Optimization and Characterization of Biodiesel Production

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Abstract: The research focused on the application of used vegetable oil as feedstock for the production of biodiesel. A thorough examination of the results obtained in this research revealed that the produced biodiesel is more environmentally friendly because it has a lower carbon residue content than standard biodiesel with minimal effects on the ozone layer depletion due to its combustion. The feedstock (used vegetable oil) was sourced from eateries and restaurants with methanol and potassium hydroxide used as alcohol and catalyst respectively. 800ml of feedstock was filtered, activated, and warmed up for efficient and proper mixing with 4.65g of potassium hydroxide (KOH) and 100ml of methanol through the trans-esterification process that yields high biodiesel product and glycerine as a byproduct. The products further undergo separation process due to density variation, and biodiesel settled as the filtrate while the denser product glycerine settled as the bottom product (residue). The biodiesel was washed to remove the impurities present and then heated at a temperature of 110°C in order to dry up any entrained water content. In addition, the produced biodiesel was characterized and evaluated to obtained its properties such as density of 0.980kgm^{-3} at 15℃, calorific valuer of 40073KJ/kg, kinematic viscosity of 4.0mm^2s^{-1} at 40ºC, moisture content of < 0.01, flash point, pour point and cloud point are 169ºC, -12ºC and -5 ℃ respectively. Also, the sulphur content of 0.0017% by mass, carbon residue of 0.18% by mass, fire point of 173, cetane number 54 and refractive index of 1.83 respectively. Comparison between produced biodiesel with diesel and petrol fuel in terms of: kinematic viscosity (mm^2s-^{1}), density (kgm^{-3}), pour point, cloud point, moisture content (% vol), sulphur content (% mass), carbon residue (% mass), heating value (KJ/kg), refractive index, and cetane number were relatively close.

Keywords: Feedstock, Biodiesel, Characterization, Trans-esterification, Optimization

I. Introduction

Biodiesel is the main alternative source of energy in Indonesia and its potentials is developed already (Adeloye & Igbagara, 2024). The estimated consumption of CPO (crude palm oil) in 2015 that produces margarine and cooking oil was around 5.9 million tons or 54.63percent of the total production (Alnuami *et al.,* 2014, Fangyuan & Haeng, 2025). Biodiesel is actually derived from fat and oil either through chemical or mechanical process. Biodiesel can be processed through four main forms namely, transesterification, micro emulsion, blending, and pyrolysis (Adeloye *et al.,* 2022). Biodiesel process that uses the two-stage processing are esterification and transesterification which requires twice consumption rate of methanol (Meng *et al.,*2008*)*. Besides used vegetable oil is usually discarded freely into the environment is now being converted into useful product (Van Gerpen*,* 2005). Biodiesel is a diesel substitute that can be gotten from different oil, fat and greases. It is defined as a form of fuel extracted from plants or animals consisting of long chain fatty acid esters. It is used alternatively for

petrol diesel production because it is renewable energy, non-toxic and biodegradable (Fadhil *et al.,* 2012; Osman *et al.,* 2024). The use of solvent is employed for extraction of the oil in the chemical method (Yujin *et al*., 2014). Solvent vaporization is used to remove the oil. In other to remove the solvent from the oil, the mixture is heated to150^oC, after which the solvent is recycled (Zullaikah *et al*., 2009). Malaysia, Thailand, Colombia, and Nigeria, are the biggest palm oil producers (Lam *et al.,* 2019). Full production of palm fruit reaches 8 years after planting palm fruit contains 50% oil. Per hectare yield within 3000-5000 kg of pulp oil and per hectare produces 600-1000 kg of kernel oil (Murillo, 2003). It has been reported that cetane number of palm biodiesel has a range value from 42 to 62 (Mittelbach, 2004), which exceeds the value of conventional diesel. However, the value of biodiesel cetane number can be limited to minimum of 41. Tropical areas with temperature range not below 25ºC, therefore a greater proportion of palm biodiesel can be used (Rashid *et al*., 2008). Both palm kernel oil and palm oil are raw materials for the production of biodiesel. Food uses less of Palm kernel oil (PKO) (Predojevic, 2008). With the rapid attention toward the causes of pollutions through fossil fuels such as petrol diesel, natural gas and coal, alternatively fuels and sources of renewable energy like biodiesel are coming in diverse fashioned (Garlapati *et al.,* 2013). It was observed that waste cooking oils is more affordable in commercial quantity than any other because of the economic value as a raw material for the production of biodiesel. (Predojevic, 2008).

Biofuel produced by micro-emulsification yields higher carbon deposits on the injectors, intake valves and cylinder liners when using engines (Liaquat *et al.,* 2013). Thermal cracking of vegetable oil to biofuels yields alkanes, alkenes, alkadienes, aromatics and carboxylic acids in different proportions (Ibrahim *et al*., 2019). Transesterification involves the transformation of fatty acid chain of triglyceride molecules in oil samples into methyl or ethyl esters in the presence of a catalyst mixture and alcohol (Folayan *et al.,* 2019). Considerable amount of glycerol is released when peanut oil is trans-esterified at 6:1 molar ratio using ethanol. However, glycerol quantity has decreased when 1:3 oil to alcohol molar ratio is utilized for transferring peanut oil into ethyl ester. (Mittelbach & Schober, 2003). Thus, the aim of this research is to optimize and characterize biodiesel production via transesterification process via biodiesel production from used vegetable oil with the application of potassium hydroxide as catalyst, produced biodiesel properties characterization, its optimization through parametric analysis and its comparative with convectional and other sources of diesel production.

II. Methods

2.1 Transesterification

This involves the transformation of fatty acid chain of triglyceride molecules in oil samples into methyl or ethyl esters in the presence of a catalyst mixture and alcohol (Folayan *et al.,* 2019). Ethyl or methyl esters are gotten with similar properties with different conventional diesel fuels. Glycerol is the main byproduct obtained. Ethanol is the most common alcohol used for the production of biodiesel, this is because of its availability and conversion rates. Plant-based ethanol, propanol, isopropanol, and butanol can also be applied.

2.2 Experimental Procedure for Production of Biodiesel

Used vegetable oil collected was first filtered into a container, after which it was placed on a hot plate for pre-heating at 60°C to eliminate or remove any moisture or water content present. Then an empty container was weighed and its weight recorded (0.84g), then KOH (potassium hydroxide) was added into the weighed empty container and re-weighed (5.49g). Thus, 4.65g of potassium hydroxide was poured into 100mls of alcohol in a separate container and stirred continuously for even mixture, stirring stops when KOH dissolved and methoxide compound formed as a result of this. After complete mixing, the methoxide compound was then introduced into a beaker which contained the pre-heated used vegetable oil, and was allowed to mix properly via a magnetic stirrer for 2 hours, within a temperature range of 55-60ºC after which the mixture was allowed to cool for 24 hours to separate into biodiesel and glycerine. Trans-esterification process resulted into biodiesel and glycerine as the byproduct and due to density differences between glycerine and biodiesel, the

bottom product is usually glycerine content while the top product contained the biodiesel, which are separated using separation funnel. Therefore, repeated washing of the biodiesel is initiated with a warm distilled water to remove the remaining contents of methoxide. Thus, washing process was carried out for a while and once the washing process is complete, the biodiesel is therefore separated from the water. The produced biodiesel is then heated to 110^{o}C in a hot plate to remove water content prior to analysis or characterization process.

2.3 Characterization of Produced Biodiesel

2.3.1 Determination of Density and Specific Gravity

About 10ml of the sample was utilized to analyze the specific gravity. The empty bottle was pre-weighted at first, before filling it to the brim with the fuel sample for final weighing of the sample and this was done at a room temperature of 15°C and 20°C, and the process was done with water and the actual density and specific gravity. This was done after the weighing balance error has being checked.

2.3.2 Determination of the Cloud Point

Part of the sample was added in a test tube and then inserted or placed inside the steam bath and the temperature was calibrated below 10°C. Checking of the sample cloud point is done at an interval of every 3°C by bringing out the sample and checked for the cloudiness of the placed sample, this was observed closely at every interval. With this process, the sample was observed to form a cloud of gel and the temperature reading was deduced and referred as the cloud point.

2.3.3 Determination of Pour Point

Ultra-low temperature refrigerator of -800°C was used to determine the pour point of the sample with a test tube, the sample was kept in it. Fluidity checking was carryout at an interval of 5°C by bringing the sample out for checking. The liquid will cease to flow at a particular temperature, and such recorded temperature is known as the pour point.

2.3.4 Determination of Flash Point

About 30ml of the sample was obtained into Pensky Martin Apparatus cup and thermometer was fitted. The thermometer was immersed in a way that it does not contact the bottom of the cup containing the sample, continuous stirring was performed during this process. In every 10°C rise in temperature, the sample vapour is exposed to a flame. The temperature at which the exposed fume got ignited by the lighted match stick is the flash point.

2.3.5 Determination of Fire Point

The fire point of a fuel is the lowest temperature at which the vapour or air mixture of that fuel will continue to burn for at least five seconds after ignition by an open flame of standard dimension. Similarly, the fire point is defined as the lowest temperature at which vapours of the material will catch fire and continue burning even after the ignition source is removed. The fire point is usually higher than the flash point because the vapours produced at the flash point are not sufficient enough to maintain ignite fume, besides flash and fire points depend upon the volatility of the biodiesel.

2.3.6 Sulphur Content

Sulfur is a natural component in oil that is present in gasoline and diesel unless removed. Fuel contains sulfur which is derives from the original oil source and can still be present after refining. After combustion in the engine burning chamber, the sulfur in fuel forms particulates which is the primary contributor to air pollution and the cause of harmful corrosion in the engine.

2.3.7 Moisture Content

Using moisture analyzer (Sartorius MA35) to determine moisture at 105^{o}C as the total moisture obtained gravimetrically through the solid sample measuring weight loss with temperature increase until there is no any weight loss within the temperature.

2.3.8 Cetane Number

The cetane number which is the ability of fatty acid methyl esters as a fuel to ignite quickly after being injected. The higher its value, the better its ignition quality, this parameter is highly considered during the selection of fatty acid methyl esters for use as a biodiesel.

2.3.9 Kinematic Viscosity Test

The viscosity of sample taken using the old oil glass viscometer, using the mouth to suck the biodiesel in the lower bulb to a point above the top white ring mark which is the second bulb of the old glass viscometer. The biodiesel meniscus was adjusted by releasing the thumb until it is at the same level with white ring mark on top of second bulb. The biodiesel was allowed to flow and a stop watch was used to take the time interval of the flow within the temperature of 40**ºC**. The kinematic viscosity is calculated from the results obtained.

2.3.10 Refractive Index

Proportionality of the speed of light in the absence of air and in oil is usually determined by refractive index. Also, the proportion existing between the sine refraction whenever the wave-length (usually 589.3mu) moves from the air into an oil. It is important in purity and identification purposes, and it is used in obtaining progress of a reaction such as isomerization and catalyst hydrogenation. Binary esters have been excessively analyzed through this process.

2.3.11 Carbon Residue

The sample is first distilled (ASTM D86) until 90% of the sample has been recovered. The residue is weighed into a special glass bulb and heated in a furnace to 550ºC. Most of the samples evaporates or decomposes under this condition. The bulb is cooled and the residue weighed.

2.4 Gas Chromatograph and Mass Selective Detector Analysis

Gas Chromatographs (GC's) is purpose-built gas monitors that provides specific information (composition of gas stream) of the sample such as qualitative (species) and quantitative (amount) of the sample. Chromatographic analysis procedure consists of four steps that include sample collection, sample injection, sample separation, and sample detection. Agilent 6890N Gas Chromatograph and Agilent 5975 Mass Selective Detector were used to further characterize the produced biodiesel b collecting the sample through gas syringe. The collected sample was then introduced or injected into the carrier gas with a syringe automatically, although it is introduced via a sample loop and analytical valve which are in-line with the carrier stream. The liquid is then volatilized into a gas, which the columns separate the sample into its constituent components and the different phases or components are separated based on boiling point, polarity, molecular weight and molecular size etc. As the separated gasses leave (or elute from) the column(s), they pass through a detector which, in turn, responds with an output signal. This signal is what generates the characteristic GC peaks in a chromatogram form in this research.

III. Results and Discussion

3.1 Production and Characterization

The properties of the biodiesel produced are presented in Table 1. The table shows results of the produced biodiesel properties such as density of $0.980 kgm^{-3}$ at 15℃, a heating value of 40073KJ/Kg, kinematic viscosity of $4.0 mm^2 s^{-1}$ at 40ºC, a moisture content of <0.01. The produced biodiesel flash, pour and cloud points are 169ºC, -12ºC and -5℃ respectively, and the content of sulphur and carbon residue (%mass) of the produced biodiesel are 0.0017 and 0.18 respectively. The fire point of 173, cetane number 54 and refractive index of 1.83 respectively. All are within the acceptable or standard value.

Table 1: Physical Properties of Produced Biodiesel

Produced biodiesel	Value

Heating value (KJ/Kg)	40073
Flash point (°C)	169
Kinetic viscosity (mm^2s^{-1}) @ 40 °C	4.0
Density (specific gravity) (15 °C, kgm^{-3})	0.980
Sulphur content (%mass)	0.0017
Carbon residue (%mass)	0.18
Pour point (°C)	-12
Cloud point	-5
Moisture content (%vol)	<0.01
Fire point	173
Refractive index	1.83
Cetane number	54

The results highlighted in Table 1 were compared with other biodegradable feedstock sources for biodiesel production as shown in Table 2. The produced biodiesel has a density of (15°C, kg/m^3) of 0.980, which has 7.1percent and 11.2percent above the density of cottonseed and mustard biodiesel whose values are 0.910 and 0.870 respectively. The produced biodiesel has a kinematic viscosity value of 4.0, which is 10percent and 7.5percent higher than the kinematic viscosity of cottonseed and mustard biodiesel. Also, the heating value of the produced biodiesel is higher than the heating value of cottonseed and mustard biodiesel with 0.18percent and 1.3percent respectively. The flash point of produced biodiesel is 5percent and 14.2percent higher than that of cottonseed and mustard biodiesel. The produced biodiesel has -12^{0}C as its pour point which is 33percent higher than that of cottonseed, but equal with that of mustard which is -12^{0}C, while the cloud point is also 40 percent and 20 percent respectively above that of cottonseed and mustard biodiesel. It is also observed from this research that both produced biodiesel, cottonseed and mustard oil with no sulphur content, while the carbon residue of the produced biodiesel has 93percent and 92percent higher than that of cottonseed and mustard biodiesel. The refractive index of the produced biodiesel is 26percent and 20percent more than biodiesel of cottonseed and mustard biodiesel, while the cetane number of the produced biodiesel is 1.9 percent lower than that of cottonseed biodiesel and same 1.9 percent higher than that of mustard biodiesel. The fire point of cottonseed and mustard biodiesel happened to be lower with 4.6 percent and 13.3 percent than that of produced biodiesel, and finally the moisture content of cottonseed and mustard biodiesel are higher than that of produced biodiesel with 50 percent each.

Table 2: Comparison of Produced Biodiesel with Biodiesel from other Bio-sources

Characteristics	**Produced biodiesel**	**Cottonseed biodiesel**	**Mustard biodiesel**
Kinematic viscosity (mm^{2}s^{-1}) 40(°C)	4.0	3.6	3.7
Density (specific gravity) 15 °C, kg/m^3	0.980	0.910	0.870
Flash point (°C)	169	160	145
Pour point (°C)	-12	-8	-12
Cloud point (°C)	-5	-3	-4
Moisture content (% vol)	< 0.01	0.020	0.020
Sulphur content (%mass)	0.0017	-	-
Carbon residue (% mass)	0.18	0.0112	0.0138

Heating value (KJ/Kg)	40073	40000	39542
Refractive Index	1.83	1.34	1.46
Fire Point	173	165	150
Cetane number	54	55	53

Similarly, the produced biodiesel properties were compared with diesel fuel and petrol fuel as highlighted in Table 3 with little deviations for most of the characterized properties. Furthermore, the heating values of diesel fuel and that of petrol fuel seems to be higher that of the produced biodiesel with 15.1percent and 6.81percent respectively, while the refractive index of the produced biodiesel is higher than that of diesel fuel and petrol fuel with 8.2 percent and 16.9 percent respectively. Both the fire point of diesel fuel and petrol fuel are very low with 166 percent each compare to that of the produced biodiesel. The cetane number of produced bio-diesel falls within 54, while that of diesel fuel and petrol fuel falls within 48 and 53 respectively, shows that the produced biodiesel has a higher cetane number. The burning quality of the produced biodiesel is 11.1percent and 1.8percent higher that diesel fuel and petrol fuel.

3.2 Gas Chromatography Analysis

From GC and MS analysis result, it was observed that some certain fatty acids dictated in the oil such as Mono-unsaturated Fatty acids (MUFA) Myristoleic 0.56 percent Palmitoleate 0.05 percent oleic acid 0.05 percent Eicosenoic acid 0.12percentErucate 0.14 percent Nervonic acid 0.04 percent which showed the element above that a perfect biodiesel would be made only from monounsaturated fatty acids (MUFAs). An ideal biodiesel composition should have fewer polyunsaturated like Eicosadienoic acid 0.28 percent Eicosatetraenoic acid 0.31 percent docosapentaenoic acid 0.19percent and docosahexaenoic acid 0.06 percent and saturated fatty acids (SFAs) like Octanoic acid 0.18 percent, Pelargonicacid 0.05 percent, Capricacid 0.16 percent, Undecylicacid 0.04percent Lauricacid 0.10 percent and Tridecylicacid 0.15 percent. High levels of polyunsaturated fatty acids (PUFAs) would negatively impact the oxidative stability, which do not suit diesel engines. On the contrary, biodiesel derived from vegetable oil has saturated fatty acids have good oxidative stability, but poor fuel properties at low temperatures, which is a disadvantage in winter operation.

Table 3: Comparison of Produced Biodiesel with Conventional Diesel

Characteristics	**Produced biodiesel**	**Diesel fuel**	**Petrol Fuel**
Kinematic viscosity (mm^2s^{-1}) @ 40 (°C)	4.0	3.9	3.8
Density (specific gravity) @ 15 °C, kgm^{-3}	0.980	0.850	0.861
Flash point (°C)	151	63	68
Pour point (°C)	-12	-16	-19
Cloud point (°C)	-5	-12	-14
Moisture content (% vol)	<0.01	0.02	0.02
Sulphur content (%mass)	0.0017	0.45	0.35
Carbon residue (% mass)	0.18	0.19	0.17
Heating value (KJ/Kg)	40073	47200	43000
Refractive Index	1.83	1.68	1.52
Fire Point	173	65	65
Cetane number	54	48	53

3.3 Parametric Effects on Biodiesel Yield Optimization

3.3.1 Effects of Methanol

Methanol used in this research contributed greatly to the proper mixing with KOH and the used vegetable oil, due to its solubility nature in KOH and its quick reaction with triglyceride which helps in reducing the reaction time of the study. Also, an improved biodiesel product yield was achieved as a result of proper mixture.

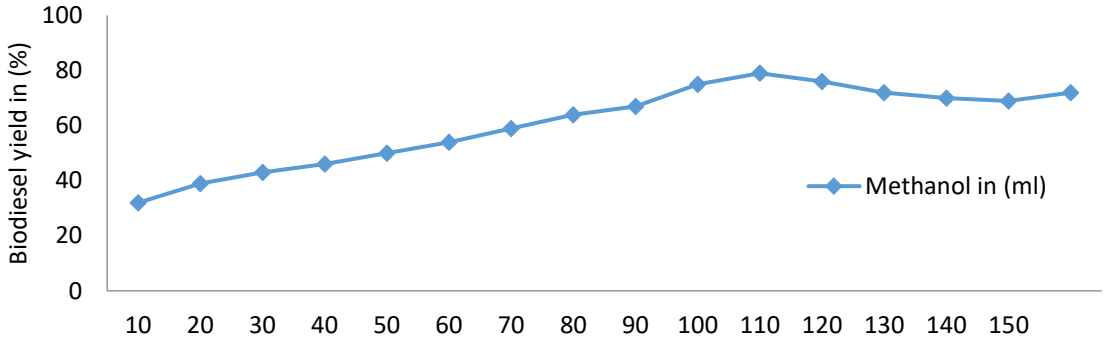

Figure 1: Effect of Methanol against Biodiesel Yield

The volume of the methanol affects biodiesel yield, although the volume of the feedstock (used vegetable oil) is of importance too. If the volume or quantity of the methanol applied is high, poor yield of biodiesel may be observed. Thus, 100ml volume of methanol was applied in this research with 75 percent yield of biodiesel product.Trans-esterification is very effective when methanol is used with potassium hydroxide as a catalyst, because the solubility of the alcohol in the oil medium increases, and enhanced yielding of the needed byproduct.

3.3.2 Temperature

The temperature progression of this research is shown in Figure 2, which shows that the temperature range of 55- 60^{o}C favoured forward reaction that produces more yield of biodiesel product. The temperature at certain degree attenuates due to the boiling point of methanol, besides a very low temperature will result in low yield of biodiesel. Therefore, the need to have a control temperature that will not be above the boiling point is of importance. Therefore, the temperature range for this research is between 55 – 60^{o}C and the average temperature applied was 58^{o}C with 75 percent yield of biodiesel observed. Also, the main factor which affect the production of biodiesel is the reaction temperature. Hence, increase in reacting temperature also speed up the reaction and reduces the reaction time because of the decrease in viscosity of the oils. If the reacting temperature is allowed to go above the optimal level such will reduce the yield of biodiesel at the end.

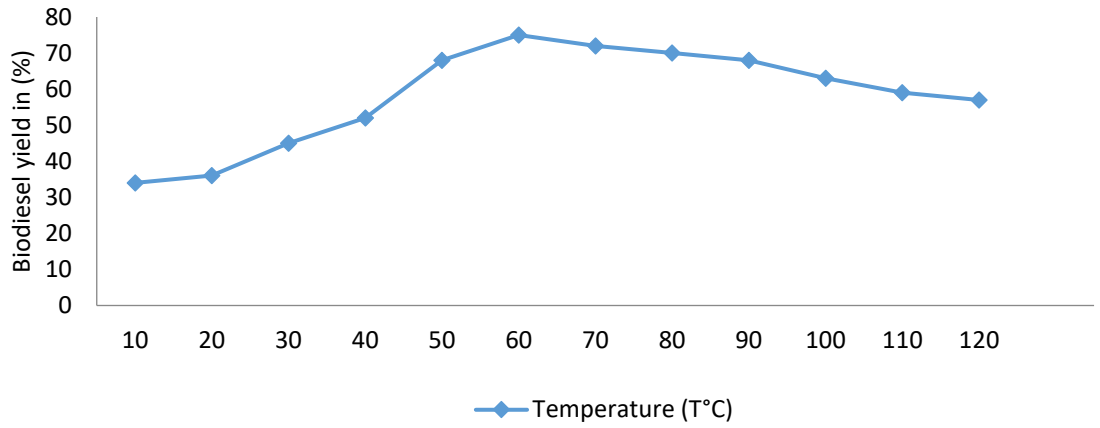

Figure 2: Temperature Effect against Biodiesel Yield

3.3.3 Potassium Hydroxide as a Catalyst in Biodiesel

Good biodiesel uses KOH as its caustic catalyst. It produces a high-quality fuel and is easy to mix with methanol, plus its by-product can be used to make quality liquid soap or fertilizer.

It is important to note that KOH is highly hygroscopic thereby absorbing moisture content from the air rapidly, and too much moisture will interfere with the biodiesel process. However, with the super-critical methanol production, this issue can be solved. In this method, the chemical process of creating bio-diesel is carried out under high temperature and pressure, which eliminates contamination of fuel with water.

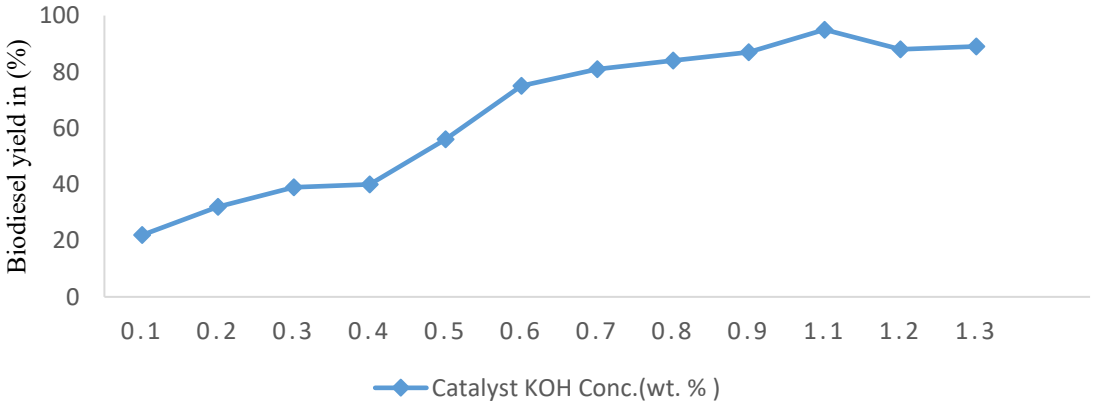

Figure 3: Effect of Catalyst (KOH) against Biodiesel Yield

It can be deduced from Figure 3 that very low KOH concentration gives small amount of biodiesel yield, but as it increases the yield, and at a point the yield concentration started attenuating due to excess amount of KOH concentration.

3.3.4 Reaction Time

Reaction time is an important parametric property with respect to the yield of produced biodiesel. The mixture was allowed to react for 2hrs, before allowing it to separate into biodiesel and glycerol through density variations. Hence, 75 percent yield of biodiesel was recovered at the total reaction time of 120mins as depicted in Figure 4

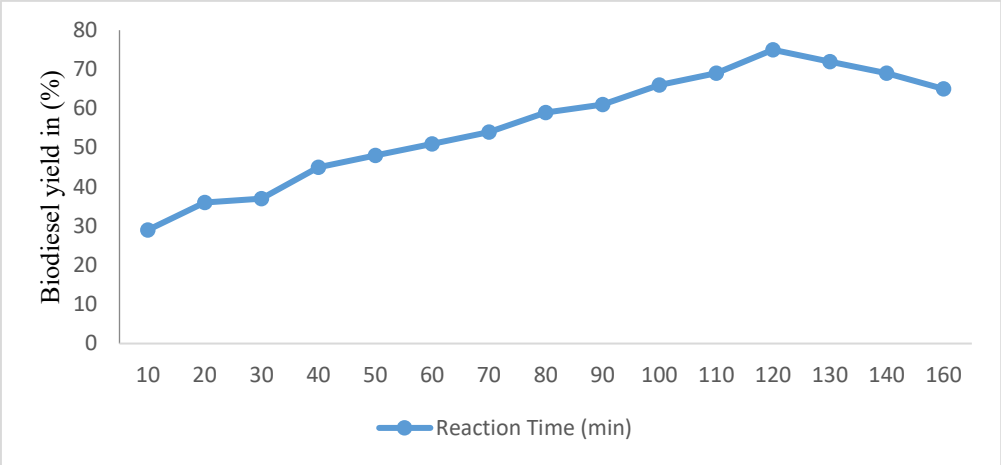

Figure 4: Effects of Reaction Time against Biodiesel Yield

3.3.5 Stirring Rate

For effective production of biodiesel, temperature and stirring speed are important parametric properties that are required to optimized biodiesel yield. The effect of stirring on biodiesel production is a factor that needs not to be neglected.

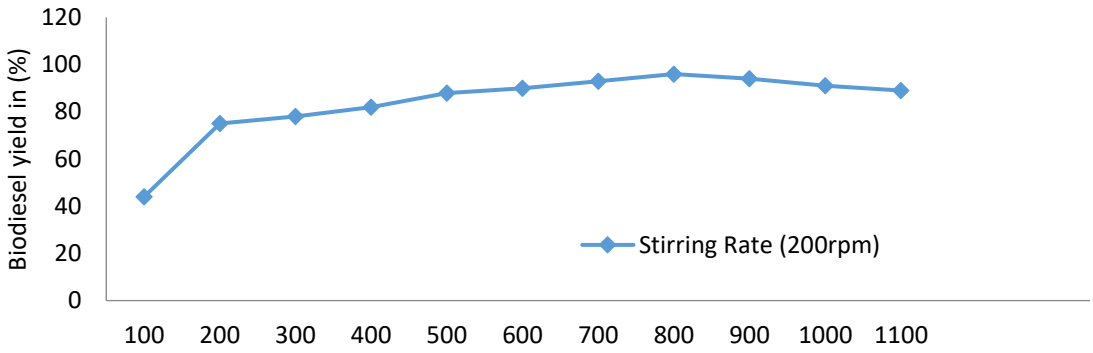

Figure 5: Effect of Stirring Rate against Biodiesel Yield

From this study, the speed velocity of 200 rpm of the magnetic stirrer was selected due to the volume of the mixture which required a moderate stirring and to avoid splitting of the samples, and also for effective and improved biodiesel yield as shown in Figure 5. Poor yield of Biodiesel may be experienced due to poor mixing of the samples and also as a result of selecting a very low or very high rpm.

IV. Conclusion

The research achieved its set objectives through the application of feedstock (used vegetable oil) used to produce biodiesel using potassium hydroxide as catalyst. This was carried out by transesterification that produced biodiesel and glycerine as the product. The produced biodiesel was then separated from the glycerine based on their density variation, washed with distilled water to remove methoxide content that acts as impurity and then heated in a hot plate at a temperature of 110^{o}C to remove moisture content before its characterization. Characterization was done on the produced biodiesel in order to determine its density (0.980 kg/m^{3}), flash point (169^{o}C), kinematic viscosity (4mm^{2}s-^{1}), sulphur content (0.0017%mass), carbon residue (0.18%mass), pour point (-12^{o}C), cloud point (-5), moisture content (< 0.01%vol), fire point (173), refractive index (1.83), cetane number (54), and heating value (40073 KJ/kg). Hence, comparison of the produced

biodiesel with biodiesel from other sources such as cotton seed and mustard biodiesel was done and it was observed that the differences in physical properties were not significantly different from each other except for carbon residue whose values for produced biodiesel, cotton seed biodiesel and mustard seed biodiesel were 0.18%mass, 0.0112%mass, and 0.0138%mass respectively. Also, produced biodiesel was compared with conventional diesel and petrol in terms of physical properties and the deviations between their values were not significant except for the flash point, sulphur content and fire point. In addition, optimization of biodiesel yield through parametric properties such as methanol, temperature, catalyst and reaction time were studied and the results obtained showed that an increase in parametric properties optimizes biodiesel yields.

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