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# SYNTHESIS OF FATTY ACID ETHYL ESTER FROM CHICKEN FAT USING ZnO/$SiO_2$ HETEROGENEOUS CATALYST

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## ABSTRACT
***The synthesis of fatty acid ethyl ester from chicken fat waste using ZnO/$SiO_2$ heterogeneous catalyst was carried out using two-step procedures of acid pretreatment by esterification and transesterification of the pretreated oil. The first step reduces the high free fatty acid in the oil to an acceptable level for transesterification using concentrated sulphuric acid at a temperature of 60 $^oC$ during 1 h of reaction time. The second step was transesterification of the preheated oil using silica promoted zinc oxide as heterogeneous catalyst. A three level, four factorial box behnken design (BBD) of response surface methodology was used whereby the biodiesel yield obtained in the range of 56–88%and a second order quadratic polynomial regression model that established the relationship between biodiesel yield and the process variables was developed. The quality of the produced biodiesel was determined by investigating its physicochemical properties which results are obtained for specific gravity at 15$^oC$ to be 0.889 g/$cm^3$, Flash point was 175$^oC$, Cloud Point was -3$^oC$, Pour point was -8$^oC$, Cetane number was 57.72 and Kinematic Viscosity at 40$^oC$ was 7.95$mm^2/s$. The results obtained are consistent and in line with ASTM standard for biodiesel; hence, chicken fat as non-edible oil can be a good renewable feedstock for biodiesel production for a sustainable green environment.***

***Keywords:*** *Chicken fat; Zn/$SiO_2$ heterogeneous catalyst, Transesterification, Ethyl ester*

## 1. INTRODUCTION
The gradual depletion of fossil fuels reserves, increasing prices of fossil derived fuels and the rise in environmental hazard associated with the use of fossil fuels has stimulated the urge to search for a more economic, renewable and eco-friendly alternatives. Biofuel has been perceived as a more suitable alternative to fossil derived fuel. Examples of biofuels include biogas, bioethanol, biomethanol and biodiesel [1].

Biodiesel or methyl ester is produced by transesterification reaction of animal fat or plant oils and alcohol [2]. Transesterification is a chemical process of reacting triglycerides with alcohol in the presence of a catalyst Several catalysts are used to obtain high yield of biodiesel [3]. Biodiesel is biodegradable, non-toxic, simple to use, eco-friendly, sulphur free, reduce exhaust emission and higher lubricity whereby it can used in diesel engines without modification [2]. The fuel is receiving attention globally as a possible replacement for petroleum diesel [4]. Most commercial processes for biodiesel in developed countries use edible oils as its raw material. In Europe and America (USA), edible oil such as soya bean oil and rapeseed oil are the common feedstock of biodiesel production [5]. According to Bugaje and Mohammed [1] using edible vegetable oil for the production of biodiesel may not be economical. This is because there is acute shortage of edible oil in most developing countries [6]. Therefore animal fat or oils, waste oil and non-edible oil are proposed as viable option for biodiesel production [7].

The free fatty acids (FFAs) and water content present in these feedstock limits their use in biodiesel production as soap is formed when homogeneous catalysts are used [8]. However, homogenous acid catalysts are known to tolerate high FFA and water content, but their use are not encouraging as equipment used in production are easily corroded. This problem can be surmounted by using solid heterogeneous catalysts on the account of their non-

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toxicity, non-corrosiveness and ease of separation for reuse [9-10].

Transesterification of triglycerides is the most common way to produce biodiesel of edible or non-edible types of oils using alcohol, in the presence of an acid or a base with a catalyst [11]. It is the displacement of alcohol from an ester by another alcohol to form Fatty Acid Methyl Ester (FAME) or Fatty Acid Ethyl Ester (FAEE) in a case where methanol or ethanol is used as alcohols, respectively [12]. Although, methanol is mostly used as alcohol for the transesterification of vegetable oils; however, ethanol was employed in the present study to make the biodiesel production process completely environmentally benign. Biodiesel Production is a bulk process; the general scheme of the transesterification reaction is presented in Equation 1, where R is a mixture of various fatty acid chains.

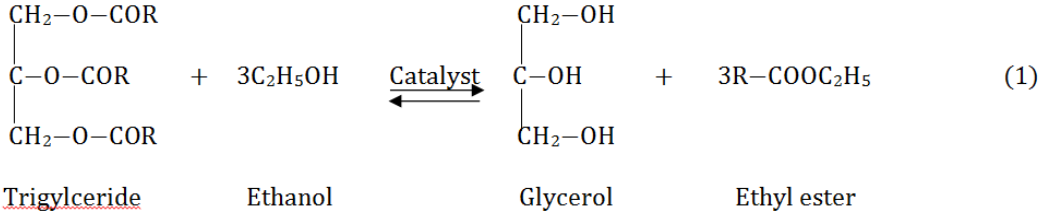
Generally, transesterification is a reversible reaction, although in the production of biodiesel, the backward reaction does not occur or is negligible because the glycerol formed is not miscible with the product, leading to a two-phase system. Nevertheless, an excess of alcohol is usually employed to force reaction towards the right side. The stoichiometry of reaction is a 3:1 molar ratio of alcohol to oil, to produce 3 moles of biodiesel and 1 mole of glycerol. Though, in practice it is usually increased from 6:1 to 1000:1 to favor the formation of products and increase its performance [13]. Bournay *et al.* [14] reported that heterogeneous catalysts (mixture of zinc and aluminium) have been used for industrial scale biodiesel production. Solid acid or basic catalyst developments for transesterification have been a pertinent issue addressed by the scientific community. This has aroused a search for various catalyst types for biodiesel production. However, heterogeneous catalysts have not been exploited at industrial level yet in the transesterification of vegetable oils and chicken fat [13].

Generally, catalysts that are to be used for synthesis of biodiesel should be selective, specific, and result in esterification/transesterification with high conversion and yield of biodiesel. A solid acid catalyst should possess high stability, numerous strong acid sites, large pores, a hydrophobic surface providing a favorable condition for reaction, and should also be economically viable [15]. Developed solid acid catalysts are cation exchange resin (that is, Amberlyst-15 and NR50), mineral salts such as (ferric sulfate, zirconium sulfate, alum phosphate and zirconium tungsten), supported solid acid and heteropolyacid catalysts [9].

Nigeria is a nation that struggles with increasing cost of petroleum products such as diesel, kerosene and gasoline fuels. Inconsistency in the supply of the products is due largely to theft and vandalization of the facilities. This negative situation further escalated with the reduction of fuel subsidy in January 2012. To check this trend, the Nigerian government has earmarked up to a billion dollars for the purpose of implementing policies and programmes to curb crude bunkering, vandalism of the gas facilities and also re-position Nigeria's major energy sources from fossil fuel to renewable alternative energy sources [16].

Besides using inexpensive feedstock for biodiesel commercialization, optimization of low cost feedstock is necessary [17]. In this research, a three level, four factorial Box-Behnken design (BBD) of response surface methodology was used to investigate the transesterification of ethyl ester. This experimental design was chosen for its fewer treatment combinations and requires fewer numbers of experimental runs than a central composite design (CCD) in cases involving three or four factors. In addition, CCD has widely been used compared to the Box-Behnken design. However, the design is a spherical and revolving design, it has been applied in optimization of media, extraction of natural active substances and other uses because of its reasonable design and excellent outcomes [18].

Chicken fat (CF) is a low cost feedstock extracted from chicken wastes such as chicken feathers, trims, offal and blood usually after rendering processes which can be used for the production of biodiesel comparable to high-grade vegetable oils [7]. Animal fats are attractive feedstock for biodiesel because their cost is substantially lower than the cost of vegetable oil.



This is partly because the market for animal fat is much more limited than the market for vegetable oil. Also, much of the animal fat produced is not considered edible by humans. In addition, the price of virgin vegetable oil is about two times more than that of animal fat, although the drawbacks of using animal fat as a raw material for biodiesel production is its physical properties which could be eliminated by adding necessary amount of alcohol and catalyst. Thus, the price of feedstock can be reduced about 50% with using low grade animal fat. Methanol, ethanol, propanol or butanol are examples of alcohols which can be used for transesterification and the monoesters are named methyl esters, ethyl esters, propyl esters or butyl esters, respectively [19].

## 2. MATERIALS AND METHODS

### 2.1 Materials

Chicken fat was bought from Chicken Centre at Kure Market, Minna, Nigeria. Ethanol (purity of 99/100%), Sulphuric acid (98% purity) phenolphthalein indicator, Di-ethyl ether and distilled water were procured for use in the experiment without any further purification. Silica promoted zinc oxide ($ZnO/SiO_2$) catalyst previously synthesized and characterized was used for the transesterification [20].

### 2.2 Pretreatment by Acid Esterification

Chicken fat oil was prepared by heating the chicken fat at 120 $^oC$ for 1 h. This was done to remove moisture and volatiles after which it was filtered. A solution of concentrated sulfuric acid $H_2SO_4$ (2% based on the oil weight) in ethanol (50% volume per volume) was heated to 60$^oC$ which was then added to the melted oil in a conical flask. The mixture was well stirred, placed into the water bath and shaker for heating and then agitated simultaneously at 60$^oC$ for 1 h. The esterified chicken fat was removed from the water bath shaker and decanted into a 500 mL separating funnel and allowed to separate for 12 h. Separation of the esterified oil from ethanol and sulfuric acid was visible, the esterified oil was at the bottom of the separating funnel while the ethanol and sulfuric acid suspended at the upper layer. The oil was collected into a 500 mL beaker and dried in the oven at 100$^oC$ for 1 h to further remove the remaining ethanol contained in the oil. The ethanol and $H_2SO_4$ at the top layer of the separating funnel can be reused and recycled for further use.

Ethanol to oil ratio of 6:1 by weight of oil was used to investigate the influence on the acid value of chicken fat oil. The choice of ethanol to oil molar ratios of 6:1, 2% of sulfuric acid based on weight of oil and 50% volume per volume of ethanol was based on previous studies [21-22].

### 2.3 Transesterification Reaction Procedure

The pretreated oil from chicken fat was measured and poured into a 150 mL conical flask and heated to 60$^oC$ using a water bath. A solution of silica promoted zinc oxide ($ZnO/SiO_2$) catalyst with ethanol was prepared by measuring a known percentage of catalyst ($ZnO/SiO_2$) pellet and calculated volume of ethanol based on each experimental run. The solution was properly stirred although the catalyst was insoluble. The ($ZnO/SiO_2$) and ethanol was poured into the warm chicken fat oil which was then fitted into the water bath shaker to rotate at 150 revolution per minute (rpm). The reaction temperature was increased to that of each experimental run, ($ZnO/SiO_2$) catalyst was measured based on the percentage of each experimental run, and the reaction time was maintained for each experimental run. After the shaking process was completed, the mixture was left to settle for 24 h in a separating funnel.

The produced biodiesel which is in the upper layer was decanted into a separate beaker while the lower layer which comprise of glycerol and catalyst was collected from the bottom of the funnel. In order to prevent ethanol leakage, closed vacuum condition was used by closing the conical flask that contains the reactants with a cork. The quantity of biodiesel (ethyl ester) collected was filtered, washed and dried by subjecting it to heating at 110°C to remove excess alcohol and water. The obtained ethyl ester was weighed and characterized in the laboratory. Plate I shows the biodiesel-glycerol-silicate separation in the separating funnel. The procedure above was repeated for different experimental runs. Production yield (%) was determined according to equation (2):

$$Production\ Yield\ (\%) = \frac{weight\ of\ ethyl\ ester\ produced}{weight\ of\ oil\ used} \times 100 \qquad (2)$$

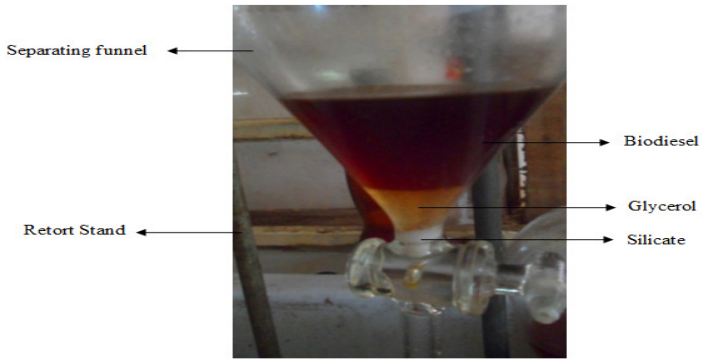



*Plate I: Biodiesel – glycerol - silicate separation*

### 2.4 Design of Experiment

In this research, a three level, four factorial box-behnken design (BBD) of response surface methodology was used to investigate the transesterification of ethyl ester and determine the optimal values of process parameters such as reaction time (A), ethanol to oil molar ratio (B), temperature (C) and catalyst loading (D).

This experimental design was chosen for its fewer treatment combinations and requires fewer numbers of experimental runs than a central composite design (CCD) in cases involving three or four factors. In addition, CCD has widely been used compared to the box-behnken design. Selection of the values for the high and low to each corresponding factor were based on results obtained from preliminary experiments and review of relevant literatures [23]. However, Box-Behnken design is a spherical and revolving design, it has been applied in optimization of media, extraction of natural active substances and other uses because of its reasonable design and excellent outcomes [18].The parameters considered for the reaction were varied according to the experimental design using the response surface methodology (RSM) provided by using Design-Expert software version 6.0.6 (Stat-Ease Inc., USA) as presented in Table 1. The table shows the real and coded independent variables of factors and corresponding levels for the response surface design.

*Table 1: Real and coded independent variables of factors and corresponding levels for the response surface design*

| Factors | Factor codes | Units | Low(-1) | Central (0) | High(+1) |
|---|---|---|---|---|---|
| Reaction time | A | H | 1 | 3 | 5 |
| Ethanol/Oil ratio | B | - | 3:1 | 6:1 | 9:1 |
| Temperature | C | $^oC$ | 60 | 80 | 100 |
| Catalyst loading | D | wt% of oil | 0.50 | 1.25 | 2.00 |

*Table 2: Physiochemical Properties of Chicken Fat*

| Parameters | Chicken Fat | ASTM D445 Standard |
|---|---|---|
| Density at 15°C ($g/m^3$) | 0.919 | 0.932 |
| Iodine value (g/100g) | 42.58 | 79.00 |
| Saponification value (mg/KOH.g) | 103.785 | 191 |
| Specific gravity ($g/m^3$) | 0.912 | 0.930 |
| Kinematic viscosity at 40°C ($mm^2/s$) | 21.50 | 59.20 |
| Acid value (mg.KOH/g) | 5.61 | 26.89 |
| Free fatty acid (%) | 2.805 | |
| Peroxide value (Mmol/kg) | 17.00 | 4.00 |

## 3. RESULTS AND DISCUSSION

### 3.1 Characterization of Chicken Fat Oil (CFO)

Chicken fat oil was used without undergoing any further refining for use as a biodiesel feedstock. Its properties were established to ascertain suitability for biodiesel production and to determine a suitable production process for this feedstock. The physiochemical properties as determined by standard methods of chicken fat prior to the production of biodiesel are presented in Table 2.

The density of the chicken fat oil of 0.919 $g/cm^3$ is consistent with previous results. Bakir and Abdelrahman [24] reported 0.922 $g/cm^3$ for fried chicken oil and Ertan and Mustafa [7] reported a value of 0.932 $g/cm^3$ for chicken fat. Iodine value and saponification value were 42.58 g/100g and 103.785 mg/KOH.g respectively. Iodine value falls in range of the ASTM D445 standard. Specific gravity and kinematic viscosity at 40°C were found to be 0.912 $g/m^3$ and 21.5 $mm^2/s$. The viscosities of pure animal fat are usually high and slightly out of the specification, this could be due to impurities and water content present in the oil. The acid value was discovered to be 5.61 mgKOH/g. An acid value below 1 mgKOH/g is required for efficient biodiesel production from plant oils [25]. This is because acid value above 1 mgKOH/g result to huge catalyst consumption rate required to offset the amount of the

free fatty acids contained in the oil. The high free fatty acid value could arise due to impurities or water present in the oil. The peroxide value of the chicken fat oil was found to be 17 Mmol/kg. These results indicate that CFO contains higher unsaturated fatty acid contents compared to other animal fats like lards and beef tallow [24].

### 3.2 Optimization of the Process and Statistical Analysis

The four main parameters reaction time, ethanol to oil molar ratio, temperature and catalyst loading studied were subjected to response analysis to assess the relationship between them. The results of experimental yield for FAEE showing the interaction between the parameters is presented in Table 3.

*Table 3: Experimental Design results from The Box-Behnken RSM design for optimizing chicken fat*

| Runs | A(h) | B (nil) | C (°C) | D (wt%) | Yield (%) Experimental |
|---|---|---|---|---|---|
| 1 | 3 | 6:1 | 80 | 1.25 | 88 |
| 2 | 5 | 6:1 | 80 | 2.00 | 81 |
| 3 | 5 | 6:1 | 60 | 1.25 | 72 |
| 4 | 3 | 9:1 | 60 | 1.25 | 76 |
| 5 | 3 | 6:1 | 100 | 0.50 | 82 |
| 6 | 1 | 6:1 | 100 | 1.25 | 80 |
| 7 | 1 | 6:1 | 60 | 1.25 | 56 |
| 8 | 3 | 3:1 | 80 | 2.00 | 86 |
| 9 | 5 | 6:1 | 100 | 1.25 | 84 |
| 10 | 1 | 3:1 | 80 | 1.25 | 80 |
| 11 | 1 | 6:1 | 80 | 0.50 | 82 |
| 12 | 3 | 6:1 | 80 | 1.25 | 88 |
| 13 | 3 | 9:1 | 100 | 1.25 | 82 |
| 14 | 5 | 3:1 | 80 | 1.25 | 84 |
| 15 | 3 | 3:1 | 60 | 1.25 | 60 |
| 16 | 1 | 9:1 | 80 | 1.25 | 80 |
| 17 | 1 | 6:1 | 80 | 2.00 | 84 |
| 18 | 3 | 6:1 | 80 | 1.25 | 88 |
| 19 | 3 | 6:1 | 80 | 1.25 | 88 |
| 20 | 5 | 6:1 | 80 | 1.25 | 86 |
| 21 | 3 | 3:1 | 100 | 2.00 | 86 |
| 22 | 3 | 6:1 | 100 | 1.25 | 84 |
| 23 | 5 | 6:1 | 80 | 0.50 | 86 |
| 24 | 3 | 6:1 | 60 | 0.50 | 76 |
| 25 | 3 | 3:1 | 80 | 0.50 | 86 |
| 26 | 3 | 9:1 | 80 | 0.50 | 88 |
| 27 | 3 | 9:1 | 80 | 2.00 | 86 |
| 28 | 3 | 6:1 | 80 | 1.25 | 84 |
| 29 | 3 | 6:1 | 60 | 2.00 | 72 |

A quadratic model of the experimental data was generated for regression analysis of experimental data and 3D surface plot. Thus, the final equations in terms of coded factors based on box-behnken design translated into the second-order polynomial model equation and final equations in terms of actual factors aredetailed in Equations3 and 4, respectively [23]. Table 4 presents the quadratic response surface model from analysis of variance (ANOVA)

$$\begin{aligned} FAEE\ content = {} & 86.80 + 2.58A + 1.50B + 7.17C \\ & - 0.42D - 3.98A^2 - 1.11B^2 - 9.61C^2 \\ & + 1.02D^2 + 0.50AB - 3.00AC \\ & - 1.75AD - 4.50BC - 0.50BD \\ & + 2.00CD \end{aligned} \quad (3)$$

Where A is the reaction time (hours), B is the ethanol/oil ratio, C is the temperature (°C) and D is the catalyst loading (wt%).

Final equations in terms of actual factors:

$$\begin{aligned} FAEE\ Content = {} & -157.561 + 14.225 * reation\ time \\ & + 8.001 * ethanol\ oil\ ratio + 4.71 \\ & * temperature - 10.907 \\ & * catalyst\ loading - 0.996 \\ & * reaction\ time^2 - 0.123 \\ & * ethanol\ oil\ ratio^2 - 0.024 \\ & * temperature^2 + 1.807 \\ & * catalyst\ loading^2 + 0.083 \\ & * reaction\ time * ethanol\ oil\ ratio \\ & - 0.075 * reaction\ time \\ & * temperature - 1.167 \\ & * reaction\ time * catalyst\ loading \\ & - 0.075 * ethanol\ oil\ ratio \\ & * temperature - 0.222 \\ & * ethanol\ oil\ ratio \\ & * catalyst\ loading + 0.133 \\ & * temperature \\ & * catalyst\ loading \end{aligned} \quad (4)$$

The analysis of variance (ANOVA), statistical significance of the model equation was evaluated by the F-value, which showed that the regression is statistically significant at95% confidence level. The model F-value of 12.27 for biodiesel production implied that the model was statistically significant as presented in Table4. There is only a 0.01% chance that a "Model F-Value" could occur due to noise because it is large.Values of "Prob > F" less than 0.0500 indicate model terms are significant (Table 4). In this case reaction time (A), temperature (C), square of reaction time($A^2$). Square of temperature ($C^2$), ethanol/oil ratio * temperature (BC) are significant model terms. The "lack of fit F-value" of 3.47 implied that the lack of fit is not significant relative to pure error. Table 5 presents coefficient of the model for biodiesel yield from chicken fat oil (CFO).

### 3.3 Effects of Parameters on the Transesterification of Chicken Fat Oil

The effects of the parameters on the biodiesel yield from chicken fat oil using response surface plots of the second order model are discussed.

#### *3.3.1 Effects of Reaction Time*

The effect of reaction time on the yield of biodiesel represented by the response surface plot in Figure 1 revealed that as the reaction time increases from 1 to 3 h, high FAEE content was obtained and the yield now starts to decrease thereafter as the reaction time further increases from 3 to 5 h. This might be due to the effect of water produced during the esterification of FFA, which prevented further reaction. Zhang *et al.,* [6] reported that FFA in *Zanthoxylumbungeanum*seed oil (ZSO) easily reacted with methanol in the first 1 h and the rate of esterification slowed from 1 to 2 h. They also observed that increasing reaction time beyond 2 h did not have much effect on reducing the acid value.

#### *3.3.2 Effects of Ethanol to*

The effect of ethanol to oil ratio on the yield of FAEE investigated revealed that there was a proportionate increase in yield as the ratio was increased as shown in Figure 2. However, above the ratio of 6:1 the conversion only slightly increased. Thus it is evident that to obtain higher conversion, excess of ethanol was effective to a certain degree. This is in agreement with the work of Srilatha *et al.*[27]. They observed the effect of methanol to oil molar ratio on the ester yield to be highly significant using ZnO/Na-Y catalyst at a reaction temperature of 65$^o$C. They also reported that an increase in the ratio of methanol to oil increases the ester yield as transesterification generally requires large excess of methanol to shift the equilibrium towards products. However, when the mole ratio exceeded the optimum ratio, the increase in yield was marginal.

#### *3.3.3 Effects of Temperature*

Temperature greatly influences the heterogeneous transesterification reaction. High conversion was obtained at high temperatures (80 to 100$^o$C) and at a catalyst concentration of 1.25 wt% in 3 has observed from the 3D plot in Figure 3.

*Table 4: Quadratic Response Surface Model from Analysis of Variance (ANOVA)*

| Source | Sum of squares | Degree of freedom | Mean square | F-value | Prob>F |
|---|---|---|---|---|---|
| Model | 1575.46 | 14 | 112.54 | 12.72 | < 0.0001 |
| Residual | 123.88 | 14 | 8.85 | | |
| Lack of fit | 111.08 | 10 | 11.11 | 3.47 | 0.1209 |
| Pure error | 12.80 | 4 | 3.20 | | |
| Correction Total | 1699.45 | 28 | | | |

Adj R-Squared = 0.8542. R-Squared = 0.9271

*Table 5: Coefficient of the Model for Biodiesel Yield from CFO*

| Factor | Coefficient Estimate | Degrees of Freedom, (DF) | Standard Error | 95%CL Low | 95% CL High | Variance Inflation factor(VIF) |
|---|---|---|---|---|---|---|
| Intercept | 86.80 | 1 | 1.33 | 83.95 | 89.65 | |
| A-reaction time | 2.58 | 1 | 0.86 | 0.74 | 4.43 | 1.00 |
| B-ethanol/oil ratio | 1.50 | 1 | 0.86 | -0.34 | 3.34 | 1.00 |
| C-Temperature | 7.17 | 1 | 0.86 | 5.32 | 9.01 | 1.00 |
| D-catalyst loading | -0.42 | 1 | 0.86 | -2.26 | 1.43 | 1.00 |
| $A^2$ | -3.98 | 1 | 1.17 | -6.49 | -1.48 | 1.08 |
| $B^2$ | -1.11 | 1 | 1.17 | -3.61 | 1.40 | 1.08 |
| $C^2$ | -9.61 | 1 | 1.17 | -12.11 | -7.10 | 1.08 |
| $D^2$ | 1.02 | 1 | 1.17 | -1.49 | 3.52 | 1.08 |
| AB | 0.50 | 1 | 1.49 | -2.69 | 3.69 | 1.00 |
| AC | -3.00 | 1 | 1.49 | -6.19 | 0.19 | 1.00 |
| AD | -1.75 | 1 | 1.49 | -4.94 | 1.44 | 1.00 |
| BC | -4.50 | 1 | 1.49 | -7.69 | -1.31 | 1.00 |
| BD | -0.50 | 1 | 1.49 | -3.69 | 2.69 | 1.00 |
| CD | 2.00 | 1 | 1.49 | -1.19 | 5.19 | 1.00 |

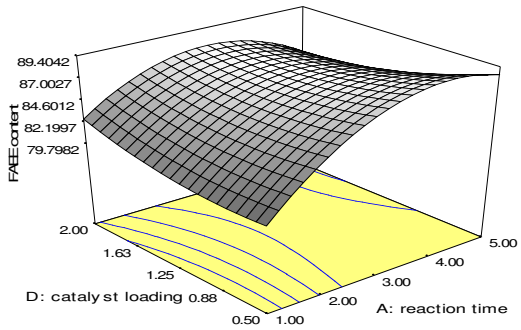



*Figure 1: Profile of response surface of biodiesel yield with catalyst loading and reaction time*

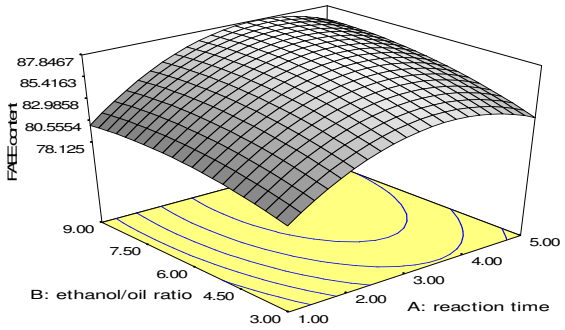



*Figure 2: Profile of response surface of biodiesel yield with ethanol/oil molar ratio and reaction time*

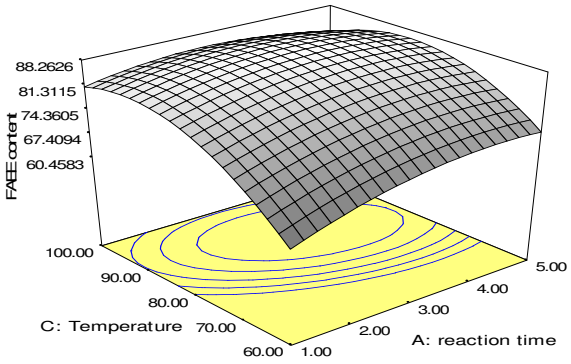



*Figure 3: Profile of response surface of biodiesel yield with Temperature and Reaction time*

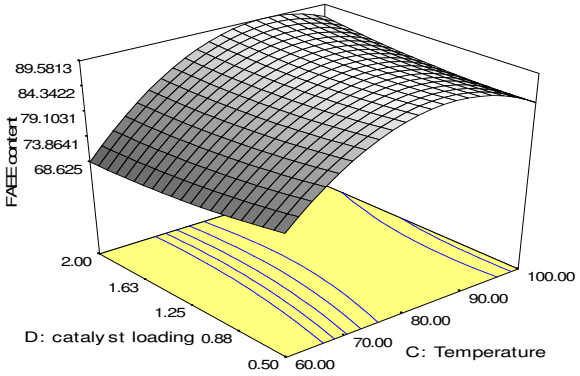



*Figure 4: Profile of response surface of biodiesel yield with catalyst loading and Temperature*

The optimum FAEE content was almost reached as ethyl esters yield increases rapidly with increase in temperature from 60 to 100°C as the yield increases from 68.62% to 89.58% as predicted. Dias *et al.*[28] reported that under similar operational conditions in the transesterification of pork lard and soya bean oil, reaction progressed much faster at 60°C than at 50°C and 40°C. At 60°C, after 4 h, the maximum ethyl ester content was almost reached, being 92.4 wt% compared to 95.8 wt% after 8 h. A great difference was observed at 4 h of reaction using the different temperatures. At 40°C, the maximum methyl ester content was obtained at the maximum reaction time (8 h), being 80.5 wt%. Although the temperature variations may be quite different in the present work done, it still compares favorably with the result, since chicken fat was used as the base feedstock.

Srilatha *et al.* [27] observed that increase in reaction temperature accelerates the reaction towards the ester formation in biodiesel production from used cooking oil by two-step heterogeneous catalysed process using methanol as solvent and ZnO/Na-Y zeolite as catalyst.

### *3.3.4 Effects of catalyst loading*

The effect of catalyst loading on the yield of biodiesel is shown in Figure 4, alongside temperature. The catalyst loading is one of the most important parameters that influence the yield of FAEE. The $ZnO/SiO_2$ heterogeneous catalyst was varied in the range of (0.5 – 2.0) wt% based on the initial mass of oil, keeping the reaction temperature at 80°C and taking the ethanol/oil molar ratio as 6:1. The optimum ethyl ester yield was obtained to be 88% which indicates high yield with increase in catalyst loading up to 1.25 wt%. There was no considerable variation in activity beyond 1.25 wt% of catalyst. This is in agreement with the work of Zhang [29] who reported that increasing catalyst loading beyond 5.27% based on the mass of oil did not have much effect on biodiesel yield.

### *3.3.5 Characterization of Chicken Fat Ethyl Ester (CFEE)*

The results for the characterization of chicken fat ethyl ester (CFEE) produced are shown in Table 6. The flash point is an important property used to determine the flammability of a fuel. It is the temperature at which the fuel will ignite when exposed to a flame. Compared to petro-diesel the flash point of biodiesel is

higher, this makes biodiesel safer for use in the transportation industries. The ethyl ester produced from chicken fat had a flash point of 175°C. This is slightly out of range of ASTM standards. Mata *et al*., [30] reported that the flash point.

*Table 6: Fuel properties of biodiesel produced compared with the ASTM standard.*

| Fuel property | Experimental Values | ASTM Standard |
|---|---|---|
| Flash point (°C) | 175 | 140 – 170 |
| Cloud point (°C) | -3 | -3 – 12 |
| Iodine value (g/100g) | 58.80 | |
| Pour point (°C) | -8 | -15 – 10 |
| Saponification Value (mg/KOH.g) | 266.47 | 140 – 180 |
| Cetane number | 57.72 | 48 – 65 |
| Specific gravity ($g/m^3$) | 0.889 | 0.88 |
| Kinematic viscosity ($mm^2/s$) | 7.95 | 1.9 – 6.0 |

from chicken fat oil (CFO) was of 171°C, which is slightly out of the ASTM specification used in this study, the flash point obtained in this study is high which showed that the CFEE is safe from handling and storage point.

The temperature at which wax first becomes visible to the naked eye when the fuel is cooled is called the cloud point. At temperatures below the cloud point, larger crystals fuse together and form agglomerations that eventually become extensive enough to prevent pouring of the fluid and consequently affecting the performance of fuel lines, fuel pumps and injectors. The molecular structure significantly influences the low-temperature behavior of biodiesel. The cloud point of CFEE was obtained to be -3°C which is in conformity with the ASTM standard although several literatures reported no limit for cloud point specification. Aransiola *et al*. [31] reported a cloud point of 6 °C from the production of biodiesel from crude neem oil feedstock.

The iodine value (IV) was gotten to be 58.80. Bakir and Abdelrahman [24] reported from the production of biodiesel from chicken frying oil that iodine values for the produced fuels ranged from 57 to 61 $mgI_2/100$ oil which compares favourably with the iodine value obtained in this study. Iodine value is the property that can be used for measuring the unsaturation degree of fuel.The process of transesterification reduces the iodine value to a small extent.

Pour point is the temperature at which crystal agglomeration is large enough to prevent free pouring of fluid [2]. The pour point was found to be −8 °C which is in line with the ASTM standards. Saponification value (SV) is the measure of soap formation in the ethyl ester formed. SV for the produced CFEE was obtained to be 266.47 mg KOH/g oil while SV for the feedstock CFO was 103.785 mg KOH/g oil, this indicates that the ester has lower molecular weight than the parent oil, since the higher the SV, the lower is the molecular weight. Also the low value of the parent oil indicates that the oil is not good for soap formation, this can be compared with the work of Bakirand Abdelrahman [24] who reported SV for the feedstock to be 191 mg KOH/g oil, while those for the produced fuel ranged from 192 to 195 mg KOH/g oil from the production of biodiesel from chicken frying oil.

The cetane number of the CFEE was 57.72. Cetane number assures good control of the combustion increasing performance and improving cold start which results into less exhaust. The cetane number measures how immediately ignition occurs upon injection of fuel into the combustion chamber and smoothness of combustion [2].The Specific gravity (SG) at 15°C of CFEE was found to be 0.889 $g/cm^3$, the result agrees with ASTM D6751 standard of 0.88 $g/cm^3$ and also European Standard EN14214 range 0.860-0.900 $g/cm^3$ for biodiesel when compared. The SG of ethyl ester produced decreased with increasing reaction temperature and ethanol to oil molar ratio. This was probably due to a decrease in residual triglycerides. The kinematic viscosity of the CFEE produced in this study was 7.95 $mm^2/s$ at 40 °C. Mata *et al,* [30] obtained a value for kinematic viscosity at 40°C to be 5.85 $mm^2/s$ from chicken fat. With regards to fuel atomization and also fuel distribution, kinematic viscosity is a very important property. High viscosity leads to a higher drag in the injection pump and thus results into higher pressures and injection volumes more especially at low engine operating temperature. Generally, viscosity increases with the number of $CH_2$ moieties in the fatty ester chain (corresponding to larger molecules), and generally decreases with the increasing number of double bonds between carbon atoms in their molecules [30].

## 4. CONCLUSION

It can be concluded that chicken fat ethyl ester (CFEE) was produced using silica promoted zinc oxide catalyst. The processes of optimization and transesterification of chicken fat oil was made feasible by four factors of box-behnken design (BBD) using RSM in 29 experimental runs. The achievement of

heterogeneous catalyst of the type silica supported on zinc oxide ($ZnO/SiO_2$) was unveiled by this research which gave 89.13% optimum yield as predicted alongside temperature at 100°C, reaction time at 3 h and catalyst concentration of 1.25 wt% under atmospheric pressure. Results from the statistical analysis of variance (ANOVA) showed that reaction time, catalyst concentration, temperature and ethanol to oil molar ratio has positive effect on the yield of biodiesel, however temperature has higher effect than the catalyst concentration. The results obtained are consistent and are in line with the appreciable standard, thus makes chicken fat a decent feedstock for the production of biodiesel which can be used to run diesel engine vehicles effectively.

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