Production and Characterization of Biodiesel from Cotton Seed Oil.

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Abstract- This paper present result of the research work conducted on the production of biodiesel by trans-esterification of cottonseed oil (new and waste) with alcohol in the presence of a catalyst (NaOH), with the aim of providing an alternative to fossil fuel which have adverse effect on the environment. To achieve these; batch transesterification process was used, which involved pre-treatment of the cottonseed oil, conversion of the oil into biodiesel, purification of the biodiesel and then its characterization. Using methanol as the alcohol and reaction temperature of 65°C gave the optimum yield of biodiesel with valuable co-product (glycerine), 99.0% and 14.67% for new oil, and 98.33% and 14.0 for waste oil respectively. The analysis of the biodiesel produced from the new oil, shows that it has the following characteristics; specific gravity of 0.859, flash point of 115°C, pour point of –5°C, Cetane index of 50.58, kinematic viscosity of 5.7cst, and sulphur content of 0.0002 %mass. While that obtained from the waste oil are; specific gravity of 0.86, kinematic viscosity of 6.53 cST, flash point of 117°C, pour point of –2°C, Cetane index of 51.22 and sulphur content of 0.0005%mass. These values fall within the range of the standard specifications for diesel fuel (ASTM and National Soy diesel Development Board), and indicate that the biodiesel produced is essentially sulphur free, has high cetane number (less knocking) higher lubricating effect than petrodiesel, and will not cause corrosion of pipes, and can effectively serve as an alternative to petrol diesel. Hence base catalyze trans-esterification method is an effective method for biodiesel production.

I. INTRODUCTION

Diesel oil is a fuel made from petroleum. It is made up of hydrocarbons boiling in the range of 250 – 350°C and incorporates hydrocarbon molecules containing 11 - 16 carbon atoms. Its uses as a fuel, covers fuel for diesel engine, domestic and industrial heating and fuel oil blending.

A variety of ester-based oxygenated fuels derived from natural, renewable biological sources such as vegetable oils are called "biodiesel" because they possess properties (both physical and chemical) and engine performance that are very close to those of petrol diesel. The process of converting vegetable oil into biodiesel fuel is new and called trans-esterification. Trans-esterification is a chemical reaction whereby an alcohol (usually methanol or ethanol) is used in the presence of a catalyst, such as sodium hydroxide or potassium hydroxide, to chemically break the molecule of the raw renewable oil into methyl or ethyl ester (biodiesel), of the renewable oil with glycerol as a co-product.

Trans-esterification is not a new process; it was conducted as early as 1853, by scientist E. Duffy and J. Patrick. One of the first uses of trans-esterified vegetable oil was for powering heavy-duty vehicles in South Africa before World War II (Roger et al, 2003). A successful trans-esterification reaction is signified by the separation

of the ester and glycerol layers after the reaction time. The heavier co-product; glycerol settles out and may be purified for use in pharmaceutical, cosmetics and soap industries. Conversion of cottonseed oil to methyl or ethyl esters (biodiesel) is an example of many chemical processes available to improve biofuel availability in the world.

About 15-20% of the embryo of cottonseed is oil, which is extracted by squeezing and rolling. The oil may be used directly in diesel engines with adjustment to the injection system, but toxic fumes are produced and deposits occurred, which lead to serious engine problems (University of Idaho, 1994). This is the reason why the oil needs to be converted into an alkyl ester. The esters produced are better engine fuel than the crude oil and the glycerol is a valuable co-product. The energy density of biodiesel is about 38 MJ/Kg while that of petrol diesel is 47 MJ/Kg.

Series of research have been carried out on the use and conversion of vegetable oil into biofuel. Roger *et al*, 2003 carried out experiments on rape seed and jojoba oil as fuel for diesel engines. Peterson *et al*, 1983 *and* Peterson *et al, 1991* carried out experiment on vegetable oil expression into a high-grade biodiesel fuel, and batch type trans-esterification process for winter rape oil. But it was observed that little has been done so far on the conversion of cottonseed oil into biodiesel.

Virgin/refined cottonseed oil was procured from Sun seed Nig. Ltd. Zaria, followed by conversion of the cottonseed oil (new and used) into biodiesel using the base-catalyzed trans-esterification method, which studies have shown to be the cheapest with very high efficiency. Sodium hydroxide was the base-catalyst used. In addition, the excess alcohol in the products was recovered by distillation and further purification of the products was carried out.

II.   LITERATURE REVIEW

A Historical Development of Biodiesel

Cotton (Grossupuium hnirsylium), grows as a perennial crop in the tropics and can attain the size of a small tree. The seeds contain oil and other by-products for use in the manufacture of edible and industrial products. The seeds are oblong in shape, pointed toward one end and large from one quarter to almost one half of an inch of length. The seed coat is dark brown or black. The embryo makes up almost the entire kernel and consists of the cotyledons or seed leaves. Throughout the cotyledons are numerous dark spots that are oil glands or rosin cavity. Until 1900, cottonseed oil was considered essentially inedible because it contained, among other things, gossypol, a bitter pigment produced in glands located throughout the cotyledons (Beryl, Simpson and Molly, 1985 and Weiss. 983).

Three basic routes to biodiesel production from oils and fats are;

-Base catalyzed transesterification of biolipid (biological oils and fats). This yields about 98% conversion at low temperatures and pressure.

-Direct acid catalyzed trans-esterification of the biolipid

-Conversion of the biolipid into its fatty acids and then to biodiesel.

The reaction between the fat or oil and the alcohol is a reversible reaction and so the alcohol must be added in excess to drive the reaction towards the right and ensure complete conversion (University of Idaho. 1994).

B. Trans-Esterification Reaction

The alcohol/catalyst mixture is charged into a closed reaction vessel and the oil or fat is added. The reaction mixture is kept just below or above the boiling point of the alcohol (around 65°C for methanol and 78°C for ethanol) to speed up the reaction. Recommended reaction time varies from 1-5 hours(*Peterson* etal,1991), and some authors recommend the reaction to take place at

room temperature (Noureddini and Zhan, 1997) Excess alcohol is normally used to ensure total conversion of the fat or oil to its esters. Care must be taken to monitor the amount of water and free fatty acids in the incoming oil or fat. If the free fatty acid level or water level is too high it may cause problems with soap formation and the separation of the glycerin. Acid-catalyzed and base-catalyzed trans-esterification reactions also known as acidolysis and alcoholysis are explained by different mechanisms (Noureddini and Zhan, 1997). With base catalyzed reactions, the basicity of the oxygen of the alcohol appears to be increased so that it attacks the carbonyl carbon directly. Lower activation energies are observed in base catalyzed reactions than in acid catalyzed ones (Noureddini and Zhan, 1997).

C. Catalysts Used

The catalyst used in trans-esterification of vegetable oil or animal fats and oils is sodium hydroxide (NaOH; Caustic soda), or potassium hydroxide (KOH). These alkalis are hygroscopic, i.e. they absorb water from the atmosphere. So fresh lye (NaOH or KOH) must be obtained and containers kept tightly sealed always. After weighing, the lye is added to the methanol as quickly as possible. These alkalis (NaOH and KOH) absorb carbon (VI) oxide from the atmosphere and become carbonated if not stored properly. Fresh lye is almost translucent. Carbonated lye can still be used, but with an added 25% of the calculated amount of lye needed. NaOH is cheaper and more readily available than potassium hydroxide (KOH). It usually comes in grades, flakes and 5mm pearls or half-pearls are 96-97% pure, small pearls (1-2mm) are 99%pure, but more expensive. Ones with lower percentage purity will not work for biodiesel. KOH is not as strong as NaOH; anhydrous grade flake purity is usually about 92%, while half-pearl 85%.It requires 3.5g of NaOH or 1.40253.5g of KOH per liter of oil to transesterify new oil (virgin, uncooked), Used oil (waste

vegetable oil, WVO) required more bases than new oil to catalyzed the neutralization of the free fatty acids'(FFA'S) formed in the cooking oil, which can slow or stop the trans-esterification process. Titration is carried out to determine the FFA content and how much bases will be required. The extra base act as a neutralizer and convert the FFA's to soap, which drops out with the glycerin layer (Noureddini and Zhan 1997).

D. Quantity of Methanol/Ethanol Required

The stoichiometric quantity of methanol is the amount needed to convert triglycerides (fats and oils) into methyl esters (biodiesel); it provides the "methyl" portion of the methyl esters. An excess of methanol is needed to push the conversion process (reaction) towards completion. Without the excess methanol, the ester and the glycerol react to provide an equilibrium mixture which is less than desirable and the process runs out (reach equilibrium) before all the triglycerides are converted to esters, resulting in to poor fuel that doesn't combust well and can be corrosive. The excess methanol acts more like a catalyst. It encourages the process but does not become part of the final product and can be recovered afterwards Fats and vegetable oils have been found to require different stoichiometric amount of methanol for their trans-esterification. typical value ranges from 11-13% by volume of oil. This depends on the type of fatty acid residues present in the fat or oil. However, excess has been found to be between 60% and 100% of the stoichiometric amount, so as said above if the stoichiometric amount of methanol to be used is 12.6%. therefore for 126 ml per litre of oil, the excess would range between 76 ml and 126 ml bringing the total amount of methanol to be used as 200-252 ml per litre of oil

For ethanol, a relationship developed by Peterson C L., et al (1983) was used. It is given as

EtOH = 0.2738 x oil, KOH = oil/85 Where EtOH = amount of ethanol in litre, oil = desired amount of oil in litre. The excess amount of ethanol required has been taken care of in this relationship.

III. EXPERIMENTAL METHODOLOGY

Standard methods of testing and characterization of hydrocarbons were used as recommended by ASTM (Annual book of ASTM, 1987, University of Idaho, 19994), and the procedures followed accordingly.

A. Production Process

500 ml of cottonseed oil was poured into a beaker and warmed gently to a temperature of 120°C (Make your own biodiesel 2 n.d); 100 ml of it was taken and mix with measured with catalyst/alcohol mixture containing 0.35g of sodium hydroxide pellets and 20ml of methanol which was thoroughly mixed to ensure complete dissolution of the pellets and was then transferred into biodiesel reactor (1l conical flask) stopper, and stirred vigorously for 1, to 3 hours using a magnetic stirrer for different runs. After the reaction time, the mixture was then centrifuged; producing two phases, methyl ester and crude glycerol. Crude glycerol, the heavier brownish liquid collected at the bottom while clear amber yellow methyl ester (biodiesel) was withdrawn (decanted) from the top. Each run of the trans-esterification reaction was carried out at room temperature and at 65°C. The same procedure was repeated using 27.38ml of ethanol at room temperature and at 78 °C for both new and waste oil. The basis of chosen this temperatures bellow the boiling point of the respective alcohols was not to allow the evaporation of the alcohols involved and to obtained optimum yield of Biodiesel since reaction tend to proceed faster at higher temperatures.

B. Pretreatment of Waste Oil

Solid bodies were filtered off using a mesh filter of 5µm. The oil was washed with hot water in a separating funnel. It was allowed to settle for 1 hour and decanted. The oil was then heated again and allowed to settle for some minutes and decanted for the second time.

C. Titration

One gram of NaOH was dissolved in 1l of distilled water. In a 250ml beaker, 1ml of the dewatered used cottonseed oil was dissolved in 10ml pure isopropyl alcohol. The beaker was warmed gently and stirred until all the oil dissolved and the mixture turned clear. Two drops of phenolphthalein solution was then added. Using a graduated syringe, 0.1% NaOH solution was added drop by drop to the oil-alcohol-phenolphthalein mixture, stirring all the time until the solution turns magenta for some seconds. The number of millimeters of 0.1% sodium hydroxide solution used was recorded.

IV. CHARACTERIZATION OF BIODIESEL FUEL PRODUCED

The analysis of the resulting biodiesel was carried out in other to compare its property with that of fossil diesel fuel. The procedure for the various test carried out are shown below and the results are tabulated in table 1 and Table 2.

A Density

The mass of the oil was measured using pyknometer, hence the density of the oil was calculated using the formula;

$$\rho_t = \frac{m_1 - m_o}{v_t} \qquad (1)$$

Where, Mo is the mass in gram of the pyknometer or density bottle.

M₁ is the mass in gram of the pyknometer filled with water.

V_t is the volume in ml of the oil in the pyknometer at a temperature t.

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B. Refractive Index

The refractive index was determined by placing a drop of the oil sample on the glass slab of the refractometer and the adjustable arms of the meter were adjusted to give reading. It was determined at a temperature of 15°C.

C. Viscosity Test

Some amount of the oil was drawn into the stem of the cannon fenske viscometer (with stokes constant of 0.03) to the mark above the upper bulb of the viscometer. The time taken for the meniscus to fall between the upper and the lower mark of the bulb through the capillary tube of the viscometer was recorded. The viscosity was determined using the following expressions:

Kinematic viscosity =Time of fall × Stokes constant   (2)

The measurement was carried out at 40°C.

D. Specific Gravity Determination

A known volume of the biodiesel fuel was weighed and the weighed recorded, and the same volume of water was also weighed and the weigh recorded. The ratio of the weight of the oil to the weight of water gives the specific gravity of the Biodiesel oil.

E. Flsah Point Test

Pensky- Martens (ASTMD93) method was used to determine the flash point of the Biodiesel fuel, the test cup was filled to a specific level with a sample of Biodiesel. The temperature of the sample was increased rapidly at first and then slowly as the flash point is approached. At 200°C a small test flame was passed across the cup, with a smooth continuous motion of the test flame across the cup, at a point the vapor above the surface of the testing sample was ignited with the aid of the test flame and the temperature at this point was noted and was recorded as the flash point .the test is continued until at temperature was reached where the flame causes the oil sample to ignite and burned consciously for 5sec. the temperature at which this occur is at the fire point.

F. Pour Point Test

The Biodiesel was poured into the test jar to the appropriate level. The cork into which the thermometer was inserted tightly closed the test jar. the position of the cork was adjusted and the thermometer fits the cork tightly. The thermometer and the cork were set coaxial and the thermometer bulb was immersed such that one end of the capillary was 3mm below the surface of the oil. The oil was heated without stirring to 48°C and maintained at this temperature. The fuel was then cooled to 35°C (95°F) in water bath. A jar ring was placed around the testing jar. 25mm from the bottom. The test jar was inserted into the ice jacket. The jacket was supported by the test jar in a vertical position in the cooling bath. After preliminary heating. the sample was cooled at a specific rate and examined at interval of 3°C for flow characteristics. The lowest temperature at which movement of the biodiesel was observed was recorded as the pour point

V. DISTILLATION CHARACTERISTICS

Heat was applied to the distillation flask contents gradually and the initial boiling point (IBP) was observed and recorded. with the tip of the condenser away from the walls of the graduated cylinder. The graduated cylinder was moved immediately. So that the tip of the condenser touches its inner wall. The heating was regulated so that the time from initial boiling point to 10% by volume recovered and the temperature was read on the thermometer and recorded. The heating was continuously regulated so that the uniform average rate of condensation for 10%-99% recovered was obtained. In the interval between the initial boiling point and ends of the distillation. all volumes in the graduated cylinder and all

thermometer readings corresponding to them were recorded. The end point, which is the final boiling point (FBP), was observed and recorded. While the condenser tube continues to drain into the graduated cylinder, the volume was measured accurately and recorded. After the flask has been cooled, its contents were poured into a 5ml-graduated cylinder. The flask was allowed to drain until no appreciable increase in the volume of liquid in the 5ml-graduated cylinder was observed. The value obtained for the percent recovery was added to the percent residue and the total recovery was obtained.

A. Sulphur Test

The sulphur content of the biodiesel produced was determined using ASTM.D 2622 method (Annual book of ASTM, 1987). The sample was placed in an X-ray beam and the intensity of the sulphur X-ray fluorescence was measured.

B. Copper Strip Corrosion Test

For this test, the ASTM.D 130 detection of copper corrosion from petroleum products by the copper strip tarnish test method was used. A polished copper strip was immersed in the biodiesel sample for 3hours at 100^0C and then removed and washed. The condition of the copper surface was qualitatively rated by comparing it with standards.

C. Ash Content

The biodiesel sample (of known weight) was placed in a crucible, ignited and allowed to burn. The carbonaceous residue was heated further in a furnace to convert the entire carbon to carbon dioxide and all the mineral salts to oxides (ash). The ash was then cooled and weighed.

D. Water Content

A known weight of the Biodiesel sample was heated at a constant temperature of 100^0C in an ovum for 50mins and weight was taken after every 10mins. The process was repeated until a constant weight was obtained. After every 10mins the sample was removed and placed in the desiccators for 20 minutes to cool the oil. The sample was then removed and re-weighed. The percentage water content in the oil was then calculated.

E. Cetane Index

Cetane index of a diesel fuel is a measure of the tendency of the fuel to ignite spontaneously. In the Cetane index scale, high values represent fuels that ignite readily and therefore perform better in diesel engines. It was calculated using equation below as recommended by ASTM D 4737(Annual book of ASTM, 1987).

$$CCI = 45.2 + (0.0892) (T_{10N}) + [0.131 + (0.901) (B)][T_{50N}] + [0.0523 - (0.420) (B)][T_{90N}] + [0.00049][(T_{10N})^2] - (T_{90N})^2] - (107)(B) + (60)(B^2) \qquad (3)$$

Where: CCI = calculated Cetane index, D = Density at 15^0C, DN = D – 0.85, B = $[e^{(-3.5)(DN)}] – 1$, T_{10N} = 10%, recovery temperature, T_{10N} = T10 – 215, T_{50N} = T_{50} – 260, T_{90N} = 90% recovery temperature, and T_{90N} = T_{90} – 310

VI.   EXPERIMENTAL RESULTS

The results of various analysis conducted on biodiesel oil production and their characterization are presented in Table 1 to 3

TABLE 1   PRODUCTION OF BIODIESEL FROM NEW AND WASTE OIL AT VARIOUS CONCENTRATION AND TEMPERATURE

PARAMETERS	Using 0.35g NaOH, 20ml methanol at 28⁰C		Using 0.35g NaOH, 20ml methanol at 65⁰C		Using 0.84g of NaOH, 27.38ml ethanol at 28⁰C		Using 0.84g of NaOH, 27.38ml ethanol at 78⁰C	
	N	W	N	W	N	W	N	W
Amount of oil Used (ml)	100	100	100	100	100	100	100	100
Amount of Bio-Diesel produced (ml)	98.25	95.67	99.00	98.33	97.50	94.00	98.50	97.00
Amount of Glycerin produced (ml)	12.63	11.37	14.67	14.00	8.33	6.00	14.60	13.67
Alcohol recovered (ml)	5.40	6.30	4.54	5.10	7.35	7.95	6.00	6.70
Process efficiency (%)	96.90	94.42	98.51	97.86	88.85	84.75	93.50	92.14

TABLE 2   COMPARISONS BETWEEN THE COST OF PETROL DIESEL AND BIODIESEL

MATERIAL	QAUNTITY (L)	COST (₦)
Petroldiesel	1	60
Biodiesel	0.9900	88.9
Glycerin	0.1467	293.4
Cottonseed oil	1	120

*The cost as at Oct 2004

TABLE 3. CHARACTERIZATION OF BIODIESEL PRODUCED

PARAMETERS DETERMINED	Standard values			Experimental values	
	Diesel fuel	Biodiesel	Low sulphur diesel oil	N	W
Density at 15°C g/ml	0.82-0.88	0.87-0.89	0.83-0.86	0.860	0.87
Refractive index at 15°C				1.479	1.46
Specific gravity at 15°C	0.82-0.87			0.859	0.86
Viscosity kinematic at 40°C(Cst)	1.6-1.65	4 -7	3.2	5.700	6.53
Ash content % mass	0.01max	<0.01		0.006	0.007
Sulphur content %	0.05max	0.01max	0.03max	0.0002	0.0005
Water content % mass	0.05	<0.05	<0.05	0.009	0.04
Flash point °C	>52	>100	>60	115	117
Cu strip corrosion (3hrs at 100°C)	No 1 strip	No 1 strip	No 1 strip	No 1 strip	No 1strip
Distillation characteristics IBP (°C)				130.75	126.50
FBP (°C)				334	336.50
% Total recovery	90	>98	>98	98.68	98.30
% Residue				0.73	1.15
% Loss , New oil = unused – refined oil	0.1-1.5			0.60	0.55
Pour point °C	-10 to -0.01	<-10		-5.00	-2.00
Cetane index (calculated)	40min	48-52	45	50.58	51.22

Key:

W = waste oil ie oil that has been used for cooking, frying etc

N= new oil

Standard values from (Geyer etal, 1984, Fuel characterization. n d and Vegetable oil Yields characterization n d)

VII. DISCUSSION OF RESULTS

Biodiesel was produced from cottonseed oil (refined and used) using a base catalyzed trans-esterification process. Temperatures and concentrations of the reacting mixture influenced the rate of the conversion. It could be observed from table 1 that at temperatures close to the boiling point of the alcohol (i.e. 66°C), a higher yield of biodiesel and glycerin were obtained, 99.0% and 14.67% for new oil, and 98.33% and 14.0 for waste oil respectively. Similarly, the overall process efficiencies were higher at high temperatures. The table also shows that using methanol as the solvent gave higher yield of biodiesel at all temperatures than ethanol. However, these could be attributed to the fact that higher temperatures significantly increase internal energy and that reactivity decreases as molecular weight increases down the homologous series.

The result of characterization presented in table 3; shows that the densities and specific gravities of biodiesel produced are (0.86g/ml and 0.859) for new oil and (0.87g/ml and 0.86) for waste oil which are within the range of standard values of (.82-0.88)g/ml and (0.82-0.87)respectively for diesel fuel,(0.88)g/ml for biodiesel and (0.83-0.86)g/ml for low sulfur diesel fuel signifying that the biodiesel produced will

have a better lubricating effect on the engine parts of compression ignition engine (Weiss, 1983).

The flashpoint of biodiesel produced was; 115°C and 117°C for new and waste oil respectively. These values are within the minimum range of: >52°C for a typical diesel fuel given by ASTM, >100°C for a typical biodiesel provided by the national soy diesel development board for biodiesel and >60 °C for a typical low sulphur fossil diesel. This higher value of flash point is advantage over fossil diesel in the event of crash (i.e. safety) in handling (Oil Seeds, n.d). The pour point for biodiesel produced from new and waste oil were found to be -5°C and-2°C respectively which is within the range of standard specification of (-10 to -0.01) for diesel fuel, and (>-10) for biodiesel which is an indication that the biodiesel can support low temperature operability. Similarly, the biodiesel produced passed the copper strip corrosion test at No: 1 strip, meaning it will not cause corrosion of pipe (Oil Seeds, n.d). The Cetane numbers were calculated using equation 3 recommended by ASTM D 4737. The values obtained were 50.58 and 51.22 for biodiesel produced from new and waste oil respectively. This values fall within the standard specification of (48-52) recommended by ASTM D975for biodiesel signifying that the biodiesel is of high ignition quality (less knocking. Distillation characteristic of a product signifies its purity, and from table 3, the distillation recovery of the biodiesel produced is 98.68% and98.30% for new and waste oil respectively. The residue was 0.73% and 1.15% and loss of 0.60% and 0.55% for new and waste oil respectively, these values are within the range of standard specification, which implies that the biodiesel is of high purity (Annual book of ASTM, 1987, Weiss, 1983).

Table 2 shows that the cost of 0.99l of biodiesel is ₦88.9 while for 0.1467l of glycerine is ₦293.4.The cost of producing the same litters of biodiesel and glyceride from 1litre of cotton seed oil is ₦ 225.3, comparing this with the selling price of the two product (₦ 382.3) shows that the process is profitable. Finally, the environmental and ecological advantages of biodiesel should be given more emphasis rather than the economic advantage.

VIII.   CONCLUSION

Production and characterization of biodiesel from cottonseed oil was performed, analysis carried out on the biodiesel produced show that it is essentially sulfur free, has a higher Cetane rating (less knocking), higher lubricating effect than petrol diesel and will not cause corrosion of pipes. Thus the biodiesel can effectively serve as an alternative to petrol diesel without modification to diesel engines and that the environmental and ecological advantages of biodiesel should be given more emphasis rather than its economic importance. Hence, the base catalyzed trans-esterification process using ethanol is an effective method for biodiesel production.

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