

Production of Biodiesel, Its Characterization and Parameteric Effects Via Used Vegetable Oil

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Abstract - The research study focused on the application of used vegetable oil as feedstock for the production of biodiesel as an alternative energy source. Thus, bio-diesel production and characterization from used vegetable oil as substrate or feedstock via transesterification process was investigated in this study. The feedstock (used vegetable oil) collected from eateries and restaurants at Rukpakwulusi New Layout, Eliozu, Port Harcourt, Rivers State, Nigeria, was sent for pre-treatment to remove dirt and decanted after which it was placed on a hot plate for pre-heating to eliminate or remove water content. The feedstock was processed through transesterification process in the presence of methanol and potassium hydroxide as catalyst to yield biodiesel. The physicochemical and fuel properties of the produced biodiesel distillates that include pH value, specific gravity, refractive index, flash point, pour point, cloud point etc were evaluated and the results are in tandem with other previous studies and within the acceptable range. The GC and MS analysis of the produced biodiesel showed the presence of polar compounds, aromatic compounds, range of hydrocarbon and fatty acid methyl ester with optimum yield of 75% after 120 minutes of operational time. In addition, parametric effects such as molar ratio of methanol, temperature, catalyst, reaction time and stirring rate were studied or investigated on their effects on the yield of produced biodiesel.

Keywords - Used oil, Transesterification, Biodiesel, Cetane number, ASTM distillation, Gas chromatography.

I. INTRODUCTION

As a result of increase in consumption of energy, future decline prediction of oil production, greenhouse effects of fossil fuel, and decline in petroleum production, these have led to the upward review of oil price worldwide (Akpan *et al*., 2023; Leke *et al*., 2023). Decrease in petroleum reserve is a noticeable challenge that is attached to this kind of energy from fossil, and there is need for sorting for an alternative source of energy due to these associated constraints of fossil fuel and its products by exploring both new and renewable energy sources (Adokiye *et al.,* 2020; Igbagara *et al.,* 2021; Adeloye *et al*., 2022; Zhang *et al*., 2002; Sreelekha *et al.,* 2024). Biodiesel serves as the main substitute or replacement for fossil fuel like diesel oil (Adeloye *et al.,* 2022). Used vegetable oil serves as feedstock or raw material for the production of biodiesel, and this is abundantly available as an estimation can be deduced from an increase in the growth in palm oil from 2004-2014 which increased about 11.09percent per year. 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*)*.* Biomass resources emits little or no pollutants due to their less content of nitrogen and sulphur and several technologies have been developed to derive bio-oil from biomass raw materials and also possible upgrading techniques such as pyrolysis, gasification, liquefaction, hydro-treatment etc. (Igbagara *et al.,* 2021)

Biodiesel from crude palm oil (CPO) has a high cetane number which is considered as the characteristic of ignition quality. Besides, 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, and with global production of palm oil within 71.7 Million tonnes (Ibrahim *et al*., 2019). Biodiesel is a diesel substitute that can be gotten from different oil, fat and greases, and defined as a form of fuel extracted from plants or animals consisting of long chain fatty acid esters. It is used alternatively for fossil diesel production because it is a renewable energy, non-toxic and biodegradable (Fadhil *et al*., 2012; Adeloye *et al*., 2022). 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; Adeloye *et al*., 2022; Leke *et al*., 2023). However, the biofuel produced by micro-emulsification yields higher carbon deposits on the injector's intake valves and cylinder liners when using engines (Liaquat *et al.,* 2013). Thermal cracking of vegetable oil to biofuels yields alkanes, alkenes, alkedienes, aromatics and carboxylic acids in different proportions (Lapuerta *et al.,* 2008). Hence, biodiesel production has few production processes which include micro emulsion, thermal cracking, blending and trans-esterification which is the most common process. Trans-esterification is common because of its yields at the end of the process.

Depletion of energy source due to high consumption and its environmental challenges have triggered interest in search for an alternative source of energy. For energy requirements to be met, there must be focus on developing alternative sources of fuel such as biodiesel that provide a stable diesel needed to promote internal combustion. Biodiesel is a good alternative source of diesel fuel, because it is a renewable resource with similarity in properties. It is a promising source of diesel, which has attracted positive recognition due to its effectiveness compared to the conventional

fuel and its environmental challenges (Farouk *et al*., 2024). Although biodiesel is widely accepted, there is a major constraint of bio-based process as feedstock costs account for a major portion of biodiesel production costs. To minimize these costs and to nip the food scarcity that may subsequently lead to famine, waste cooking oils have been considered to be ideal feedstocks. With reference to raw material or feedstock, the quality and quantity of biodiesel produced are influenced by the raw material applied, methanol-to-oil molar ratio, the temperature and time of the reaction, amount of catalyst, and agitation speed. Use of liquid fuel like biodiesel derived from used vegetable oil by transesterification shows the most promising options that can be used as an alternative to conventional diesel (Rocha-Meneses *et al*., 2023). Waste cooking oil has emerged as a valuable resource for sustainable biodiesel production, offering an effective solution to two critical environmental challenges namely waste management and renewable energy generation. Waste cooking oil sourced from plant-based oils represents an important resource for biodiesel manufacturing. This oil is available in large quantities worldwide especially in developing countries, but improper disposal often leads to environmental contamination. In the United States, nearly 100 million gallons of waste cooking oil are generated each day, with the average individual contributing around 9 pounds of waste cooking oil. In Canada, with a population of 33 million, the total waste cooking oil production is estimated at 135,000 tons annually, while in European countries, it is between 700,000 and 1,000,000 tons per year. The high content of free fatty acids in waste cooking oil makes it a viable source for biodiesel production via the transesterification process (Sreelekha *et al.,* 2024). Therefore, biodiesel production via used or waste vegetable oil will serve as an alternative feedstock or substrate or raw material for its production. This research study focused on biodiesel production by transesterification process via the application of used or waste vegetable oil as feedstock and its characterization. This is achieved by transesterification process using potassium hydroxide and methanol as catalyst, characterization of produced biofuel, comparison of produced biodiesel properties with other biodiesel from other sources and parametric effects on produced biodiesel yield.

II. MATERIALS AND 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. 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 due to its cheap availability and conversion rates. Plant-based ethanol, propanol, isopropanol, and butanol can also be used.

2.2 Production of Biodiesel

Used vegetable oil collected from eateries and restaurants at Rukpakwulusi New Layout, Eliozu, Port Harcourt, Rivers State was first filtered into a container, after which it was placed on a hot plate for pre-heating at 60°C to eliminate the water content. Then an empty container was weighed and recorded as 0.84g, after then KOH (potassium hydroxide) was added into the weighed empty container for final weighing and the total weight was 5.49g and after mathematical or algebraic analysis, the weight obtained was 4.65g that was added or poured into 100mls of alcohol in a separate container, which was followed by efficient stirring or mixing process. Stirring process stops when KOH dissolved and methoxide compound was formed as a result. After the completion of the stirring, the methoxide compound was then introduced into a beaker which contained the pre-heated used vegetable oil, and it was allowed to stirred in magnetic stirrer for 2 hours, within a temperature range of $55{\text -}60^{\circ}\text{C}$ 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 due to their small density differences between glycerine and biodiesel. The bottom is usually glycerine content while the top contained the biodiesel in a separation funnel which enhances products separation. Then, repeatedly washing of the biodiesel is initiated with distilled water that is warm to remove the remaining content of methoxide and the washing process was performed repeatedly up to four cycles and gently, water (warm) was added. Once the washing process is complete, the biodiesel is then separated from the water. The produced biodiesel is then heated to 110° C to remove water content before analysis or characterization process.

2.3 Characterization of Produced Biodiesel

2.3.1 Determination of Density and Specific Gravity

The specific gravity of the biodiesel produced was evaluated by weighing a dry empty density bottle and its mass recorded. The bottle was then filled with water, followed by the sample, and its mass was measured accordingly. Thus, the specific gravity of the produced biodiesel was evaluated in sequential with other previous studies of Rabiu *et al.,* 2021 and Adeloye *et al.,* 2022 respectively.

Specific Gravity of Biodiesel

$=\frac{Density\ of\ Produced\ Biodiesel}{Dumits\ of\ Watsu}$ Density of Water

2.3.2 Determination of the Cloud Point, Pour Point and Calorific Valuer

The determination of the produced biodiesel cloud point and pour point were carried out using ASTM D2500 and ASTM D5853 instruments respectively in agreement with other previous studies of Rabiu *et al*., 2021 and Adeloye *et al.,* 2022, while the evaluation of the biodiesel calorific valuer was achieved through the application of ASTM D 4806 technique in tandem with other studies of Yusuf and Inambao, 2019 and Adeloye *et al.,* 2022

2.3.3 Determination of Flash Point

About 30ml of the produced biodiesel sample was added into Pensky Martin apparatus cup and thermometer was fitted. The

thermometer was immersed such that it does not contact the bottom of the cup containing the sample and the produced biodiesel sample was stirred continuous. At every 10°C rise in temperature, the sample vapour was exposed to a flame and the temperature at which the exposed fume got ignited by the flame source is termed the flash point.

2.3.4 Cetane Number

The cetane number which is the ability of fatty acid methyl esters as a fuel to ignite quickly after being injected, is a measurement of the diesel engine performance or quality. The greater its value, the superior its ignition quality, and it is akin to the octane rating in that it represents a rating given to a fuel to evaluate the quality of burning or combustion. Biofuels with a larger carbon number frequently have a larger specific gravity, a higher melting point, and a higher energy density As the carbon numbers of gasoline and diesel fuel typically range between 6–10 and 11–20 respectively, the carbon number is one of the significant factors for determining the combustion application of biofuel composed of fatty acid esters (Adu-Mensah *et al.,* 2019; Cheng-Yuan & Xin-En, 2022). Hence, ASTM D613 was applied in this study to determine the produced biofuel cetane number engine test, and the cetane number of a fuel is equal to that of a blend calculated using the expression thus.

 $Ceta$ Number = % $ceta$ + 0.15(iso – cetane) In addition, an alternative technique to the ASTM D613 standard method is the determination of the cetane number using empirical formula according to the ASTM D4737 or ASTM D976 techniques

 $CN = 0.016G^2 - 420.34 + 0.192G(\log T_{50})$ + 65.01($log T_{50}$)² - 0.0001809 T_{50} ²

Where $G = \frac{141.5}{55}$ $\frac{41.5}{sg}$ – 131.5 (API), sg is the specific gravity, and T⁵⁰ is the corresponding distillation temperature to 50 wt.% liquid fuel vaporized and condensed.

2.3.5 Viscosity Measurement

The produced biodiesel viscosity was determined using the procedure of Adeloye *et al.,* 2022, which measured viscosity by using Brookfield viscometer that uses the speed range of 0.1rpm to 100rpm (1rpm= 1.7035-1) with spindle size number 1 to 7. The measurement was carried out at spindle number 7 and an angular speed of 50rpm. The procedure involved insertion of viscometer spindle number 7 into the shaft of the instrument. The biodiesel sample at room temperature of 25°C was poured into the sample up to the marked level so that the spindle head was completely immersed. The spindle was rotated clockwisely inside the oil sample. The pointer of the dial deflected and stabilized on a given dial value and the reading was taken.

2.3.6 Determination of Refractive Index

The refractive index of the produced biodiesel from waste or used vegetable oil was measured using Abbe refractometer in line with other studies of Joshua *et al*. (2019), Gidigbi *et al*. (2019) and Adeloye, (2022) respectively. Hence, the average value of quadricate analysis was determined.

2.3.7 Carbon Residue

The carbon residue of the produced biodiesel sample was determined with 90% recovery rate using ASTM D86. The residue was weighed into a special glass bulb and heated in a furnace to 550° C, until most of the samples evaporates or decomposes under this condition. The bulb is cooled and the residue weighed.

2.3.8 ASTM Distillation Analysis

The ASTM distillation analysis of the produced biodiesel was performed in line with the study of Adeloye *et al.,* 2022. This process involved measurement of 100ml of produced biodiesel using graduated receiver, and round bottom flask was removed from the apparatus and fresh biodiesel transferred directly into it. The sample was left to drain for 30 minutes since the produced biodiesel is viscous and ensuring that oil does not enter the vapour tube, and the round bottom flask containing the produced biodiesel inserted into the condenser through the vapour tube of the flask. Thermometer (2CA) of ASTM type was inserted in the ASTM distillation apparatus and a 250ml beaker was placed at the outlet of the condenser tube in such a position that the condenser tube extents into the graduated cylinder. Also, the operating conditions for heat application to first drop of distillate is between 5 and 10 minutes while the rise duration of vapour column in neck of flask 60 side arm was 2.5 to 3.5 minutes. The heat input was adjusted so that the distillation proceeds normally, reading of the first drop of distillate was taking from the thermometer and volume of distillates at each temperature range recorded. Without changing the heater setting, the distillation was continued until dry point was observed with final boiling temperature recorded and amount of residue also measured. Thus, the different distillates derived were recorded and amount measured in percentage fractional conversion.

2.3.9 Gas Chromatography Analysis

Adeloye *et al.,* 2022 mode of gas chromatography analysis was applied in this study, which involved measurement of 40ml of produced biodiesel via a measuring cylinder and 10m of hexane was mixed with the biodiesel. The sample was vapourized and injected onto the head of the chromatographic column and transported via the column by flow of inert gaseous phase. The column prior to the analysis was charged with liquid stationary phase (helium) that was absorbed into the surface of the inert solar and the temperature of the sample port was maintained at 58°C higher than the boiling point of the least volatize component of the produced biodiesel.

III. RESULTS AND DISCUSSION

The results of the characterization properties of biodiesel produced from waste or used vegetable oil are highlighted in Table 1. The produced biodiesel pH value is 6.8, which is within the ASTM D6423 standard range and in agreement with other previous studies of biodiesel of Yusuf and Inambao, 2019, Sebayang *et al., 2016* and Adeloye *et al.,* 2022. The pH value trend to neutrality implies that good quality biodiesel was produced of high market value. The specific gravity of the produced biodiesel from used vegetable oil is 0.98, which is

slightly above the ASTM standard (0.785 to 0.8099). This implies that there is minimal moisture content in the produced bioethanol distillates and this is in agreement with other studies of Rabiu *et al.* (2021) and Saka *et al.* (2015), therefore the produced biodiesel is suitable and useful as fuel. In addition, the characteristic appearance of produced biodiesel is clear and colourless. The characterized flash point property of produced biodiesel is within the ASTM standard range, therefore attesting to similar flammability property of biodiesel. Also, the pour point value (- 12° C) of produced biodiesel is below the ASTM standard value of -5°C as shown in Table 1, and this attests to the valuability of produced biodiesel for operations either in temperate or polar regions.

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The cloud point value $(-5⁰C)$ of produced biodiesel is above ASTM standard value (-23°C), thereby showing minimal tendency for wax formation within the produced biodiesel since formation of wax is favoured at lower cloud point temperature with its consequences as thicken of fuel, clogging of filters and injectors in engines.

Also, the characterized properties of produced biodiesel from waste or used vegetable oil was compared with biodiesel produced from other feedstocks or raw materials such as cotton and mustard seeds respectively as depicted in Table 2

It can be deduced from Table 2 that the characterized properties of produced biodiesel from waste or used vegetable oil is similar or falls within the same value range as of biodiesel produced from cottonseed and mustard seed respectively. This attests to the viability and valuability of produced biodiesel as an alternative fuel or energy source. Also, the characterized properties of produced biodiesel were compared with fossil fuel of diesel and petrol as shown in Table 3. These properties of biodiesel produced form waste or used vegetable oil are generally higher in values when compared with diesel and fuel oil owing to the biological feedstock applied in produced biodiesel. Thus, it is worthy to note that despite the slight high value of these properties, they are within the acceptable standard range. Besides, sulphur content of produced biodiesel is extremely minimal or low in comparison with fossil fuel feedstock owing to their different feedstock's properties and constituents.

3.1 Gas Chromatography

The gas chromatography result shows the presence of polar compounds, aromatic compounds, range of hydrocarbon and fatty acid methyl ester in the produced biodiesel structure as shown in Figure 1.

An ideal biodiesel composition should have fewer polyunsaturated such as Eicosadienoic acid (0.28%), Eicosatetraenoic acid (0.31%), docosapentaenoic acid (0.19%), and docosahexaenoic acid (0.06%) and saturated fatty acids (SFAs) such as Octanoic acid (0.18%), [Pelargonic acid](https://en.wikipedia.org/wiki/Pelargonic_acid) (0.05%), [Capric acid](https://en.wikipedia.org/wiki/Capric_acid) (0.16%), [Undecylic acid](https://en.wikipedia.org/wiki/Undecylic_acid) (0.04%), [Lauric acid](https://en.wikipedia.org/wiki/Lauric_acid) (0.10%) and [Tridecylic acid](https://en.wikipedia.org/wiki/Tridecylic_acid) (0.15%). Therefore, high levels of polyunsaturated fatty acids (PUFAs) would negatively impact the oxidative stability of produced biodiesel and thereby making it unsuitable for diesel engines. On the contrary, biodiesel derived from vegetable oil has saturated fatty acids, good oxidative stability but poor fuel properties at low temperatures, which is a constraint in winter operation. In addition, within the biodiesel residence time, produced biodiesel yielded the presence of benzene, toluene, xylene and phenols, and the chromatogram highlighted the presence of gasoline, kerosene, diesel and distillates, thus the gas chromatograph analysis was able to detect

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hydrocarbon and unresolved hydrocarbon complex mixture in the produced biodiesel.

3.2 Biodiesel Yield Variation with Methanol

Methanol effects in the operational production process of biodiesel enhanced proper or adequate mixing with KOH and the feedstock (used vegetable oil) due to its solubility nature in KOH and quick reaction with triglyceride, which in turns improves the operational rate of the reaction process by lowering the reaction or process time. Thus, better yield of biodiesel was achieved due to efficient mixing and the results show that increasing methanol to oil molar ratio increased the yield of biodiesel production as depicted in Figure 1. Thus, oil to methanol molar ratio has a significant impact on the biodiesel yield. The yield of produced biodiesel increases gradually as the amount or quantity of methanol increases in the process due to its strong affinity or solubility with feedstocks, thereby increases rate of biodiesel production. This trend can be seen steadily in Figure 1 until there is maximum yield of biodiesel production at 110ml of methanol and followed by steady decline in biodiesel production due to depletion of feedstocks

3.3 Variation of Biodiesel Yield with Temperature

Temperature has a significant effect on produced biodiesel operational process. As highlighted in Figure 2, maximum yield of the produced biodiesel is 75% at optimum temperature of 60° C followed by steady decline in produced biodiesel yield as a result of continuous vapourization of methanol thereby methanol remained in the gas phase in the reflux leading to a decline of methanol in the reaction media.

3.4 Biodiesel Yield Variation with Potassium Catalyst

An important factor that showed the effectiveness of the transesterification process is the application of KOH as an operative catalyst. It can be deduced from Figure 3 that there is increase in produced biodiesel yield with corresponding increase in the amount of catalyst applied.

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This showed that loading or increase of catalyst improves the catalyst active surface area that are involved in the transesterification process, thereby improving or enhancing the yield of produced biodiesel from waste or used vegetable oil. Also, there is gradual decline in the yield of produced biodiesel after the optimum yield even as the quantity of catalyst increases in the process owing to larger amount of catalyst in the process exceeding the mean or relative quantity that enhances transesterification process, thereby making produced biodiesel to be stickier and thus, impeding the mass transfer operation in the produced oil-methanol -potassium hydroxide structure. Also, low amount of catalyst favoured low yield of produced biodiesel due to the insufficient quantity or amount of catalyst available for transesterification leading to the formation of methyl ester.

3.5 Biodiesel Yield Variation with Reaction Time

Reaction time is a major factor that affects the rate of any process, and its effect on the transesterification process of using waste or used oil to produce biodiesel is shown in Figure 4.

Fig.4: Effects of Reaction Time against Biodiesel Yield

It can be seen that the yield of produced biodiesel increases with increase in reaction time with maximum yield achieved after reaction time of 120 minutes. After the maximum or optimum reaction time, there is decline in the yield of produced biodiesel due to the longer reaction time that enhances the reversible reaction of transesterification, hydrolysis of esters and additional production of fatty acids for soap making.

3.6 Effects of Stirring Rate

The effects of the rate of stirring or mixing of feedstocks to yield biodiesel is also a pertinent factor for consideration in the operational production of biodiesel from waste or used oil as highlighted in Figure 5.

It can be deduced from the above Figure 5 that a desired stirring or mixing rate of 200rpm is applicable for the produced biodiesel operational process. It should therefore be emphasized that improper stirring or incomplete mixing of the feedstocks or raw materials can lead to poor or low yield of produced biodiesel.

VI. CONCLUSION

This research study focused on the application of used oil as a feedstock or raw material for the production of biodiesel as an alternative energy source to crude oil or fossil fuel with its degrading environmental effects through the transesterification process. The produced biodiesel was characterized to determine its physicochemical and fuel properties, which are useful comparison data for understanding the standard quality and applicability of the produced biodiesel. The characterized properties such as _PH, flash point, specific gravity, calorific value (heating effect), refractive index, moisture content etc are within the acceptable standard range. The presence of polar compounds, aromatic compounds, range of hydrocarbon and fatty acid methyl ester were confirmed present in the produced biodiesel through the GC and MS analysis, and the produced biodiesel gave an optimum yield of 75% after 120 minutes of operational time and

functional parametric effects on the yield of produced biodiesel was studied. The parametric effects showed the variation or dependency of produced biodiesel yield with parameters such as molar ratio of methanol, temperature, catalyst, reaction time and stirring rate.

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Author Contribution

Adeloye Olalekan Michael: Conceptualization, methodology, validation, formal analysis and writing—review and editing *Igbagara Princewill Wonyibrakemi*: Investigation, data curation, writing—original draft preparation and supervision

All authors have read and agreed to the published version of the manuscript.

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The data presented in this study are contained within this article. *Conflicts of Interest*

The authors declare no conflict of interest.

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