



EFFECT OF CATALYST CONCENTRATION AND REACTION TIME ON THE QUALITY OF BIODIESEL PRODUCED FROM USED VEGETABLE OIL



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Abstract: The transesterification of used cooking oil with short-chain alcohols, in the presence of base catalyst sodium hydroxide (NaOH) and methanol as solvent, by means of single step batch process in order to obtain biodiesel fuel was studied using a reaction ratio of 6:1 for alcohol: oil. The process variables that were investigated are catalyst concentration and reaction time. The biodiesel yield for the samples (Sample 1, Sample 2 and Sample 3) were 58 mL, 79 mL and 70 mL, respectively while the glycerine yield for the samples (Sample 1, Sample 2 and Sample 3) were 19 mL, 19 mL and 20 mL, respectively. The reaction times for the three samples (Sample 1, Sample 2 and Sample 3) were 60, 90 and 120 min, respectively. The best result for highest yield and highest purity was at 90 mins reaction time and 1.5 g catalyst concentration. Sample 2 (sample with 1.5 g catalyst concentration, heated at 65°C for 90 min) was found to have the highest cetane rating closer to the ASTM standard which implies that sample 2 will be a more efficient fuel than the other two samples, with guaranteed smooth running of diesel engine as well as clean burning.

Keywords: Biodiesel, cetane rating, characterization, transesterification, used vegetable oil, yield

Introduction

There is a rising demand to globally provide renewable energy by means of a sustainable and ethical approach (Meher *et al.*, 2006). Biodiesel is defined as mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats which conform to American Society for Testing and Materials, ASTM D6751 specifications for use in diesel engines. It is a clean burning alternative fuel, produced from domestic, renewable resources. Biodiesel contains no petroleum, but it can be blended at any level with petroleum diesel to create a biodiesel blend. Biodiesel is simple to use, biodegradable, nontoxic, and essentially free of sulphur and aromatics.

Biodiesel is produced through a chemical process called transesterification whereby the glycerine is separated from the fat or vegetable oil (Fukuda *et al.*, 2001). The process leaves behind two products: methyl esters or biodiesel and glycerine. Biodiesel is much cleaner than fossil-fuel diesel. It can be used in any diesel engine with no need for modifications. In fact diesel engines run better and last longer with biodiesel. Moreover, it can easily be made from common waste product, used cooking oil. Biodiesel is better for the environment because it is made from renewable resources and has lower emissions compared to petroleum diesel (Ramadhas *et al.*, 2005). It is less toxic than table salt and biodegrades as fast as sugar. Biodiesel has physical properties very similar to petroleum-derived diesel fuel, but its emission properties are superior (Demirbas, 2008). Using biodiesel in a usual diesel engine substantially reduces emissions of unburned hydrocarbons, carbon monoxide, sulphates, polycyclic aromatic hydrocarbons, nitrated polycyclic aromatic hydrocarbons, and particulate matter (Lakshmi *et al.*, 2010). Diesel blends containing up to 20% biodiesel called B20 can be used in nearly all diesels powered equipment, and higher-level blends and pure biodiesel, B100 can be used in many engines with little or no modification. Lower-level blends are compatible with most storage and distribution equipment, but special handling is required for higher-level blends. One of the most used renewable energy is biodiesel which is the most common biofuel in Europe.

Biodiesel can be used in any diesel engine when mixed with mineral diesel. In some countries manufacturers cover their diesel engines under warranty for 100% biodiesel use. Many people have run their vehicles on biodiesel without problems.

However, the majority of vehicle manufacturers limit their recommendations to 15% biodiesel blended with mineral diesel. In many European countries, a 5% biodiesel blend, B5 is widely used and is available at thousands of gas stations. Virgin vegetable oils can be used in modified diesel engines. In fact, the diesel engine was originally designed to run on vegetable oil rather than fossil fuel. There are also studies and efforts to commercialize biodiesel from algae (Ramadhas *et al.*, 2009).

Petroleum processes are associated with incessant oil spillage that affect land and water resources of the environment, gas flaring, global warming and climate change which has threatened the health of human beings living in the areas where these crude is being refined and very unstable Petroleum price. It has become necessary to sought for alternative means to source for energy. A major benefit of biodiesel is lower emissions. Biodiesel is environmentally friendly and renewable.

Most used vegetable oil (UVO) ends up in the sewers and landfills where it does no good and doesn't offset any fossil-fuel use. Converting it to biodiesel is a much better option, a social service. Reduce, reuse, and recycle. The US produces an estimated 4.5 billion gallons a year of used cooking oil, and most of it goes to waste.

According to Adepoju (2014), Waste cooking oil (WCO) obtained from Mr. Biggs in Ibadan, Nigeria, is a potential raw material for biodiesel production which are biodegradable, economical, environmental friendly and always available. While efforts has been made to convert waste cooking oil from well-organized places such as Mr Biggs, little or no effort has been made to reach out to the numerous bean and groundnut cake sellers in the local markets and streets in Nigeria. In this study, Bosso Market in Minna, Niger state, Nigeria was carefully selected being a place where we have several people generating WCO or Used Groundnut or Cooking Oil. Hence, the objective of this paper is the production of high quality methyl ester and environmental friendly biodiesel, from Used Cooking Oil.

Materials and Methods

Materials

Materials used in this study include used vegetable oil, Sodium hydroxide, Methanol, Pipette and Pycnometer bottle.

Others are Electric weighing balance, Beaker, Measuring cylinder, Magnetic hot plate and Water bath.

Sampling

The used groundnut oil was sourced from local bean-cake sellers at Bosso market, Minna, Nigeria. The methanol (boiling point of 65°C and 99.5% purity) and sodium hydroxide used as catalyst were manufactured by Sigma Aldrich Chemicals Co. Ltd, England. The semi-colloidal oil was preheated, cooled and then filtered to remove the impurities.

Experimental procedure

Three samples of the used vegetable oil were put in three different beakers to produce the biodiesel at varying temperature, time and quantity of catalyst used using the procedure below.

Characterization of used vegetable oil

Specific gravity: This experiment was carried out in accordance with description reported by Onwuka (2005). A 50 mL pycnometer bottle was washed thoroughly with detergent, water and petroleum ether, it is then dried and weighed. The bottle was filled with water and weighed, the bottle was then dried and filled with groundnut oil sample and weighed. The temperature at which the density was being measured was known, as density changes with changes in temperature (Ibitoye, 2005).

$$\text{Specific gravity} = \frac{\text{weight of groundnut oil (ml)}}{\text{weight of water (ml)}}$$

$$\text{Density} = \frac{\text{weight of oil (g)}}{\text{volume of oil (ml)}}$$

Refractory index: A few drops of the oil was placed on the face of the prism of the refractometer and allowed to gently spread, closed and tightened for sometimes so as to allow for the oil and the prism to attain a steady temperature. The refractive index was read from the demarcation line after adjusting to where it coincides with diagonal crossing (Ibitoye, 2005).

Free fatty acid (FFA): The free fatty acid was estimated by titrating 10 g of waste vegetable oil against sodium hydroxide (NaOH) using phenolphthalein as indicator (Ibitoye, 2005). The acid value is the quantity of NaOH in mg required for neutralizing the free fatty acids present in one gram of the sample. It is expressed as oleic acid (octadec-9-enoic acid) equivalent. The acid value in mg/NaOH/g oil was obtained by using the equation below:

$$AN = \frac{56.1 \times V \times N}{W_{oil}}$$

where;

V= volume of standard alkali used

N= normality of standard alkali used

W_{oil}= weight of oil used

$$FFA = \frac{AN}{2}$$

Iodine value (IV): This is the measure of the degree of unsaturation in a given volume of oil. 0.25 g of the oil was weighed into a glass stopper bottle and 10 cm³ added. It was shaken so as to dissolve the oil. 25 cm³ of iodine monobromide was added from a burette to stopper and then kept in dark for exactly 30 min. After 30 mins during which iodine absorption had taken place, 15 cm³ and 1000 cm³ of distilled water were added. The mixture was titrated against standard sodium sulphate solution until the colour becomes faint yellow. At this point, 1cm³ of starch solution was added. A blank titration was also carried out using the same reagents and distilled water in exactly the same quantity but with the oil (Ibitoye, 2005).

$$\text{Iodine Value} = \frac{0.1269 \times (B-A) \times N \times 100}{W_{oil}}$$

where;

B= volume of standard Na₂CO₃ used in blank titration.

A= volume of standard Na₂CO₃ used in titration with oil.

N= normality of standard Na₂CO₃.

W_{oil}= weight of oil used.

Saponification value: Saponification value is the amount (mg) of alkali required to neutralise definite quantity (1g) of oil. A known quantity of oil is refluxed with an excess amount of alcoholic KOH. After saponification, the remaining KOH is estimated by titrating it against a standard acid. The saponification value of the groundnut oil was determined in accordance to the experimental procedure reported by Onwuka (2005). The alcoholic KOH is freshly prepared by dissolving KOH pellet in ethanol. 2 g of groundnut oil was measured and poured into a conical flask. 25 mL of the alcoholic KOH was added to it. A blank is also carried out. The sample was well covered and placed in a steam water bath for 30 min shaking it periodically, 1 mL of phenolphthalein was added to the mixture and titrated against 0.5M HCl (Ibitoye, 2005).

$$\text{Saponification value} = \frac{(B-A) \times 28.05}{\text{weight of groundnut sample (g)}}$$

Peroxide value: The peroxide value of the groundnut oil was determined in accordance to the experimental procedure reported by Onwuka (2005). 1 g of groundnut was weighed into a clean drying boiling tube, 1 g of powdered potassium iodide and 20 mL of solvent mixture (2 volume of glacial acetic acid + 1 volume of chloroform) were added, the tube was placed in boiling water so that the liquid boils within 30seconds and was allowed to boil vigorously for not more than 30seconds. The content was quickly poured into a flask containing 20 mL of potassium iodide solution; the tube is washed out with 25 mL of distilled water and is titrated with 0.002M sodium thiosulphate solution using starch as indicator (Ibitoye, 2005). A blank titration was also carried out at the same time.

$$\text{Peroxide value} = \frac{T \times M \times 1000}{\text{weight of sample (g)}}$$

Filtration

The used vegetable oil collected from the bean-cake fryers was first properly filtered using a filter paper and transferred into a clean beaker so as to remove completely every food particle present in the oil in order to obtain a very clean, clear and particle free oil which guarantee a good and acceptable result.

Transesterification process

Sodium Hydroxide (1.0 g of NaOH) crystals was put into a beaker and 100 mL of methanol was used to dissolve the NaOH crystals by heating it on a magnetic hot plate with a magnetic stirrer inside the mixture to obtain a Sodium methoxide solution. 100 mL of the used vegetable oil was measured and poured into a separate beaker and was purified by heating in a water bath at a temperature of 45°C for 60 min. 25 mL of the meth oxide solution was then mixed with the 100 mL of purified oil and the mixture was heated for 30 min on the magnetic hot plate with a magnetic stirrer inside the mixture (Schuchardt *et al.*, 1998). This was labelled as sample 1.

Also, Sodium Hydroxide (1.5 g of NaOH) crystals was put into a beaker and 100 mL of methanol was used to dissolve the NaOH crystals by heating it on a magnetic hot plate with a magnetic stirrer inside the mixture to obtain a meth oxide solution. 100 mL of the used vegetable oil was measured and poured into a separate beaker and was purified by heating in a

water bath at a temperature of 65°C for 90 min to remove the free fatty acid content which may alter the result. 25 mL of the meth oxide solution was then mixed with the 100 mL of purified oil and the mixture was heated for 30 min on the magnetic hot plate with a magnetic stirrer inside the mixture (Schuchardt *et al.*, 1998). This was labelled as sample 2.

Moreover, Sodium Hydroxide (2.0 g of NaOH) crystals was put into a beaker and 100 mL of methanol was used to dissolve the NaOH crystals by heating it on a magnetic hot plate with a magnetic stirrer inside the mixture to obtain a meth oxide solution. 100 mL of the used vegetable oil was measured and poured into a separate beaker and was purified by heating in a water bath at a temperature of 70°C for 120 min to remove the free fatty acid content which may alter the result. 25 mL of the meth oxide solution was then mixed with the 100 mL of purified oil and the mixture was heated for 30 mins on the magnetic hot plate with a magnetic stirrer inside the mixture (Schuchardt *et al.*, 1998). This was also labelled as sample 3.

Settling

The resulting samples were kept in desiccators for 48 hours to allow for separation of the biodiesel from glycerine after which the biodiesel gotten was separated by a decantation process and was washed with water and heated to obtain pure biodiesel.

Characterisation of the biodiesel produced

Determination of specific gravity by hydrometer method (ASTM D1298)

This procedure is used to measure the specific gravity of the biodiesels. A clean dry empty (50 mL capacity) density bottle was weighed and the mass recorded as *m*, it was then filled up with distilled water and subsequently with the samples. The mass of the bottle and water was taken and recorded as *m*₁ and that of biodiesel as *m*₂ respectively hence, the specific gravity was evaluated.

$$\text{Specific gravity (biodiesel produced)} = \frac{m_1 - m}{m_2 - m}$$

Determination of flash point by Pensky-Martens closed cup tester (ASTM D 93)

A sample of the biodiesel was heated in a close vessel and ignited. When the sample burns, the temperature was recorded; the Pensky-Martens cup tester measures the lowest temperature at which application of the test flame causes the vapour above the sample to ignite. The biodiesel was placed in a cup in such quantity as to just touch the prescribed mark on the interior of the cup. The cover was then fitted onto the position on the cup and Bunsen burner was used to supply heat to the apparatus at a rate of about 5°C per min. During heating, the oil was constantly stirred. As the oil approaches its flashing, the injector burner was lighted and injected into the oil container after every 12 second intervals until a distinct flash was observed within the container. The temperature at which the flash occurred was then recorded, it was repeated three times and the average value was taken (Van Gerpen, 2005).

Determination of cloud point (ASTM D 2500)

A sample of the biodiesel was placed in a test jar to a mark and then placed inside a cooling bath. The temperature at which the biodiesel starts to form cloud was taken as the cloud point (Van Gerpen, 2005).

Determination of kinematic viscosity (ASTM D 445)

A viscometer was inserted into a water bath with a set temperature and left for 30 min. The sample was added to the viscometer and allowed to remain in the bath as long as it reaches the test thermometer. The sample was allowed to flow freely and the time required for the meniscus to pass from the first to the second timing mark was taken using a stop watch. The procedure was repeated a number of times and the average value was taken which was then multiplied with the

viscometer calibration to give the kinematic viscosity (Van Gerpen, 2005).

Determination of pour point (ASTM D 97)

A sample of the biodiesel kept in the freezer at about 5°C was removed and then placed in a heating mantle to melt. The temperature at the bottom of the test jar that is the temperature at which the biodiesel starts to pour is taken as the pour point (Van Gerpen, 2005).

Cetane number of diesel fuel oil ASTM D 613

Cetane Number is a measure of the fuel's ignition delay; Higher Cetane numbers indicate shorter times between the injection of the fuel and its ignition. Higher numbers have been associated with reduced engine roughness and with lower starting temperatures for engines (Van Gerpen, 2005).

Acid number of petroleum products by titration ASTM D 664

The biodiesel sample was measured and poured in a beaker. A neutral solvent (a mixture of petroleum ether and ethanol) was prepared and 50 mL of it was taken and poured into the beaker containing the biodiesel. The mixture was stirred vigorously for 30 min. 0.56 g of potassium hydroxide (KOH) pellet was measured and placed in a separate beaker and 0.1M KOH was prepared, 3 drops of phenolphthalein indicator added to the sample and was titrated against 0.1M KOH till the colour change observed turned pink (Van Gerpen, 2005).

Results and Discussion

Table 1 shows a comparison of chemical properties and fatty acid composition (%) of used cooking oil (UCO). UCO has higher specific gravity because it has much residue such as water has mixed inside the UCO because of condensation of cooking process. Therefore, the density of UCO is high. From Table 2, some significant differences were found between the Reference (REF) and biodiesel fuel. The reference fuel (REF) is a typical low sulphur diesel fuel similar to those available in Spanish petrol stations in winter (Magin *et al.*, 2008).

Table 1: Chemical properties and fatty acid composition (% of used cooking oil (UCO))

Property	Fatty acid % composition UCO
Palmitic acid C16:0	16.00
Stearic acid C18:0	5.21
Oleic acid C18:1	34.28
Linoleic acid C18:2	40.76
Specific gravity	0.92

Table 2: Specifications of biodiesel fuels

Properties	REF	UCO
Density at 15 °C (Kg/m ³)	834	887
Kinematic viscosity at 40°C (cSt)	2.72	5.16
Gross heating value (MJ/kg)	45.54	39.26
Lower heating value (MJ/kg) _a	42.49	36.59
Acid Number (mg KOH/g)	0.10	0.55
% C (wt.)	86.13	76.95 _b
% H (wt.)	13.87	10.91 _b
% O (wt.)	0	12.14 _b
Sulphur Content (ppm wt.)	34	0 _b
Water Content (ppm wt.)	57	466
IBP (°C)	172	320
T10 (°C)	211	325
T50 (°C)	270	333
T90 (°C)	340	356
Molecular weight	211.7 _c	293.2 _b
Stoichiometric fuel/air ratio	1/14.67	1/12.55
CFPP (°C)	-18	-6
Iodine Number	-	97.46
Renewable fraction	0	90.11 _d

Source: Extract from Mahfuzah B. M.; *a*- Calculated from composition and gross heating value; *b*- Calculated from speciation; *c*- Calculated by Aspen-Advisor software; *d*- Calculated from used cooking oil composition

Table 3: Characterization of used vegetable oil

Properties	Used Vegetable Oil
Acid value	17.391 mgOH/g
FFA	8.70 mg/g
Iodine value	119g
Peroxide value	10
Saponification value	191.388 mg/g
Specific gravity	0.912
Refractive index	1.463

Table 4: Biodiesel produced and its yield

	Quantity of oil (mL)	Quantity of catalyst (g)	Temp (°C)	Time (Mins)	Biodiesel Produced (mL)	Glycerine Produced (mL)
Sample 1	100	0.5	45	60	58±0.04	19±0.01
Sample 2	100	1.5	65	90	79±0.05	19±0.03
Sample 3	100	2.0	70	120	70±0.02	25±0.02

The results of the characterization of the used vegetable oil and the quantity of biodiesel produced are as shown in Table 3 and 4, respectively. The results of the characterization of the produced biodiesel are shown in Tables 5 to 7.

Effect of catalyst concentration on purity

The concentrations of catalysts used vary for the three samples with the same quantity of oil. When 0.5 g of the catalyst was used for the first sample, it was discovered that the purity of the biodiesel obtained did not conform to the acceptable standard and this could prove costly for its use in automobiles using diesel engines. This is because the viscosity of the diesel, cetane rating among other properties were below the recommended values and this could lead to excessive use of the diesel by automobiles and smoky exhaust. This is not advisable as smoky exhaust causes the release of carbon monoxide to the environment, which could prove harmful to the people living in that area and the excessive increase in the cost of maintaining the vehicle for the user (Jose *et al.*, 2005).

Effect of catalyst concentration on yield

The yields obtained are of three different volumes despite using the same quantity of oil for the production. The yield of the first sample was the smallest of the three. The third sample has a catalyst concentration of 2.0 g followed by the second with a catalyst quantity of 1.5 g. The second sample produced more biodiesel because of the reaction ratio of the oil sample to the catalyst. From this, it can be deduced that a biodiesel produced using 6:1 for alcohol: oil will yield more product and less glycerine (Jose *et al.*, 2005).

Effect of reaction time on purity

The reaction time of 90 min produced the biodiesel that is in its purest state than the other two at 60 and 120 min. This implies that at either lower reaction or higher reaction time than 90 min, the biodiesel produced may likely be of low quality as well as contain some form of impurities. This can be checked by ensuring that diesel produced at acceptable reaction ratio is allowed to completely react at corresponding time, which will enhance the purity of the diesel, produced (Jose *et al.*, 2005).

Table 5: Characterization of Biodiesel produced (Sample 1)

Test	Unit	Test Method		Limit	Result
		IP	ASTM		
Specific gravity	kg/l	160	D1298	0.95 max.	0.88±0.001
Total sulphur	% wt	107	D4294	0.5 max.	0.006±0.001
Flash point	°C	54	D93	150 min.	ND
Pour point	°C	219	D97	70 max.	ND
Kinematic viscosity	c.s.t	71	D445	26 max.	4.65±0.01
Diesel index	°C	21	-	47 min.	18.0±0.01
Cetane number	-	-	D975	40 min.	52.5±0.03
Free Glycerine	% mass	-	-	0.02	0.019±0.01
Total Glycerine	% mass	-	-	0.24	0.17±0.02
Cloud point	°C	219	D2600	40 max.	20±0.00
Water by Distillation	% vol.	53	D95	0.5 max.	Trace
Acid value	mgKOH/g	65	D108	0.5 max.	0.43±0.01

Table 6: Characterization of biodiesel produced (Sample 2)

Test	Unit	Test Method		Limit	Result
		IP	ASTM		
Specific gravity	kg/l	160	D1298	0.95 max.	0.89±0.01
Total sulphur	% wt	107	D4294	0.5 max.	0.006±0.01
Flash point	°C	54	D93	150 min.	ND
Pour point	°C	219	D97	70 max.	ND
Kinematic viscosity	c.s.t	71	D445	26 max.	4.65±0.02
Diesel index	°C	21	-	47 min.	19.0±0.01
Cetane number	-	-	D975	40 min.	53.5±0.03
Free Glycerine	% mass	-	-	0.02	0.019±0.01
Total Glycerine	% mass	-	-	0.24	0.18±0.01
Cloud point	°C	219	D2600	40 max.	20±0.00
Water by Distillation	% vol.	53	D95	0.5 max.	Trace
Acid value	mgKOH/g	65	D108	0.5 max.	0.46±0.01

Table 7: Characterization of biodiesel produced (Sample 3)

Test	Unit	Test Method		Limit	Result
		IP	ASTM		
Specific gravity	kg/l	160	D1298	0.95 max.	0.88±0.01
Total sulphur	% wt	107	D4294	0.5 max.	0.006±0.01
Flash point	°C	54	D93	150 min.	ND
Pour point	°C	219	D97	70 max.	ND
Kinematic viscosity	c.s.t	71	D445	26 max.	4.65±0.02
Diesel index	°C	21	-	47 min.	19.0±0.01
Cetane number	-	-	D975	40 min.	53.1±0.02
Free Glycerine	% mass	-	-	0.02	0.019±0.01
Total Glycerine	% mass	-	-	0.24	0.17±0.01
Cloud point	°C	219	D2600	40 max.	20±0.00
Water by Distillation	% vol.	53	D95	0.5 max.	Trace
Acid value	mgKOH/g	65	D108	0.5 max.	0.45±0.01

Effect of reaction time on yield

The results obtained shows that the highest quantity of biodiesel produced was at a reaction time of 90 min. This implies that the biodiesel produced at 60 and 120 min have lower yield which suggest that with a reaction time of 90 min, the second sample produced more biodiesel with good combustion properties and less glycerine than the other two samples with a reaction time of 60 mins and 90 mins. This result agrees with the findings of Jose *et al.* (2005).

Conclusion

The biodiesel produced at a temperature of 65°C, heating time of 90 min and 1.5 g (NaOH) catalyst was found to be of higher quality and generates more yield than the rest. Also, the biodiesel produced at this condition has a good reaction ratio (oil: catalyst). In conclusion, high yield of quality biodiesel can be produced using used vegetable oil as feedstock with a good reaction ratio, appropriate concentration of catalyst, temperature and time of heating.

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