Production of Biodiesel from Jatropha Oil (Curcas Oil)

Ogunwole O.A.

Department of Mechanical Engineering, Federal University of Technology, Minna, Niger State, NIGERIA

Available online at: www.isca.in

Received 11th July 2012, revised 25th July 2012, accepted 31st July 2012

Abstract

This work deals with the production of Biodiesel from Jatropha oil also known as Curcas oil. Material used for the production was sourced for locally. Basic test for the suitability of the Jatropha oil was carried out in the lab. The test carried out to determine if the produced Biodiesel met standards set was carried out at the NNPC Kaduna Refinery. The production consisted of very few materials which makes the fuel easily produced without much complication. Jatropha oil was heated to 60°C, a solution of Potassium methoxide also at 60°C was added and stirred continuously for one hour. It was left to settle for 24 hours. Glycerine is then removed from the sample by decantation. The sample is then washed and dried. Test carried out on the biodiesel found it well within limits set for biodiesel's properties by ISO 14214 specification on Biodiesel.

Keywords: Biodiesel, jatropha oil, refinery, potassium methoxide, glycerine, by-product, filtering.

Introduction

Side effect of petroleum based fuels is that over the years there has been a steady increase in the amount of pollution produced by these fuels¹. The use of these energy sources over many years have resulted to the rise in global temperature levels also known as global warming. This is due to high levels of carbon that are released as by-products and exhaust gases. Their methods of extraction have also led to serious cases of oil spills which tend to destroy the immediate or remote environment where such resources are found. This also had led to many political and socio-economic problems the world over. With these problems it became necessary to find other sources of fuel, as early as the 1800's during the early production of the combustion engine. During this period various oils where used to run the engines until the mass production of various fuels eventually overtook the older fuels. In Nigeria there is a huge potential for the development of bio-fuels as an alternative energy source. This is due to the fact that there are a wide variety of plants produced in the country that could be used as Bio-fuel sources. Plants such as oil palm, Jatropha, melon, soya bean, corn and sugar cane can be produced in large quantities for use as biomass for use in fuel production. Among these plants jatropha is of particular importance since is a non-edible plant and will not be competing for human food as the others. However, one of the problems associated with the use of biodiesel is that biodiesel is less oxidatively stable than petroleum diesel fuel. Old fuel can become acidic and form sediments and varnish². Energy efficiency and energy balance are problems that were raised by³ they explained that the development of biomass for fuel requires energy and these equally produce environmental problems. This problem is shared by others who explain that for fuels such as biodiesel from various sources they tend to use up more energy to

producing the fuels than their input energy⁴. The only advantage of biofuels is their carbon content and the little amount of carbon waste they expel into the air after they are used when compared with that of fossil fuel.

Theoretical Analysis: The conversion of biomass depends of the category or the bio fuel. The production of biodiesel is basically a Trans-esterification reaction. Triglycerides are reacted with an alcohol such as ethanol to give ethyl esters of fatty acids (biodiesel) and glycerol. Production process include ultra and high-shear in-line and batch reactors process, ultrasonic reactor process, microwave process, lipase-catalyzed process, super critical process⁵. According to Nicholas⁶ microwave ovens for the microwave method of production of biodiesel can be used to provide the heat needed for trans-esterification process. For lipase-catalyzed method enzymes are used as catalyst for the trans-estarification. Researchers have found that very good yield could be obtained from crude and used oils using lipases⁷.

Material and Methods

The ripe jatropha fruits were plucked from the trees and sundried. To prepare the seeds for oil extraction, they were roasted for ten minutes. The heating process does the extraction by breaking down the cells containing the oil and eases the oil flow. The heat also liquefies the oil, which improves the extraction process. 300ml of the jatropha oil was measured using measuring cylinder, then poured into a 500ml conical flask and heated to a temperature of 60° C.

In a 250ml beaker a solution of potassium methoxide was prepared using 0.25g (catalyst concentration of 0.5%) of sodium hydroxide pellet and 10.5ml (mole ratio of oil to methanol, 1:6)

Res. J. Chem. Sci.

of methanol. The solution was properly stirred until the potassium hydroxide pellet was completely dissolved. The solution was then placed in the oven to bring its temperature to 60°C. The potassium methoxide solution was then poured into the warm jatropha oil and stirred vigorously for one hour using a magnetic stirrer. The mixture was then allowed to settle for 24 hours in a separating funnel. Thereafter, the upper layer, the biodiesel was decanted into a separate beaker while the lower layer which comprised glycerol and soap was collected from the bottom of the separating funnel. To remove any excess glycerol and soap from the biodiesel, lukewarm water was used to wash it and then allowed it to remain in the separating funnel until clear water was seen below the biodiesel in the separating funnel. The washed sample was then dried by placing it on a hot plate and excess water still in the biodiesel removed.

Characterization of Jatropha Oil: Determination of Free Fatty Acid: The free fatty acid in the oil was estimated by titrating it against potassium hydroxide (KOH) using phenolphthalein as indicator. 2g of the oil was dissolved in 50 ml of the neutral solvent in 250 ml conical flask, 3 – 4 drops of phenolphthalein indicator was then added and titrated against 0.1 M KOH the content was constantly strired until a pink colour which persist for fifteen seconds was obtained.

Determination of Iodine Value: 0.5 g of the oil was weighed into a 250 cm³ glass stoppered bottle. 15 cm³ of chloroform was added to dissolve it, followed by 25 cm³ of wiji's iodine solution. The bottle was placed in the dark and allowed to stay for 30 minutes, after which 20 cm³ of 15% KI solution was added, the bottle was stoppered and shaken vigorously. This solution was titrated against a standard 0.1M sodiumtrisulphate solution. Titration was done with constant shaking until yellow colour of iodine almost disappears. 2 cm3 of 1% starch indicator was added and titration continued, when the colour disappears, bottle was stoppered and shaken vigorously, so that any iodine in the organic solvent layer will pass into the water layer. Finally, when the titration was completed the titre values were recorded. Blank determination was carried out on 5ml of chloroform and of equal proportion of wiji's solution allowing the precipitate to dissolve for same length of time as for the sample analysed.

Determination of Peroxide Value: The peroxides present were determined by titration against thiosulphate in the presence of KI using starch as indicator. 1g of the oil was weighed into a clean dry boiling tube, 1 g of powdered KI and 20ml of solvent mix was then added and transferred into a boiling water and allowed to boil vigorously for not more than 30 seconds. The content was transferred quickly to a conical flask containing 20 ml of 5% KI solution, the tube was washed twice with 25 ml of water each time and collected into the conical flask. 0.002M Na₂S₂O₃ was titrated with the solution until the yellow colour almost disappears. Also, 0.5ml of starch was added and mixed vigorously, it was carefully titrated until blue colour just disappears, a blank was also set at the same time.

Determination of Saponification Value: A known quantity of oil was refluxed with an excess amount of alcoholic KOH, after saponification the remaining KOH was estimated by titrating against a standard acid. The oil sample was filtered to remove any impurities and last traces of moisture, 5g of the sample was then weighed into a flask and 50 ml of alcoholic KOH was added from burette by allowing it to drain for the same duration of time. A reflux condenser was connected to the flasks and allowed to boil gently for one hour. After the flask and condenser gets cooled, they were rinsed down the inside of the condenser with a little distilled water and then the condenser was removed, about 1ml of indicator was added and titrated against 0.5M HCL until the pink colour disappears.

Determination of Specific Gravity: Density bottle was used to determine the density of the oil. A clean dry bottle of 25 ml was weighed (w₀) and then filled with oil, a stopper was inserted and then reweighed to give (w_1) . The oil was substituted with water after washing and drying and weighed to give (w2).

Specific gravity =
$$\frac{\text{weight of sample }(w_2 - w_1)}{\text{weight of water }(w_2 - w_0)}$$

Determination of moisture content: The oil sample was weighed and the mass taken as (w₁), this was then dried in the oven and the weight after drying was taken as (w₂). The percentage moisture in the oil was then calculated using the formula % moisture content.

% moisture content =
$$\frac{(w_1 - w_2)}{w_1} x$$
 100

Where w_1 = weight of oil sample before drying, w_2 = weight of oil sample after drying

Determination of Viscosity: The oil sample was filtered through a sintered glass (fine mesh screen), to eliminate dust and other solid materials. The viscometer was charged with the sample by inverting the tube's thinner arm into the liquid sample and suction force was drawn up to the upper timing mark of the viscometer, after which the instrument was turned to its normal vertical position. The viscometer was placed into a holder and inserted to a constant temperature bath set at 30°C, the sample was left for some minutes until it attain 30°C. The suction force was then applied 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 timing mark to the lower timing mark was recorded.

Results and Discussion

The results of the standardization of the jatropha oil is presented in table-1, table-2 and table-3.

Table-1 Standard Specification of The Jatropha Oil

Standard Specification (
Property	Value
Specific gravity	0.9186
Flash point	240/110°C
Carbon residue	0.64
Cetane Value	51.0
Distillation Point	295°C
Kinematics Viscosity	50.73cs
Sulphur %	0.13%
Calorific Value	9470kcal/kg
Pour Point	8°C
Colour	4.0
Acid Value	1.0 - 38.2
Iodine Value	90.8 – 112.5
Saponification Value	188 – 198

The results can be conveniently discussed under the following groups of solutions. Analysis of the jatropha oil sample used in Biodiesel production. Production and yield of Biodiesel from Jatropha sample. Analysis of the results from the final production of Biodiesel. Analysis of the jatropha oil sample used in Biodiesel production.

Table 1 shows the various properties of the Jatropha oil to be used in production of biodiesel and when compared to Standard specification as laid out in Table 2 it can be observed that specific gravity of the sample given at 0.914 is within range of

the standard. Another important value is saponification value of the jatropha oil. The value for the sample is given as 195. The standards indicate a range of 188 to 198. This indicates the sample has high soap content but is still within range. The outcome of this was that the sample had to be washed with a large amount of water to clear the glycerine residue after the trans-esterification reaction. Other values such as the Iodine value of 103 g iodine/100 g was within the specified range of 130 g iodine/100 g, the moisture content was also very low which was given at 0.21%. The moisture content is also important in the reaction process because pure samples of methanol, the catalyst KOH, and Jatropha oil are needed to produce a complete reaction and eventually a high yield.

Table-2 Specification of ISO 14214 Standard Biodiesel oil

Property	Value
Specific Gravity	0.82-0.845
Flash point	50°C
Carbon residue	0.25 or less
Cetane value	50.0
Distillation Po	350°C
Kinematics viscosity	2.7cs
Sulphur %	1.2%
Calorific value	1017kcal/kg
Pour point	10°C
Colour	4.0

Analysis of manufactured biodiesel from jatropha oil

Test	T T *4	Test m	Test method		D a cres 14
	Unit	IP	ASTM	Limit	Result
Specific gravity	Kg/L	160	D1298	0.95 max	0.89
Flash point (pensky martens)	0 F	54	D 93	150 min	176
Distillation IBP	⁰ C	128	D 86	-	132
10 %	⁰ C	-	-	70 max	318
50 %	°C	-	-	125 max	324
90 %	⁰ C	-	-	180 max	330
E.B.P	°C	-	-	205 max	-
Total sulphur " (by X-ray)	%wt	107	D 4294	0.5 max	0.05
Pour point	⁰ F	219	D 97	70 max	-
Redwood no.1 viscosity (100°F)	Sec.	Calculate	-	1500 max	-
Kinematic Viscosity (180 ⁰ F)	c.s.t	71	D 445	26 max	4.93
Ash content	%wt	4	D 482	0.1 max.	ND
Carbon residue (Conradson)	%wt	74	D 189	15 max	ND
Diesel index	$^{0}\mathrm{F}$	21	-	47 min	48.0
Cetane Number	-	-	D 975	40 min	50.1
Free glycerine	% mass	-	-	0.02	0.03
Total glycerine	% mass	-	-	0.24	0.22
Cloud Point	⁰ F	219	D 2600	40 max	38
Water by distillation	% vol	53	D 95	0.5 max	Trace
Bottom sediment& water(BS&W)	% vol	-	D 1796	0.5 max	Trace
Gross calorific value	Cal/gm	Calculate	D 240	10600 min	-

Production and yield of jatropha sample: During the production of the biodiesel care was taken to ensure that the samples are kept at the right temperature. The purity of the reagents used is also of vital importance in obtaining a hitch free and total reaction. Due to the high saponification value of the jatropha sample and if the required catalyst used is not properly measured there is the tendency for emulsification to occur. This is when the soap molecules encase the biodiesel molecules. The emulsified sample will have a milky colour with traces of yellow particles. It is also common to have a layer of emulsion form between the biodiesel and water layers after the completion of the reaction or during washing of the sample. Heat can be used to break up the emulsion. After it is heated, it should be placed to settle to allow completion of the reaction. Also the use of table salt, NaCl to break up the particles could also be used. Hot salt water is added to the emulsion which will bring about quicker separation. The heat decreases the density of the oil, while the salt increases the density of the water making them separate easily. This will ensure that the highest possible yield is obtained from the sample. In biodiesel production, 1 litre of Jatropha oil should also give 1 litre of biodiesel.

Analysis of the results from the final production of Biodiesel: Analysis of the final biodiesel was carried out at the NNPC Refinery in Kaduna. The result is placed with the corresponding limits set for ISO 14214 biodiesel standards (table 3). One of the most important characteristics of any fuel is its flash point. This is defined as is the lowest temperature at which it can vaporize to form an ignitable mixture in air. The biodiesel sample has a higher flash point of about 81°C as compared to the standard diesel flash point of about 50°C. This makes the biodiesel sample safe for use and storage. Fuels with lower flash point which tend to ignite at lower temperatures making them highly dangerous if not stored and used properly. The cetane number is another important property of fuels. This is a measurement of the combustion quality of diesel fuel during compression ignition. Generally diesel engines will run with values of between 40 and 55. The biodiesel fuel produced had a value of 48 which is well within range and indicates that it is suitable to run in diesel engines. Another important value is the viscosity of the fuel. Viscosity, which is a measure of resistance to flow of a liquid due to internal friction of one part of a fluid moving over another is of utmost importance in use as fuel, the higher the viscosity, the greater the tendency of the fuel to cause problems such as clogging engine components. The esterification process reduces the viscosity to that closer to fuels required by a vast majority of engines. With a very low viscosity value of 4.93c.s.t against the maximum 26c.s.t this particular sample of biodiesel has an ideal value to be used as biodiesel or combined with regular diesel to be used as a blend for diesel engines.

Conclusion

The manufacture and use of biodiesel using the locally available Jatropha oil from the seed is viable for use as biodiesel in the form of B100 as 100% biodiesel or in combination of regular diesel. Materials for use in the manufacture of biodiesel are readily available without the need of highly specialized equipment or scarce chemicals. This enables anyone who is interested in the production of biodiesel from Jatropha oil to do so without the use of very highly specialized and technical equipment. The trans-esterification process carried out on the Jatropha oil sample brought it to the standards set for biodiesel. If a high level of accuracy is used in the manufacture process there is the benefit of producing high quality biodiesel with a good yield which in turn minimises the amount of waste of both the Jatropha oil and chemicals and reagents used.

References

- 1. Perkin Elmer, Differentiation between Fossil and Bio fuels by Liquid Scintillation Beta Spectrometry Direct Method, 6, (2009)
- 2. Hansen Alan C., Combustion and Emissions Characteristics of Biodiesel Fuel, CABER Seminar, Department of Agricultural and Biological Engineering University of Illinois, 6, (2008)
- 3. Divan Deepak and Kreikebaum, Frank Organic (But not Green), IEEE Spectrum, North American, 46(11), 49-53 (2009)
- **4.** Shapouri, The Energy banalce of corn ethanol: An update, united states department of Agriculture, Agricultural Economic Report, **813**, 8 (**2002**)
- 5. Vera C.R., D'Ippolito S.A., Pieck C.L. and Parera J.M., Production of biodiesel by a two-step supercritical reaction process with adsorption refining, 2nd Mercosur Congress on Chemical Engineering, 4th Mercosur Congress on Process Systems Engineering, *Rio de Janeiro. Brazil*, (2005)
- 6. Nicholas E., Fast, Easy Preparation of Biodiesel Using Microwave Heating, *Energy & Fuels*, **20**, 2281-2283 (2006)
- 7. Du, Wei Comparative Study on Lipase-Catalyzed Transformation of Soya-bean Oil for Biodiesel Production with Different Acyl Acceptors, *Journal of Molecular Catalysis B: Enzymatic*, 30(3-4), 125-129 (2004)