86.39982 | -1.26982 |
| 20 | 0 | 0 | 0 | 60 | 6 | 1 | 85.13 | 86.39982 | -1.26982 |

Table 3 illustrates the time (A), catalyst (C), quadratic term methanol to oil molar ratio (B^2), quadratic term catalyst amount (C^2) and time- catalyst amount (AC) were significant as the p- value < 0.05. There exist four insignificant values which were higher than p- value > 0.005, linear methanol to oil ratio (B), quadratic term time (A^2), time- methanol to oil molar ratio (AB) and molar ratio – catalyst amount (BC). This indicates that the model could be utilized to predict biodiesel yield. Figure 4 again shows that the regression model equation provided a very accurate description of the experimental data, indicating that it was successful in capturing the correlation between the process parameters to the yield of TSO biodiesel. This is further supported by the correlation coefficient, R^2 which was found to be very close to unity, 0.961.

Table 3. ANOVA table for response surface quadratic model

| Source | Prob.> F | Remark |
|-------------|----------|-----------------|
| Model | < 0.0001 | Significant |
| A | 0.0007 | Significant |
| B | 0.3594 | Not significant |
| C | < 0.0001 | Significant |
| A^2 | < 0.5884 | Not significant |
| B^2 | 0.0029 | Significant |
| C^2 | < 0.0001 | Significant |
| AB | 0.0582 | Not significant |
| AC | 0.0401 | Significant |
| BC | 0.1351 | Not significant |
| Lack of fit | 0.4553 | Not significant |
| Pure error | | Not significant |
| R^2 | 5.53 | |
| Adj R^2 | 0.9672 | |
| Pred R^2 | 0.9138 | |

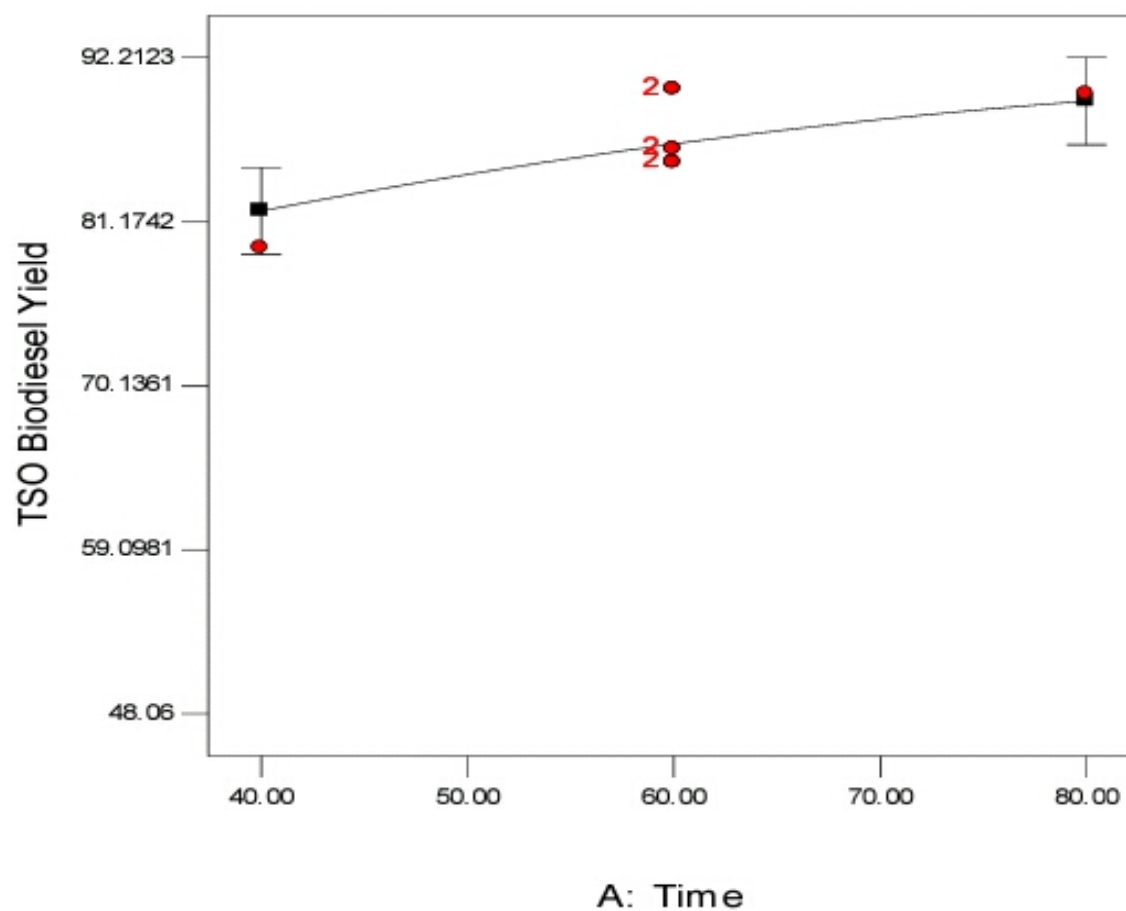


Figure 5. Effect of reaction time on TSO biodiesel yield.

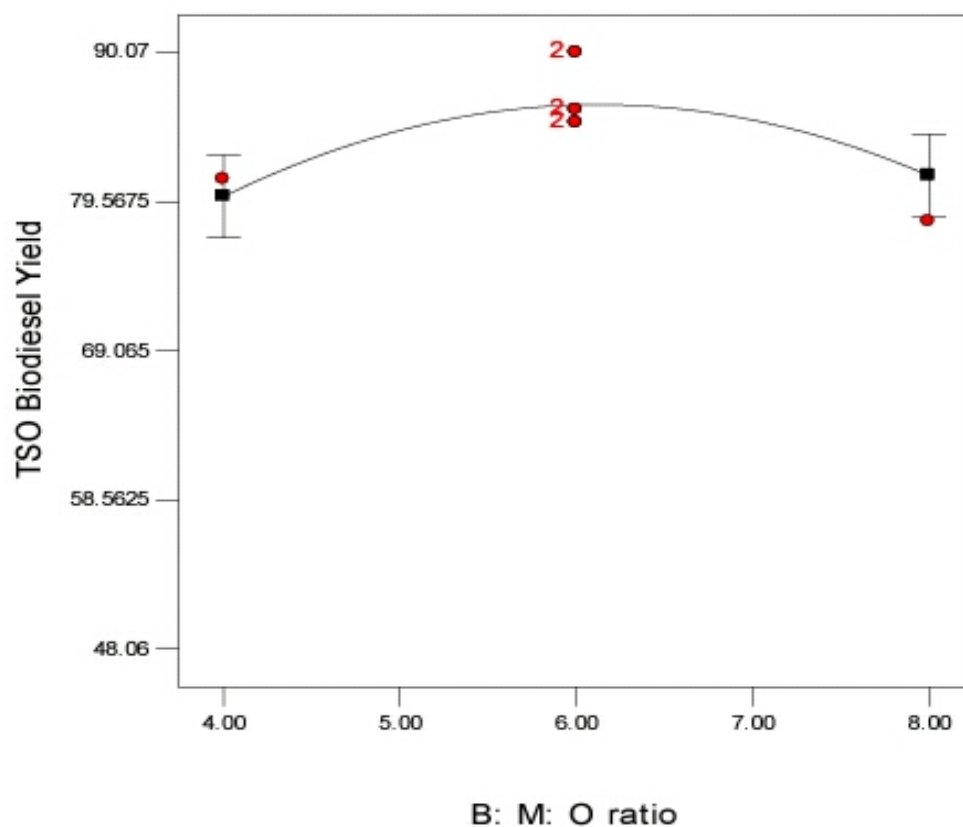


Figure 6. Effect of methanol-to oil molar ratio on TSO biodiesel yield.

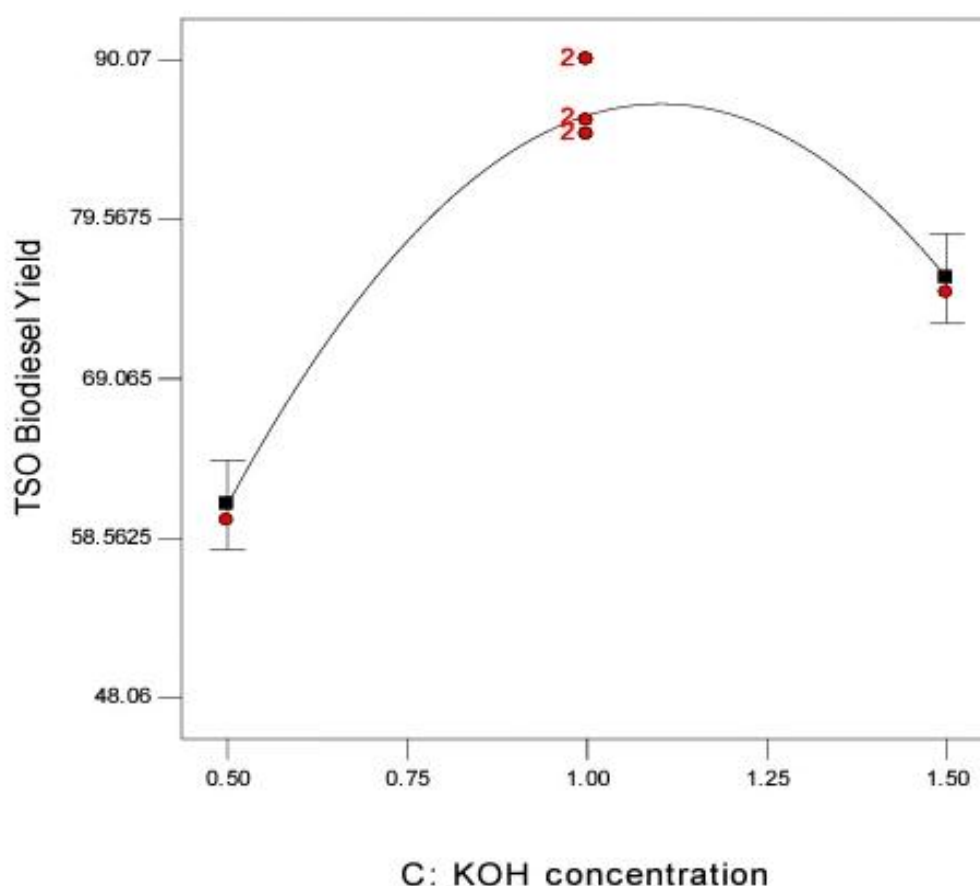


Figure 7. Effect of amount of catalyst on TSO biodiesel yield.

3.3. Effect of Transesterification Process Variables

Based on the analysis of variance, the transesterification reaction was remarkably influenced by various interactions between the process variables. However, significant individual process variables that affect the transesterification reaction are reaction time (A), methanol-oil molar ratio (B), and catalyst amount (C). The effect of reaction time on biodiesel yield is shown in Figure 5. It is seen from one factor plot that the yield of biodiesel increased with increase in reaction time from 40 to 80 minutes. Above 40 minutes, the biodiesel yield greatly increased and maximum yield was obtained at a reaction time of 80 minutes.

The ratio of methanol to oil is one of the important factors that affect the conversion of triglyceride to fatty acid methyl ester (FAME). The results obtained in this chapter as revealed in Figure 6 shows that the yield of biodiesel increased with increase in methanol-oil molar ratio from 4 to 6 mol/mol. Above 6 mol/mol, the biodiesel yield slightly decreased. Therefore, maximum yield is obtained at a molar ratio of 6, showing that biodiesel yield increased as methanol-to-oil molar ratio increases up till an optimum value after which it decreased. Aworanti et al. [22] reported that higher ratio of methanol used could also minimize the contact of excess triglyceride molecules on the catalyst's active sites which could decrease the catalyst activity. They further stressed that an increase in the ratio of methanol could also lead to the increase in the purity of the biodiesel layer which would also be responsible for the observed increase in biodiesel yield.

Figure 7 illustrates influence of the dosage of KOH catalyst on biodiesel yield. When the mass of catalyst was increased from 0.5% to 1%, the active sites of KOH was increased; accelerating transesterification reaction, thereby enhancing biodiesel yield. However, optimum yield was obtained at a mass of 1%.

3.4. Effect of Interaction between Process Variables

Three-dimensional surface plots of the predicted yield of the biodiesel production are given in Figures 8-10. Each surface was generated by keeping one factor constant at the central point of CCD and varying the other two within their experimental ranges. The figures show the yield of biodiesel increases with the increase of time, oil to methanol molar ratio and catalyst loading.

Figure 8 shows the effect of interaction of methanol-to- oil molar ratio and reaction time on methanolysis of TSO to biodiesel at a constant amount of KOH dosage (1%). There was a high significant effect of reaction time on alkali-catalyzed transesterification. Under such conditions, methanol-to-oil molar ratio of about 6: 1 gave the suitable conversion to biodiesel. Also, there was negative significant interaction between methanol-to oil molar ratio and reaction time. This shows that TSO biodiesel yield decrease beyond an optimum value, whereas it increases up till an optimum value (6:1).

Figure 9 shows the interaction effect of catalyst amount and reaction time on TSO biodiesel production at a constant methanol to-oil molar ratio of 6:1 dosage of KOH and reaction temperature significantly influences TSO biodiesel production. There was a positive significant interaction between catalyst and reaction time. This indicated that TSO biodiesel increases with increase in temperature an increase of catalyst. However, at longer reaction time (80min), an increase of catalyst amount from 0.5wt. % to 01.5 wt. % only slightly increases in the yield of TSO biodiesel.

Effects of interaction of methanol-to oil molar ratio and catalyst amount on transesterification of TSO to biodiesel at a constant reaction duration is showed in Figure 10. At lower methanol-to-oil molar ratio, the conversion to biodiesel increased with increase in KOH amount. It appears increase in methanol-to-oil molar ratio had remarkable influence on the conversion at different catalyst dosage.

3.5. Model Evaluation of Alkaline Transesterification

The optimum condition of these three parameters was obtained by optimization of the Equation (3) using Design Expert 6.0.6. Table 4 shows the summary of optimization conditions with high and low limit to optimize based on the previous model. In this case, reaction time and catalyst amount were in the range. The optimal condition for this study was 1.13 wt. % of KOH catalyst, 6.09 methanol/ oil molar ratio and time of 78 min (Table 5).

To evaluate the accuracy of the developed model, base catalyzed transesterification was conducted according to the optimum condition as in Table 5. Table 5 shows the results obtained and the yield was 90.11% if compared to the predicted yield, 90.17%. Both of the values are close with smaller error of 0.06% and this shows that this model was in agreement with the actual value.

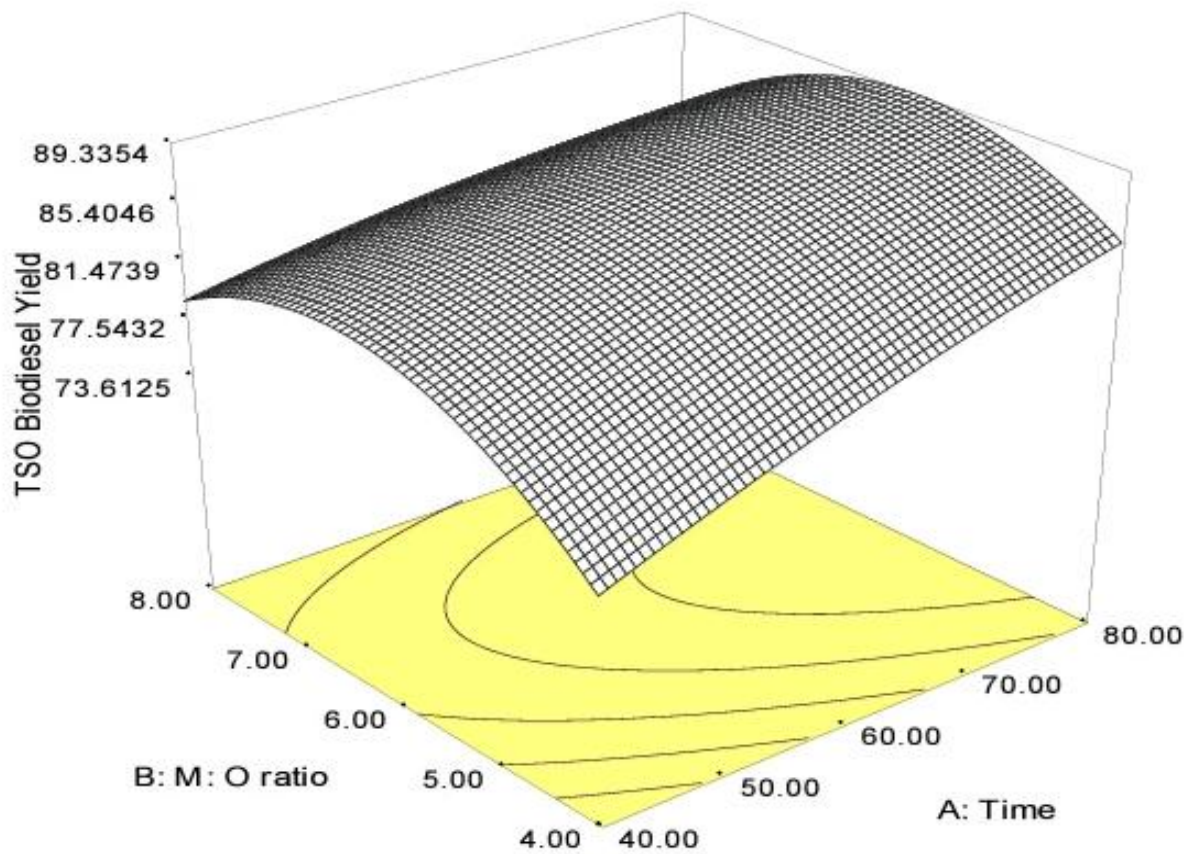


Figure 8. Response surface plot of the interaction of time and methanol-to oil ratio on TSO biodiesel yield when the amount of catalyst is 1 percent.

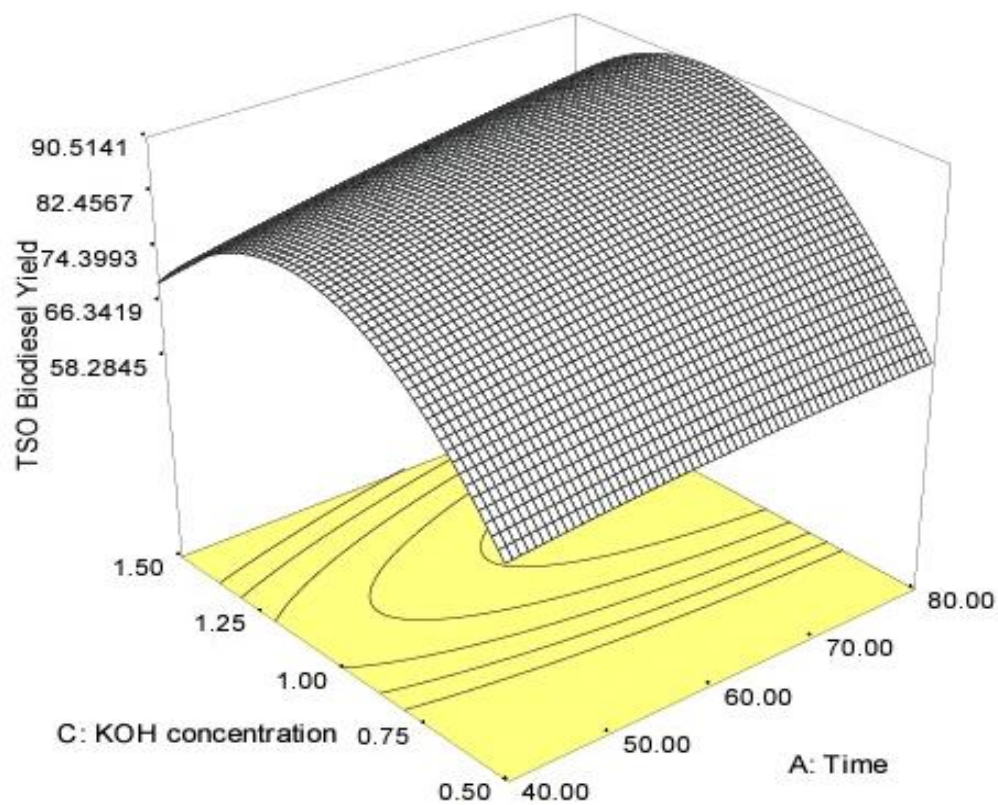


Figure 9. Response surface plot of the interaction effect of catalyst amount and reaction time on TSO biodiesel yield when the molar ratio of methanol to oil is 6:1.

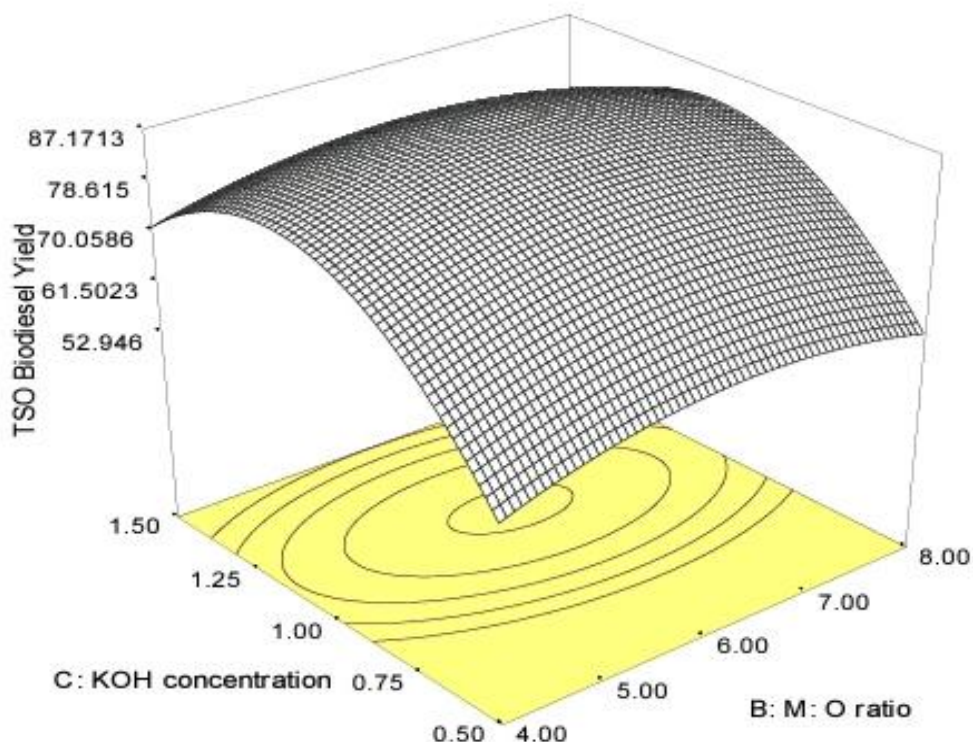


Figure 10. Response surface plot of the interaction effect of methanol-to oil molar ratio and catalyst amount on TSO biodiesel yield when the reaction time is 60 minutes.

Table 4. Summary of optimization conditions

| Parameter | Objective of optimization | Lower limit | Upper limit |
|---------------|---------------------------|-------------|-------------|
| Reaction time | Is in range | 40 | 80 |
| M: O ratio | Is in range | 4 | 8 |
| KOH amount | Is in range | 0.5 | 1.5 |
| Yield | To maximize | 48.06 | 90.07 |

Table 5. Results of modeling/optimization of biodiesel yield

| Experiment | Time, A (min) | Methanol/ oil ratio, B (mol) | Catalyst amount, C (wt. %) | Yield (%) (predicted) | Yield (%) (Experimental) |
|------------|---------------|------------------------------|----------------------------|-----------------------|--------------------------|
| 1 | 78 | 6.09 | 1.13 | 90.17 | 90.11 |

Table 6. Properties of TSO biodiesel

| Fuel Characteristics | Unit | TSO methyl ester | ASTM Standard D6751 | EU Standard EN 14214 Corrected value | IS 15607 |
|-----------------------------|--------------------|------------------|---------------------|--------------------------------------|-------------|
| Specific gravity@ 50°F/50°F | g/cm ³ | 0.891 | 0.880 | 0.86-0.90 | 0.860-0.900 |
| Density | Kg/m ³ | 890 | | 860-900 | |
| Kinematic viscosity @40°C | mm ² /s | 3.87 | 1.9-6.0 | 3.5-5.0 | 2.5-6.0 |
| Flash point | °c | 126 | >120 | > 120 | - |
| Acid value | Mg KOH/g | 0.420 | <0.8 | <0.50 | - |

Validation of the Final Model

Through the *t*-test, no significant differences are observed between the experimental and predicted values ($P \geq 0.05$). Moreover, the closeness of actual and predicted values in Figure 11 shows that the regression equation is adequate (Mirhosseini et al., 2008).

3.6. Biodiesel Properties

Table 6 shows the summary of the properties of the biodiesel of tobacco seed oil. The properties of TSO biodiesel were within the mentioned biodiesel standards, ASTM D6751, EN 14214 and IS 15607. The result of few standard fuel test such as specific gravity, viscosity, flash point and acid value of the TSO biodiesel are within biodiesel specification.

CONCLUSION

Central Composite Design was used for optimization of methanol/oil ratio, catalyst amount, and reaction time on the transesterification of Nigerian tobacco seed oil. Thus, research work gave an optimal value of 90.17% biodiesel, in 78 minutes of reaction time with 1.13 wt.% of catalyst loading and methanol/oil ratio of 1:6.09. Quadratic polynomial models were obtained to predict yield of biodiesel. On the basis of ANOVA; reaction time, catalyst amount, quadratic term methanol to oil molar ratio, quadratic term catalyst amount and time-catalyst amount were significant. Reaction time, catalyst amount, quadratic term of methanol/oil ratio, quadratic term catalyst amount, reaction time - catalyst amount had a significant effect on TSO biodiesel yield. The result showed that the predicted value was in agreement with the experimental value, which was established with additional experiments to confirm the optimized parameters. The prepared TSO biodiesel gave promising results as alternative diesel fuel with fuel properties in good agreement within limit set by international biodiesel standard. Therefore production of biodiesel from Nigerian tobacco seed oil is a feasible process.

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