



Comparative Biodiesel Production from Coconut and Palm Kernel Oils

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ABSTRACT: This research work was carried out to produce biodiesel from two lauric oils; coconut oil and palm kernel oil. Biodiesel was made from these biodiesel feedstocks by a bench scale production. It was observed that the choice of biodiesel production method largely depended on the amount of free fatty acid contained in the feedstock oil. Coconut oil biodiesel was produced by a single-step base method of transesterification while palm kernel oil biodiesel was produced by a two-step acid-base method of transesterification because of its high free fatty acid (FFA) content. The result of ASTM standard test analysis on coconut oil biodiesel gave cloud point of -9.9°C, kinematic viscosity of 12.54 cSt, flash point of 155°C, bottom sediment and water 0%, carbon content of 0.003%, total sulphur content of 0%, and copper strip corrosion of 1b (slightly tarnished). The result of ASTM standard test analysis on palm kernel oil biodiesel gave cloud point of 13.8°C, kinematic viscosity of 10.12 cSt, flash point of 93°C, bottom sediment and water of 0%, carbon content of 0.002%, total sulphur content of 0%, and copper strip corrosion of freshly polished copper strip. The results obtained aligned with published data for other biodiesels as well as ASTM standards.

Keywords: biodiesel, biofuels, bioethanol, biogas, transesterification, alkyl esters, coconut oil, palm kernel oil, free fatty acid (FFA), ASTM.

INTRODUCTION

Biodiesel is a renewable low-emission petroleum diesel substitute fuel made from vegetable oil or animal fat. It can also be described as a mono-alkyl-ester biofuel for unmodified diesel engine vehicles produced by chemically reacting a vegetable oil or animal fat with an alcohol (usually methanol or ethanol) in the presence of a catalyst (NaOH or KOH) (Demirbas, 2005; Atadashi *et al.*, 2010). This process is referred to as “transesterification”. In other words, transesterification or alcoholysis is the chemical reaction between triglycerides and alcohol in the presence of catalyst to produce mono-alkyl esters and glycerin. The resulting product, biodiesel (fatty acid methyl or ethyl esters) is thinner and less viscous than the original vegetable oil or fat and thus works better in a diesel engine (Zhang *et al.*, 2018).



Biodiesel is not the same as straight vegetable oil or straight animal fat fuel. Vegetable oil is much more viscous than either biodiesel or petrol-diesel and cannot

be used in a diesel engine without the engine’s modification (Demirbas, 2005). It has been shown that engines would fail prematurely when operating on fuel blends containing vegetable oil that is not transesterified with alcohol. Vegetable oils are problematic due to their greater viscosity which affects engine piston, injector deposits and cause fuel thickening. Engines burning biodiesel, however, exhibited no such problems and even perform better by some measures than engines using petroleum diesel (Kaufman, 2019). On its own biodiesel has much lower emissions than petrol-diesel and it can be mixed with petrol-diesel to reduce emissions. B20 for example is a fuel containing 20% biodiesel and 80% petrol-diesel. Pure biodiesel is B100. When the weather drops below 50°F, it is recommended to blend biodiesel with petrol-diesel or add anti-gel additives to prevent biodiesel from gelling (Pandey *et al.*, 2012).

Apart from biodiesel, other well-known biofuel materials are bioalcohols, biogas and solid biofuels. Bioalcohols are biologically produced alcohols from sugars, starches or cellulose by the action of microorganisms and enzymes through fermentation. Most commonly got product is ethanol, while propanol and butanol are less commonly obtained (Onuguh *et al.*,

2022a; Onuguh *et al.*, 2022b). Ethanol can be mixed with gasoline to any percentage (Iodice *et al.*, 2018). An advantage of ethanol is that it has a higher-octane rating than ethanol-free gasoline available at roadside gas stations. Biogas contains methane and is made by anaerobic digestion of organic or biodegradable waste materials. Solid biofuels include wood, sawdust, grass cuttings and charcoal. Commonly cited advantages of biofuels are that: (1) they reduce the emission of gases responsible for global warming, (2) they promote rural development, employment and wealth creation, (3) they contribute toward the goal of energy security, (4) they are renewable and (5) they reduce pollution, and can be used without modification in engines currently in use (Shah and Sen 2011).

Global warming is one of the justifications for the development of biofuels. Global warming entails the measurable increase in the average temperature of Earth's atmosphere, oceans and landmasses (Onuguh *et al.*, 2022c; Onuguh *et al.*, 2022d). Scientists believe that Earth is currently facing a period of rapid warming brought about by rising levels of heat-trapping gases, known as greenhouse gases, in the atmosphere. Greenhouse gases retain the radiant heat provided to Earth by the Sun and warm it in a process known as the greenhouse effect. Though they occur naturally and make the earth not to be too cold; combustion of fossil fuels, industrialization and increase in human activities has increased the world's average temperature. The rate of this increase is skyrocketing yearly. These greenhouse gases (carbon IV oxide, nitrous oxide, methane and synthetic chemicals like chlorofluorocarbons) are generated by burning of fossil fuels such as coal, oil and natural gas in industries and by automobiles. It is argued that, biofuel is environmentally friendly and reduces greenhouse gas emissions because carbon dioxide released from burning them is balanced by carbon dioxide intake by growing plants from where biofuels are made (Bessou *et al.*, 2011). This process reduces greenhouse gas emissions. Biofuel industry also raises domestic and international income. It creates wealth, employment and accelerates rural infrastructural development. Depletion of our traditional fossil fuels is also a huge problem facing the world at this time (Guy *et al.*, 2022). As fossil fuels are coming to a shortage, measures are being taken in order to conserve the irreplaceable energy resources. Alternative energy sources are being sought so that if these fossil fuels were to run out now, there would be a near-suitable replacement for them that are equally as efficient at producing similar amount of energy (Onuguh *et al.*, 2022c; Onuguh *et al.*, 2022d).

Biodiesel Processing

Titration of Oil for the Free Fatty Acid Content: This is done to determine the relative amount of free fatty acid (FFA) in the oil and to calculate the amount of NaOH catalyst that will be used. Free fatty acids (FFA) can slow or stop the transesterification process.

Fresh or processed cooking oil contains a standardized amount of FFA. Waste or raw vegetable oil contain free fatty acids, and the amount of FFAs in the oil is very important because it determines the quality and quantity of the fuel produced, as well as the amount of chemicals required and the cost per litre. Percentage free fatty acid (%FFA) is the number in grams of fatty acid per 100ml of oil. From acid number, the percentage free fatty acid present in the oil can be calculated. Titration with KOH is a simple method used to determine the acid number. In the process, 0.1 to 10 g of oil is weighed and dissolved in about 50 ml of a suitable solvent. Methanol, ethanol and ether are some normally used solvents. It is then heated gently for some time followed by the addition of a small drop of phenolphthalein indicator. The solution is then titrated with shaking, with the KOH solution to the end point when the pink colour persists for 15 seconds. The amount of KOH required, in milligram (mg) to neutralize the free fatty acid in one gram of oil is known as the acid number (Bazina and He 2018).

$$\text{Acid value} = \frac{56.1 \times N \times V}{M}$$

Where, V is the number of ml of KOH, N is the normality of KOH and M is the mass in grams of sample.

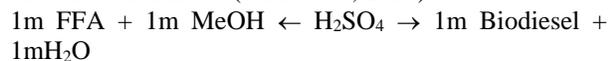
$$\text{Acid value} = 1.99 \times \% \text{FFA}$$

%FFA calculated is added to the basic amount of lye needed to process one litre of freshly refined vegetable oil (3.5 grams for NaOH or 4.9 grams for pure KOH). The total will be the amount of lye in grams needed per litre of the oil to be transesterified (Olagbende *et al.*, 2021).

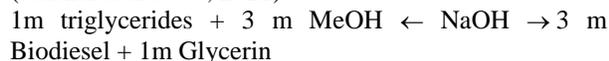
Transesterification: The reaction between oil and methanol-catalyst mixture, known as methoxide, can take place in any container resistant to the corrosive nature of the chemicals involved in a moderately hot temperature. On a small scale, a glass beaker, a mini-processor, a two litre High Density Polyethylene (HDPE) plastic bottle or gallon are generally safe bench-top vessels. The amount of methanol used should be 25% of the volume of the oil (Maceiras *et al.*, 2009). In processing one litre of vegetable oil/waste oil, methoxide is first made in a clean glass bottle by dissolving appropriate amount of lye catalyst (got from titration) in 250 ml of methanol. Methoxide mixture should be stirred until the catalyst is completely dissolved. In a typical processing unit, the oil to be processed is pre-heated to 50°C in a beaker with fitted condenser and the prepared methoxide poured into it. The mixture is heated and stirred for one hour while maintaining the temperature of 50±5°C. It has been shown that at this temperature range, there is minimum alcohol loss and maximum yield of alkyl esters. The recommended reaction time varies from 1 to 8 hours. A condenser is often used to prevent the evaporative losses of the alcohol. A heater with mechanical stirrer is normally used with this method. Care must be taken not

to create a closed system which can explode if temperature is not regulated. The system can be closed in the absence of a condenser if there will be no further heating after pre-heating the oil and adding the methoxide to it (Maceiras *et al.*, 2009).

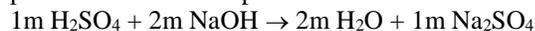
Free fatty acids (FFAs) pose a problem when raw unrefined oils are used (Suraj *et al.*, 2017). A free fatty acid is one that has already separated from the glycerol molecule. High FFA's constitute three major problems in making biodiesel: (1) soap (fatty acid salt) is formed and in some cases turns to jelly that can render the whole process useless. Soap formation reduces biodiesel yield; (2) water is formed which will retard the main reaction and (3) more catalyst will need to be used leading to higher cost of production. These problems are negligible when the oil has less than 2.5% FFA; single-stage base method procedure can be used though others have reported good results up to 4% FFA. A good way of FFA removal is a two-step process involving acid-catalysed esterification followed by base-catalysed transesterification. The first step, acid catalysed esterification reduces the FFA content of the oil to less than 2.5% (Chai *et al.*, 2014).



To annul the effect of water by-product made, more methanol percentage wise must be added to compensate for the water. To overcome this, industrial producers use counter current reactors that enable a continuous flow of high FFA oil in and water out. The second step, alkaline catalysed transesterification process converts the triglycerides to their mono-alkyl esters and glycerol (Salaheldeen *et al.*, 2021).



Acid neutralization also takes place and gives a by-product of sodium sulphate.



This procedure has the advantage of increasing biodiesel yield by eliminating soaps and the disadvantage of increased requirements for catalyst, alcohol and energy.

After reaction, the solution is allowed to sit and cool in a separating funnel for at least eight hours to enable the biodiesel and the glycerin by-product to be gravity separated. Biodiesel normally floats on top while glycerin sinks to the bottom of the container forming a gelatinous mass. After separation, washing with water is used to remove contaminants like soaps, residual glycerol, methanol and lye by an osmosis type of action. Contaminants more readily dissolve in water than in biodiesel. Unwashed biodiesel is caustic and may damage diesel engine components. Soap is not a fuel and will reduce fuel lubricity and cause injector coking and other deposits. A small batch of biodiesel can be washed using a separating funnel. Warm water can be sprayed on the diesel in the funnel using a syringe. Cloudy water settles and it is drained off. The

process is continued until wash water is clear or colourless. After the biodiesel is washed, it should be dried until it is crystal clear. This can be done by letting the biodiesel sit uncovered in a sunny location for a few days, or it may be heated to about 120°F for a few hours. Reacted, washed and dried biodiesel may be used in any diesel engine. It should have a pH of close to 7, and have no methanol left in it (Stojković *et al.*, 2014).

Titration of Biodiesel for the Iodine Value: The iodine value or the iodine number is the amount of iodine in grams absorbed by 100 grams of a given oil. It is used to measure the chemical stability properties of oils. The higher the iodine value, the more unsaturated (the greater the number of double bonds) the oil is and the higher the potential for the oil to polymerize. High iodine value oils polymerize when the double or triple bonds are broken by atmospheric oxygen and converted to peroxides. At high temperature commonly found in the internal combustion engines, the process is accelerated and the engine can quickly become gummed-up with the polymerized oil. Biodiesel made from high iodine value oils should be stored carefully and used quickly (Odoom and Edusei 2015).

The objective of the study was to produce biodiesel from two lauric oils; coconut oil and palm kernel oil and determine the appropriate production method suitable for them. Thereafter, their biodiesel properties were analyzed using ASTM standard and were compared with both petroleum diesel and biodiesel standards.

MATERIALS AND METHODS

Titration of Oils for %FFA: This was done to determine the relative amount of free fatty acid (FFA) in the oils and to calculate the amount of NaOH catalyst that will be used. The acid value of coconut oil was determined in order to calculate the %FFA.

Materials and Reagents: Solvent mixture (95% ethanol/diethyl ether, 1/1, v/v), 0.1M KOH in ethanol accurately standardized with 0.1M HCl, 1% phenolphthalein in 95% ethanol, weighing balance, beakers and 25ml burette.

Procedure: 1.763g of coconut oil was weighed into a beaker and was dissolved with 50ml of solvent mixture followed by addition of 5 drops of phenolphthalein indicator. It was titrated with shaking, with the KOH solution (in a 25ml burette graduated in 0.1 ml) to the end point when the pink colour persisted for 15 seconds. The same procedure was repeated using 0.882g palm kernel oil.

Table 1: Coconut Oil Titration Values.

Sr. No.	Initial reading (mL)	Final reading (mL)	Titre (mL)
1.	0.00	1.10	1.10
2.	0.00	1.10	1.10

$$\text{Acid value} = \frac{56.1 \times N \times V}{M}$$

Where, V is the number of ml of KOH, N is the normality of KOH and M is the mass in grams of sample.

Thus, the Acid value of the coconut oil = $\frac{56.1 \times 0.1 \times 1.10}{1.763} = 3.50 \text{ mg KOH/g}$

Acid value = $1.99 \times \% \text{FFA}$

$\% \text{FFA of coconut oil} = \frac{3.50}{1.99} = 1.76$

This indicates low titration oil which will only need single-stage base method of transesterification.

Table 2: Palm Kernel Oil Titration Values.

Sr. No.	Initial reading (mL)	Final reading (mL)	Titre (mL)
1.	0.00	4.00	4.00
2.	0.00	4.00	4.00

$$\text{Acid value} = \frac{56.1 \times N \times V}{M}$$

Where, V is the number of ml of KOH, N is the normality of KOH and M is the mass in grams of sample.

Thus, the Acid value of the palm kernel oil = $\frac{56.1 \times 0.1 \times 4.00}{0.8882} = 25.27 \text{ mg KOH/g}$

Acid value = $1.99 \times \% \text{FFA}$

$\% \text{FFA of palm kernel oil} = \frac{25.27}{1.99} = 12.70$

This indicates high titration oil that will need two step acid-base method of transesterification.

Biodiesel Production from Coconut Oil

Equipment: 4-litre round bottom flask, heater with regulation, thermometer, mechanical stirrer, water bath, reflux condenser, Teflon stopper, weighing balance, litmus papers and separatory funnel.

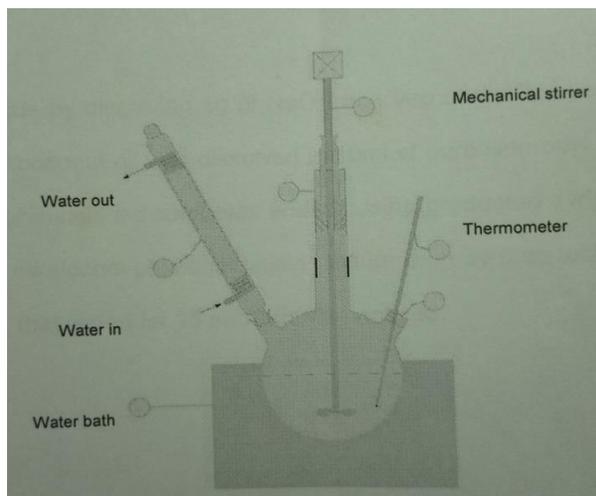


Fig. 1. Biodiesel Production Apparatus (IJEST, 2009).

Procedure:

Methoxide mixture was made by dissolving 5.26g of NaOH in 250ml of methanol in a 1 litre HDPE plastic bottle with tight lid. The mixture was swirled around until all the particles of NaOH dissolve in methanol. The mixture was also tightly covered to prevent evaporative loss of methanol and to avoid inhaling its poisonous fumes.

1 litre of coconut oil was fed into a 4 litre round bottom flask fitted with condenser and was preheated to 50°C. The prepared methoxide mixture was then poured into it and the system closed from the atmosphere. The mixture was heated and stirred for one hour while maintaining the temperature at 55°C. After the reaction, the biodiesel was poured into the separatory funnel for settling and washing.

The biodiesel was allowed to cool and settle for 8 hours in the separatory funnels clamped to retort stands after which two distinct layers of glycerol and biodiesel were observed. The glycerol was removed and the biodiesel washed with warm water. The biodiesel was sprayed on the surface with a fine mist of water using a 20ml syringe. These tiny droplets of water fell through the biodiesel and picked up impurities on the way down and became cloudy. The cloudy water was drained off after settling. Washing was continued until wash water was clear and colourless. The washed orange juice coloured biodiesel was dried in an oven at 105°C for 12 hours to remove water, make it crystal clear and to remove any remnant methanol. The ASTM parameters, pH, percentage yield, and iodine value of the product were determined.

Biodiesel Production from Palm Kernel Oil

Equipment: 4-litre round bottom flask, heater with regulation, thermometer, mechanical stirrer, water bath, reflux condenser, Teflon stopper, weighing balance, litmus papers and separatory funnel.

Procedure: The First Step (Acid Catalysed Esterification): This was used to reduce the FFA content of palm kernel oil. One litre of raw palm kernel oil required 250ml of methanol for the acid esterification process. The palm kernel oil was poured into the round bottom flask and was pre-heated to 35°C. Methanol was added and the mixture was stirred for 5 minutes after which 1 ml 95% conc. H₂SO₄ was introduced using a syringe. Care was taken to avoid any physical contact or spillage of the acid. The reaction temperature was then raised and maintained at 55°C. Stirring was continued for 30 minutes. On completion of the reaction, the product was poured into a separating funnel to sit and settle for 8 hours. At this point the sweet aroma of methyl esters was smelt. The excess methanol, with sulphuric acid and impurities moved to the top surface and were removed. The lower layer was separated for further processing (alkaline transesterification).

The Second Step (Alkaline Catalysed Transesterification): This was used to convert the

triglycerides to their mono-alkyl esters and glycerol. 4 grams of NaOH was dissolved in 250 ml of methanol to make methoxide. The product from the first step was poured into the reactor and was preheated to 50°C. The prepared methoxide was then poured in the system was closed from the atmosphere. The reaction temperature was then raised and maintained at 55°C. The mixture was stirred for 40 minutes. The reaction was stopped, and the product was poured into separatory funnels to sit and settle for 12 hours. Palm kernel oil biodiesel was separated from glycerin, washed and dried using the

same method that was used for coconut oil biodiesel. The ASTM parameters, pH, percentage yield, and iodine value of the product were also determined.

RESULTS AND DISCUSSION

The table below shows the ASTM parameter results of the manufactured coconut oil and palm kernel oil biodiesels compared with standards. This was carried out at Petroleum Training Institute (PTI); Effurun, Delta State, Nigeria.

Table 3: Biodiesel Test Results Compared with ASTM Standards.

Parameter	Test Method	Coconut Oil Biodiesel	Palm Kernel Oil Biodiesel	Petroleum Diesel (AGO) Limit	Biodiesel Standard ASTM D6751
Cloud Point, °C	ASTM D97	-9.9	13.8	4.0 max	Report
Kinematic Viscosity, Cst	ASTM D445	12.54 @ 100°F	10.12 @ 100°F	1.6 – 5.5 @ 100.4°F	1.9 – 6.0 @ 100.4°F
Flash point, °C	ASTM D93	155	93	63 min	93 min
B, S & W, %	ASTM D96	NIL	NIL	<0.05	<0.05
Carbon Content, %	ASTM D189	0.003	0.002	0.15max	<0.05
Sulphur Content	ASTM D1266	NIL	NIL	0.5max	<0.05
Copper Strip Corrosion	ASTM D130	(1b) Slightly Tarnished	Freshly Polished	No. 1 Strip max	No. 3 max

It was observed that the colours of the biodiesels were seen to be lighter and more transparent compared to their raw oils. Printed materials could be read through them.

The cloud point data of coconut oil biodiesel (-9.9°C) shows that it will have a good cold weather performance compared to its counterpart, palm kernel oil biodiesel (13.8°C). In Nigeria, these two biodiesels are trouble-free and cannot wax or gel since the average Nigeria temperature ranges from 23°C to 32°C (Nigeria-Atlapedia Online, 1993).

The flash points of these biodiesels were higher than that of petroleum diesel which is not flammable but rather combustible under extreme pressure and heat. Coconut oil biodiesel had a more promising flash point of 155°C compared to its palm kernel oil biodiesel counterpart with a flash point of 93°C (just on the standard minimum).

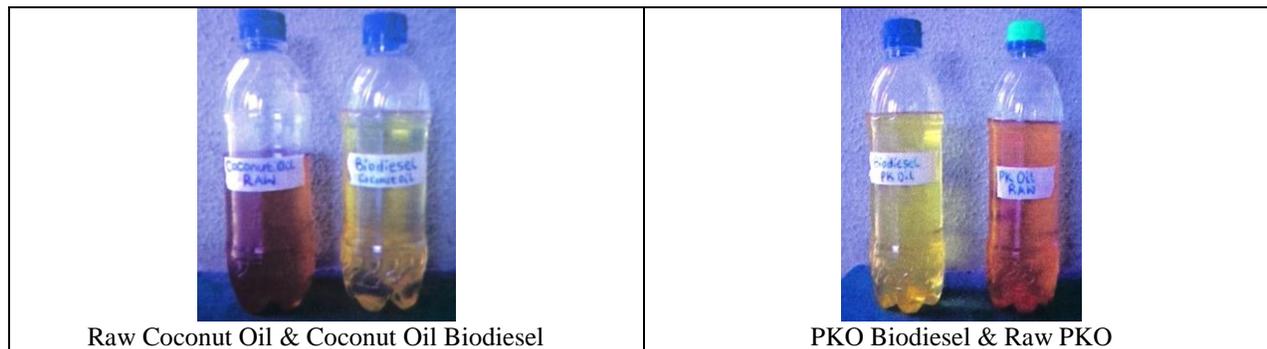
There were no bottom sediment and water content in both biodiesels produced. This shows that the fuels are clean and they will not be susceptible to any microbial growth or disintegration caused by water. It also indicates that there will not be corrosion and rust problems caused by water (Bhawna *et al.*, 2020).

The carbon content or carbon residue in both biodiesels produced was very minute or approximately zero. This showed that there were virtually no materials left over after combusting the fuels.

The sulphur content in both biodiesels produced recorded zero. This shows that they are safe from emissions that will make sulphur (IV) oxide, an acid rain precursor. Finally, the copper strip corrosion tests on both biodiesels were very satisfactory. Slightly tarnished (1b) was got for coconut oil biodiesel while freshly polished was got for palm kernel oil biodiesel. These indicate absence of acid in the samples especially FFAs that normally cause problems.

The iodine value of coconut oil biodiesel determined was 10.15 while that of its counterpart, palm kernel oil biodiesel was 45.60. This shows that both biodiesel fuels will be stable and will not polymerize with time or even in hot temperatures. Europe's EN 14214 specification allows a maximum of 120-gram iodine/100g for iodine value while Germany's DIN 51606 tops out at 115.

Both biodiesels were found to be neutral on litmus paper. The coconut oil percentage yield of biodiesel was 92.9% while that of palm kernel oil biodiesel was 96.1%. The two-step acid-base transesterification method increased the biodiesel yield by converting FFAs to biodiesel, though more methanol was consumed. Single step base catalysed transesterification was simpler for biodiesel production though FFAs were lost as soaps, and more NaOH catalyst was used.



Raw Coconut Oil & Coconut Oil Biodiesel

PKO Biodiesel & Raw PKO

CONCLUSIONS

Biodiesel production is a rewarding thing to be part of as it enables one to make fuel for one's diesel-powered vehicles. Coconut and palm kernel oils are nice and potential feedstocks for large scale production of biodiesel. Their biodiesels were light, transparent, safe to handle and mixable with petroleum diesel fuel in any proportion. They are alternative biofuels that can lead to a cleaner environment.

It was observed that the choice of biodiesel production method largely depended on the amount of free fatty acid contained in the feedstock oil. The cost of producing coconut oil biodiesel was more than its palm kernel oil biodiesel counterpart. This was because coconut oil was more expensive than palm kernel oil. Palm kernel oil biodiesel was more cost competitive with petroleum diesel fuel.

FUTURE SCOPE

The future scope of this research is to expand these productions from laboratory scale to industrial scale. Sustained technological advancement will also increase their production efficiency and cost effectiveness.

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