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PRODUCTION OF BIODIESEL FROM COCONUT (Coco nucifera) OIL USING TRANS-ESTERIFICATION METHOD

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Abstract: **This study investigates the potential of producing biodiesel from coconut oil through trans-esterification reaction. The experiment was carried out by varying the molar ratio of methanol to oil (6:1), reaction temperature at 80 °C, and a reaction time of 2 hours. The produced biodiesel was found to have good fuel properties such as high peroxide value and low viscosity, making it a potential alternative to petro-diesel. The quality of the produced biodiesel was analyzed based on its physical and chemical properties. The results showed that the optimal reaction conditions for the trans-esterification of coconut oil were at a molar ratio of 6:1 (methanol to oil), a reaction temperature of 80 °C, and a reaction time of 2 hours. The produced biodiesel was found to have good fuel properties such as high peroxide value and low viscosity, making it a potential alternative to petro-diesel.**

Keywords: **Production, Biodiesel, Coconut Oil, Trans-esterification.**

1. INTRODUCTION

Fossil fuels and energy crisis are the major concern of the society for the depleting world's non-renewable energy resources which led to a renewed interest in the quest for alternative fuels. The first use of vegetable oil in a compression ignition engine was first demonstrated through Rudolph who used peanut oil in his diesel engine (Huynh et al. 2011). The use of oils from soybean, sunflower, peanut, linseed, rapeseed and palm oil amongst others have been attempted. As the world populations continue to increase annually, the demand for petroleum is also, increasing with each day. Due to the limited resources of petroleum crude, it becomes a necessity to search for an alternative fuel which is renewable. The world requires fuel that can be easily transported with low toxic emission, low greenhouse gases, greater efficiency of energy use, less dependence on foreign oil imports and affordable. Using trans-esterification method, the oil is converted to the alkyl esters of the fatty acids present in the coconut oil. These esters are commonly referred to as biodiesel. Biodiesel is an alternative fuel that is renewable in the sense that its primary feedstock has a sustainable source (Meher et al. 2006; Frierre et al. 2011). By adopting and increasing the use of biodiesel, Nigeria will also be free from her over-dependence on crude oil reserves. The emissions produced from biodiesel are cleaner compared to petroleum-based diesel fuel. Particulate emissions, soot, and carbon monoxide are lower since biodiesel is an oxygenated fuel (Yusuf et al. 2011).

The world is depending on fossil fuel as the source of energy in which these sources of energy are not renewable and also deliver high toxic emission, high greenhouse gases leading to global warming. In fact, because of the increase in world population annually, the concentration of fossil fuel deposit is forecasted to finish in few decades from now. These problems pose effects to our environment and at the same time create an unfriendly habitat for both man and plants.

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Trans-esterification is the most common method used currently for the production of biodiesel (Meher et al. 2006). Coconut oil is edible oil extracted from the kernel or meat of mature coconut harvest from the coconut palm. It has various applications as food, cosmetics and in the production of biofuel. Because of its high saturated fat content, it is slow to oxidize and thus resistant to rancidification lasting up to six months at 24°C (75F) without spoiling. Coconut oil as other vegetable oils are compounds consisting of various kinds of triglyceride fatty acids and about 90% are saturated fatty acid compounds (C8-C12), especially lauric acid and myristic acid. Coconut oil as a raw material has advantages than other vegetable oils, it contains medium chain fatty acids about 70% making it possible to obtain other fuels such as kerosene or jet fuel (Benzard, 1971). Although various oil seeds from different geographical regions have been used as raw materials for biodiesel production, the coconut is the most abundant oil seed crop in the Pacific Island Countries and may be the best material for biodiesel production in the Region. The first mature coconuts can be produced after 5-6 years following plantation, and about 50 to 80 fruits per year are produced from a fruit bearing palm with each endosperm yielding up to 40% oil. Approximately 8-10 coconuts are required to produce 1 litre of coconut oil. The productive lifespan of such palms is about 80 years. The use of coconut oil has not been widely reported in the literature as a raw material for biodiesel production although, Benzard (1971) reported that coconut oil contains more than 90% saturated fatty acids with the remainder being unsaturated. About 62% of the saturated fatty acids are medium chain length (C8 – C18) and 29% are characterized as long chain fatty acids (above C18). Coconut oil has excellent solubility and solvency, such features make "coco biodiesel", a perfect biodiesel for developing countries (Canakei and Gerpen, 1999). The coconut oil is a natural oil that is extracted from the endocarps or meat of coconut fruit using a blending or extraction method. Coconut oil is composed of the fatty acids, caprylic acid C -8:0 (8%), capric acid C-10:0 (7%), lauric acid C-12:0 (49%), myristic acid C-14:0 (8%), palmitic acid C-16:0 (8%), stearic acid C-18:0 (2%), oleic acid C-18:1 (6%) and 2% of C-18:2 linoleic acid (Canakei and Gerpen, 2001).

The coconut tree (*cocos nucifera*) the cocnut friut the endocarp of coconut

Figure 1.1 Structure of the coconut palm and fruit

Most plants utilize solar energy to create sugars, starting from simple substances such as water and carbon dioxide. This energy is stored in molecules of glucose, starch, oil, etc. Biofuels could include ethanol, biodiesel, and bio-methanol among others. Most developed and used are bioethanol and biodiesel (Abigor et al. 2000). One of the major benefits of biodiesel is its low content of sulphur. Due to the presence of oxygen in their chemical composition, its combustion is more complete, reducing the particulate emissions, carbon monoxide and unburnt hydrocarbons, among other contaminants. During its production process the by-product, glycerine is obtained. The glycerine after purification can be used in pharmaceutical and cosmetic industry. Biodiesel is usually obtained by trans-esterification (Meher et al. 2006) of oils or fats by reacting a short-chain alcohol, like methanol, in the presence of a homogeneous base- catalyst (typically NaOH). Trans-esterification is the general term used to describe the chemical reaction for the production of biodiesel from the various triglycerides. Typical triglycerides (Fig. 1.2) react with an alcohol in the presence of an acid, base, acid/base-hybrid or enzyme catalyst resulting in a mixture of fatty acids, esters and glycerol. Amongst the alcohols, methanol or ethanol are commonly used due to their low cost and relative abundance (Yusuf et al. 2011; Ma et al. 1998).

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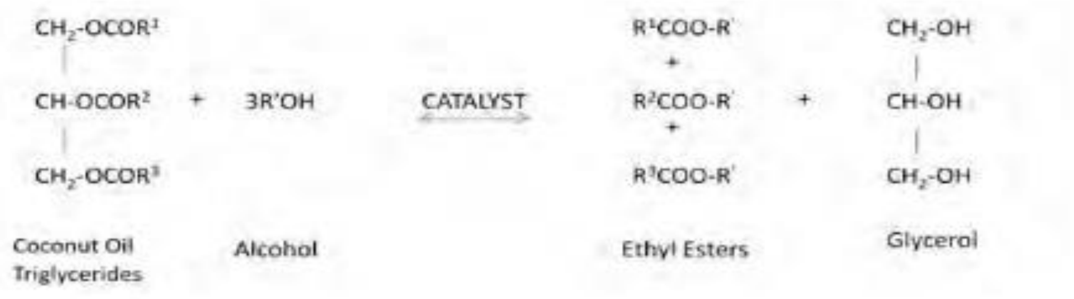

Figure 1.2 Chemistry of trans-esterification process

The stoichiometric trans-esterification reaction involves 1 mole of triglyceride and 3 moles of alcohol to produce of 1 mole glycerol and 3 moles of fatty acid esters, while the associated esterification reaction stoichiometrically produces 1 mole of fatty acid ester and 1 mole of water from 1 mole of fatty acid and 1 mole of alcohol in the presence of a catalyst. (Fig. 1.3)

Ethanol is often the preferred alcohol instead of methanol because it can be produced from a bio-refinery using renewable biomass. However methanol is often used commercially because of its lower cost (Ma et al. 1998).

Figure 1.3 Basic chemistry of an esterification process

There are commonly three main catalysts used for biodiesel production processes from various oil feedstocks, viz. acid, alkali and enzyme catalysts. These catalysts facilitate both the trans-esterification and esterification processes shown above. The most commonly used acid catalysts are sulphuric acid, hydrochloric acid, or sulfonic acid (Demirbas, 2009; Du et al. 2004). Acid catalysts can be used for both free fatty acid (FFA) and high FFA oil feedstock conversions using either methanol or ethanol (Huynh, 2011; Abigor et al. 2000). The acid catalysts have an advantage over alkali catalyst particularly for high FFA oils, as they avoid unwanted side reactions such as saponification (soap formation) caused by alkali catalysts. Canakei and Gerpen (1999) have used alcohol to oil molar ratios ranging from 3:1 to 30:1 to investigate the effectiveness of an acid catalyst for biodiesel production, and showed that an increase in the molar ratio resulted in an increase in the ester conversion of the tri glycerides with the highest conversion being 98.4% for a 30:1 molar ratio alcohol to oil.

2. MATERIALS AND METHODS

The coconut sample was purchased at Eketa main market in Umuine Igbodo, Etche Local Government Area of Rivers state, Nigeria. The coconut was dried using sun drying method and later extracted using chemical method of analysis. Sodium methoxide was prepared by dissolving 20g of NaOH in 160 ml of methanol. 300 ml of coconut oil was mixed with 160 ml of sodium methoxide and stirred at 600 rpm and at reaction temperature of 80°C for 2 hours in a conical flask. The mixture was poured into a separating funnel and allowed to stay overnight for the reaction to be completed and for the mixture to separate into two layers of biodiesel and denser glycerol at the bottom. The glycerol was drained off and the biodiesel was washed with distilled water stir gently to remove impurities such as di-glycerine and mono-glycerine, catalyst, soap and excess methanol, which can affect combustion and exhaust emission (Canakei and Gerpen, 1999). It was allowed to settle for 2 hours to separate into two layers of pure biodiesel and hydrated methanol, which was separated using separating funnel. This process is called trans-esterification method. In order to speed up the reaction, the coconut oil was heated. The ideal temperature range was 120-140F. The reaction took days at room temperatures and was inhibited above 140F. Heating with electric elements was the easiest way to bring up the oil up to temperature required. The purpose of mixing methanol and the catalyst (NaOH) is to react the two substances to form methoxide. Methanol and NaOH are dangerous chemicals. Therefore care was taken not to touch the skin when handling. NaOH does not readily dissolve in methanol and NaOH was slowly added to it. The methoxide was readily used when the particles of NaOH cannot be seen. The mixing process was achieved between 10-20 minutes.

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After the trans-esterification reaction, the glycerol was allowed to settle at the bottom of the separating funnel. This happened because glycerol is heavier than biodiesel. The settling begins immediately but the mixture was left for a minimum of twenty four (24) hours to make sure all of the glycerol has settled out. The glycerol volume was approximately 20% of the original oil volume.

- The product of the trans-esterification reaction contains some impurities like un-reacted methanol; potassium methoxide and possibly potassium alkylate (soap). Therefore, it needs some form of purification before it can be used in diesel engine (Frierre et al. 2011). Since all the impurities are polar group, water was a suitable solvent for dissolving. The following procedure was used in washing the biodiesel. 20 ml of water was measured using a measuring cylinder and poured gently on the product sample.
- The mixture was gently stirred to avoid foam formation. Shaking rigorously is not advised.
- The mixture of water and biodiesel was left for 16 hours to settle into two phase via; water-impurities phase and biodiesel phase.
- The two phase mixture was then separated using a separating funnel.
- The biodiesel layer was then heated to about 100°C for 1 hour to evaporate the remaining water molecules in it.

The properties of biodiesel produced from coconut oil can vary depending on the quality of the feedstock, the type of catalyst used, and the reaction condition (Yusuf, 2009).

Determination of iodine value using Wijs method

The iodine value is a measure of the degree of unsaturation in a biodiesel sample, which is an important quality parameter for biodiesel. 0.3 g of the biodiesel sample was weighed into a clean and dry Erlenmeyer flask and 10 ml of chloroform was added and swirled to dissolve the biodiesel sample. 25 ml of the iodine mono-chloride solution and 2 g of potassium iodide was added to the flask. The flask was swirled and allowed to stand in the dark for 15 minutes. 50 ml of distilled water was added to the flask and the excess iodine titrated with 0.1 N sodium thiosulfate solution, using starch solution as an indicator. The titration was repeated without the biodiesel sample to determine the blank value.

The iodine value of the biodiesel sample was calculated using the following formula:

Iodine value = [(volume of sodium thiosulfate used for sample titration - volume of sodium thiosulfate used for blank titration) × 12.7] / weight of biodiesel sample in gram.

Where: 12.7 is the iodine equivalent weight of the iodine mono-chloride solution.

Determination of saponification value (ASTM D1962 Titration method)

The saponification value is another important quality parameter for biodiesel that is used to determine the amount of free fatty acids in the biodiesel sample. 2 g of the biodiesel sample was weighed accurately into a 250 ml Erlenmeyer flask. 25 ml of a 1:1 mixture of ethanol and 0.5 N potassium hydroxide solution was added into the flask, with few drops of phenolphthalein indicator. The flask was heated on a hot plate with gentle swirling until the contents of the flask reach a steady boil. There was continued heating and swirling for an additional 10 minutes to ensure complete saponification of the free fatty acids. The flask was removed from the hot plate and allowed to cool to room temperature. 50 ml of distilled water was added to the flask and the excess potassium hydroxide was titrated with 0.5 N hydrochloric acid solution, using the burette. The titration was repeated without the biodiesel sample to determine the blank value.

The saponification value of the biodiesel sample was calculated using the following formula: Saponification value = [(volume of potassium hydroxide used for sample titration - volume of potassium hydroxide used for blank titration) × 56.1] / weight of biodiesel sample in grams.

Where: 56.1 is the saponification equivalent weight of potassium hydroxide.

Determination of acid value (Green Titration method)

The acid value is a measure of the amount of free fatty acids present in a biodiesel sample, and it is an important quality parameter for biodiesel. 2 g of the biodiesel sample was weighed into a 250 ml Erlenmeyer flask. 25 ml of a 1:1 mixture of ethanol and distilled water was added to the flask. A few drops of phenolphthalein indicator was also added into the flask and the solution was titrated with 0.1 N potassium hydroxide solution, while swirling the flask gently until the pink color of the phenolphthalein persists for at least 30 seconds. The volume of the potassium hydroxide solution used to reach the endpoint was recorded. A repeat of the titration without the biodiesel sample was made to determine the blank value. The acid value of the biodiesel sample was calculated using the following formula:

Acid value = [(volume of potassium hydroxide used for sample titration - volume of potassium hydroxide used for blank titration) × 56.1] / weight of biodiesel sample in grams.

Where: 56.1 is the equivalent weight of potassium hydroxide.

Note that the acid value is expressed in milligrams of potassium hydroxide per gram of biodiesel (mg KOH/g).

Determination of Peroxide value (OAIC method 965:33)

The peroxide value is a measure of the amount of peroxides in a biodiesel sample, which can indicate the level of oxidation or degradation of the sample. 5 g of the biodiesel sample was accurately weighed into a 250 ml Erlenmeyer flask. 30 ml of a 3:2 mixture of acetic acid and chloroform was added to the flask, and 0.5 g of potassium iodide was added to the flask and gently swirled until it dissolves. 30 ml of distilled water was then added to the flask and swirled gently. The solution was titrated with 0.1 N iodine solution, with constant swirling, until the yellow color persists for at least 30 seconds. A few drops of starch indicator solution was added and the titration continued until the blue color disappeared. The volume of iodine solution used to reach the endpoint was recorded. The titration was repeated without the biodiesel sample to determine the blank value.

Calculate the peroxide value of the biodiesel sample using the following formula:

Peroxide value = [(volume of iodine solution used for sample titration - volume of iodine solution used for blank titration) × 1000] / weight of biodiesel sample in grams.

Noting that the peroxide value is expressed in milli-equivalents of peroxide per kilogram of biodiesel (meq O^2/kg).

Determination of specific gravity (density)

Specific gravity is another important property to measure for biodiesel, as it can affect the density and the energy content of the fuel. A clean 50ml specific gravity bottle was weighed (W0). Then the bottle was filled to the brim and the stopper was inserted. The water on the stopper was carefully wiped off and reweighed (W1). The same process was repeated, but using the biodiesel instead of water and weighed again (W2). The specific gravity of biodiesel was calculated using the following formula:

Specific gravity of the sample = W2-W0/W1-W0 (g/ml)

Determination of Viscosity (ASTM D445 test method)

Viscosity is an important parameter to consider for biodiesel since it affects its flow properties and can impact the performance of engines that use it as a fuel. The sample of biodiesel that has been properly stored and prepared for testing was used. The sample was heated to a specific temperature (typically between 40°C and 100°C) to reduce the viscosity and make it easier to handle and was placed in a calibrated viscometer, which is a device that measures the flow rate of the liquid. The time it took for the liquid to flow through the viscometer was measured.

The viscosity of the biodiesel using the flow rate, the dimensions of the viscometer, and the properties of the biodiesel were calculated.

Determination of flash point (Pesky Marten flash point)

The flash point of a biodiesel is the lowest temperature at which it gives off enough vapor to form an ignitable mixture with air. The determination of the flash point of biodiesel is important for assessing its potential fire hazard and for ensuring safe handling and transportation.

The flash point was determined by the use of the automatic Pesky Marten flash point tester. The samples was poured into the cup of the tester and covered. A flame or electric spark of specified size was directed to the cup at interval until the vapor above the sample ignited. The thermometer reading was recorded and the flash point corrected.

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3. RESULTS AND DISCUSSION

Mean results of the physicochemical analysis of biodiesel from coconut oil are as presented in the table below:

Table 1.0: Mean Concentration of physicochemical parameters of Coconut Oil Biodiesel

Sample	Coconut oil biodiesel (with solvent n-hexane)	Coconut oil biodiesel (without solvent) – Frying method
Density (g/ml)	0.804	0.811
Kinetic Viscosity @40^0C (mm^2S^{-1})	6.33	6.29
Iodine value ($gI_2/100g$)	8.833	8.834
Peroxide value (mgO_2/g)	10.59	10.61
Saponification value (mgKOH/g)	128.05	128.11
Acid value (mgKOH/g)	14.025	14.022
API Gravity	44.495	44.493
Flash Point (^0C)	155	153
Cloud Point (^0C)	8	7
Pour Point (^0C)	11	12

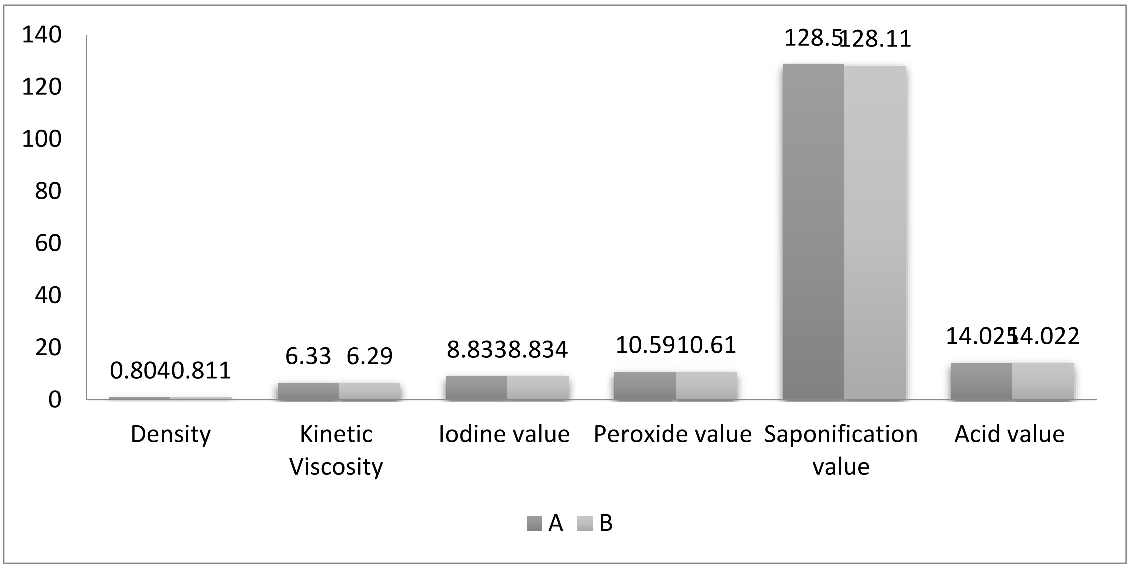

Figure 1.4 Mean Levels of coconut oil biodiesel extracted with solvent (n-hexane).

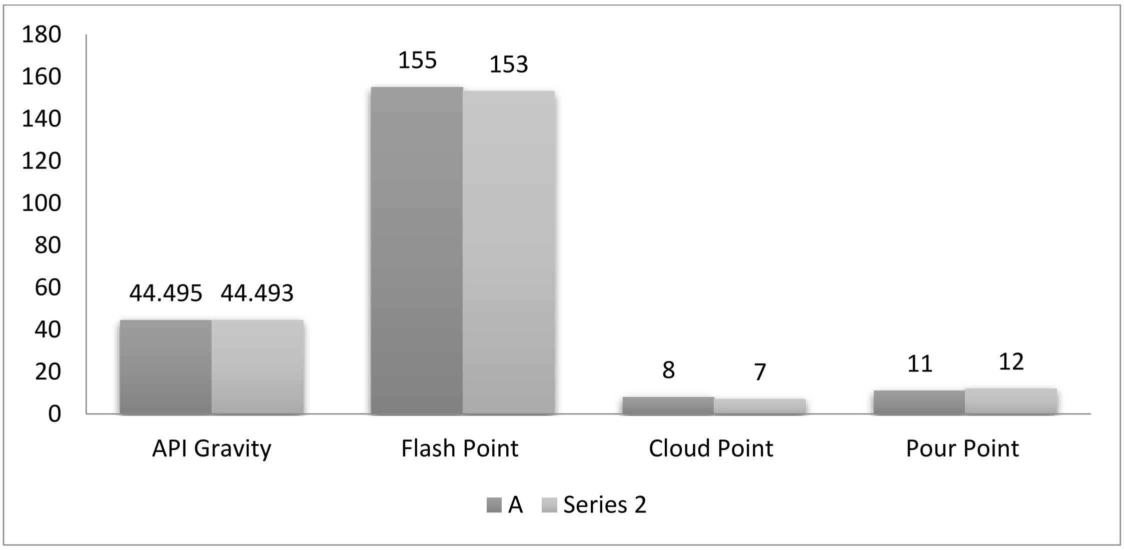

Figure 1.5 Mean Concentrations of coconut oil biodiesel extracted without solvent

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4. DISCUSSION

Coconut oil is one of the potential feedstocks for biodiesel production due to its high content of saturated fatty acids and the availability of the raw material in many regions. The production of biodiesel from coconut oil involves a trans-esterification process, which converts the triglycerides in the oil into methyl esters (biodiesel) and glycerol. The results obtained in Table 1.0, show the production of biodiesel using coconut oil extracted with a solvent (n-hexane) and production of biodiesel using coconut oil extracted using frying techniques.

The density, which is an important physical property that is often measured in biodiesel production may affect the quality and performance of the fuel. In the case of this study, the method of production and the degree of purification had effect on the density. Hence, the density of the biodiesel produced using coconut oil extracted by frying technique tends to be higher with a mean value of 0.811g/ml than that of the biodiesel extracted using a solvent (n-hexane) which was recorded as 0.804g/ml. The density parameters of biodiesel produced from coconut oil are influenced by the degree of saturation of the oil, as well as the presence of impurities such as free fatty acids. Biodiesel produced from refined coconut oil tends to have a lower density and viscosity than that produced from unrefined or virgin coconut oil, due to the removal of impurities during the refining process. Additionally, the use of catalysts and different reaction conditions during the trans-esterification process also affected the density parameters of the biodiesel (in agreement with Narasimharao et al. 2007).

Viscosity is a measure of the resistance of a fluid to flow and is influenced by a variety of factors, including the degree of purification, chemical composition and degree of saturation of the oil used to produce the biodiesel. From the results obtained the mean viscosity of the biodiesel produced using coconut oil extracted using a solvent was 6.33mm^2S^{-1}, which is higher than in the biodiesel produced using coconut oil extracted by frying technique having a concentration of 6.29 mm^2S^{-1}. These values are higher than the permissible standards (3.5-5.5 mm^2/s at 40°C) stipulated by the World Health Organization (WHO). Biodiesel produced from coconut oil has a different fatty acid composition compared to other vegetable oils commonly used for biodiesel production, such as soybean or rapeseed oil. Coconut oil contains a higher proportion of saturated fatty acids and lower levels of unsaturated fatty acids, which can contribute to a higher viscosity compared to other types of biodiesel. However, the viscosity of the biodiesel can be reduced by refining the coconut oil to remove impurities and by using the appropriate production method and reaction conditions.

The iodine value is a measure of the degree of unsaturation in a biodiesel sample, which is an important quality parameter for biodiesel. The mean concentration of the iodine value of the biodiesel produced using coconut oil extracted using a solvent was 8.833 (g$I_2/100g$), while the concentration of the biodiesel produced using coconut oil extract by frying technique was 8.834(g$I_2/100g$), this shows that the degree of unsaturation in the biodiesel is normal. The iodine value of coconut oil used for biodiesel production should be less than 10 g$I_2/100g$ oil. A higher iodine value indicates that the oil has more unsaturated fatty acids, which can lead to lower oxidative stability of the biodiesel (Ma et al. 1998).

The peroxide value is a measure of the oxidative stability of a substance and is an important quality parameter for biodiesel produced from coconut oil. The peroxide value reflects the amount of peroxides present in the biodiesel, which are formed as a result of the oxidation of unsaturated fatty acids in the oil. High peroxide values indicate that the biodiesel is more susceptible to oxidative degradation, which can lead to the formation of harmful compounds and reduced fuel quality. The result obtained indicates that the mean concentration of biodiesel produced using coconut oil extracted using a solvent and biodiesel produced using coconut oil extracted by frying technique ranges between 10.59 and 10.61mgO_2/g. The peroxide value of biodiesel produced from coconut oil can be influenced by a variety of factors, including the degree of saturation of the oil, the presence of impurities, and the production method. Biodiesel produced from virgin coconut oil tends to have a higher peroxide value compared to that produced from refined or bleached coconut oil, due to the higher level of unsaturated fatty acids present in the virgin oil. However, the use of antioxidants or the addition of hydrogen peroxide during the production process may help to reduce the peroxide value and improve the oxidative stability of the biodiesel.

The saponification value is a measure of the amount of potassium hydroxide (KOH) required to saponify a certain amount of the biodiesel, and it can be used to estimate the average molecular weight of the fatty acids in the oil. The World Health Organization saponification value of coconut oil is in the range of 245-265 mg KOH/g, but from the result obtained the mean saponification values ranged from 128.05mgKOH/g, for biodiesel produced using coconut oil extracted using a solvent to 128.11mgKOH/g, for biodiesel produced using coconut oil extracted by frying technique. This shows that coconut oil have a low saponification. The saponification value of biodiesel produced from coconut oil can be influenced by a variety of factors, including the degree of saturation of the oil, the presence of impurities, and the production method. Biodiesel produced from virgin coconut oil tends to have a lower saponification value compared to that produced from refined or bleached coconut oil, due to the lower level of unsaturated fatty acids present in the virgin oil. However, the saponification value can be adjusted during the production process by controlling the amount of catalyst used or the reaction time, to achieve the desired molecular weight of the fatty acids.

The mean acid value which ranged between 14.025mgKOH/g for biodiesel produced using coconut oil extracted using a solvent and 14.022 mgKOH/g for biodiesel produced using coconut oil extracted by frying technique is an important parameter in biodiesel production as it measures the amount of free fatty acids present in the feedstock, which can have a negative impact on the biodiesel production process and the quality of the final product. In the case of coconut oil, which is a commonly used feedstock for biodiesel production, the acid value can vary depending on the quality and freshness of the oil. The permissible limit for acid value of coconut oil used for biodiesel production is less than 0.5 mgKOH/g (WHO). If the acid value is higher, it indicates that the oil is not suitable for biodiesel production or may require additional pre-treatment steps to lower the free fatty acid content.

The flash point is another important parameter in biodiesel production as it measures the temperature at which the fuel will produce enough vapor to ignite in the presence of an ignition source. A high flash point is desirable in biodiesel production as it reduces the risk of accidental fires during transportation, storage, and handling of the fuel. The permissible limit for flash point of the biodiesel is >130°C to ensure safe handling and storage of the fuel. The result obtained shows that the mean flash point is 155°C for biodiesel produced using coconut oil extracted using a solvent and 153°C for biodiesel produced using coconut oil extracted by frying technique which is above 130°C.

5. CONCLUSION

In conclusion, the production of biodiesel using coconut oil can be a viable and sustainable alternative to traditional fossil fuels. Coconut oil is a readily available and renewable resource that can be converted into a high-quality fuel source through a relatively simple chemical process. Biodiesel produced from coconut oil has several benefits, including reduced greenhouse gas emissions, improved engine performance, and a lower environmental impact than traditional petroleum-based fuels. Additionally, the use of coconut oil as a feedstock for biodiesel production can provide economic benefits for coconut farmers and support the development of rural communities. However, like any alternative fuel source, there are also challenges and limitations to consider, including the availability of feedstock, the cost of production, and the need for infrastructure to support widespread adoption. Summarily, the production of biodiesel from coconut oil is a promising avenue for sustainable energy production that warrants further research and investment.

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