
COMPARATIVE ASSESSMENT OF BIODIESEL PRODUCED FROM MICROALGAE, USED VEGETABLE OIL AND FOSSILS

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ABSTRACT: Biodiesel was produced from two sources; microalgae oil and used vegetable oil and compared with conventional fossil diesel. The microalgae were collected from an open pond where they constitute nuisance while the used vegetable oil was gotten from roadside fried food sellers as waste products. Trans-esterification was carried out to give the corresponding mono alkyl ester (biodiesel). Quality assessment of the biodiesel produced was carried out via determination of chemical characteristics; Density, viscosity, flash point, pour point and acid value. The density of the biodiesel from the two sources were 0.882 kg. L⁻¹ and 0.870 kg. L⁻¹ respectively and higher than the conventional diesel. Flash points of the biodiesel produced from microalgae (165⁰C) and used vegetable oil (181⁰C) were significantly higher than the conventional diesel. Pour points of the biodiesel produced from microalgae oil and used vegetable oil were -10⁰C and -15⁰C respectively while viscosity values at 35⁰C were 5.2 and 4.5 respectively. The acid value of the biodiesel produced from the microalgae oil (0.394) and the used vegetable oil (0.290) were lower than that of the conventional diesel fuel (0.5). The chemical characteristics of the biodiesel produced were in line with standard specifications. The biodiesel produced when compared with the conventional diesel fuel based on their different parameters may be fit and greener replacement for fossil diesel fuels, which are nonrenewable and not biodegradable.

KEYWORDS: biodiesel, microalgae, used vegetable oil, renewable energy, waste management

INTRODUCTION

Significant growth in population and consequent change in lifestyle has greatly increased the consumption of energy. Over the years, fossil fuels have been used to provide energy for this ever increasing population. These fossil fuels are the major factors responsible for global warming; this is due to large-scale carbon dioxide emissions (Maximino *et al.*, 2008). Secondly, there is an increasing price of oil while its supply is depleting on daily basis as they are nonrenewable. They are also seen and considered among the major sources of environmental pollution (Vellguth, 1983).

There is an urgent need for an alternative sustainable, renewable and even environmentally friendly source of energy. Allah1 and Alexandru (2016) stated that biodiesel is a non-toxic renewable fuel and can replace diesel fuel. It is clean, environmental friendly (because it is biodegradable and releases lesser emissions) and renewable (Marina *et al.*, 2017, Meka *et al.*, 2007). Biodiesel can be produced from vegetable oil (Nabanita *et al.*, 2014; Teresa *et al.*, 2009) or microalgae (Marc *et al.*, 2012).

Microalgae are found plentifully in the Southeastern part of Nigeria as in other tropical rainforests (Kadiri 2010). They are more abundant during the rainy seasons and many times, cause a nuisance in the environment especially in puddles, ponds, fresh water (streams and rivers). They are most times destroyed to avoid their nuisance. Harnessing them for production of biodiesel will ensure cheap and readily available raw materials/substrate for the biodiesel industries while keeping our environments cleaner.

Vegetable oil can be used to produce biodiesel however, using waste cooking oil instead will ensure a cheaper and more environmentally friendly option (Allah1 and Alexandru, 2016). Used vegetable oil are waste products from frying of different kinds of foods. Most times, they are thrown away as waste or used by roadside traders of fried foods for rekindling of fire. Fried foods (especially commercial) are among the most common quick ready-to-eat foods in the Southeast. Hence, a good quantity of used vegetable are wasted on daily basis. Harnessing the energy in these used oil for biodiesel production becomes a great strategy for turning waste into wealth. This can be done by a process known as trans-esterification; reaction of animal fats or vegetable oil with alcohols in the presence of a catalyst to produce glycerol and alkyl ester (Rabu and Honnery 2013).

This study was aimed at producing biodiesel from microalgae and used vegetable oil and ascertaining their comparability with conventional diesel fuel as well as international acceptable standards. Attempts were made at deciphering which of the two raw materials produced better quality of biodiesel.

METHODOLOGY

Sample Collection and Preparation:

The microalgae were harvested from Otamiri River in Ihiagwa and from an open pond in Nekede both in Imo State, Nigeria. The algae were retrieved from the stagnant areas of the water bodies using perforated bag to enable the water drain out. The weight of the wet algae was recorded. After collection, the algae were adequately sun dried to remove the water content completely. The dried algae were disintegrated by rubbing them between palms and the weight of the dry algae was recorded and appropriately stored.

The used vegetable oil was obtained from roadside fried food sellers after the oil has been used to fry foods such as plantains, fish, meat, yam, potatoes. The oil collected was checked for colour and possible food particles as well as quantity. Where present, food particles were appropriately removed. The oil was then stored in sterile grease-free container prior to use.

Conventional diesel was obtained from a filling station using a clean grease-free container.

Production of Biodiesel from Samples**Oil Extraction and Production of Microalgae Biodiesel:**

The oil present in the algae was extracted using Solvent Extraction Method according to Niraj *et al.*, (2011). N-hexane and petroleum ether were used for the extraction in a Soxhlet extractor. The algae were immersed in N-hexane for three (3) days and placed in a mechanical shaker to blend the solvent with the sample. On the fourth day, the solvent and the oil were recovered using a rotary evaporator. The recovered oil was put in the oven so as to completely remove the solvent.

The Soxhlet apparatus was subsequently used in extracting oil by placing the sample in the thimble (a compartment in the Soxhlet apparatus) while the flat bottom flask was half filled with petroleum ether. The temperature of the petroleum ether was increased by heat from the heating mantle and the condenser cooled the vapour to its liquid state. This cycle continued until the thimble containing the sample got filled and the sample returned to the flat bottom flask with oil from the algae in the thimble. The quantity of oil obtained was recorded and stored for further use.

After extraction, trans-esterification of the microalgae oil was carried out using the method of Ayhan (2001) using Sulfuric acid as catalyst. The flask was heated to reaction temperature by using a constant temperature water bath. The standard reaction mixture comprised algal oil and methanol in the ratio of 1:10 and 1% concentrated sulfuric acid. The reaction mixture was heated to 65°C without stirring for two (2) hours, after which it was cooled to room temperature and the solid phase was separated by filtration using a separating funnel under vacuum condition. Finally, the bottom layer of glycerin was separated from the mixture biodiesel and methanol layer (top layer), which was then washed with warm water to remove excess methanol, traces of catalyst and any soap that may have formed during trans-esterification process in line with Karaosmanoghet *et al.*, (1996); Lang *et al.*, (2001); Molla and Nigus (2014).

Production of biodiesel using already used vegetable oil

The first step in the production of the biodiesel from used oil was filtration. The oil was poured in a large, clean cooking pot and heated to 95°F over an electric burner to make the oil easier to pour for filtration. Clean grease-free cheesecloth was placed into a funnel and the heated oil was carefully poured through this prepared funnel into another clean container. The filtered oil was reheated to allow water separate from the oil. The oil was then placed in a clean container and allowed to settle for 24 hours. Afterwards, the oil was separated carefully to prevent water from mixing with it. The presence of water inhibits transesterification processes (Theresa and Antonio 2010) as it accelerates the hydrolysis reaction, simultaneously reducing the amount of ester formation (Gnanaprakasam *et al.*, 2013)

The next step was to determine the acidity of the oil. A gram of Potassium hydroxide was dissolved into one liter of distilled water producing 0.1% Potassium hydroxide solution used as a testing device for the pH level of the oil. In a separate container, 1 ml of the filtered oil was added to 10 mls of methanol. The mixture was warmed gently by placing the container into hot water and stirring until the mixture was clear. Two drops of phenolphthalein were added to the oil and methanol mixture. This was done to determine the right level of Potassium hydroxide to be added to the oil in order to create an ideal level. The Potassium hydroxide

solution was slowly added using a graduated pipette into the container with oil, methanol and phenolphthalein. The mixture was stirred continuously until a pink/magenta colour was seen. The quantity of Potassium hydroxide used to achieve the colour change was recorded (this marked the acidity of the oil).

The actual production process was done using the method of Leung and Guo (2006). Methoxide solution was prepared by dissolving 2 g of Potassium hydroxide in methanol and agitated until it was completely dissolved, then, 20 ml of the oil was placed in 1 L beaker and heated (making sure the temperature was not above 55°C). The methoxide was carefully added to the oil and was shaken vigorously for 10 mins. The mixture was placed in a separating funnel and allowed to stand for 24 hours to separate the biodiesel from the crude glycerol. Sulphuric acid was used to neutralize the residual Potassium hydroxide to make it easier to wash the biodiesel. Water was then used to wash out all the residual chemicals and glycerol in the biodiesel and then allowed to stand in a separating funnel for 24 hours for any residual water to settle out. The biodiesel recovered was oven-dried to completely remove the solvent. This was necessary to prevent fire outbreak during analysis of the oil (Leung and Guo 2006).

Determination of the properties of the biodiesel obtained from the samples and conventional diesel

Biodiesel produced as a result of trans-esterification of microalgae oil and used vegetable oil as well as conventional fossil diesel were analyzed for density, kinematic viscosity, flash point, acid value and pour point.

Pour Point: this measures the temperature point at which a sample (liquid) becomes too viscous and loses its flow characteristics. ASTM D97 method was employed. The samples were poured into a test jar to a set mark, a thermometer was fixed to the cork and was fitted into the test jar containing the samples to be tested. The thermometer was allowed to attain a steady temperature of the samples. The samples were then pre-heated. Afterwards, they were cooled in a cooling bath till the sample was at a temperature of 9°C above its expected pour point. This allowed formation of paraffin wax crystals. The sample was then inspected at this point and at every 3°C drop in temperature by bringing out the test jar to check if the sample was still flowing i.e. tilt it to see if there was any surface movement. The temperature at which the sample stopped flowing was recorded and 3°C to it to get the pour point.

Flash Point: the flash point of a volatile material is the lowest temperature at which vapours of the material will ignite in the presence of an ignition source. The flash points of the three types of diesels were determined using the method of Lance *et al.* (1978). The appropriately labelled sample cup was filled with the sample to be tested. The lid was placed on the cup and the thermometer set. The test flame was lit and adjusted. The stirrer was turned on and heat applied at every 3°C rise in temperature. The temperature at which the fuel ignited was recorded.

Acid Value/Free Fatty Acid (FFA): Acid value can be seen as the weight of KOH in mg that is needed to neutralize the organic acids in 1g of oil (Kardash and Tur'yan 2005). The Acid Value of the samples were determined using the method of Kardash and Tur'yan in (2005) which was based on a titration in ethanol using phenolphthalein as an indicator. Aliquots of

the samples were each measured into beakers. A neutral solvent was prepared and 50 ml of it was poured into the beakers containing the biodiesel and was stirred vigorously. Potassium hydroxide of 0.56 g pellet was measured and 0.1 M KOH prepared from it. Three drops of phenolphthalein indicator were added to the sample and was titrated against 0.1 M KOH until a colour change (from colourless to light pink) was observed. The Acid Value was calculated as:

$$\text{Acid Value (AV)} = \frac{5.61 \times V \times N}{W_{\text{OIL}}}$$

Where; V = volume of standard alkali

N = Normality of standard alkali

W_{OIL} = Weight of oil used

Density of biodiesel was measured in the beam balance with a specific gravity bottle. Kinematic viscosity at 30°C was measured with the help of Ostwald viscometer with water as the reference liquid.

RESULTS AND DISCUSSIONS

The volumetric values of biodiesel produced from the microalgae oil and the used vegetable oil were analyzed and tabulated as follows:

Table 1: Volumetric analysis of biodiesel produced from microalgae oil and used vegetable oil.

Volume	Biodiesel Yield	
	Microalgae oil (ml)	Used Vegetable oil (ml)
Sample	22.5	20
Biodiesel produced	31.8	30.3
Biodiesel after drying	26	28.3

Table 2. Comparison of quality control parameters between the biodiesel from the two sources, conventional diesel and ASTM biodiesel standard.

Properties	Quality Control Parameters			
	Microalgae oil	Used vegetable oil	Conventional diesel	ASTM standard biodiesel
Density (kg.L ⁻¹)	0.882	0.870	0.838	0.84 – 0.90
Viscosity at 35°C	5.2	4.5	3.6	3.5 – 6.0
Flash Point (°C)	165	181	80	Min 100
Pour Point (°C)	-10	-15	-20	
Acid Value (mgKOH.g ⁻¹)	0.394	0.290	0.5	Max 0.8

DISCUSSION

From Table 1 above, the volume of biodiesel obtained after it was oven-dried was lower than that gotten before drying. This shows that the initial biodiesel produced contained solvent. This agreed with the work of En dang *et al.*, in 2018. Drying the biodiesel is very important in order to remove water or risk the possibility of water damaging the engine of machines. There was no significant difference in volume of the biodiesel produced from the microalgae and used vegetable yield. However the biodiesel produced from Microalgae had more moisture (5.8 ml) than that produced from used vegetable oil (2 ml) as with drying the biodiesel produced from microalgae lost 5.8 ml while that from the used oil lost only 2 ml. Used vegetable oil may therefore be better in terms of yield.

Acid catalyst was used in this study because acids have higher tolerance to water and free fatty acids to oil. They have also been found to catalyze both the esterification and transesterification processes. Canakci and Gerpen (1995) opined that acid catalysts are best for oils with high FFA contents without soap formation but can be corrosive to the process equipment (Akoh *et al.*, 2007). Sulfuric acid is commonly used (Tyagi *et al.*, 2010) just as in the present study. Acid trans-esterification was carried out and this method presented a mild and simple biodiesel production in a short reaction time with high conversion rate. There was no need to retreat the oil for acidity and only required high methanol to oil molar ratio (Tyagi *et al.*, 2010).

Biodiesel density mainly depends on its ester content and the remnant quantity of alcohol (Felizardo *et al.*, 2006). It had been reported that density is very important because it influences the efficiency of atomization of fuel (Nabanita *et al.*, 2014). The products obtained from microalgae oil and used vegetable oil had densities of 0.882 kgL^{-1} and 0.870 kgL^{-1} respectively, thus, corroborates the results of Nabanita *et al.* (2014) who obtained a density of 0.87 g/cm^3 from biodiesel produced from used vegetable oil. Samir and Rehab (2015) also produced biodiesel of density $0.878 - 0.883 \text{ gcm}^{-3}$. The conventional fuel diesel had a density of 0.838 kg.L^{-1} , thus the biodiesel produced in this study had densities higher than the conventional diesel. This is as a result of a higher degree of unsaturation present in the biodiesel (Aworanti *et al.*, 2012). Density is a very important property of any fuel. This is because injection systems, pumps and even injectors must deliver an amount of fuel accurately adjusted to provide proper combustion or burning (Samir and Rehab 2015; Dzida and Prusakiewicz 2008). From Table 2, the biodiesels and the conventional diesel met the density value specified by the ASTM Standard which is between $0.84 - 0.90 \text{ kg.L}^{-1}$.

The viscosity at 35°C of the biodiesel produced from microalgae oil and used vegetable oil were 5.2 and 4.5 respectively while the viscosity of the conventional diesel at the same temperature was 3.6. This is in line with the biodiesel produced from soybean oil by Tat and Van (1999) which had viscosity value $4.2 - 4.6$ at 40°C . Although they all fall within the ASTM specified range, the conventional diesel will definitely burn much faster than the biodiesels produced in this study. Viscosity is the property of lubricant due to which it offers resistance to its own flow (Anand *et al.*, 2009). The biodiesel produced in this study, because of its viscosity may also be able to function as a lubricant in machines, reducing its wear and tear. The viscosity of a good diesel must be high enough to provide sufficient lubrication for

engine parts but low enough to flow at operational temperature as high viscosity can plug the fuel filter and injection system in engines (Tat and Van 1999).

The flash points of the biodiesel produced from the two sources (microalgae oil and vegetable oil) were 165°C and 181°C respectively while the flash point of the conventional diesel was 80°C. The biodiesels produced in this study had higher flash points than the regular diesel implying that the biodiesels will not ignite so easily as compared to the latter (Zhendi *et al.*, 2005) and this is a big advantage over the petroleum diesel. The reason for the higher flash points of the biodiesel produced in this study can be attributed to the increased molecular weight of the biodiesel (Aworanti *et al.*, 2012).

Pour point of the two types of biodiesel were measured to be -10°C (from microalgae oil) and -15°C (from vegetable oil). The pour point is the lowest temperature at which a fluid ceases to flow or pour when cooled and tested under prescribed temperature. The pour point of the biodiesel from microalgae oil was the highest (-10°C) followed by that from used vegetable oil (-15°C), the conventional diesel had higher pour point (-20°C) than both produced in this study. This indicates that during very cold weather, the produced biodiesel may not be advisable for use as they will thicken more than diesel.

Acid value is a measure of free fatty acids present in fat or oil. The acid value was found to be 0.394 in biodiesel from microalgae and 0.290 in biodiesel from vegetable oil while that of the conventional diesel was 0.5 mgKOH.g⁻¹. A higher acid value indicates that there will be more corrosion of the machine surfaces, more wear and tear and obviously more cost of maintaining the machine. The biodiesels produced in the study had lower acid values than the conventional diesel hence will be more machine-friendly.

CONCLUSION

Waste management has become a global utmost concern owing to the fact that the population is ever on the increase. This ever growing population needs sustainable, renewable and environmental friendly source of energy. Microalgae most often constitute a nuisance in the environment and often times, are being disposed as waste. Used vegetable oil are waste products from frying and are mostly discarded by these roadside traders and even households on a daily basis; hence a good quantity of used vegetable oil is wasted on daily basis. The good news from this study is that both types of wastes can actually be turned into wealth by harnessing them for biodiesel production and thus improving waste management.

From the study, used vegetable oil seems to be a better substrate than microalgae for biodiesel production. Biodiesel from vegetable oil had a lower density, viscosity and acid value when compared to biodiesel produced from microalgae. The conventional diesel though the commonly used is not an ideal source of energy owing to the fact that it had lower viscosity, flash point and greater acid value than the biodiesels. In other words, it can easily ignite and even cause corrosion as when compared to the biodiesels. It is also not environmentally-friendly due to emission of greenhouse gases. Biodiesel from the study substrates can therefore be appropriately harnessed as a replacement for fossil diesel availing mankind of cheaper and greener source of energy.

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