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# Optimization of Biodiesel Production from Thevetia Peruviana Seed Oil using Transesterification Method

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***Abstract:*** *Globally there is a rekindled interest to find alternative fuels to fossil fuel because of the issues of pollution and climate change associated with fossil fuels. Therefore, one of the alternatives is plant oil from non-edible sources. So, this study investigated the synthesis and optimization of biodiesel production from a novel non-edible feedstock (Thevetia Peruviana Seed Oil) using NaOH catalyst and methanol through transesterification route. The influence of parameters catalyst loading and alcohol-to-oil molar ratios on the yields of the biodiesel was checked. A mixture method of experimental design was adopted for the study. The results showed an optimal biodiesel yield of 98.87 % comprising of 80.02 % methyl esters and 18.85 % ethyl esters from the experiment with the reaction conditions; concentration of catalyst 0.25%, 9:1 methanol/oil molar ratio, 60 $^0$C reaction temperature, 60 minutes reaction time and 150 rpm agitation speed. This study developed an optimal catalyst concentration, alcohol-to-oil molar ration, reaction temperature, reaction time and agitation speed for synthesizing biodiesel from Thevetia Peruviana Seed Oil and it also identifies the methyl esters %, ethyl esters % and free fatty acid (FFA) % present in the biodiesel. The study showed biodiesel can be produced from Thevetia Peruviana Seed Oil with a very high yield, this is a potential alternative to diesel fuel and can be applied to diesel engines to produce power for use in automobiles, farm machineries and electricity generation.*

***Keywords:*** *Biodiesel, Seed Oil, Methyl esters, Transesterification, Optimization, Mixed Method Design of Experiment*

## 1. INTRODUCTION

Global population expansion and increase in industrialization has heightened the dependence on fossil fuel for energy [1-2]. According to reports, the negative contribution of burning fossil fuel in engines to global warming [3-4]. Therefore, it becomes a matter of necessity to seek for fuels from alternative environmentally friendly sources to support better climate-change conditions [5-7]. One of these sources is from non-edible plants that are capable of providing clean energy that can also be replenish and affordable [4, 8]. Over 350 crops that bear oil have been found and regarded as having the potential to substitute conventional diesel fuel in engines [9-10]. Biodiesel is a renewable alternative fuel derived from plant or animal sources. The advantages of biodiesel over conventional diesel includes: it is renewable, non-toxic, biodegradable, possesses high cetane number, has lower aromatic and sulphur content, gives off reduced emission of unburnt hydrocarbon and carbon monoxide [11-12].The methods of producing biodiesel are: micro-emulsion, transesterification and pyrolysis [13]. Among these methods, the suitable and most popular way is the transesterification method, which can be homogenous or heterogenous catalyzed using acid, base or enzyme [14]. The parameters that affect the yield and quality of biodiesel during transesterification includes:, percentage loading of catalyst, reaction temperature, reaction time, free fatty acids (FFA) content, alcohol-to-oil ratio, presence of water in oil [2].

Mixture design of experiment in the field of biodiesel synthesis are closely relying on response surface methodology, though other techniques of experimental design exist such as artificial neural networks (ANN), Taguchi, response surface method (RSM) and adaptive neuro-fuzzy inference systems (ANFIS), genetic algorithm (GA) [15-16]. Mixture design is a combination of techniques that are mathematical and statistical which are important in the development, alteration and optimization of many processes when the variables to be studied are in some proportions or composition of the whole system, such as in the synthesis of biodiesel [7, 16]. The advantages of mixture design are that; it can handle various constraints; it can handle both continuous and categorical components and it is very effective in handling the interactions between components.

Several researches in the area of optimizing biodiesel production includes: Ahmad et el., optimized the production of biodiesel using flaxseed oil via transesterification. The yield of 99.5 % at 59 $^{o}$C, 0.51 % catalyst, 33 minutes reaction time and 5.9:1 methanol/oil molar ratio was predicted by the model. The experimental verification gave a yield of 98±2 % [2]. Also, Bitire & Jen, optimized biodiesel production from seed oil using KOH catalyst. The predicted yield was 98.18 % and the experimental yield was 98 % at 60 $^{o}$C, 30 minutes and 6:1 alcohol-to-oil molar ratio [4]. Saqib et al., optimized the production of biodiesel from rapeseed oil [7]. The optimal yield was 97.5 % at optimal conditions of 0.30 % catalyst, 55 $^{o}$C, 6.75 alcohol/oil molar ratio and 60 minutes reaction time. Biodiesel production optimization from Manilkara zapota (L.) seed oil was studied by Kumar et al., [12]. The optimal parameters were found to be 50 $^{o}$C, 6:1 methanol-to-oil molar ratio, 90 minutes reaction time and 1 wt% catalyst concentration. 94.83 % was obtained as optimal yield. Biodiesel production optimization from camelina oil was investigated by [17]. The optimal yield was between 95.8-98.4 % and the optimal conditions were 8:1 methanol/oil ratio, 70 minutes reaction time, 50 $^{o}$C, and 1 wt. % catalyst concentration. Gulum & Bilgin, investigated biodiesel production from Safflower oil [13]. The optimal conditions were 8:1 alcohol/oil molar ratio, 0.75 % catalyst concentration and 56 $^{o}$C for methanolysis; 12:1 alcohol/oil molar ratio, 1.00 % catalyst concentration, and 70 $^{o}$C for ethanolysis. Sivaprakasam & Saravanan carried out biodiesel production optimization from Jatropha oil [14]. The optimal conditions were: 90 mL of methanol, 500 mL of vegetable oil, 1.0-hour reaction time and 8 g of KOH. Farobie & Hartulistiyoso investigated production of biodiesel from crude palm oil with various free fatty acid [18]. The results showed a yield of 97.91 % at 40 $^{o}$C, 7:1 methanol-to oil molar ratio. Bouaid et al., investigated free fatty acids effects on the quality of biodiesel [19]. The result showed that yield of methyl esters decreases from 97.2 % to 95 % when the content of free fatty acid of the oil was increased from 0 % to 4 %. Olalere et al., studied the up scalability and Techno-economic perspectives of non-conventional essential oils [20]. Falowo et al., studied the base catalyst derived from ripe and unripen plantain peels for application in the transesterification of waste cooking oil [21]. Dube et al., developed a sustainable grease from Jojoba oil for application in rolling bearings [22]. Khater et al., investigated the improvement of biodiesel from Egyptian Jatropha seeds [23]. None of these studies investigated the optimization of Thevetia peruviana (TP) biodiesel production. This study uses mixture design of experiment to investigate the optimization of biodiesel synthesis from Thevetia peruviana (TP).

This study investigated the optimization of biodiesel yield from *Thevetia peruviana* (TP) using transesterification reaction while varying the reaction parameters that is; methanol/oil molar ratio and catalyst loading. TP seeds were used to produce biodiesel, because it is a non-edible feedstock. Previous works have established that methanol/oil ratio and loading of catalyst influences the yield of biodiesel, hence the choice of these factors to investigate their influence on TP biodiesel yield. Mixture design of experiment was selected for this study because it combines both elements of qualitative and quantitative research.

## 2. MATERIALS AND METHOD

### 2.1 Materials

The major materials used for this study includes Thevetia peruviana seeds, Petro-diesel, Methanol ($CH_3OH$), Potassium hydroxide (KOH) Sodium hydroxide (NaOH), Tetraoxosulphate (iv) acid ($H_2SO_4$), Phenoiphthalein, Propan-2-ol ($C_3H_7OH$). While key equipment used are Expeller Oil Press 6YL-100 - GB/T19007-2000idt ISO 9001:2000 (Input 7.5kW, 28-38 rpm, O/p 150-300kg/h), GC-MS Machine - Gas Chromatograph-Mass Spectrometre (GC-MS), 79-1 Magnetic Stirrer with Heater (230V, 9A), Weighing balance (Min. 5g – Max 6kg), Hot plate (AMSTAT, MS300), Thermometers (Range of 0 to 100 $^{o}$C), Stop clock, Measuring cylinder (Maximum graduated -250, 500 and 1000 millilitres), Beakers (1,2 and 5 litres), 50ml Burettes, 250ml Conical flasks, Test tubes and Corks (9, 15, 20 ml), Retort stand, 500ml Separating funnels, Air tight desiccators and Rolls of Aluminium foil paper (8 Packs).

### 2.2 Methods

The following processes were applied for the study. The simplistic schematic approach for the production of the Thevetia Periviana (TP) biodiesel is presented in Figure 1.

#### 2.2.1 Design of experiment

The factors usually considered for biodiesel production are temperature of the reaction, time, alcohol-to-oil molar ratio, concentration of catalyst and agitation speed or rate. Any design of experiment with 3 levels and above is relatively large and will require huge resources to execute. Thus, the experiments in this study were reduced from full factorial design ($5^5$ = 3125 runs) to a mixed-level fractional factorial design of 2-factors and varied levels for biodiesel production (55-3 = 52 = 25 runs at most) to reduce the complexities to a moderate level. Mixture design of experiment is of interest because it can handle various constraints and can statistically estimate the effects of components and interactions efficiently, this method is preferred to Taguchi method because it cannot handle constraints [24]. Furthermore, some runs were eliminated because of the information gotten from chemical characteristic, which varies from crude oil to crude oil [25]. Thus, carried out experiments were conducted based fatty acids compositions profiling of the raw oils as indicated by GC-MS result of each oil in agreement with reviewed literatures [26].

For the design of experiment in this study; the process factors; reaction temperature, reaction time and agitation speed or intensity were kept constant at 60 $^{o}$C, 60 minutes and 150 rpm respectively. These values were considered because 64.7 $^{o}$C is the boiling temperature of methanol, so as to avoid loss by evaporation. 60 minutes is the half-life oil to esters (Second-order reaction) conversion to reach a significant level [27]. In all experimental runs for the biodiesel production,

40 grammes of the esterified oil was used and the amount of catalyst and alcohol for each of the run are presented accordingly in Table 1. The results were expressed as mean ± standard deviation of three repeated tests.

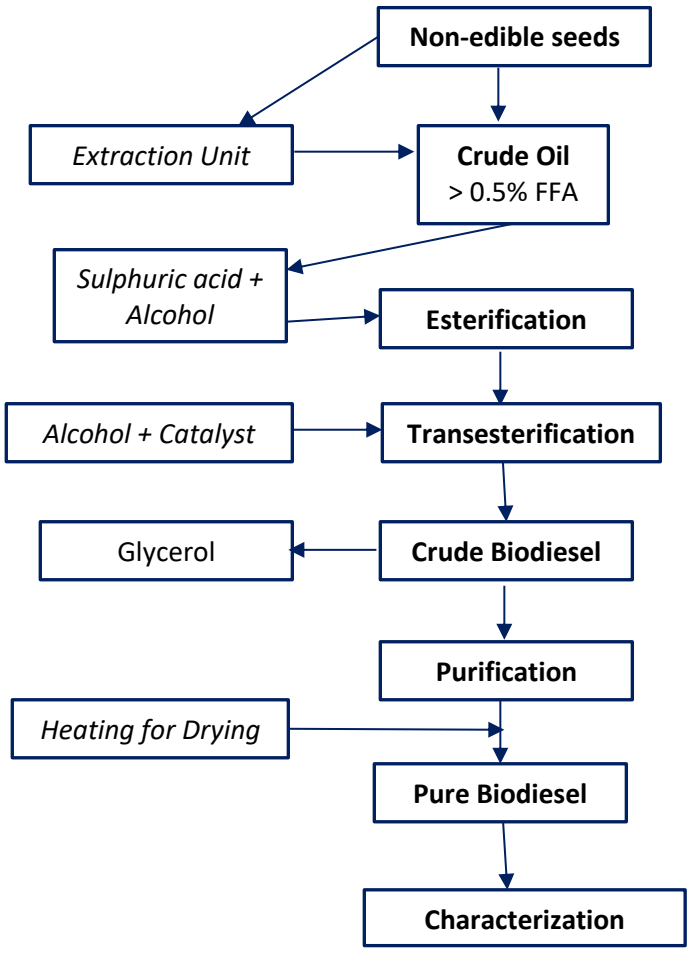


Figure 1: Diagrammatic view of experimental set-up.

Table 1: TP Batch - Experimental design matrix.*

| Run Order | Design Code | Temperature T (°C) | Time t (min) | For Mass of oil at 40g | |
|---|---|---|---|---|---|
| | | | | Mass of alcohol (Molar ratio) (g) | Mass of Catalyst (Catalyst Conc.) (g) |
| **1** | TP1A | 60 | 60 | 8.68 | 0.1 |
| **2** | TP1B | 60 | 60 | 13.03 | 0.1 |
| **3** | TP2A | 60 | 60 | 8.68 | 0.2 |
| **4** | TP2B | 60 | 60 | 13.03 | 0.2 |
| **5** | TP2C | 60 | 60 | 17.37 | 0.2 |
| **6** | TP3C | 60 | 60 | 17.37 | 0.3 |
| **7** | TP5C | 60 | 60 | 17.37 | 0.5 |

*Nomenclature: TP1A Number = Catalyst Conc. %, Alphabet = Molar ratio
Key 1 = 0.25 %, 2 = 0.5 %, 3 = 0.75 %, 4 =1.0 % and 5 =1.25 % catalyst concentration by weight of esterified oils.
While A = 6:1, B = 9:1 and C = 12:1 alcohol to oil molar ratio.

**2.2.2 The gas chromatography-mass spectrometry (GC-MS) analysis**

GCMS-QO2010 Shimadzu Japan was used in this analysis. A fused silica column, filled with Elite-5MS (95 % dimethylpolysiloxane 5 % biphenyl, 30 m × 0.25 mm ID × 250 µm df). Helium as carrier gas flowing constantly at 1 mL/min was used to separate the components. As outlined in **[1]** Injector temperature was set at 250 $^{o}$C for running the

chromatography. When the temperature was 70 $^{o}$C (0 min) then the extract (1 µL) was injected into the instrument, followed by 280 $^{o}$C at the rate of 10 $^{o}$C min$^{-1}$; and 280 $^{o}$C, where it was kept for 5 minutes. The conditions for mass detector were; set at ion source temperature of 200 $^{o}$C, transfer line temperature at 250 $^{o}$C, and ionization mode electron impact of 70 eV, a scan time of 0.5 seconds and scan interval of 0.1 seconds respectively. In this study, compounds were detected in the crude oil (veggie oil in stage 1), biodiesel in stage 13 and their respective quantities with GC-MS spectrometer.

### 2.2.3 Compounds Identification

National Institute of Standard and Technology (NIST) library was used as a reference to compare and interpret results. Compounds were identified according to molecular mass, structure and fragments calculated. The unknown compounds spectrum was compared with that of the NIST library, and the closest match is taken.

## 3. RESULTS AND DISCUSSIONS

### 3.1 GCMS Spectra of Biodiesels

#### 3.1.1 TP 1A biodiesel

The data showing the composition of the spectrum in Figure 2 are shown in Table 2.

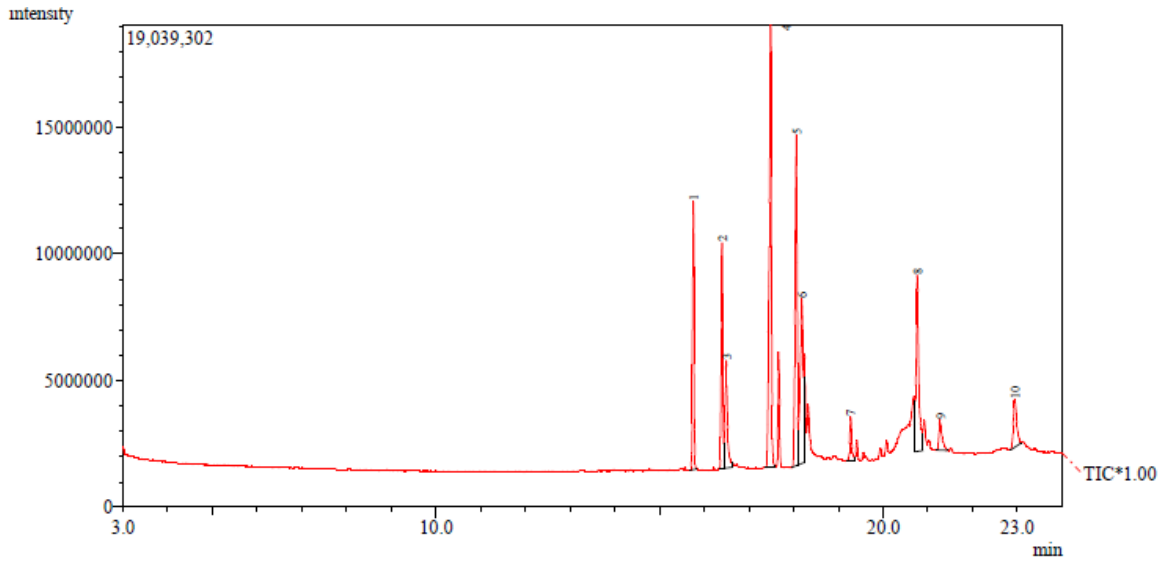


Figure 2: GC-MS spectra of *Thevetia peruviana* biodiesel at 6:1 methanol/oil molar ratio and 0.25 % catalyst concentration

#### 3.1.2 TP 1B biodiesel

The data showing the composition of the spectrum in Figure 3 are presented in Table 2.

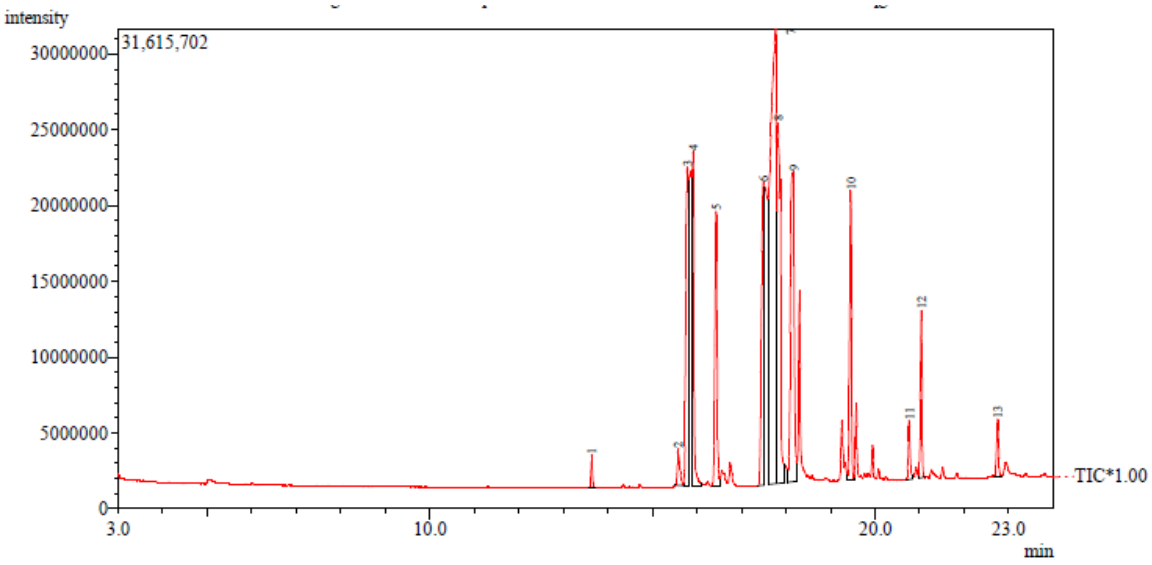


Figure 3: GC-MS spectra of *Thevetia peruviana* biodiesel at 9:1 methanol/oil molar ratio and 0.25 % catalyst concentration

### 3.1.3 TP 2A biodiesel

The data showing the composition of the spectrum in Figure 4 are in Table 2.

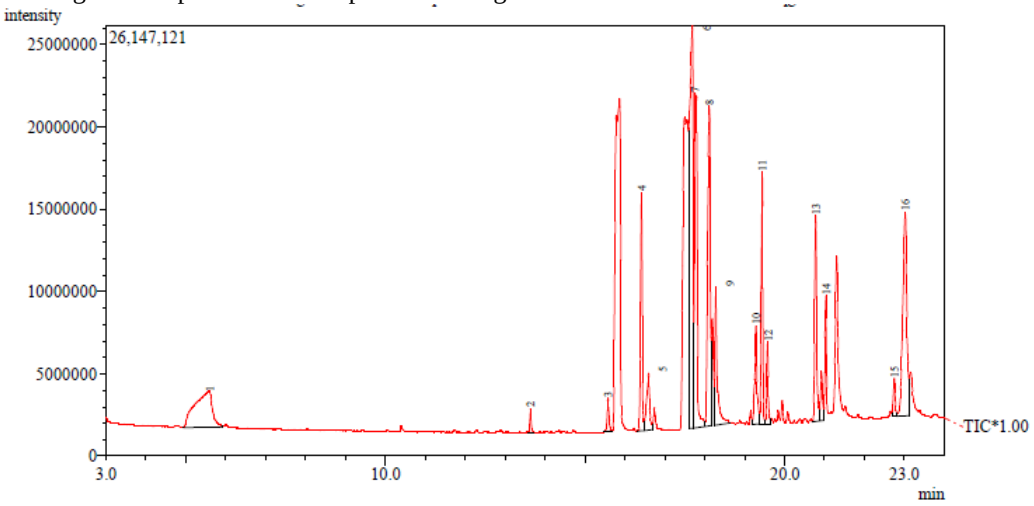


Figure 4: GC-MS spectra of *Thevetia peruviana* biodiesel at 6:1 methanol/oil molar ratio and 0.5 % catalyst concentration

### 3.1.4 TP 2B biodiesel

The data showing the composition of the spectrum in Figure 5 are indicated in Table 2.

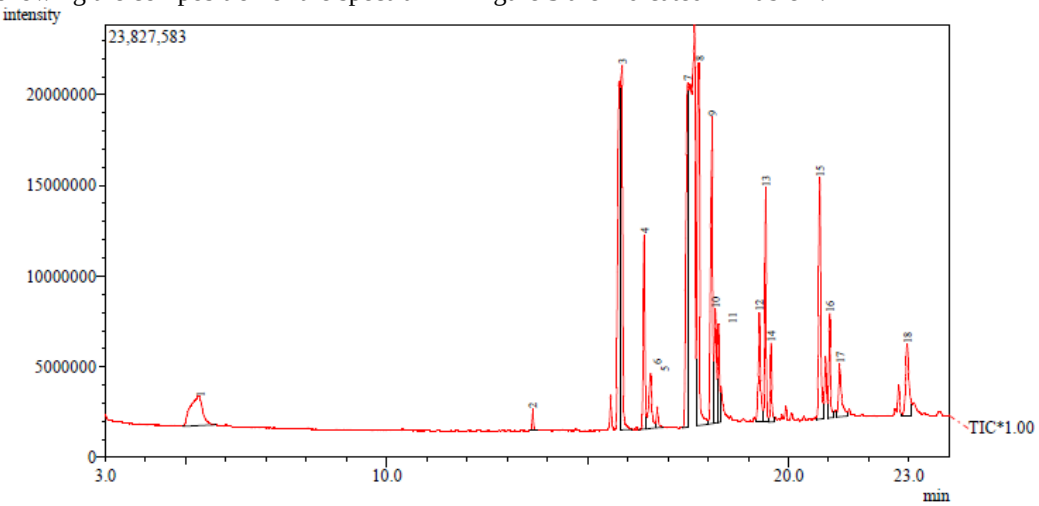


Figure 5: GC-MS spectra of *Thevetia peruviana* biodiesel at 9:1 methanol/oil molar ratio and 0.5 % catalyst concentration

### 3.1.5 TP 2C biodiesel

The data showing the composition of the spectrum in Figure 6 are shown in Table 3

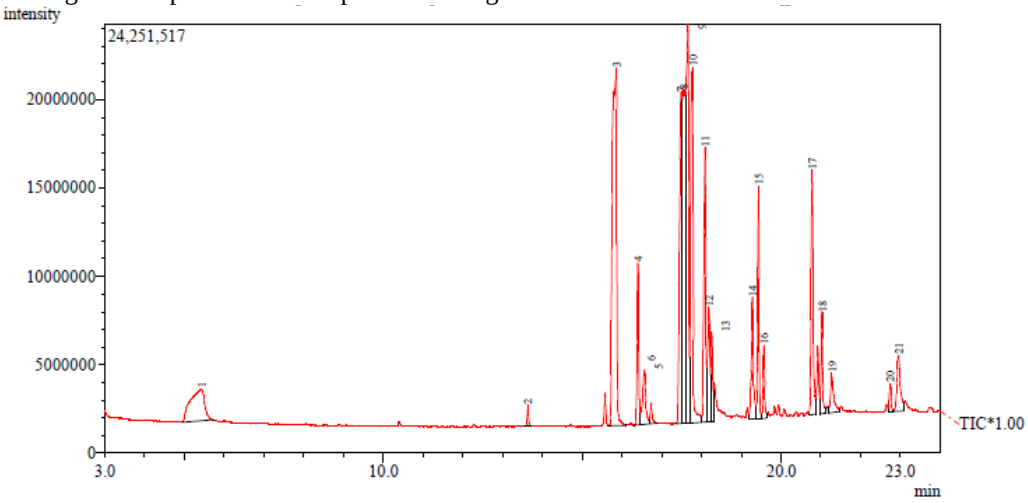


Figure 6: GC-MS spectra of *Thevetia peruviana* biodiesel at 12:1 methanol/oil molar ratio and 0.5 % catalyst concentration

### 3.1.6 TP 3C biodiesel

The data showing the composition of the spectrum in Figure 7 are in Table 3.

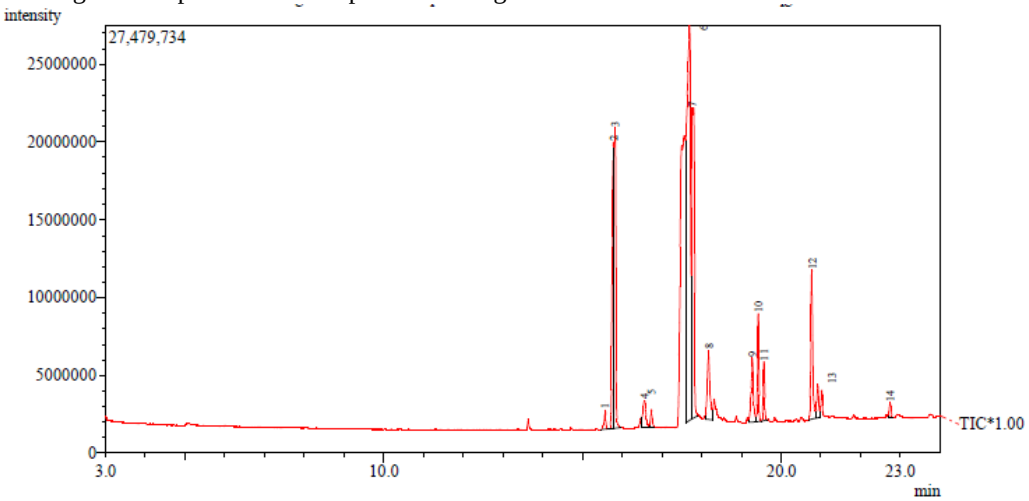


Figure 7: GC-MS spectra of *Thevetia peruviana* biodiesel at 12:1 methanol/oil molar ratio and 0.75 % catalyst concentration

### 3.1.7 TP 5C biodiesel

The data showing the composition of the spectrum in Figure 8 are presented in Table 3.

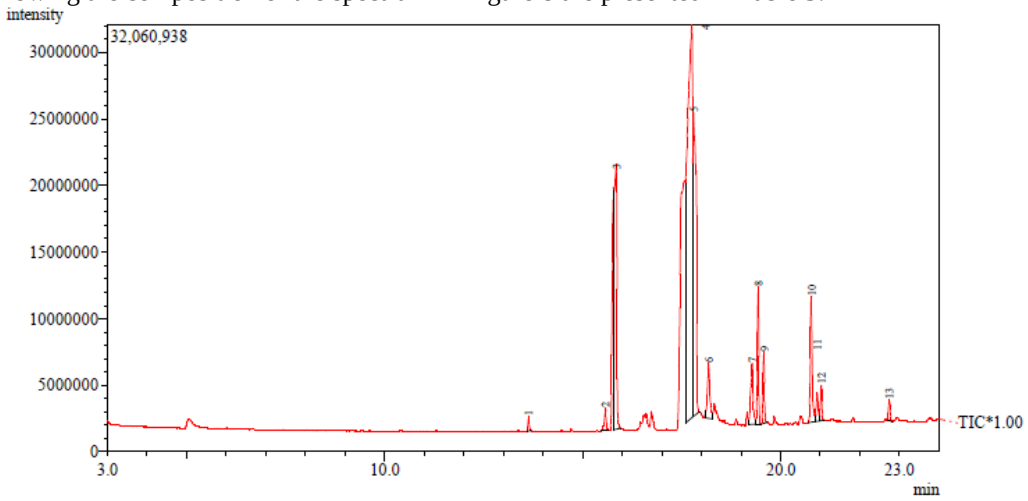


Figure 8: GC-MS spectra of *Thevetia peruviana* biodiesel at 12:1 methanol/oil molar ratio and 1.25 % catalyst concentration

### 3.2 GC-MS Compounds Identification in the Biodiesels

The spectra data of the composition of the *Thevetia peruviana* (TP) biodiesels as synthesized are shown in in Table 2 and Table 3, while that of the composition of the fatty esters and free fatty acid is as summarized in Table 4.

Table 2: Compounds present in *Thevetia peruviana* (TP) biodiesels

| S/N | TP 1A | % A | TP 1B | % A | TP 2A | % A | TP 2B | % A |
|---|---|---|---|---|---|---|---|---|
| 1 | Palmitic acid methyl ester | 10.16 | Hexadecanoic acid, 15-methyl-,methyl ester | 0.5 | Glycerol | 9.02 | Glycerol | 5.43 |
| 2 | Palmitic acid ethyl ester | 8.4 | 11-Octadecenoic acid, methyl ester | 0.98 | Myristic acid methyl ester | 0.45 | Myristic acid methyl ester | 0.43 |
| 3 | Palmitic acid | 6.92 | Palmitic acid methyl ester | 8.73 | 11-Octadecenoic methyl ester | 0.89 | Palmitic acid methyl ester | 12.79 |

| S/N | TP 1A | % A | TP 1B | % A | TP 2A | % A | TP 2B | % A |
|---|---|---|---|---|---|---|---|---|
| 4 | 11-Octadecenoic acid, methyl ester | 25.19 | Palmitic acid methyl ester | 6.95 | Docosanoic acid, ethyl ester | 5.36 | Palmitic acid ethyl ester | 4.32 |
| 5 | 9-Octadecenoic acid, ethyl ester | 16.75 | Palmitic acid ethyl ester | 6.26 | Palmitic acid | 2.51 | Palmitic acid | 2.53 |
| 6 | Oleic acid | 12.36 | 16-Octadecenoic acid, methyl ester | 10.64 | 9-Octadecenoic acid, methyl ester | 20.73 | Hexadecanoic acid 15-methyl-methyl ester | 0.61 |
| 7 | Stearyl vinyl ether | 2.28 | 9-Octadecenoic acid, methyl ester | 29.09 | Octadecenoic acid, methyl ester | 14.12 | 15-Octadecenoic, methyl ester | 15.8 |
| 8 | 9-Octadecenal | 11.42 | 13-Docosenoic acid, methyl ester | 12.85 | 9-Octadecenoic acid, ethyl ester | 11.82 | Octadecanoic acid, methyl ester | 15.19 |
| 9 | Hexadecanoic acid, 2,3-dihydroxypropyl ester | 2.37 | 9-Octadecenoic acid, ethyl ester | 12.59 | Octadecanoic acid, ethyl ester | 4.38 | 9-Octadecenoic acid, ethyl ester | 9.68 |
| 10 | 2-Methyl-Z,Z-3,13-Octadecadienol | 4.14 | Eicosanoic acid, methyl ester | 6.27 | Decanoic acid,2-ethyl-hexyl ester | 3.29 | Oleic acid | 4.1 |
| 11 | | | 9-Octadecenal | 1.13 | Eicosanoic acid, methyl ester | 5.77 | 9,12-Octadecadien-1-ol | 2.26 |
| 12 | | | Docosanoic acid, methyl ester | 2.84 | 11-Octadecenoic acid methyl ester | 1.97 | Decanoic, decyl ester | 3.71 |
| 13 | | | Heneicosanoic acid, methyl ester | 1.17 | 9-Octadecenal | 5.55 | Eicosanoic acid, methyl ester | 5.49 |
| 14 | | | | | Eicosanoic methyl ester | 1.26 | 11-Octadecenoic acid, methyl ester | 1.9 |
| 15 | | | | | Docosanoic acid, methyl ester | 0.99 | 1,3-Propanediol,2-dodecyl | 7.12 |
| 16 | | | | | 2-methyl-Z,Z-3,13-Octadecadienol | 11.89 | Eicosanoic acid, methyl ester | 2.54 |
| 17 | | | | | | | Glycerol 1-palmitate( an ester) | 2.39 |
| 18 | | | | | | | 2-methyl-Z,Z-3,13-Octadecadienol | 3.71 |
| 19 | | | | | | | | |

The information contained in Table 2 is summarized in Table 4, where A= GC-MS spectral area.

Table 3: Compounds present in *Thevetia peruviana* (TP) biodiesels

| S/N | TP 2C | % A | TP 3C | % A | TP 5C | % A |
|---|---|---|---|---|---|---|
| 1 | Glycerine | 4.9 | 11-Octadecenoic acid, methyl ester | 0.72 | Myristic acid,methyl ester | 0.4 |
| 2 | Myristic acid methyl ester | 0.3 | Palmitic acid, methyl ester | 13.46 | 11-Octadeceoic acid,methyl ester | 0.91 |
| 3 | Palmitic acid methyl ester | 17.51 | Palmitic acid, methyl ester | 9.01 | Palmitic acid methyl ester | 12.43 |
| 4 | Palmitic acid ethyl ester | 2.52 | Palmitic acid | 1.68 | Oleic acid, methyl ester | 46.48 |
| 5 | Palmitic acid | 1.87 | Hexadecanoic acid, 15-methyl-methyl ester | 0.69 | 13-Docosenoic acid,methyl ester | 19 |
| 6 | Hexadecanoic acid 15-methyl-,methyl ester | 0.44 | 9-Octadecenoic acid,methyl ester | 34.22 | Linoleic acid | 2.96 |
| 7 | 6-Octadecenoic acid,methyl ester | 10.12 | Stearic acid methyl ester | 19.7 | Oxalic acid, 2-ethylhexyl hexyl ester | 3.33 |
| 8 | Trans, trans-2,8-decadiene | 9.27 | Oleic acid | 4.1 | Pentadecanoic acid, 14-methyl-methyl ester | 4.28 |
| 9 | Oleic acid methyl ester | 12.49 | 1-Octanol,2-butyl | 3.3 | 11-Octadecenoic acid, methyl ester | 2.37 |
| 10 | Stearic acid methyl ester | 10.78 | Eicosanoic acid,methyl ester | 3.31 | 9-Octadecenal | 4.92 |
| 11 | 9-Octadecenoic acid,ethyl ester | 5.97 | 11-Octadecenoic acid, methyl ester | 1.97 | Stearyl vinyl ether | 1.1 |
| 12 | Linoleic acid | 3.05 | 9-Octadecenal | 5.96 | Docosanoic acid, methyl ester | 1.08 |
| 13 | Stearic acid, ethyl ester | 1.48 | Stearic acid, octadecyl ester | 1.31 | Heneicosanoic acid, methyl ester | 0.74 |
| 14 | Glycerol1-palmitate | 2.9 | Heneicosanoic acid, methyl ester | 0.56 | | |
| 15 | Eicosanoic acid, methyl ester | 3.97 | | | | |
| 16 | 11-Octadecenoic acid,methyl ester | 1.34 | | | | |
| 17 | 7-tetradecenal, (Z)- | 5.3 | | | | |
| 18 | Docosanoic acid, methyl ester | 1.78 | | | | |
| 19 | Glycerol 1-palmitate | 1.36 | | | | |
| 20 | Heneicosanoic acid, methyl ester | 0.57 | | | | |
| 21 | 2-methyl-z,z-3,13-Octadecadienol | 2.09 | | | | |

The information contained in Table 3 is summarized in Table 4, where A= GC-MS spectral area.

Table 4: Compositions of fatty esters and free fatty acid in *Thevetia peruviana* biodiesels

| S/N | SAMPLE | Biodiesel Yield % | Methyl Esters % | Ethyl Esters % | FFA % |
|---|---|---|---|---|---|
| 1 | TP 1A | 62.87 | 35.35 | 25.15 | 19.28 |
| 2 | TP 1B | 98.87 | 80.02 | 18.85 | |
| 3 | TP 2A | 80.05 | 46.18 | 24.85 | 2.51 |
| 4 | TP 2B | 80.28 | 54.75 | 9.68 | 6.63 |
| 5 | TP 2C | 78.42 | 59.3 | 9.97 | 4.92 |
| 6 | TP 3C | 84.96 | 83.64 | | 5.78 |
| 7 | TP 5C | 91.02 | 87.69 | 3.33 | 2.96 |

Table 4 shows the data summary of biodiesel yield, methyl and ethyl esters, FFA -free fatty acid, of the different biodiesel's treatment as contained in Table 2 and 3. The data in Table 4 is discussed in sections 3.3-3.5.

**3.3 Biodiesel Yield**

From Figure 8, as catalyst loading increases from TP1 to TP5, the yield of biodiesel gradually increases in the reaction, even though the catalyst loading at TP 1B did not follow the trend, it gave the highest yield of esters due to differences in

alcohol/oil molar ratio. This trend was observed in this study because as the percentage of catalyst increases, more catalyst participated in lowering the activation energy during the biodiesel production, therefore favouring and accelerating the forward reaction. This is consistent with Ahmad et al., reports [2].

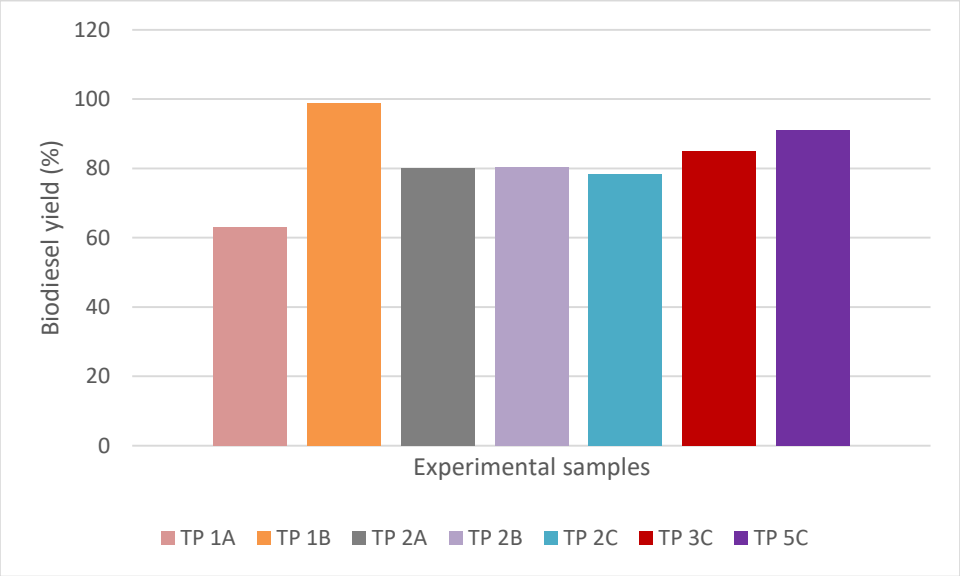


Figure 8: Percentage yield of biodiesels with varying catalyst loading and methanol/oil molar ratio.

Again from Figure 8, at constant catalyst and varying alcohol/oil molar ratio i.e. between TP1A and TP1B, by observation the higher the molar ratio, the higher the biodiesel yield. This is because as the concentration of alcohol is increased, inter-atomic distance between reacting species is reduced and more collision of reacting species takes place that favour's the yield of esters. Between TP 2A to TP 2C was also at constant catalyst loading, the trend shows that there was very little or no differences in ester yield followed by a slight decrease at TP 2C. It can be said that the reaction at this point has reached a maximum at this catalyst loading and started to decline. Therefore, increasing the alcohol/oil at this point does not have any effect on the reaction. This shows that the yield of the biodiesel followed a pattern defined by some relationship between catalyst loading and methanol/oil ratio, this is consistent with studies by Ahmad et al., [2] and Ishaya et al., [1]. However, between TP 3C and TP 5C the catalyst loading was higher than TP 2 and an increased in yield was observed. This shows that different results are obtained from different reacting conditions.

From each three repeated tests, the optimum condition for maximum yield of 98.87 % was found to be TP 1B at 9:1 alcohol/oil molar ratio and 0.25% catalyst loading. This reaction condition ensures the maximum lowering of activation energy favours forward reaction and the maximum collision between reacting species. The outcome of this study is comparable to Ahmad et al., [2] that reported an optimal yield of 98±2 % at 0.51 % catalyst and 5.9:1 methanol/oil molar ratio, similarly Dube et al., [22] showed an optimal yield of 98% at $60^0$C, 30 minutes and 6:1 alcohol/oil molar ratio. Again, this study is in agreement with Kumar et al., [12] that reported an optimal yield of 94.83 % at 6:1 methanol/oil molar ratio and 1wt% catalyst concentration.

### 3.4 Methyl Esters Yield

From Figure 9, it was observed that as catalyst concentration increased from TP 1A to TP 5C, the formation of methyl esters also increases. Though out-of-trend increase was recorded at TP 1B and it due to the combine effect of the higher alcohol/oil molar ratio at that point, that was found to be the optimum condition for the experiment. The observed trend was due to increased catalytic activity that lowers the activation energy, therefore favouring forward reaction. Similarly, keeping catalyst concentration constant and increasing the alcohol/oil molar ratio also increases the methyl ester yield as observed between TP 1A and 1B, and from TP 2A to 2C. This is attributed to the decrease in emulsifier ability of the hydroxyl group. According to Gulum & Bilgin [13] the higher the alcohol/oil molar ratio the lower the biodiesel viscosity and vice versa, whereas at lower concentration of catalyst $\leq 0.75$ % the change in viscosity of methyl esters is small. It is undesirable to have higher viscosity in a biodiesel due to issues of poor atomization, poor combustion and higher emissions. Comparing Figures 9 and 10, it was observed that more methyl esters were formed than ethyl esters and this is because the alcohol used for the transesterification was methanol and also due to increase in catalyst loading activity as activation energy of methyl esters are much lower than that of the ethyl esters. This observation is consistent with studies by Ishaya et al., and Juliana [1, 28]. Though methyl esters can absorb moisture from the atmosphere and retain it as suspension in the fuel. This can lead to poor combustion properties as well as fuel contamination.

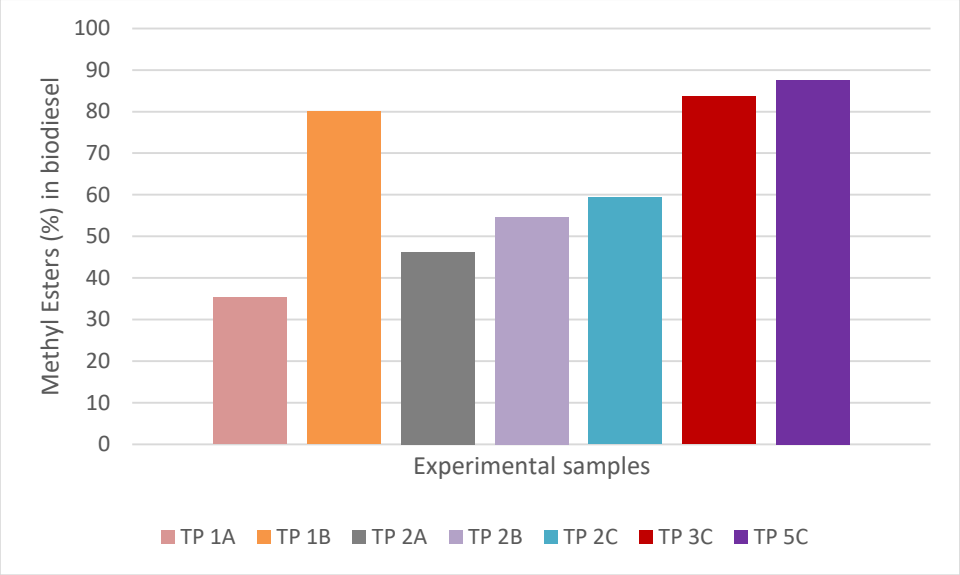


Figure 9: Percentage yield of methyl esters in the biodiesel with varying catalyst loading and methanol/oil molar ratio.

### 3.5 Ethyl Esters Yield

It was observed from Figure 10, that catalyst loading varies inversely with ethyl ester formation. It means that as catalyst concentration increases, ethyl esters production decreases. This is because more catalyst loading continues to lower the activation energy such that more methyl from the methanol is able to react to form methyl esters. Therefore, it means that ethyl's need more energy to react than methyl. A non-conformity to observed trend was recorded at TP 2A and a zero-ethyl ester was observed at TP 3C and this is attributed to optimum loading of catalyst at a particular alcohol/oil molar ratio. Similarly, keeping catalyst concentration constant and increasing the alcohol/oil molar ratio also decreases the ethyl ester yield as observed between TP 1A and TP 1B, and from TP 2A to 2C. This is because more and more methyls are introduced into the system and as concentration increases, it favours the formation of methyl esters rather than methyl esters. According to Ulu et al., [3] ethyl esters are better in oxidation stability, cold flow property, improved lubricity and lower iodine values than methyl esters, but has longer lengths during spray penetration and narrow spray cone angles

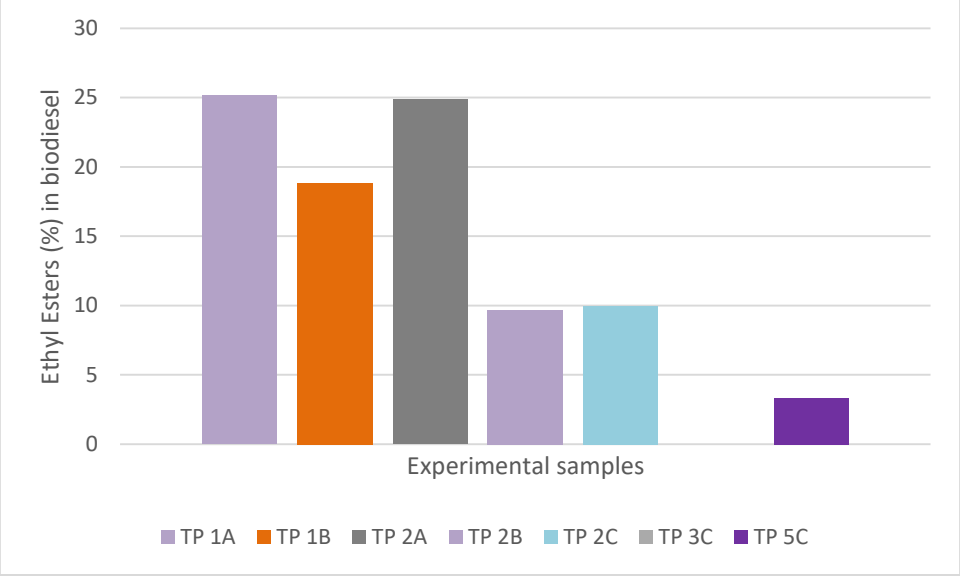


Figure 10: Percentage yield of ethyl esters in the biodiesel with varying catalyst loading and methanol/oil molar ratio

### 3.6 Free Fatty Acids Remaining

Similarly, from Figure 11; it was observed that as catalyst concentration increased from TP 1A to TP 5C, the percentage of free fatty acid decreases non-uniformly. Though no free fatty acid was recorded at TP 1B and it is due to the nearly complete conversion of TAG and FFA to esters as shown in Figure 8. This was because the optimum condition for

biodiesel formation settled in the region of TP 1B. Again, keeping catalyst concentration constant and increasing the methanol/oil molar ratio also sharply decreases the FFA in the biodiesel as observed in Figure 11 between TP 1A and TP 1B, though from TP 2C to TP 5C the decrease in FFA is not sharp by keeping methanol/oil molar ratio constant and increasing the catalyst concentration. This observation implies that the reaction is most influenced by methanol/oil molar ratio. The higher the FFA in a biodiesel the higher the acid value which leads to rancidity of the oil and corrosion of engine parts when used in internal combustion engines.

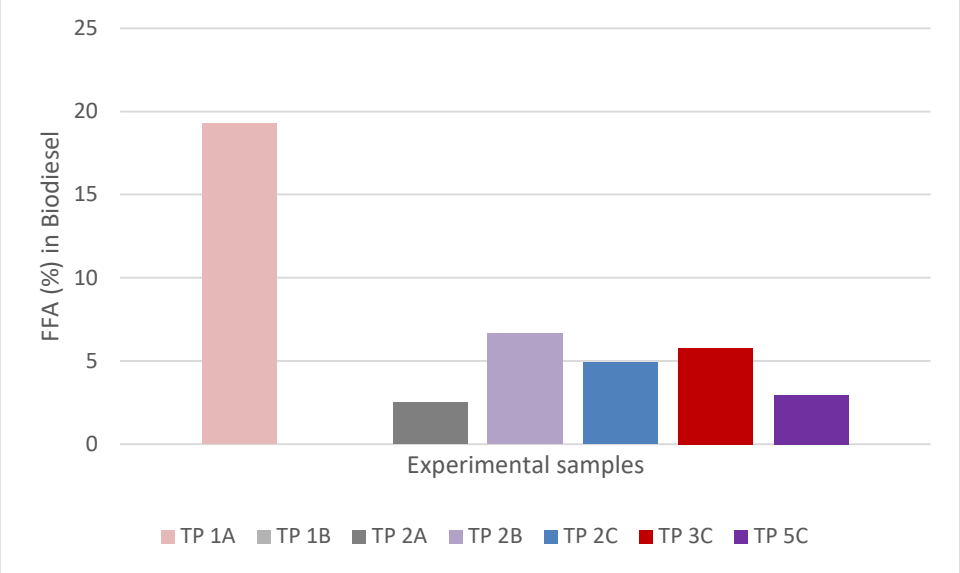


Figure 11: Percentage free fatty acid remaining in the biodiesel with varying catalyst loading and methanol/oil molar ratio

## 4. CONCLUSION

In this study; biodiesel production optimization from *Thevetia peruviana* (TP) seed oil using transesterification method was investigated. The following conclusions are drawn from the results:
The best conditions for this study gave a biodiesel yield of 98.87 % having 80.02 % methyl esters and 18.85 % ethyl esters were as follows; the concentration of catalyst 0.25 %, 9:1 methanol/oil molar ratio, $60^0$C reaction temperature and 60 minutes reaction time. It was noted to be TP 1B. It was observed that holding catalyst concentration constant and increasing the alcohol/oil molar ratio increases the methyl ester yield. The study showed that alcohol/oil molar ratio has the most influence on the yield of methyl esters from Thevetia peruviana seed oil, and alcohol had a higher molar ratio compared to oil (9:1). Alcohol can be sourced from renewable sources and Thevetia peruviana seed oil is a renewable raw material. This implies that following this route to produce biodiesel is sustainable and this would greatly reduce the well to wheel carbon dioxide emissions from automotive sources.

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