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Kinetics of Alkali Catalysed Transesterification Reaction of Palm Kernel Oil and physicochemical Characterization of the Biodiesel Product

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ABSTRACT: *The palm kernel oil (P.K.O) biodiesel was produced through alkali catalysed transesterification of crude palm oil (P.K.O) with methanol and NaOH as a catalyst. After four experimental runs of esterification reaction to reduce the FFA value from 23.8425% to 0.8976%, and transesterification reaction was followed. The biodiesel produced was characterized, and its physicochemical properties were estimated and compared to standard values of American Society of Testing and Material (ASTM) and European (EN14214) biodiesel standards. After the fuel characterization, the kinetics of the alkali catalysed transesterification process was studied varying the catalyst concentration and time at constant temperature (for 0.2, 0.3, 0.5, 1.0 and 1.2g at 30, 40, 50, 60 and 120seconds, for a methanol to oil ratio of 4:1). These parametric effects of the kinetic test were carried out for 25 different runs, and the results obtained and analysed. After the analysis of the kinetic studies result, it was discovered that an increase in reaction temperature speeds up the reaction rate and shortens the reaction time. Hence, Biodiesel yield increases with reaction time and its best for the condition 0.5g and 1g at increased temperature of 60^{o}C with maximum Fatty Acid Methyl Ester (FAME) content obtained ranged from 80 to 90%. The limited fuel characterization carried out demonstrated that the PKO biodiesel produced can fuel a diesel engine after much comparison with standards.*

KEYWORDS: *P.K.O, FFA, FAME, Transesterification*

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NOMENCLATURE

P.K.O – Palm kernel oil, FFA – Free fatty acid , EIA - Energy Information Administration , BTU - British thermal units, ULSD - Ultra low sulphur diesel, FAME – Fatty acid methyl ester, API – America petroleum institute, AAS - Atomic absorption spectrophotometer, EN14214 - European Biodiesel Standard, ASTM - American Society of Testing and Materials, %w/w – Weight by weight percent of volume.

I. INTRODUCTION

Human endless search for energy is great, and her inquisitiveness has made her to delve into alternative sources of energy other than the sun. The emergence of alternative sources (either renewable or not) like thermal energy, wind energy, Geo-thermal energy and even energy from fossil fuels have changed man's view about energy and energy generation. Though energy generation is necessary, but maintaining a sustainable environment is paramount. The main advantages of using biodiesel fuels as 100 % methyl or ethyl esters of vegetable oil and animal fat or biodiesel blends (up to 20 % blend to the diesel fuel) are producing less smoke and particulates, having higher cetane numbers and producing lower carbon monoxide and hydrocarbon emissions (Diwani et al., 2009). Most of the world's energy comes from fossil fuels, but issues have arisen from the use of fossil fuels over the years. The environmental degradation from the use of fossil fuels and from human activities is alarming, and this ranges from the release of toxic pollutants and green house gases from the use of coals, to the release of hydro carbonaceous gases. Therefore, the expected scarcity of petroleum supplies and the negative environmental consequences of fossil fuels have spurred the search for renewable transportation bio fuels. Exploring new energy resources such as Biodiesel fuel is of growing importance over the years. This new fuel is a perfect and sustainable substitute for petroleum diesel; hence it's been of interest to

researchers lately. The raw material for any biodiesel produce is an alkyl ester group (i.e. vegetable oils). The costs of raw materials for biodiesel production accounts for large percent of the direct biodiesel production costs required. Thus, one way of reducing the biodiesel production costs is to use the less expensive raw material containing fatty acids such as animal fats, non edible oils, and waste cooking oils and by products of the refining vegetables oils (Ogunwole, 2012). Several vegetable oils can be used in the production of biodiesel, Soya bean, rapeseed, sunflower and palm oils are most studied. The major importers of vegetable oils are China, Pakistan, Italy and the United Kingdom. Few countries such as Netherlands, Germany, United States and Singapore are both large exporters as well as importers of vegetable oils. Global vegetable oil exports rose modestly from 29.8 million tons in 1997/1998 to 31.2 million in 1998/1999 (Demirbas, 2005). In addition, burning of vegetable oil based fuel does not contribute to net atmospheric CO_2 levels because such fuel is made from agricultural materials which are produced via photosynthetic carbon fixation (Sukjit and Punsuvon, 2013).

The need to contribute to research and development to produce eco-friendly and readily available bio fuels, for human domestic and industrial activities is of great importance. And raw material that can be easily sourced for is important for any process aimed at given a lasting and sustainable product. Hence, for an effective reactor design and transesterification reaction condition of fatty esters into biodiesels, this research project investigates "Kinetics of alkali catalyzed transesterification reaction of palm kernel oil, and physicochemical characterization of the biodiesel product." The research study covers the following scope: Physicochemical analysis and tests of the biodiesel products, Kinetic study and analysis of varied parametric effect on the alkali catalyzed reaction.

II. BIODIESEL TRANSESTERIFICATION (ALKALI CATALYSED)

"Bio" indicates a source of energy that is biological and renewable; "diesel" means it can be used only in diesel engines (Zhang et al., 2003). Biodiesel refers to a vegetable oil or animal fat based diesel fuel consisting of long-chain alkyl (i.e. methyl, ethyl, or propyl) ester, biodiesel is considered to be one of the potential renewable alternatives to petroleum since it is biodegradable, non-toxic, and has low emission profiles. It is produced typically by chemically reacting lipids e.g. vegetable oil, soybean oil, animal fat with OH group producing fatty acid esters. According to Johnston and Holloway (2007), the global demand for petroleum is predicted to increase 40% by 2025. The United States Energy Information Administration (EIA) reported that total world energy consumption was 406 quadrillion British thermal units (Btu) in 2000 and is projected to increase to 769.8 quadrillion Btu by 2035. The challenge of using low quality feedstocks for biodiesel production is that the low quality feedstock contains a large amount of FFAs, which can have a side reaction with the alkali-catalyst used in the transesterification process to produce undesirable soaps, inhibiting the separation of biodiesel from glycerol. Soap formation can also produce water that will hydrolyze the triglycerides and aggravate the soap formation. This undesirable side reaction will add a fixed cost due to the use of an additional unit for removing soaps and also lead to a reduction of the yield. When using a low quality feedstock for biodiesel production, a pretreatment step, i.e., esterification is required. In the esterification process, the FFAs are converted into biodiesel without forming soaps, which increases the final yield. It can take place without any catalyst due to the weak acidity of carboxylic acids, but the reaction is extremely slow and requires several days to complete at typical reaction conditions. Previous research results showed that either homogenous mineral acids, such as H_2SO_4, HCl, or HI, or heterogeneous solid acids, such as various sulfonic resins, can effectively catalyze the esterification reaction. The homogenous catalyst is more effective than the heterogeneous catalyst in the esterification reaction, and the reaction kinetics using heterogeneous catalysts are more complicated than those using homogenous catalysts since the restriction of both absorption and dis-absorption rates in the pore of the catalyst needs to be considered in the overall reaction rate. (Zhou, 2013)

Transesterification, also called alcoholysis, is a traditional technology to produce biodiesel. It is the most effective process to transform the big triglyceride molecules into small and straight-chain molecules of fatty acid esters. It can reduce the molecular weight to one-third that of the oil and the viscosity by a factor of eight, and it can increase the volatility. (Zhou, 2013)

In the transesterification of vegetable oils, a triglyceride reacts with an alcohol in the presence of a strong acid or base, producing a mixture of fatty acids alkyl esters and glycerol. The stoichiometric reaction requires 1 mol of a triglyceride and 3 mol of the alcohol. However, an excess of the alcohol is used to increase the yields of the alkyl esters and to allow its phase separation from the glycerol formed. Several aspects, including the type of catalyst (alkaline, acid or enzyme), alcohol/vegetable oil molar ratio, temperature, water content and free fatty acid content have an influence on the course of the transesterification. In the transesterification of vegetable oils and fats for biodiesel production, free fatty acids and water always produce negative effects, since the presence of free fatty acids and water causes soap formation, consumes catalyst and reduces catalyst effectiveness, all of which result in a low conversion. When the original ester is reacted with an alcohol, the transesterification process is called alcoholysis. The transesterification is an equilibrium reaction and the transformation occurs essentially by mixing the reactants. (Demirbas, 2003)

In the biodiesel transesterification process, triglycerides react with an alcohol in the presence of some catalyst to produce esters (biodiesel) and another glycerol.

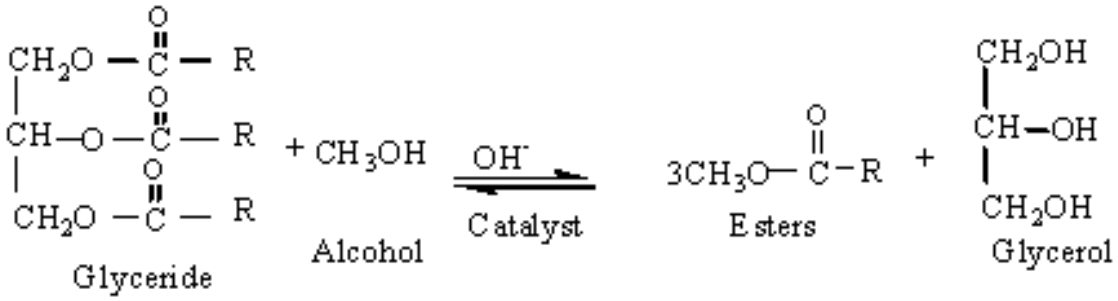

Figure 2.1: General reaction for the transesterification of triglyceride

The overall reaction of transesterification is expressed as follows:

Triglyceride + 3 R' OH ⇨ Glycerol (GL) + 3R′COOR3

The alkali-catalyzed transesterification of vegetable oils proceeds faster than the acid-catalyzed reaction. In the mechanism of the base-catalyzed transesterification of vegetable oils, the first step is the reaction of the base with the alcohol, producing an alkoxide and the protonated catalyst. The nucleophilic attack of the alkoxide at the carbonyl group of the triglyceride generates a tetrahedral intermediate, from which the alkyl ester and the corresponding anion of the diglyceride are formed. The latter deprotonates the catalyst can react with a second molecule of alcohol and starts another catalytic cycle. Diglycerides and monoglycerides are converted by the same mechanism to a mixture of alkyl esters and glycerol. Alkaline metal alkoxides (CH_3ONa) are the most active catalysts, since they give very high yields (>98%) in short reaction times (30 min) even if they are applied at low molar concentrations (0.5 mol %). The presence of water gives rise to hydrolysis of some of the produced ester, with consequent soap formation. Potassium carbonate, used in a concentration of 2 or 3 mol% gives high yields of fatty acid alkyl esters and reduces the soap formation. This can be explained by the formation of bicarbonate instead of water, which does not hydrolyze the esters.

Transesterification is the most widely used process, as FFA esterification is only used for productions of biodiesel from feedstock that contains high levels of FFA. The transesterification process involves the reaction of triglycerides in the oleaginous feedstocks with alcohol (alcoholysis) to form fatty acid alkyl esters, through the interchange of alkoxy moieties (Schuchardt *et al.*, 1998).

III. MATERIALS AND METHODOLOGY

The extracted oil (i.e. crude Palm kernel oil- P.K.O.) was obtained from Bro Kenn Agro allied industry limited, makers of crystal fresh vegetable oil plot D5/130 Onitsha road industrial layout, Irete, Owerri west, Imo state for the research project. And the experiments (i.e. physicochemical analysis and parametric effect analysis) were carried out in the chemical engineering laboratory at Federal University of Technology, Owerri (FUTO).

3.1 FREE FATTY ACID (FFA) DETERMINATION

The standard titrimetry method was used for the FFA determination, where 1g of the oil was dissolved in a conical flask containing 25cm^3 of propanol i.e. isopropyl alcohol. Then 3drops of an indicator preferably phenolphthalein was added to the dissolved oil. Next the oil-indicator solution was titrated against 0.1M KOH. The average titre value after three titrations was used to calculate the acid value and FFA value of the oil using the following formula adopted from Kabiru et al., 2016:

$$\text{Acid value} = \frac{\text{Titre value x conc. Of KOH (i.e. 0.1) x 56.1}}{\text{Wt. of the oil i.e. (1g)}}$$

$$\text{FFA} = \frac{\text{Acid value}}{2}$$

3.2 ESTERIFICATION REACTION

When calculated, the FFA was beyond 1%, the FFA has to be reduced to 1% or even below

Reagent needed for Esterification, adopted from Kabiru et al., 2016.

For a 400g of oil, the amount of methanol measured was 20% w/w of the weight of the oil. While the sulphuric acid measured was 5% w/w of the entire weight of oil. The acid and methanol were poured into a beaker and the mixture was poured into already stirring oil and maintains the heating at 60^{0}C. Stirring continued for 60mins.

The product at this stage was poured into a separating funnel and allowed to stay for some times to ensure proper separation. Two layers were expected. The upper layer is the methanol acid mixture while the lower layer is the oil. The below was noted properly.

3.3 TRANSESTRIFICATION REACTION

The transesterification of biodiesel was carried out using procedure adopted from Ojolo et al., 2011. 200ml of palm kernel oil and 40 ml of methanol (i.e. 20% by volume of oil) were utilized in the test batch production. 200 ml of palm kernel oil was pre-heated to a steady temperature of 60^{o}C using a magnetic heater/stirrer. With the aid of the measuring cylinder 40 ml of methanol was measured and poured into the beaker. 0.7g of NaOH pellet was measured using the weighing balance and added to the methanol. The content of the beaker was stirred vigorously using the second magnetic stirrer until the NaOH was completely dissolved in the methanol. The mixture formed is called sodium Methoxide. The Methoxide was poured into the conical flask containing the preheated oil. The content of the conical flask was stirred with the magnetic stirrer at a steady speed and temperature of 55^{o}C. Then heating and stirring was stopped after 1 hours and the product was poured into a separating funnel mounted on a clamp stand.

3.4 PHYSICOCHEMICAL ANALYSIS/ CHARACTERIZATION

After the production of biodiesel from the crude Palm kernel oil- P.K.O, the products (biodiesel) are subjected to the following tests to know their physical and chemical characteristics.

- **Colour Test:** The colour test was carried out by visual inspection.

- **Viscosity:** The viscosity of the biodiesel was determined in the Chemical Engineering department laboratory, FUTO using the angler viscometer, and the formula:

  $V = 0.226t - \frac{19.5}{t}$

Where t = time the biodiesel drops from the viscometer

- **pH Test:** The pH meter was measured using the pH meter at room temperature in Chemical engineering laboratory.

- **Density and Specific Gravity/API Gravity:** This was calculated using the pycnometer.

  Density was calculated as:

  $\text{Density} = \frac{wt\ of\ biodiesel}{vol\ of\ biodiesel}$

  Specific gravity was calculated as using:

  $S.G = \frac{Density\ of\ biodiesel}{Density\ of\ equal\ vol\ of\ water}$

  The API gravity was calculated as:

  $API = \frac{141.5}{S.G} - 131.5$

- **Flash point, Pour point and cloud point:** These were calculated for the biodiesel and compared with standards.
- **Acid value, Iodine value, peroxide value, saponification value, Cetane number:** These were calculated for the biodiesel and compared with standards.

The acid value of the biodiesel was calculated:

$A.V = \frac{5.61*T}{W}$

Where T = volume in ml of 0.5N NaOH required for titration in ml

W = weight in grams of sample taken

Iodine value was calculated using:

$I.V = \frac{12.7\ (B-S)}{Weight\ of\ sample\ \ (g)}$

Where S = volume of thiosulphate used with oil sample

B = volume of thiosulphate without oil/ blank

The peroxide value was calculated using:

$P.V = \frac{(S-B)*N*1000}{Weight\ of\ sample\ \ (g)}$

Where S = volume of thiosulphate used with oil sample

B = volume of thiosulphate without oil/ blank

N = molarity of NaOH required for titration in ml

The saponification value was calculated using:

S.V = $\frac{28.05*(T2-T1)}{W}$

Where T_2 = volume in ml of 0.5N acid required for the blank

T_1 = volume in ml of 0.5N acid required for the sample

W = weight in gram of the sample taken

(Kumar and Kant, 2013)

Cetane Number was calculated as CN = $\frac{46.3+5458}{S.V-(0.225xI.V)}$, where S.V = Saponification value and I.V = Iodine Value.

- **mSpectroscopic analysis:** The spectroscopic analysis was done in the Department of chemical engineering (FUTO) laboratory using the UV Visible Spectrophotometer (Atomic absorption spectrophotometer (AAS)) to determine the amount of metal present.

3.5 PARAMETRIC EFFECTS

The produced biodiesel is subjected to variations of several parametric effects to know their behaviours. Examples of these effects are

- Effects of Free Fatty Acid (FFA)
- Effects of reaction temperature, catalyst concentration and time

The aim of the kinetic study is to observe the variation of amount of biodiesel yield at specified parametric conditions (temperature, catalyst concentration and time), to know the best conditions for the transesterification process. The kinetics of the alkali catalysed transesterification process was studied varying the catalyst concentration (0.2, 0.3, 0.5, 1.0, and 1.2g) at constant temperature of 60^{o}C. These parametric conditions were carried out for 30, 40, 50, 60 and 120 minutes at a methanol to oil molar ratio of 4:1 for 25 different runs, with the following experimental conditions below:

Table 3.1 Kinetic experimental parameters and condition

Experimental parameter	**Condition**
NaOH Concentration (g)	0.2, 0.3, 0.5, 1.0 and 1.2
Temperature (^{o}C)	60
Time (min)	30, 40,50, 60 and 120
Agitation speed (rpm)	200
Weight of oil (g)	50
Methanol to oil molar ratio	4:1

IV. RESULTS AND ANALYSIS

4.1 Physicochemical characterization of biodiesel produced from crude P.K.O

Table 4.1 Physiochemical characterization table of biodiesel from crude P.K.O

Characteristics	**Value**
1. Weight % yield (%)	85.03
2. Viscosity (@40^{o}C) (mm^{2}/s)	1.5398
3. Density (g/cm^{3})	0.87992
4. Colour	Light Brown
5. Specific Gravity (@60^{o}F/60^{o}F)	0.9144
6. Flash point (^{o}C)	185
7. API Gravity (^{o})	23.246
8. pH test	8.34
9. Pour point (^{o}C)	17
10. Cloud point (^{o}C)	21
11. Acid value	2.5245
12. Iodine value	0.0127
13. Peroxide value	32
14. Saponification value	199.5
15. Cetane number	27.6

Table 4.2 Fuel characterization result for crude P.K.O and petroleum diesel fuel

Fuel characteristics (Properties/Parameters)	**P.K.O biodiesel**	***Petroleum diesel**	***EN14214 European**	**#American Society of Testing**

			Biodiesel Standard	and Materials (ASTM) diesel standard
1. Viscosity (@40^{o}C) (mm^2/s)	1.5398	2.847	3.50-5.00	1.6-6.5
2. Specific Gravity (@60^{o}F/60^{o}F)	0.9144	0.853	0.86-0.90	0.82-0.87
3. Pour point (^{o}C)	17	-16	-	-
4. Cloud point (^{o}C)	21	-12	-	-
5. Flash point (^{o}C)	185	74	>120	66
6. API Gravity	23.24	30-42	-	150
7. Cetane number	27.6	40-55	51-60	47

*(Alamu et al., 2008)
(Eze et al., 2013)

Table 4.3 Metal contents presents in P.K.O biodiesel and values reported in literature

Metals	AAS results obtained for P.K.O biodiesel (%)	*Literature values (%)
Iron (Fe)	2.0	1.22
Copper (Cu)	0.001	-
Lead (Pb)	33.9	-

* (Aladetuyi et al., 2014)

4.2 Results from kinetic demonstration
4.2.1 Results from parametric effect
4.2.1.1 Effects of free fatty acid

The oil should be tested to ascertain reduction in FFA and this is done by esterification, if the FFA is still greater than 0.5%-1%, another round of esterification should be done and if the FFA less than 0.5%-1%, then transesterification can occur. The need for acid catalysed esterification reaction is to avoid the formation of soap while carrying out alkali catalysed transesterification while carrying out alkali catalysed transesterification on reaction.

Table 4.4 FFA Experimental runs after Esterification

FFA EXPERIMENTAL RUNS AFTER ESTERIFICATION	FFA VALUE
1ST RUN (%)	23.8425
2ND RUN (%)	5.3295
3RD RUN (%)	2.805%
4TH RUN (%)	0.8976%

Therefore, with an FFA (0.8976) greater than 1%, the transesterification reaction was carried out.

4.2.1.2 Effects of reaction temperature, catalyst concentration and time

The variations of parametric effect (example temperature, time, catalyst concentration and FFA etc) on the reaction, helped to determine the optimum condition for transesterification reaction. The below is the condition for the parametric effect:

Table 4.5 Experimental parameters and condition

Experimental parameter	Condition
NaOH Concentration (g)	0.2, 0.3, 0.5, 1.0 and 1.2
Temperature (^{o}C)	60
Time (min)	30, 40,50, 60 and 120
Agitation speed (rpm)	200
Weight of oil (g)	50
Methanol to oil molar ratio	4:1

4.2.2 Variation of biodiesel yield with time

After the experimental runs it was discovered that an increase in reaction temperature speeds up the reaction rate and shortens the reaction time, hence, the biodiesel yield increases in reaction time. The plots of yield (%) against time (min) are as follows:

Table 4.6 Results from kinetic runs

Experimental runs	Catalyst conc. (g)	Time (min)	Mass of biodiesel (g)	Yield of biodiesel (%)
1	0.2	30	28.210	56.420
2	0.2	40	26.440	52.880
3	0.2	50	29.081	58.162
4	0.2	60	28.887	57.774
5	0.2	120	31.116	62.232
6	0.3	30	33.122	62.244
7	0.3	40	36.033	72.066
8	0.3	50	41.005	82.070
9	0.3	60	40.780	81.560
10	0.3	120	43.522	87.044
11	0.5	30	42.100	84.220
12	0.5	40	43.001	86.002
13	0.5	50	43.061	86.122
14	0.5	60	43.121	86.242
15	0.5	120	43.480	86.962
16	1.0	30	44.510	89.020
17	1.0	40	45.010	90.020
18	1.0	50	44.681	89.362
19	1.0	60	46.110	92.220
20	1.0	120	46.215	92.430
21	1.2	30	30.520	61.040
22	1.2	40	31.507	63.014
23	1.2	50	29.210	58.420
24	1.2	60	32.123	64.246
25	1.2	120	34.301	68.602

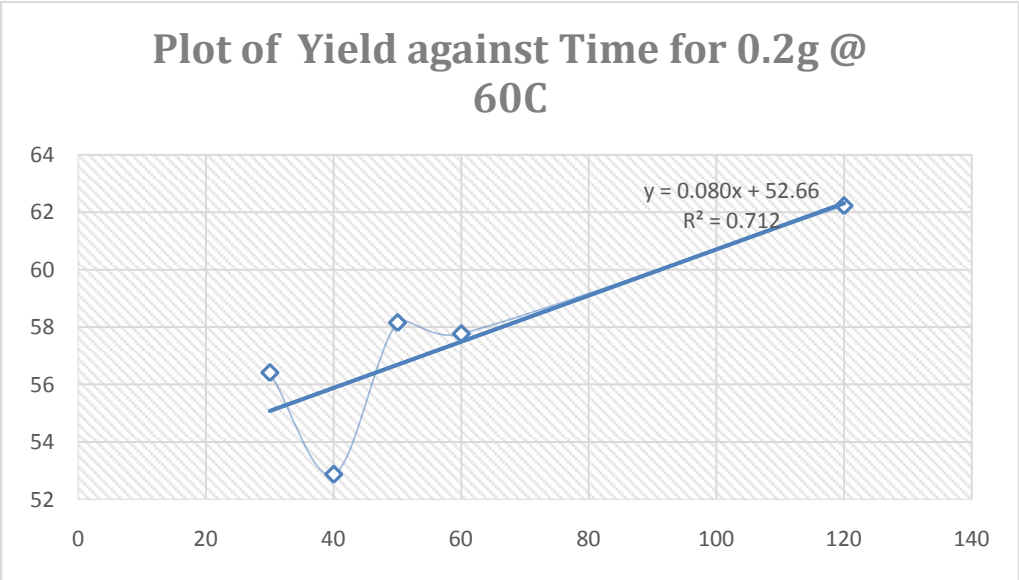

Figure 4.1: Graph of 0.2g NaOH @60^{o}C

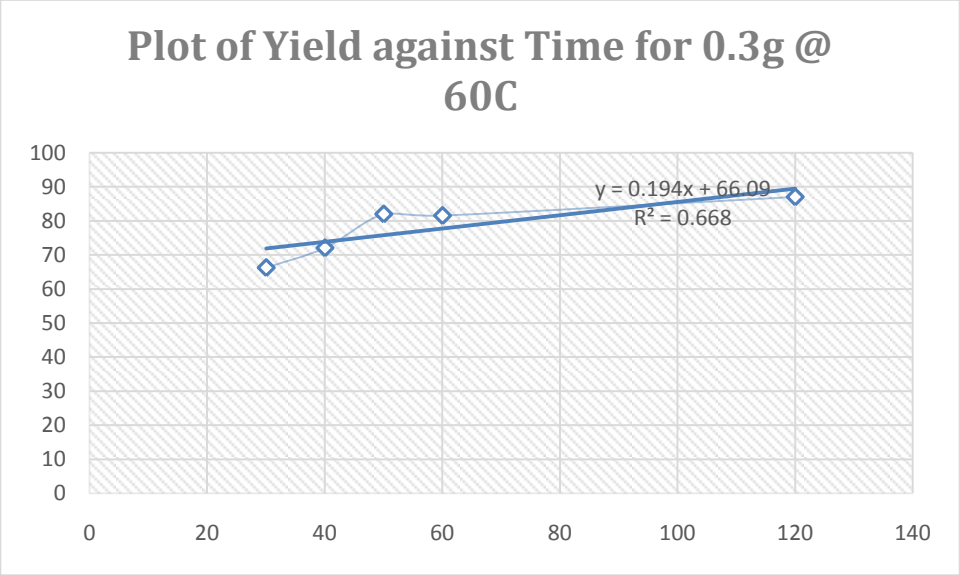

Figure 4.2: Graph of 0.3g NaOH @60^{o}C

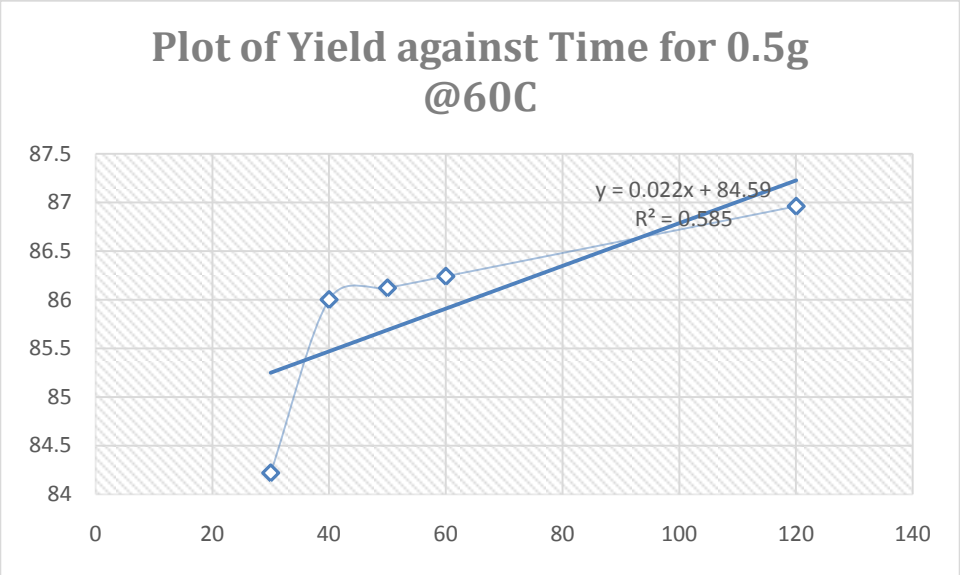

Figure 4.3: Graph of 0.5g NaOH @60^{o}C

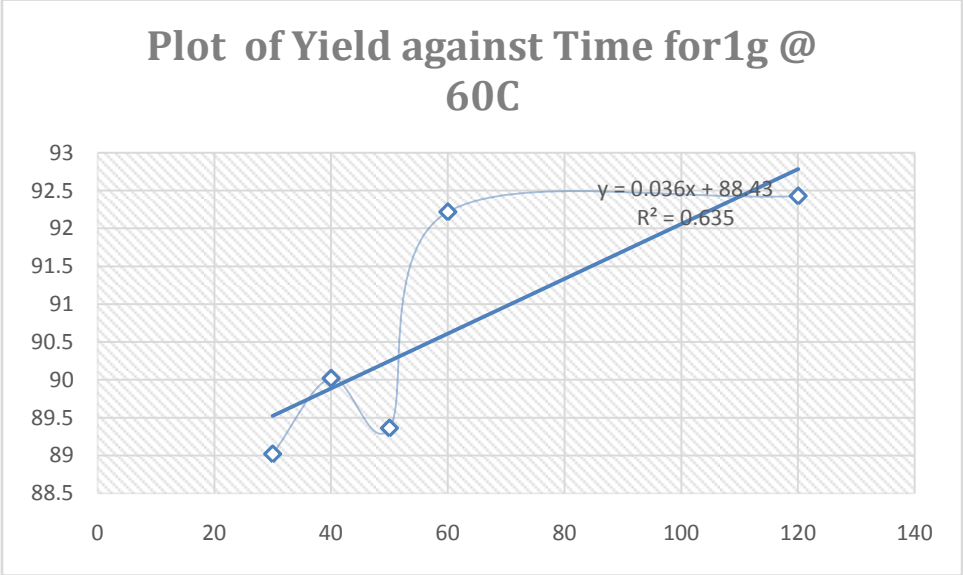

Figure 4.4: Graph of 1g NaOH @60^{o}C

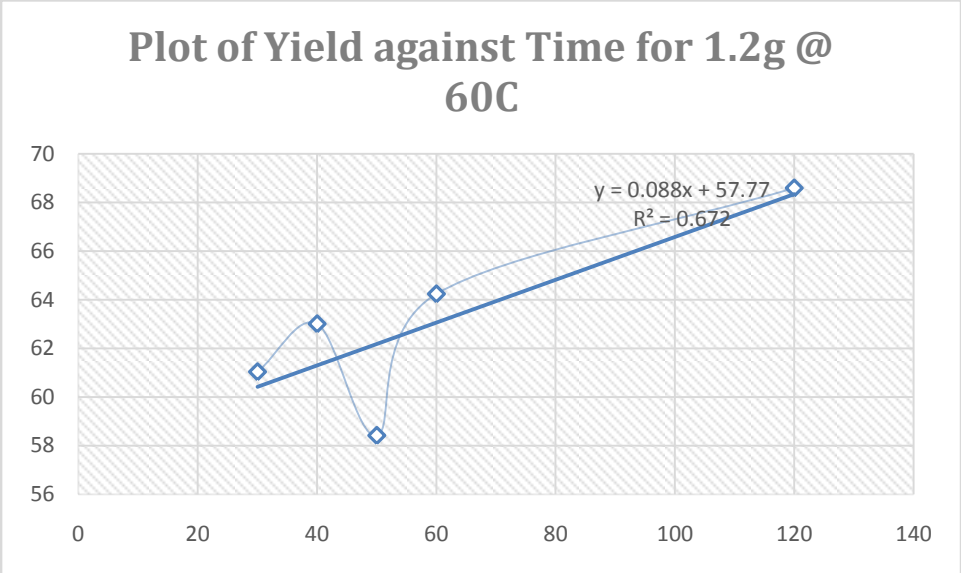

Figure 4.5: Graph of 1.2g NaOH @60^{o}C

Table 4.7: Summary from kinetic plots

Plots	**Temperature (^{O}C)**	**Concentration (g)**	**R^2**
1	60	0.2	0.7120
2	60	0.3	0.6686
3	60	0.5	0.5850
4	60	1.0	0.6350
5	60	1.2	0.6720

From the kinetic plots, figure 4.1 to figure 4.5 shows the relationship between yield and time. The plots shows that there is an increase in biodiesel yield with time, There is a slight difference in the biodiesel yield for a catalyst concentration of 0.5g and 1g. The yield of a 0.5g and 1.0g catalyst concentration, for a methanol to oil ratio of 4:1 ranges from 80 to 90%. Hence, the best optimum parameter for a methanol to oil ratio of 4:1 is a catalyst concentration 0.5g and 1g at a constant temperature of 60^{o}C. Moreso, since the high content of FFA in the palm kernel oil will greatly reduce the biodiesel production rate in an alkali-catalyzed transesterification

process; esterification was used to effectively decrease the FFA content prior to the alkali-catalyzed transesterification. This reduction in FFA helped avoid producing an unwanted product (soap), formed as a result of excess fatty acid and NaOH catalyst. Hence, the presence of FFA caused the conversion rate to drop and made the separation process difficult because of the soap formation.

V. CONCLUSION/RECOMMENDATION

From the kinetic study results, it was observed that biodiesel yield from palm kernel oil (P.K.O), increases with reaction time and is best for the condition 0.5g and 1g for increased temperature of 60^{O}C. The reaction conversion rate increased as the concentration of catalyst increased. The maximum FAME content obtained ranged from 80 to 90% regardless of the catalyst concentration and temperature. This work proposed a new reaction system for transesterification of biodiesel production from palm kernel oil (P.K.O). The data obtained and used for analysis were completely empirical, and they can be used in industrial design and for kinetic studies of parametric effect of transesterification reaction. Hence, hydrodynamics, catalyst strength and nature of feedstock are essential to take proper note of before transesterification commences.

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