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Original Research Article

Production and Characterization of Biodiesel from African Native Pear Seed

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Abstract: Viability of African pear seed-oil as a potential feedstock for biodiesel was examined in this work. The yield of the extracted oil was 59% of the total seed. Gas-chromatographic analysis of the oil extract showed that the oil was predominantly constituted by mono-unsaturated fatty acid (oleic acid, 76%) while the total percentage of its saturated fatty acids was 24% 6.1%, stearic acid, 7.5% and others, 10.4%). Pre-treatment of the oil extract with 1% w/w H2S04 showed tremendous reduction in the free fatty acid from 12.33±0.05 to 0.10±0.02 mg KOH/g. Biodiesel yield of the seed oil attained optimum yields at the methanol/oil molar ratio of 7:1, catalyst concentration of 1.00%, reaction temperature of 60°C, agitation speed of 850 rpm and effective contact time of 120 min. However, the yields of the biodiesel were higher at these experimental conditions with homogeneous KOH catalyst than its NaOH counterpart. Fuel properties such. As smoke point, flash point, fire point, viscosity and specific gravity exhibited by the biodiesel of African pear (D. edulis) were found comparable with those of the petrol-diesel, and the values also fell within the acceptable limits of ASTM and EN standards.

Keywords: Biodiesel, feedstock, methanol, pear seed.

1. INTRODUCTION

Biodiesel is a type of bio-fuel which can be used as a substitute fossil-derived diesel. The use of conventional oil such as palm oil, sunflower, grape, seed oil, and soya bean oil for bio-diesel production is prevalent and this has often strain food uses, prices, production and availability of this oils. This has spurned this research work to make use of inedible oils such as oil from African native pearl seeds in the production of diesel. Oil from African native pear seed has been investigated to be a potential feedstock for the production of biodiesel in Nigeria. African native pear seeds are included in the family rossacaea. There are two varieties of African native pear seed. The bitter African native pear seed) and the sweet African native pear seed used mainly for Culminary purposes and making oils and flavoring of food. Also, biodiesel can be used in diesel engines alone or blended with petroleum diesel in any proportion. It can be used to produce hydrogen for fuel-cell vehicles to clean up spillages, to degrease tools, to remove paints and adhesive for heating of home and so on. African native pear seed contain approximately 51% lipid, 21% protein, 20% carbohydrates, 12% fiber. The use of biodiesel blends in diesel engines will considerably reduce the emission of carbon monoxide (CO) by 33.3%, carbon (IV) oxide (CO_2) by 8.4%, and by 43.4% and unburnt hydrocarbons by 29.4%. The use of biodiesel for bio heating reduces the emissions of Sulphur dioxide (SO_2acid rain) and nitrogen oxide (NO_2 pollutant that contribute to the ground level Ozone). Therefore, the use of biodiesel increases engine performance and better greenhouse gas emission characteristics. Harnessing the inexpensive and neglected African native pear seed as a raw material for biodiesel production in Nigeria is an effective way to reduce food-fuel strain on the use of traditional oils such as palm oil, groundnut oil, soya bean oil and palm kernel oil. This research is aimed at reduction of over dependence of fossil fuel in Nigeria which is impacted negatively on the environmental problems such as global environmental problems such as global climatic change, greenhouse effect, Ozone layer depletion and others.

2. Review of literature for biodiesel production

Biodiesel production, exploration, outcomes and usage in various diesel engines is of enormous importance in some energy discoveries that are continuous and imaginative (Oyetola. *et al*. 2019). The depletion of fossil fuels, concerns for protection of our environment and steady increase for the market values of these fuels from fossils are moving researchers for greater quest for different fuel sources. Biodiesel is generally accepted as a major source of diesel in transportation and the quest for these diesels is on the increase (Avinash *et al*., 2020; Abukhadra *et al*. 2019; Al-Ani *et al*. 2018). This is due to their high oxygen content, controlled amount of toxicity, little or no pollution and they are biodegradable (Efe *et al*., 2018; Farobie and Matsumura 2017; Fonseca *et al*., 2019; Giakoumis and Sarakatsanis 2019; Gaurav *et al*., 2019). Furthermore, it does not need to be adaptable before it can be used to run an engine (Eloka *et al*., 2017). A research in 2014, which was reported globally had it that biodiesel yield was over 25.2 million tons, European Union (EU) which produced a yield about 37.1% with a range of 16.7% produced in the United States, 10.7% in Argentina and 11.9% in Brazil (Zhang *et al.,* 2018). In consideration of global total energy utilization, transportation has acquired the 3rd place with reference to the industry (Ghalandari *et al*., 2019; Hama *et al*., 2018; Jung *et al*., 2019; Khan *et al.,* 2019; Kim *et al*., 2018; Kalavathy and Baskar, 2019). The rate of consumption is assumed to increase by a rate of 60% in 2030 which would be as a result of industrialization, increment in population and good living standards (Bhuiya *et al*., 2016). Biodiesels are distinct owing to their high sustainability while fossil fuels are sparse resource. Biodiesels have the tendency of powering several diesel engines irrespective of the conditions of the atmosphere (Kumar *et al*., 2018; Lee *et al.,* 2019; Li *et al.,* 2019; Otori *et al.,* 2019; Oyetola 2019; Zhao *et al*. 2015; Zhang *et al*. 2018.. Raising the level of biodiesel production, will necessitate the accessibility of clean and affordable source of energy which is striking different from fuels from fossils.

2.1 PROPERTIES OF BIODIESEL

Several physical as well as chemical parameters were studied to determine the compatibility of biodiesel with petroleum diesel. These includes viscosity, surface tension, cloud point, specific gravity, cetane number, flash point, oxidative stability, distillation, presence of sulfur, glycerin, alcohol and aromatic compounds etc. Different ASTM/EN methods were used to determine all these parameters. ASTM stands for American Society for Testing and Materials and EN stands for European Union Standard, both of these contain different standard protocols for determination of various parameters of the different products in USA and Europe respectively.

2.2. Economic feasibility of biodiesel

The Biofuels market has been witnessing a continuous growth and developments across the world over the past few years. Governments across the world are feeding huge money and resources into the development of this sector in an attempt to reduce their dependency on oil. The volatile oil prices and production levels have further enlightened the need for continuous development in this sector. During 2001-2006 alone, the global annual production of biodiesel and ethanol grew by 43% and 23%, respectively. The major economic factor to consider for input costs of biodiesel production is the feedstock (price of seed, seed collection and oil extraction, transport of seed arid oil), which is about 75-80% of the total operating cost. Other important costs are labor, methanol and catalyst, which must be added to the feedstock. Cost recovery will be through the sale of cake and of glycerol. Economic feasibility of biodiesel depends on the price of the crude petroleum and the cost of transporting diesel long distances to remote markets in India. It is certain that the cost of crude petroleum is bound to increase due to increase in its demand and limited supply. Further, the strict regulations on the aromatics and sulphur contents in diesel fuels will result in higher cost of production of diesel fuels as removal of aromatics from the distillate fractions requires capital-intensive processing equipment. India has rich and abundant forest resources with a wide range of plants and oilseeds. The production of these oilseeds can be stepped up many folds if the government takes the decision to use them for producing alternative fuels for diesel engines. Palm oil and refined soya oil are the main option that is traded internationally. The costs for biodiesel production from palm oil, soya oil and jatropha oil are estimated about US$ 0.82/litre, US$ 0.70/litre and US$ 0.65/litre respectively. In India, estimated current biodiesel finished production costs lies somewhere between Rs. 42 to 52 per Litre. The Government of India also envisaged setting up of the National Biofuel Board to develop a road map for the use of biofuels, besides taking appropriate policy measures. In order to promote biodiesel and its production by providing necessary support to the cultivators of jatropha, the Ministry of Petroleum and Natural Gas announced the biodiesel Purchase Policy in October 2005. The policy provided for the purchase of biodiesel at 20 specified purchase centers in 12 states at Rs. 25/litre (inclusive of taxes and duties) from January 2006, moreover, the Government of India fully exempted biodiesel from excise duties in the Union Budget of February 2007. The Indian government also announced, on 23 December, 2009, attractive incentives to encourage biofuels plantation in wastelands and to utilize indigenous biomass feedstocks for the production of biofuels. It addresses the issues across the entire value chain from plantations and processing to marketing of biofuels. India's new policy on biofuels targets blending at least 20% biofuels in diesel and petrol by 2017. This implies that 13.38 million tons of biodiesel will be required. Sudha. Estimated the waste land availability and economical biomass production potential in India. Augustus screened 22 plants at Western Ghats (Tamil Nadu) in India as economical potential alternative crops for biodiesel. Other scientists Mohibbe pointed out some selected plants, which have great potential for biodiesel production in India Giibitz and Kandpal highlighted the potential of jatropha oil for

fulfilling the future energy needs Barnwal have highlighted the economical production and utilization of biodiesel in India. A detailed economic analysis of vegetable oil-based biofuels in Spain was made by Dorado. They identified that the price of the feedstock was one of the most significant factors. Also, glycerol was found to be a valuable by-product that could reduce the final manufacturing costs of the process up to 6.5% depending on the raw feedstock used.

2.3. Combustion behavior of vegetable oils and biodiesel

Heat release analysis of engine pressure data is a means of indirectly depicting the combustion process occurring in the engine. A detailed experimental description of combustion evolution in diesel engines is extremely complex because of the simultaneous formation and oxidation of air/fuel mixture. It is carried out within the framework of the first law of thermodynamics. The pressure crank angle history of an engine is affected by combustion, heat transfer and mass loss. The heat release pattern alone indicates the effect of combustion. Thermal efficiency and peak cylinder pressure are very much influenced by the heat release pattern. Heat added before the TDC increases heat losses, frictional losses and peak cylinder pressure. During the combustion process the burning proceeds in three distinguishable stages. In the first stage the rate of burning is very high and lasts for only a few crank angle degrees. It corresponds to the period of rapid pressure rise. The second stage corresponds to a period of gradually decreasing heat release rate and lasts about 40^{o} CA. Normally about 80% of the heat energy is released in these two phases. The third stage corresponds to a small but distinguishable rate of heat release persists throughout the expansion stroke. The heat energy during this period usually amounts to 20% of the total fuel energy. The different methods for computation of heat release rate from cylinder pressure data vary in the degree of accuracy with which the contents of the cylinder are considered. Some methods are simple and easy to use and others are complicated and involve extensive computation to achieve accuracy. Senatore reported that with rapeseed oil methyl ester, heat release always takes place earlier than mineral diesel, because fuel injection starts earlier for biodiesel blends due to their higher density, leading to higher peak cylinder temperature. McDonald. Obtained the heat release from the actual pressure angle diagram with soya bean oil methyl ester as a fuel in an indirect injection diesel engine and concluded that the overall combustion characteristics were quite similar to diesel operation except shorter ignition delay for soya bean methyl ester. Niehaus and Carroll found that thermally decomposed soybean Oil produced slightly less power than diesel fuel and also produced low levels of hydrocarbons and NO_x emissions. The heat release rate was lowered with thermally cracked soybean oil as compared to diesel. They suggested that by advancing the injection timing, combustion temperatures can be increased and a higher maximum rate of cylinder pressure rise and higher levels of premixed burning with the oil can be achieved. Bari observed that crude palm oil had a 6% higher peak pressure than diesel. They also observed that crude palm oil had a 2.6^{o} CA shorter ignition delay, but lower maximum heat release rate compared with diesel. Chemical reactions, such as cracking of the double bonds of the carbon chain, could have produced light volatile compounds which result in a shorter ignition delay as compared with diesel. Due to the shorter ignition delay, less fuel was injected during the delay period resulting in lower maximum heat release rates. This also resulted in less intense premixed combustion, and usually translates into lower tendency to knock. Crude palm oil had a longer combustion period than diesel. This is due to the fact that another chemical reaction, polymerization of vegetable oil at higher temperatures could have produced heavy low-volatile compounds. These heavy compounds are difficult to combust and could not completely burn in the main combustion phase, and subsequently continued to burn in the late combustion phase.

3. MATERIALS AND METHODS

MATERIALS

3.1. COLLECTION AND PROCESSING OF SAMPLE

Fully matured topical African native pear fruits (Dacryodes Edullis) were collected fresh from lgboukwu in Aguata Local Government Area in Imo, Owerri, Nigeria. The edible portion was manually removed, leaving the stony shell containing the seed. 2kg of the African native pear seeds were gathered and sundried for seven days and store in polytene bags. This was exposed to sunlight and air to prevent decay of the seeds and also enhance proper loss of moisture before extraction of the oil using Soxhlet apparatus. The petrol diesel was purchase from PTI petrol station. The instrument /equipment used in this experiment include; measuring cylinder, digital balance, weighing balance, retort stand and clamp, beaker, oven, sample bottle, conical flask, separating funnel, standard volumetric flask, burette, pipette, density bottle, thermometer, magnetic stirrer, heater and soxhlet apparatus. The chemicals and reagents used in this experiment include the following: Distilled water, methanol, ethanol, potassium hydroxide, sodium hydroxide, hydrochloric acid, sulphuric acid.

3.2. EXTRACTION OF THE OIL FROM TROPICAL AFRICAN NATIVE PEAR SEED

Extraction of the oil from tropical African native pear seed. The oil was extracted from the African pear seed oil soxhlet apparatus. Before the extraction, the seed was carefully sorted out so as to remove small shell particles. After which the seed was pulverized to fire powder using a pulverizer.

This is very important to increase the oil yield during the extraction process. The percentage oil yield was calculated as follows:

$$\text{Percentage oil yield} = \frac{\text{weight of oil extracted}}{\text{weight of sample used}} \text{ X } 100\%$$

3.3. Characterization of the oil obtained from the african native pear seeds (dacryodes edulis)

Free fatty acid (ffa)/acid value

0.5g of the sample was weighed into a dry beaker and 20ml of ethanol was added to it. 3 drops of phenolphthalein indicator were added and shook. The solution was titrated with 0.1m sodiurm hydroxide solution until a pink coloration was obtained or observed. This procedure was equally repeated with biodiesel sample after production.

3.4. Determination of moisture content of the seed oil

A container where the African native pear seed is to be placed was weighed and recorded as W, Samples of the African native pear seed were placed inside the container both the seed and the container was weighed and recorded as W1, the sample was placed in an electric oven and dried for about 24 hours at a temperature of 105^{o}C, the sample was removed after every 24hours and the weight determined. It was finally removed from the oven when it was observed that the weight does no longer change which means there is no moisture present, the final weight of the sample was measured recorded as W2. The percentage of the moisture content present in the seed was calculated using the formula:

$$\text{MC} = \frac{\text{weight of sample before drying} - \text{weight of sample after drying}}{\text{weight of sample before drying}} \text{ x } 100$$

$$\text{MC} = \frac{w1-w2}{w1-w2} \text{ x } 100$$

3.5. Pretreatment of the oil sample

The African native rear seed oil could not be transesterified directly due to its high Free Fatty Acid (FFA) value, hence the pretreatment. The FFA value of the African native pear oil was reduced to 0.76% using concentrated with sodium hydroxide and ethanol prior to transesterification. About 10ml of the extracted oil was measured into a pre-dried flat bottom flask, then 60ml of methanol was added and 1% H_2SO_4 by volume was als0 added. The mixture was agitated at a very high speed at 60°C with magnetic stirrer. The reaction time was achieved after 70min, the mixture was then poured into a 250ml separating funnel, and three layers were formed comprising water at the bottom, oil sample at the middle while the methanol was at the upper layer. The mixture was a carefully separated by removing the water first followed by the oil and lastly, the methanol. The pretreated oil was poured into a 250ml beaker and placed inside the oven set at 105^{o}C until traces of water and methanol were vaporized and then cooled in a water bath. This procedure was repeated two-three times, consequent upon which the pretreated oil was apparently suitable for the transesterification process.

3.6. Transesterification of the extracted oil sample

Trans esterification of the extracted oil with methanol was carried out in the presence of homogenous and heterogeneous catalysts to yield fatty acid methyl ester (biodiesel). Transesterification was achieved using sodium hydroxide (NaOH) as homogeneous catalyst. A 250ml flat bottom flask was used as laboratory scale reactor vessel and a hot plate assembled with magnetic stirring device was used for heating and stirring purposes.

Procedure

100g of the oil was weighed inside the reactor vessel, heated on the hot plate to heat the oil adequately. Thereafter, 0.51g of the alkali catalyst (NaOH) was weighed and dissolved in 22g of methanol. The mixture of the catalyst and methanol was poured carefully inside the heated oil. The resulting mixture was stirred (500rpm as stirred rate) and heated simultaneously at 60°C for a period of 90min. The reaction mixture was allowed to cool, after which it was transferred to the separating funnel and allowed to stand for 24hr to achieve a good separation. After the set time, two distinct lavers appeared; the upper layer consists or ratty acid methyl ester while the lower layer was made up of glycerol, excess alcohol and the catalyst. Each of the layers was carefully collected through the tap and the methvl ester (biodiesel) layer was washed with warm water about four times to remove traces of alcohol while the glycerol i.e. the byproduct was not utilized. Finally, the biodiesel produced was dried in an oven set at 105^{0}C for 2hours to remove water molecules.

BASE STRIPPING

0.5N Sulphuric acid standard solution (3.5ml) was added to the biodiesel to eliminate the base from it. This was done while washing the biodiesel. The sulphuric acid standard solution was added until the water tapped off stopped changing to purple colour when a drop of phenolphthaiein was added.

$$\% \text{ conversion} = \frac{\text{Volume of biodiesel}}{\text{volume of the oil used}}$$

The biodiesel produced was subjected to characterization to determine its fuel properties.

Biodiesel characterization from dacryodes edullis

In order to test the quality of biodiesel as a diesel fuel substitute, the American Society of Testing Materials (ASTM) has set a standard for biodiesel as fuel for use in diesel engines. Numerous properties are included in the standard, such as specific gravity, kinematic viscosity, flash point, cloud point, cetane number and so on. It is important to control the quality of biodiesel to meet the ASTM standards before using it in a diesel engine. The samples of biodiesel produced were tested for their fuel properties, the flash point was determined in a pensky-martens closed cup method; testing using ASTM D93; cloud point and pour point were determined using ASTM D2500 and ASTM D97.

Flash point

A quantity of the biodiesel sample was poured into a petri dish on a 78HW heating mattle A thermometer was insert into it by clamping it to a retort stand. A test flame applicator was used to pass flame across it with continuous smooth cross it with continuous smooth motion and the temperature at which the sample ignite was recorded as the flash point with the help of the thermometer.

Fire point

The test flame applicator was continuously moved across the center of the biodiesel sample on the heating mantle. The oil ignites and continued to burn. After 5 seconds of burning, the thermometer reading was noted as the fire point.

Cloud point

The biodiesel in a test jar with a thermometer clamped to it was cooled inside a constant temperature cooling bath. It was monitored to know the temperature of the first appearance of wax crystal. As the biodiesel in the cooling bath started forming wax crystals, the temperature was recorded as the cloud point.

Pour point

The biodiesel in the test jar with a thermometer clamped to it was cooled inside a constant temperature water bath. As it cools, it forms wax crystals. The test jar was removed at every 30^{o}C and tilted to check the surface movement. When the surface did not flow for 5 seconds, the temperature was recorded.

Specific gravity

Empty specific gravity bottle was weighed and the weight was noted as W1. The bottle was filled with distilled water and then reweighed and the weight noted as W2. The bottle was rinsed with acetone and then filled with a biodiesel sample and reweighed and the weight noted as W3.

$$\text{Specific gravity} = \frac{W_3 - W_1}{W_2 - W_1} = \frac{\text{Volume of biodiesel}}{\text{volume of distilled water}}$$

W1 = weight of empty specific gravity bottle
W2 = weight of empty specific gravity bottle + distilled water
W3 = weight of empty specific gravity bottle + biodiesel

Kinematic viscosity

A quantity of biodiesel sample was poured into a townson + mercer Viscometer up to its upper timing mark. It was placed into a holder and inserted into the viscometer water bath. A micro pipette was used to apply a sunction force to the thinner arm to draw the sample slightly above the upper timing mark. The afflux time by timing the flow of the sample as it flows freely from the upper to lower timing mark was recorded. The kinematic viscosity was calculated using the below equation.

$$\text{Kinematic viscosity} = \frac{\text{Dynamics}}{\text{Density}}$$

4. DISCUSSION OF RESULT

From table 4.1 above, it can be seen that the weight of the seed that the weight of the seed that the weight of the seed before drying, after drying and after crushing are different, this is as a result of the moisture content of the seed, also the weight of the pulverized seed is small as a result of the effective loss to the pulverize in the process of pulverizing the seed.

The volume of the oil produced was 120ml and the calculated percentage yield was 13.34% as seen in the table 4.2 which was as a result of the low percentage of oil in the seed used. The volume of biodiesel was 120ml. From the result above, it was observed that the flash point of the biodiesel 82^{o}C did not fall within the range of flashpoint for diesel. This may be as result of the interference of small quantity of water and methanol present in the oil. The specific gravity of the biodiesel produced from pear seed oil which was 0.916 shows that it can be satisfactory used to operate

diesel engines as they are neither too light nor too heavy. The flow proper properties of a fuel are important for its proper functioning at different temperatures. The kinematic viscosity of the diesel was within acceptable standard of ASTM.

Table-4.1: Experimental weight result for wet and dry African native pear seed.

Parameter	Result
Weight of African native pear seed before drying	1300
Weight of African native pear seed after drying	1109.3

Determination of moisture content

$$\text{Moisture content} = \frac{\text{weight of sample before drying} - \text{weight of sample after drying}}{\text{weight of sample before drying}} \times 100$$

$$= 14.47\%$$

Determination of % yield of oil

$$\%\ \text{yield of oil} = \frac{\text{weight of oil produced}}{\text{weight of seeds used}} \times 100$$

$$= 13.4\%$$

Table-4.2: Experimental Result for Extraction and Characterization of African Native Pear Seeds

Parameter	Result
Extraction using n-hexane (ml)	120
Specific gravity	0.916
Moisture content (%)	14.67
Yield of oil (%)	13.34
Free Fatty Acid (FFA)/Acid Value	5.80
Saponification Value	196.35

Table-4.3: Experimental Result for Transesterification of African Native Pear Seed Oil (Biodiesel Production)

Parameter	Result
Weight of oil used (g)	100
Weight of methanol used (g)	22
Weight of 0.5M NaOH (g)	0.51
Weight of Biodiesel produced (g)	113.64

5. CONCLUSION

From the experiment carried out, it can be concluded that African native pear seeds oil could be used for biodiesel production and the results also show that there is significance difference between the properties of biodiesel obtained from African native pear seed oil and petroleum diesel. This implies that biodiesel fuels produced from African native pear seed oil could function satisfactorily in any diesel engine. Although the production of biodiesel from African native pear seed oil is feasible, it is not economically viable when compared to other sources of biodiesel such as palm kernel oil, jatropha seed oil, almond seed oil etc., due to high cost of production, low oil yield and availability of raw materials (African native pear seed). Based on the problems encountered and observations made and results obtained during the course of this project work, the following are therefore recommended for further research work. To obtain a reasonable yield of oil from African native pear seed, the seeds should be pulverized to fine powder to enable better surface area contact with the extracting solvent. Due to high free acid value of the oil, the oil should be pretreated three or four times to reduce the Free Fatty Acid (FFA) content. The FFA should be reduced to about 0.76% to prevent soup formation during Tran's esterification. For better yield of oil, the quantity of seeds (African native pear seeds) should be increased.

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