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Methyl Ester Fatty Acids Profiles of African Mahogany Biodiesel Produced at Varied Molar Ratios, 0.50% NaOH Catalyst Concentration, Constant Time and Temperature

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Abstract: *World environmental degradation and energy supplies insecurities occasioned by climate change, air pollution and coronary diseases has contributed to the increased interest in new energy sources. This study focused on the production and characterization of the methyl esters fatty acid (MEFA) of African Mahogany (AM) at varied oil to methanol molar ratios (MR) of 1:6, 1:9 and 1:12, while other key parameters of 0.50 % NaOH catalyst concentration at 60 ° C, for 1-hour were kept constant. The oil from the African mahogany seed kernel was extracted using a Mechanical expeller and a yield of 44.88% - by weight was obtained. AM percent free fatty acid was reduced from 17.39 to 0.46 mg (KOH)/ g (of the oil) by 4-runs of H_2SO_4 acid esterification. This was followed by alkaline (NaOH) transesterification conducted and the biodiesels were produced at the varied oil to methanol molar ratios. The chemical composition cum profile of the produced biodiesels were determined using the gas chromatograph and mass spectrometer (GCMS). The results indicated increasing yields of MEFA from AM at molar ratio (MR) of 1:6 was 55.99 %, then AM. at MR of 1:9 was 65.97 %, and AM. at MR of 1:12 to 79.48 %. Similarly, the principal composition of Octadecenoic acid methyl ester increased from 16.55% in crude AM oil to 30.22 %, 39.89 % and 46.62 % in the biodiesel produced as the molar ratios were increased from 1:6 to 1:12. Thus, a mathematical model $y = 1.763x2 + 1.167x + 46.604$ ($MR = 1 < x < 5$) having the predictability $R^2 = 1$ for AM feedstock was established.*

Keywords: *Methyl ester fatty acid, Biodiesel, African mahogany, Molar ratio, Catalyst concentration.*

1.0 INTRODUCTION

The growing interest in new energy sources is due to energy supply insecurities promoted by environmental issues including climate change, air pollution and coronary diseases like COVID-19. Liquid biofuels are majorly used in the transport sector to power mobility solutions. Thus, biodiesel from non-edible sources has been competitively attractive because it crashed the high production expense of which edible feedstock account for over 75 % of the overall cost [1 - 3]. Seeds from African mahogany (AM) also botanically known as *Khaya senegalensis* is a source of tropic oil. The seed hard casing commonly burst to spread its kernels as waste. The kernel can be gathered to extract sufficient bio oil. Three set of biodiesels were experimentally produced from oil of AM seed kernel in this study. The molar ratio (MR) of the oil to alcohol was set as the process variable factor, while catalyst concentration, temperature and time of reaction are the process constant factors. The aim here was to determine the impact of molar ratio on the yield of biodiesel production from the seed kernel of the AM feed stock. The chemical composition as measured using gas chromatography and mass spectrometer (GC-MS) of each oil is impacted by the reaction parameters, chemicals interaction and responses with key reagents.

Reducing the percentage free fatty acid (% FFA) content of crude oil by esterification is required to fast track other processes. The acid-esterification type is preferred because its reactivity is faster compared with the alkaline type. This is subsequently followed by alkaline based transesterification of the oil using the appropriate ratio of alcohol. Normally, the stoichiometric reaction requires one (1) mole of oil (triglyceride) and three (3) moles of alcohol (Methanol) as expressed in Equation 1 [4]. The reversible reaction will result to three (3) moles of methyl esters and one (1) mole of glycerol.

$$\underset{\textit{Triglyceride}}{\text{R-CO-OR'}} + \underset{\textit{Alcohol}}{\text{3R"-OH}} \xleftrightarrow{H^+ \text{ or } R"O^-} \underset{\textit{Methyl Esters}}{\text{3R-CO-OR"}} + \underset{\textit{Glycerol}}{\text{R'-OH}} \quad (1)$$

Studies showed that crude *Calophyllum inophyllum* oil (CCIO) acid value of was 59.30 mg KOH/g. It went through the degumming, esterification, neutralization and transesterification process to reduce the acid value to 0.34 mg KOH/g. The optimum yield was obtained at 9:1 methanol to oil ratio with 1wt.% NaOH catalyst concentration at 50 ^{o}C for 2-hours [1]. The FFAs content was reduced from 21.8 % to 3.0 % using ethanol:oil at molar ratio of 8:1, and 3 % ferric sulphate ($Fe_2(SO_4)_3$) as catalyst on the oil extracted from feedstock of abundantly available degummed rice bran. The esterification reaching 85.8 % conversion of the FFA to ethyl esters fatty acid (EEFA). Further process of transesterification increased the conversion to 96.6 % of a mixture of EEFA and methyl esters fatty acid (MEFA). The variable parameter was catalyst concentration varied from 1.0 to 1.75 % potassium hydroxide (KOH), and the constants parameters were the molar ratio of methanol to oil at 6:1, temperature at 60 ^{o} C and time of 1-hour [5]. Similar outcomes were obtained using Brassica juncea oil for biodiesel production [6]. Also, oil extracted from microalgae was converted to MEFA and EEFA with a yield greater than 90 % in 24-hours using Lipase enzyme as the catalyst [7]. For the above studies, high yields of alkyl esters fatty acid (AEFA) that is biodiesel were produced at fixed catalyst and molar ratio values. In another case, catalyst was varied from 1.0 % to 1.75 %, while molar ratio was fixed at ratio 6:1. This study is to close the gap of establishing the impact of fixed catalyst concentration and varied molar ratios on the yields of the biodiesel.

2.0 MATERIALS AND METHOD

The feed stock as key material for this research was the African Mahogany seed kernel harvested from trees in Kagoro of Kaura Local government of Kaduna State, Nigeria. The analytical grades solutions of methanol (CH_3OH), propan-2-ol (C_3H_7OH), sulphuric acid (H_2SO_4), 0.1 Mole sodium hydroxide (NaOH) pellets and 0.1 Mole potassium hydroxide (KOH) pellets were the major reagents used for this experiment. Also, the major equipment used for this research are GC-MS machine, mechanical oil expeller, conical flasks, hot plates, magnetic stirrer, thermometers, stop clock, separating funnels, aluminium foil paper, etcetera.

The mechanical expeller was used to extract the crude oil [8]. A 5-microns filter was used to filter the extracted oil and its chemical content profiled using the GCMS system machine. The American standard for testing materials ASTM D6751 standards approach was used in the execution of the oil extraction, biodiesel production, qualification and quantification processes.

The methodology of the processes for this research includes the biodiesel production, which involves pretreatment method of the oil for transesterification. The sample procedure was using 10.0 g of African mahogany oil added to 240.0 g of propan-2-ol. The mixture was titrated using 0.1 Mole of potassium hydroxide and a blank titration was conducted. The FFA value and acid value (AV) of the oil were evaluated using equations 2 and 3. The process cycle was repeated until the AV value of ≥ 0.5 % was achieved [9].

$$\text{Free Fatty Acid (FFA)} = \frac{\text{KOH Titre Value x Molarity of KOH x Molar Mass of KOH}}{\text{Mass of oil}} \quad (2)$$

% FFA = (v x 0.1 M x 56.1 g /mol) / 10.0g Where, v is the titre value.

$$\text{Acid Value (A.V.)} = \frac{\%\ \text{FFA}}{2} \quad (3)$$

The transesterification was conducted by the use of 240 g of the esterified AM oil with 52.08 g of methanol for an oil to alcohol molecular mass ratio of 1:6 at the first instant, 1.2 g (0.50 % m/m of oil) of NaOH as catalyst loaded for 1-hour at 60 ^{0}C to produce the AM biodiesel. Similar process was repeated for biodiesel production at oil to methanol ratios of 1:9 and 1:12, which translate to 78.18 g and 104.22 g of methanol solutions, respectively.

The qualitative and quantitative chemical composition of each biodiesel sample was analysed using GC-MS: QP2010nc PLUS system. One micro-litre (1 µl) from each biodiesel also called "*Analyte*" was injected into the column via the Pre-column for band broadening of the solutes. The Carrier/Purge gas was $^{2}_{4}$Helium (for its very light weight and inert properties) set at a column (trap) flow of 1.80 ml/min at pressure of 116.9 kPa, with the total flow (analyte) rate of 40.8 ml/minute and linear velocity of ion was at 49.2 cm/second as components bleed or elute the test-sample in the GC Unit. Injector split ratio was set at 20:1 to narrow and focus the positively charged ions as a beam, based on the boiling point or vapour pressure of solutes to elute. The total scan time was set at 24 minutes with equilibrium time of 3 minutes, having event time of 0.5 seconds at scan speed of 666 Hz. These are set not to compromise the resulting resolution of 2 M max FWHM.

The generated report indicates the retention time for each peak or component (in minutes). To determine the area and percent composition for each peak (e.g. as for peak 1 to 10 indicated in Figure 1), the triangularisation method as in Equation (4) was used by the computer system to find the area under each curve or peak (which has triangle-like shape) using:

$$\text{Area} = (\text{height}) \times (\text{width at } ½ \text{ height}) \quad (4)$$

Thus, the areas under the peak-curve can be used to calculate percent of a component in the biodiesel composition using Equation (5) [9].

$$\% \text{ Component } X = \frac{\text{Area under peak } X}{\text{Total graph area}} \quad x \quad 100\% \tag{5}$$

The typical comprehensive full scan report generated from the GC-MS analysis for each biodiesel includes the chromatograms, peaks report, mass spectra, and spectrum comparison search results reports. These are used in for further analysis. Also, the Scheffé's mathematical polynomial relations as Equation (6) for the yield y of order k in variable x [10] as defined by

$$Y = \text{Co} + C_1X^1 + C_2X^2 + \ldots + C_kX^k \tag{6}$$

Where ($0.0 \leq x \leq 1.0$) i.e. $x = 0.0, 0.1, 0.2, \ldots. 1.0$. ($x \equiv$ % Biodiesel in each blend).

The correlation coefficient model may include predicted R-squared, which indicates the prediction strength of the developed mathematical model for the experiment as used for simulations.

3.0 RESULTS AND DISCUSSION

The oil was extracted from African mahogany seed kernels using the mechanical expeller, the oil yield was 47.36 % by weight (Ishaya *et al*., 2015). The obtained yield makes it a higher volume non-edible feed stock source for commercial production of biodiesel, compared with seed oil from jatropha (43 %), tobacco kernel (38 %) and similar plant seeds [2, 11].

The crude oil %FFA was found to be 34.78 mg (KOH)/ g (of the oil). Thus, its AV was 17.39 mg (KOH)/ g (of the oil). Subsequently, four runs of esterification were executed to obtain the ASTM D6751 standards acceptable value for %FFA of ≤ 0.50 mg (KOH)/ g (of the oil) for base catalyst transesterification. The % FFA values of the runs were 11.22, 4.57, 1.66 in this order and the % FFA of the fourth run or iterations was 0.92 mg (KOH)/ g (of the oil); hence its AV was 0.46 mg (KOH)/ g (of the oil).

The results of the GC-MS analysis of the AM crude oil and the biodiesels produced at constant base catalyst concentration of 0.50 % NaOH, at the three varied esterified oil to alcohol (methanol) MR of 1:6, 1:9 and 1:12 are presented and hereafter referred to as AM.1:6, AM.1:9 and AM.1:12 respectively hereafter. The Gas chromatographs indicating the identified peaks for the four oils are presented in Figures 1 to 4. Tables 1 to 4 are synthesis of these graphs, providing some details of each peak per graph as well as the methyl ester fatty acid yield for each MR biodiesel produced.

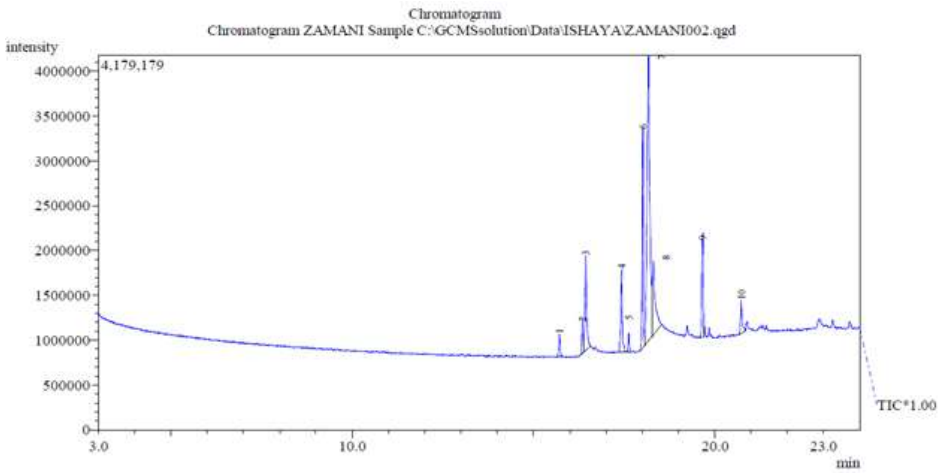

Figure 1: Chromatograph of crude oil African mahogany - mass spectra showing the scan peaks.

Table 1: African mahogany crude oil peaks report (TIC) including percent biodiesel of components

Peak No.	Retention Time (min)	Peak Area (A)	Area %	Biodiesel Fraction	% Biodiesel Area	Compound Systematic Name (Fatty Acid)
1	15.713	75,110	1.41	0.80	1.128	Decanoic acid, methyl ester
2	16.357	805,931	1.98	1.00	1.980	Hexadecanoic acid, methyl ester
3	16.440	3,691,107	9.05	0.00	0.000	n-Hexadecanoic acid
4	17.427	2,628,082	6.45	1.00	6.450	11-Octadecenoic acid, methyl ester
5	17.628	502,863	1.23	1.00	1.230	Octanoic acid, methyl ester
6	18.013	6,029,130	14.79	0.60	8.874	E)-9-Octadecenoic acid methyl ester

7	18.170	17,863,165	43.81	0.00	0.000	9-Octadecenoic acid (Z)-
8	18.304	4,704,971	11.54	0.20	2.308	10-Undecenoic acid, octyl ester
9	19.665	2,690,419	6.60	0.00	0.000	6-Octadecenoic acid, (Z)-
10	20.727	1,285,996	3.15	0.00	0.000	9-Octadecenal
Crude African Mahogany AM		40,776,774	100	***Biodiesel Total %=***	**21.970**	

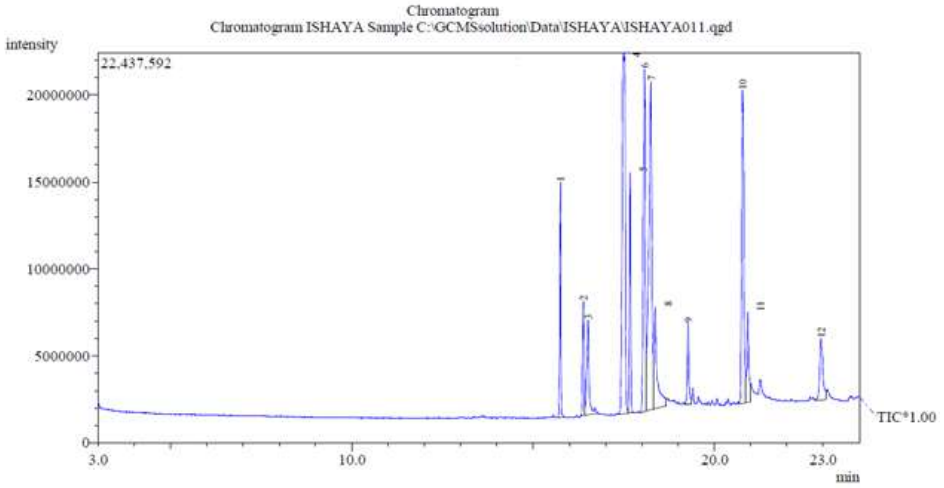

Figure 2: Chromatograph of biodiesel sample-AM.1:6 - mass spectral showing the scan peaks

Table 2: Sample-AM.1:6 peaks report (TIC) including percent biodiesel of components.

Peak No.	**Retention Time (min)**	**Peak Area (A)**	**A/H**	**Biodiesel Fraction**	**% Biodiesel Area**	**Compound Systematic Name (Fatty Acid)**
1	15.750	33,093,385	5.81	1.00	5.810	Pentadecanoic acid, 14-methyl-, methyl ester
2	16.386	14,990,096	2.63	1.00	2.630	Tridecanoic acid methyl ester
3	16.514	25,239,855	4.43	0.00	0.000	n-Hexadecanoic acid
4	17.494	120,870,098	21.21	1.00	21.210	11-Octadecenoic acid, methyl ester
5	17.676	33,759,394	5.92	1.00	5.920	Docosanoic acid, methyl ester
6	18.073	73,425,463	12.89	0.60	7.734	9-Octadecenoic acid, ethyl ester
7	18.247	111,667,784	19.60	0.40	7.840	E-11-Hexadecenoic acid, ethyl ester
8	18.366	33,548,030	5.89	0.20	1.178	Octadecanoic acid, methyl ester
9	19.272	14,290,926	2.51	0.40	1.004	Pentafluoropropionic acid, tridecyl ester
10	20.784	68,326,711	11.99	0.00	0.000	1,2-15,16-Diepoxyhexadecane
11	20.922	18,992,608	3.33	0.80	2.664	Pentafluoropropionic acid, tridecyl ester
12	22.946	21,615,034	3.79	0.00	0.000	(Z)-9-Tetradecenal
AM2a		569,819,384	100	***Biodiesel Total %=***	**55.990**	

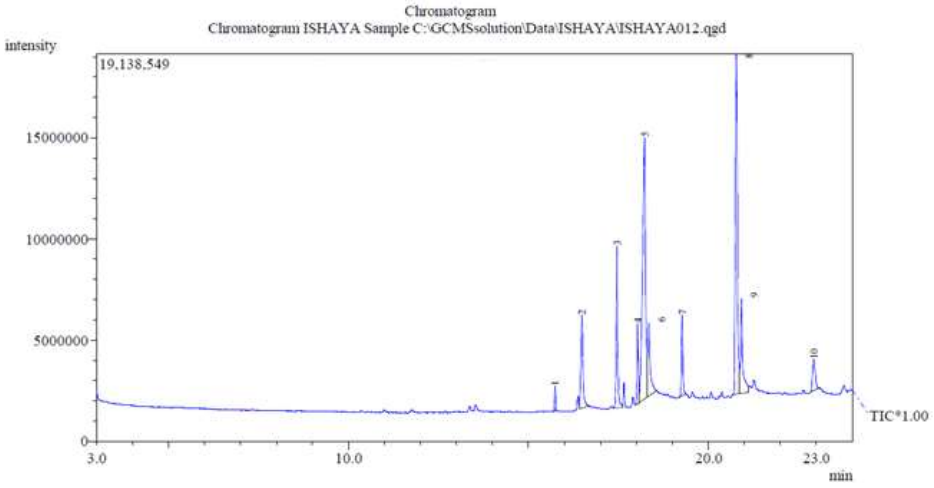

Figure 3: Chromatograph of biodiesel sample-AM.1:9 - mass spectral showing the scan peaks

Table 3: Sample-AM.1:9 peaks report (TIC) including percent biodiesel of components

Peak No.	Retention Time (min)	Peak Area (A)	Area %	Biodiesel Fraction	% Biodiesel Area	Compound Systematic Name (Fatty Acid)
1	15.735	2,854,518	1.15	1.00	1.150	Pentadecanoic acid, methyl ester
2	16.486	19,003,233	7.67	0.00	0.000	n-Hexadecanoic acid
3	17.460	23,241,136	9.37	1.00	9.370	11-Octadecenoic acid, methyl ester
4	18.035	9,990,744	4.03	0.40	1.612	E-11-Hexadecenoic acid, methyl ester
5	18.225	75,665,009	30.52	1.00	30.520	9-Octadecenoic acid (Z)-
6	18.345	17,104,487	6.90	0.00	0.000	Octadecanoic acid
7	19.268	11,096,837	4.48	0.40	1.792	Pentafluoropropionic acid, tridecyl ester
8	20.780	61,669,294	24.87	0.60	14.922	Pentanoic acid, 10-undecenyl ester
9	20.919	19,125,036	7.71	0.60	4.626	Pentafluoropropionic acid, tridecyl ester
10	22.927	8,170,437	3.30	0.60	1.980	Pentanoic acid, 10-undecenyl ester
	AM2b	247,920,731	100	**Biodiesel Total %=**	**65.972**	

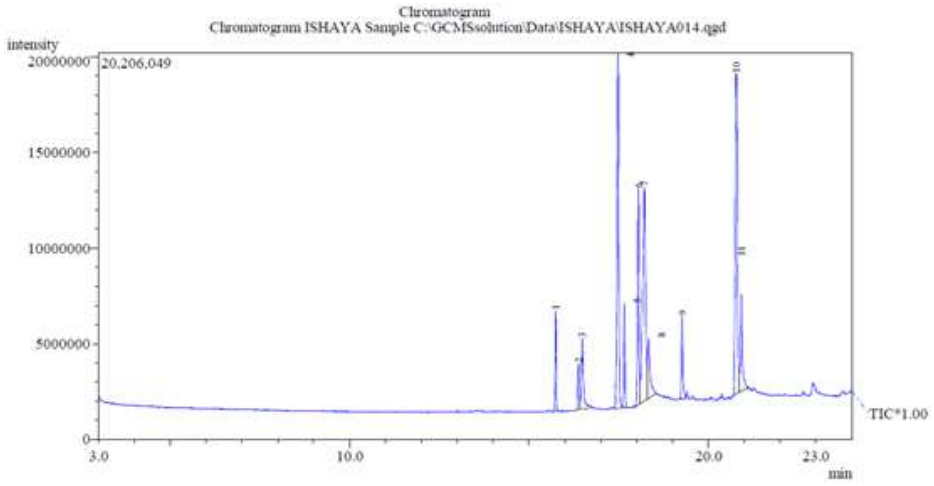

Figure 4: Chromatograph of biodiesel sample-AM.1:12 - mass spectral showing the scan peaks

Table 4: Sample-AM.1:12 peaks report (TIC) including percent biodiesel of components.

Peak No.	Retention Time (min)	Peak Area (A)	Area %	Biodiesel Fraction	% Biodiesel Area	Compound Systematic Name (Fatty Acid)
1	15.743	11,936,172	3.82	1.00	3.820	Pentadecanoic acid, methyl ester
2	16.383	5,394,388	1.73	1.00	1.730	Tridecanoic acid methyl ester
3	16.486	16,359,501	5.24	0.00	0.000	n-Hexadecanoic acid
4	17.483	62,630,539	20.05	1.00	20.050	11-Octadecenoic acid, methyl ester
5	17.662	12,840,361	4.11	1.00	4.110	Docosanoic acid, methyl ester
6	18.051	30,354,990	9.72	0.60	5.832	(E)-9-Octadecenoic acid methyl ester
7	18.218	64,990,266	20.80	1.00	20.800	9-Octadecenoic acid (Z)- methyl ester
8	18.341	14,456,119	4.63	0.00	0.000	Nonadecanoic acid
9	19.270	12,429,371	3.98	0.60	2.388	Pentafluoropropionic acid, tridecyl ester
10	20.787	63,569,814	20.35	0.80	16.280	Pentanoic acid, 10-undecenyl ester
11	20.924	17,423,116	5.58	0.80	4.464	Pentafluoropropionic acid, tridecyl ester
	AM2c	312,384,637	100	***Biodiesel Total %=***	**79.474**	

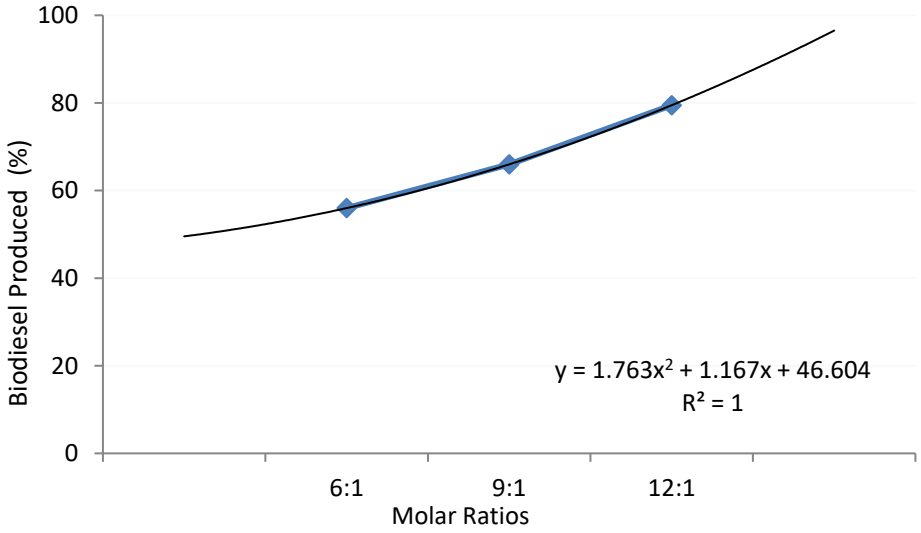

Figure 5: AM Yield Optimization at Constant CC = 0.50 % with varied Molar Ratio.

African mahogany seed crude oil has a yield of 21.97 % MEFA as shown in table 1. Its composition was made up of mainly E)-9-Octadecenoic acid and 11-Octadecenoic acid as the methyl ester fatty acids. The low MEFA yield in crude oil was consistent with known results [1, 12]. Normally, this is so because plant base crude oil has high values of free fatty acid. The reversible chemical reaction in equation 1 indicated that the stoichiometric molar ratio of oil to alcohol is 1:3 for biodiesel production. Experimentally for completeness of this second order reaction, methanol is supplied proportionally in excess to aid reactivity for increased yield. The number of distinct components of the oil are indicated by the number of peaks 10, 12 10 and 11 as shown in figures 1, 2, 3 and 4 respectively. As the oil to methanol molar ratios was varied and catalyst concentration of 0.50 % NaOH kept constant, the resulting yields of AM.1:6 was 55.99 %, AM.1:9 was 65.97 % and AM.1:12 was 79.48 % as presented in tables 1, 2, 3 and 4. The yields were increasing in a progressive manner. The results showed similar trend for rice bran oil, when potassium hydroxide (KOH) as the catalyst was varied from 1.0 to 1.75 % and methanol to oil at molar ratio of 6:1 was kept constant [5]. Also, similar profiling was observed on the results from other feedstocks [2, 8, 9, 13].

The principal MEFA compound being Octadecenoic acid methyl esters that are elute within the retention time of 17.4 to 18.3 minutes, having corresponding larger peaks areas, moved from 16.55% composition in the crude AM oil to 30.22 %, 39.89 % and 46.62 % in the produced biodiesels as the molar ratios are proportionally increased. Also, yields of other types of methyl esters fatty acids were increasing at similar rates, as analysis of information from tables 1, 2, 3 and 4 indicates. These MEFAs increases were due to the availability of methanol in excess that fast track the conversion of triglycerides to diglycerides, then to monoglycerides, as free fatty acids are being converted to biodiesel. Also, it enhances the

transesterification process of converting some esters to other types of desirable esters. The MEFA production trend or mathematical model from Figure 5 showed a Mean root squared of unity for AM feed stock as Equation (7) expressed. The esters contents in the triglycerides are increased on the bases of model:

$$y = 1.763x^2 + 1.167x + 46.604$$
at CC = 0.50 %, (MR = 1 < x < 5, i.e. x is ratio 1:3 =1 to 1:15 = 5) (7)

4.0 CONCLUSION

The African mahogany seed oil mechanically extracted from its kernel has a yield of 47.36 % by weight. The methyl ester fatty acids content in the AM crude oil was 21.97 %. The esterification of free fatty acids to biodiesels and subsequent transesterification to generate new esters at constant 0.5 % NaOH catalyst condition, were increasing with proportional increases of oil to methanol molar ratios. The MEFA yields after the production were 55.99 %, 65.97 % and the highest was 79.48 % at a highest molar ratio of 1:12 oil to methanol, recommended as the production process conditions for AM biodiesel mass production. The major MEFAs in the biodiesel yield of 79.48 % are 9-Octadecenoic acid (Z)- methyl ester 20.80 %, 11-octadecenoic acid, methyl ester 20.05 %, pentanoic acid, 10-undecenyl ester 16.28 % and (E)-9-octadecenoic acid methyl ester 5.82 %. Thus, the results further verified that the production yield of AM biodiesel increasing with increases of oil to alcohol molar ratio as other reaction conditions of temperature, time and catalyst concentration are kept constant. Thus, African mahogany seed oil is suitable for biodiesel production.

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