PHYSICOCHEMICAL PROPERTIES OF BIODIESEL PRODUCED BY TRANSETERIFICATION OF VARIOUS ESTERIFIED WASTE FRYING OILS

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ABSTRACT

This study aims at producing biodiesel from esterified waste frying oils (EWFOs), determine the physicochemical properties of the EWFO derived biodiesel (EWFO - Bs) and compare their properties to ASTM standards. The free fatty acid (FFA) contents of the EWFOs were analysed while parameters of the EWFO-Bs analysed included specific gravity at 20°C (specific gravity bottle), kinematic viscosity at 25°C (Oswald viscometer), moisture content (oven drying method), iodine value, saponification value, acid value (by titrimetry), flash point (flash point tester), cloud point and pour point (instrumental methods). The free fatty acid (FFA) contents of only 4 EWFO samples were below 0.5% while others ranged between 0.70% and 2.59%. The trans esterification reactions were maintained at the optimum process conditions of methanol to oil molar ratio of 6:1; at 60°C; for 40 minutes; at 200rpm and the varying catalyst concentration was calculated on the basis of 100 ml of oil reacting with 0.35 g of catalyst. The highest yield of biodiesel obtained was 83.07%. The results of the kinematic viscosity (18.275±1.095 – 82.124±5.729 mm^2/s) and moisture content (0.249±0.057 – 1.333 ±0.049 %) did not conform to the ASTM D6751 standard but the results of the specific gravity, iodine value, saponification value and the flash points of all the biodiesels produced conformed with the ASTM D6751 standard. It is concluded that biodiesel yield was highest if the FFA content of the EWFO is $\leq$ 0.70.

Key Words: Biofuel, esterified oil, trans esterification, biodegradable, saponification and flash point

INTRODUCTION

The negative environmental impacts of fossil fuels and unstable oil prices have led to the search for alternative fuels Saravanan *et al.,*.[1], Jitputti *et al.,*.[2]. Nigeria is one of the top-ten petroleum producers in the world and a leading gas exporter but it suffers perennial energy crisis and it is important that other alternative sources of energy that will supplement fossil fuel, natural gas and hydro sources are explored Akande and Olorunfemi[3]. Biodiesel is an alternative fuel for diesel engines and it is regarded as non-flammable, non-toxic and biodegradable. Biodiesel can be used as the pure product or can be blended with petroleum derived diesel (petro diesel). Many countries already have biofuel and bioenergy programs and devote a lot of resources in research and development of newer methods of biomass exploitation Coronado *et al.,*. [4]. For instance, the US has more than 125 biodiesel plants and production increased from about 25 million gallons in early 2000s to 2.6 billion gallons in 2018 National Biodiesel Board[5]. The industry supports over 60,000 jobs and generates billions of dollars in GDP, household income and tax revenues Biodiesel basics. Biofuel supplements a substantial part of the energy requirements in countries like Brazil, China, India and Taiwan.

The most common method for biodiesel production is by chemically reacting triglycerides contained in vegetable oils or animal fats with an alcohol like methanol or ethanol, a process called trans esterification. Trans esterification can be catalysed by bases, acids and enzymes or may be carried out without a catalyst. Vegetable oils like palm oil, palm kernel oil (PKO), soybean oil, physic nut, ground nut oil, cotton seed oils and coconut oil have been extensively studied as raw materials for biodiesel production Alamu *et al.,*.[6], Hasibuan *et al.,*.[7], Aransiola *et al.,*.[8], Akoh *et al.,*.[9]. However, the use of edible vegetable oils for biodiesel production competes with their use as food and in the production of food forms. Focus is currently on the use of waste raw materials like waste frying oil (WFO) for biodiesel production. The major drawback with WFO is its relatively high free fatty acid (FFA) content as a result of repeated use in frying. Its major advantage is availability given the widespread existence of fried food vendors in every nook and cranny of Nigeria. However, despite the overwhelming WFO and biomass resources of Nigeria, biofuel and biodiesel production is yet to make the required impact. Agbro and Ogie[10] concluded that given the large availability of biomass resources in Nigeria there is immense potential for biofuel production from these biomass resources and it offers the prospect for employment generation for the huge army of unemployed youths. Simonyan and Fasina[11] estimated that Nigeria is capable of producing about 2.01 EJ (47.97 million tonnes of oil equivalent, MTOE) of energy from the approximately 168.49 million tons of agricultural residues/wastes that is generated annually. This study aims to produce biodiesel from various esterified waste frying oil (EWFO) samples and to compare percentage yields from the EWFOs.

MATERIALS AND METHODS

Sample Collection

The samples used for this study were 10 esterified waste frying oils obtained from the Department of Chemistry, Michael Okpara University of Agriculture, Umudike. The free fatty acid (FFA) contents of the oil samples before and after esterification were also obtained.

Transesterification of the Esterified Oil

The weight of the oil was determined and used to calculate the required volume of the oil to be used, the required volume of the methanol and the amount of NaOH needed. The oil was then pre-heated using a Thermo Fisher electric hot plate with magnetic stirrer (model 4100 – PP - 01) to 60^{o}C in a closed reaction vessel containing a submerged thermometer probe (Controls digital thermometer, model D1227). 20 ml Methanol was mixed with 0.35 g of NaOH and the mixture was stirred until NaOH dissolved to give a sodium methoxide solution. Methanol (20 ml) was mixed with NaOH (0.35 g) and the mixture was stirred until NaOH dissolved to give a sodium methoxide solution. When 100 ml of oil (EWFO 1) was at 60^{o}C (below the boiling temperature of methanol), the methoxide solution was added into the heated oil in the vessel and stirred rapidly and continuously with the magnetic stirrer on the hot plate at 200rpm. This was carried out for 40 min and stopped. The mixture was transferred into a separating funnel and was allowed to cool. It separated out into two distinct layers; the lower glycerol phase and the upper phase containing the fatty acid methyl ester (FAME) or biodiesel. The weight of the biodiesel product and glycerol were taken and recorded respectively. This procedure was also carried out for samples EWFO 2 – EWFO 10 and their weights were taken and recorded.

Washing of the Biodiesel

The biodiesel produced was washed thrice with 10 % phosphoric acid water (55^{o}C) and stirred; the biodiesel-water mixture separated into the upper biodiesel and lower phase of the milky water. The water was run off from the separating funnel. This was repeated thrice until the water became clearer.

Drying of the Biodiesel and Calculation of Percentage Yield

The biodiesel was dried with the addition of anhydrous sodium tetraoxosulphate (VI), Na_2SO_4. This was allowed to stand for 10 min and filtered using a muslin cloth to remove the salt (Na_2SO_4). The biodiesel was then heated to 110^{o}C using a heating mantle, to remove any trace of water. The biodiesel was weighed and then stored in an air-tight green covered sterilized bottle pending analysis. Percentage yield was calculated as:

$$\%\text{yield} = \frac{\text{Weight of biodiesel x 100}}{\text{Weight of pre heated esterified oil}}$$

Physico-chemical Analyses of the Biodiesel

Specific Gravity was determined with a Hecht Karl pycnometer by relating the weight of each oil sample with the weight of an equivalent volume of water. Moisture Contents were determined by heating 2 g of each sample, cooling and weighing until constant weight was achieved and calculating the moisture content based on difference in weights. pH of the waste frying oils was determined with a pH meter while viscosity was determined using an Thomas Ostwald oil viscometer at the laboratory room temperature of $37^{o}C$. Saponification value was determined by the titration of a mixture of oil and ethanolic KOH with 0.5M HCl solution with phenolphthalein as indicator. Acid value was determined by warming and titrating a mixture of the sample and industrial methylated spirit (IMS) previously neutralized to a phenolphthalein end point with 0.1M alcoholic KOH to a pink end-point. Free fatty acid (FFA) determination was done by boiling a mixture of the oil sample and neutral alcohol on a hot plate, stirring with a magnetic stirrer until the oil dissolved and titrating with 0.1M NaOH using phenolphthalein as indicator until a pink colouration appeared. The iodine value which is the number of milligrams of iodine absorbed per gram of oil sample was determined by titrating with 0.1M sodium thiosulphate solution with and without the test substance while peroxide value determination was carried out in terms of milli equivalent of peroxide per 1000g (1 kg) of sample which oxidizes potassium iodide under the conditions of the test.

Determination of Flash, Cloud and Pour Points of Biodiesel

Flash Point was determined with a Tanaka Scientific Flash Point Tester which was connected to the source of power and the gas line was opened. All parts of the cup and its accessories were carefully cleaned and dried before commencing the analysis. The cup was filled with the biodiesel sample as indicated by the filling mark and a syringe was used to reduce the sample in the cup when the filling mark was exceeded. The lid was placed in the cup and the cup was set inside the stove, while ensuring that the locating groove was properly aligned. The thermometer probe was inserted into the thermometer port and the stirrer was connected to the stirrer motor by turning the placement screw counter clockwise and inserting the flexible stirrer.

The placement screw was turned clockwise to tighten while ensuring that the flexible stirrer had a smooth curve before being inserted into the motor. The test flame was lighted with an external ignition source (lighter) and the flame was adjusted using the coarse adjustment screw.

The value screw was used to adjust the flame so that it was 5/32 in diameter, the same size as the bead provided for comparison. The stirrer motor was shut off during the application of test flame which was applied at each temperature reading by turning the screw knob clockwise. The temperature at which the flame flashes light was noted and recorded as the flash point. The procedure was done thrice and average flash point readings of the biodiesel samples were tabulated with the corresponding viscosity.

Cloud Point was determined by half-filling a 50 ml beaker with the biodiesel sample and immersing the probe of a Controls digital thermometer (Model D1227) in the biodiesel. The 50 ml beaker was placed inside a 500 ml beaker containing ice blocks. The thermometer was carefully observed to note when the biodiesel had started becoming cloudy and was no longer transparent. The temperature at which the cloudiness starts to form was noted as the cloud point of the biodiesel (EWFO 1). The procedure was repeated twice with the EWFO 1 sample and the average was taken at 11^{o}C. The procedure was repeated for sample EWFO 2 – EWFO 10 and their average readings were recorded.

Pour Point was determined by continuing the immersion of the beaker containing the biodiesel into the ice block after cloud point determination. The flow of the biodiesel was checked at 5 s intervals and the point when the congealed biodiesel stops flowing and formed an immovable solid was carefully observed. The temperature at which this had occurred was recorded. The procedure was repeated thrice for each biodiesel samples, average of the three readings was taken as the pour point and the results were tabulated.

Prolonged Storage of Biodiesel

The EWFO derived biodiesel samples were stored, after physicochemical analyses, in a green covered sterilized bottles normally used for storing blood. The bottle was initially saturated with EDTA but this was rinsed off with distilled water prior to storing the biodiesel. The biodiesel produced was stored for 11 months.

RESULTS AND DISCUSSION

Result

Table 1 below shows the FFA contents of the oils before and after esterification.

Table1: FFA Contents of Waste Frying Oil Samples

Sample ID	% FFA values of WFO before esterification	% FFA of esterified oil
EWFO 1	3.12	0.26
EWFO 2	12.09	0.96
EWFO 3	4.29	2.59
EWFO 4	2.38	0.26
EWFO 5	8.58	0.86
EWFO 6	1.95	0.34
EWFO 7	3.12	0.44
EWFO 8	1.95	2.34
EWFO 9	3.12	2.26
EWFO 10	3.51	0.70

EWFO = esterified waste frying oil

Results of Transesterification Reaction

The amounts of oil, methanol and NaOH used for the transesterification reaction are shown in Table 2 below

Table 2. Quantities of Chemicals used for the Transesterification Reaction

Sample ID	Volume of esterified oil (ml)	Weight of preheated oil (g)	Volume of methanol (ml)	Weight of NaOH (g)
EWFO 1	100.00	93.493	20.00	0.35
EWFO 2	100.00	98.940	20.00	0.35
EWFO 3	100.00	95.700	20.00	0.35
EWFO 4	100.00	93.297	20.00	0.35
EWFO 5	100.00	91.214	20.00	0.35
EWFO 6	100.00	91.170	20.00	0.35
EWFO 7	100.00	95.370	20.00	0.35
EWFO 8	100.00	84.433	20.00	0.35
EWFO 9	100.00	93.105	20.00	0.35
EWFO 10	100.00	95.504	20.00	0.35

Pre-heating led to the varying of weights with EWFO 2 having the highest weight of 98.94 g while EWFO 8 had the lowest 84.433 g.

Table 3 shows the transesterification reaction conditions, amount of EWFO derived biodiesels (EWFO-Bs) produced and percentage yield of biodiesel.

Table 3. Transesterification Reaction Conditions, amount of Biodiesel Produced and Percentage Yields

Sample ID	Reaction temp.(^oC)	Reaction time(min)	Stirring rate(rpm)	Weight of Biodiesel(g)	Weight of Glycerol(g)	Percentage Yield (%)
EWFO 1	60	40	200	73.252	20.241	78.35
EWFO 2	60	40	200	62.807	36.133	63.48
EWFO 3	60	40	200	16.786	78.914	17.54
EWFO 4	60	40	200	68.881	24.416	73.83
EWFO 5	60	40	200	65.820	25.394	72.16
EWFO 6	60	40	200	69.772	21.398	76.53
EWFO 7	60	40	200	75.495	19.875	79.16
EWFO 8	60	40	200	30.700	53.733	36.36
EWFO 9	60	40	200	47.754	45.351	51.29
EWFO10	60	40	200	79.335	16.169	83.07

Percentage yield was highest for the biodiesel derived from EWFO 10 and lowest for biodiesel derived from EWFO 3. Indeed EWFO-Bs from EWFOs 3, 8 and 9 had the lowest percentage yields corresponding to their relatively high FFA contents (> 1%).

Results of the Physico-chemical Analyses of Biodiesel

The results of the physico-chemical analyses of the esterified waste frying oil derived biodiesels (EWFO-Bs) are presented in Table 4. Mean specific gravity of the biodiesel had a range of 0.862 ±0.077 – 0.876 ±0.084 which showed conformance with the ASTM D6751 standard (0.86 – 0.90). The specific gravity of biodiesel depends on the composition of fatty acid compounds and the densities of biodiesel will vary with their fatty acid composition and their purity according to Tat and Van Gerpen[12]. It is an important fuel property, because injection systems, pumps and injectors must deliver an amount of fuel precisely adjusted to provide proper combustion Dzida and Prusakiewicz[13]. The specific gravity values must be maintained within tolerable limits to allow optimal air-to-fuel ratios for complete combustion. High specific gravity biodiesel can lead to incomplete combustion and particulate matter emissions Galadima *et al.*,[14].

Table 4. Mean Physicochemical parameters of Biodiesel Produced from the WFOs

Analysis	**EWF-B1**	**EWFO B2**	**EWFO B3**	**EWFO B4**	**EWFO B5**	**EWFO-B6**	**EWFO B7**	**EWFO B8**	**EWFO B9**	**EWFO B10**	**ASTM D6751 Standard**
Physical state	Liquid	liquid	Liquid	Liquid	liquid	liquid	Liquid	Liquid	liquid	Liquid	Liquid
Colour	Dark brown	Dark brown	Pale brown	Pale brown	Dark brown	Amber yellow	Amber yellow	Amber yellow	Amber yellow	Amber Yellow	Amber yellow
Specific gravity@20°C	0.868 ±0.051	0.871 ±0.037	0.876 ±0.084	0.866 ±0.039	0.862 ±0.077	0.867 ±0.058	0.869 ±0.027	0.870 ±0.065	0.869 ±0.044	0.867 ±0.028	0.86-0.90
Kinematic Viscosity (mm^2/s) @25°C	20.753 ±2.117	36.311 ±2.870	82.124 ±5.729	53.883 ±3.634	46.286 ±1.980	47.933 ±3.615	78.764 ±4.116	60.662 ±3.221	80.113 ±2.969	18.275 ±1.095	1.9-6.0@ 40°C
%Moisture content	0.249 ±0.057	0.881 ±0.065	1.333 ±0.049	0.639 ±0.104	0.998 ±0.133	0.246 ±0.060	0.397 ±0.032	1.077 ±0.221	1.205 ±0.093	0.246 ±0.068	0.05% (maximum)
Iodine value ($gI_2/100g$)	5.457 ±0.289	3.807 ±0.150	6.726 ±0.821	9.264 ±0.879	7.995 ±0.316	1.269 ±0.254	9.644 ±0.763	3.934 ±0.305	2.919 ±0.171	0.381 ±0.062	120 max EN14214

Kinematic viscosity is one of the parameters which mostly affects the performance of the automobile engine and the emission characteristics Alptekin and Canakci[15]. In this study, the mean kinematic viscosity of the biodiesel measured at 25°C had a range of 18.275 ±1.095 – 82.124 ±5.729 mm^2/s which does not conform to the ASTM D6751 standard (1.9 – 6.0 mm^2/s) at 40°C. These values are higher than that of a petroleum diesel (2.03mm^2/s), indicating that biodiesel has large molecular mass Jaichandar and Annamalai[16]. Also, increase in temperature decreases the viscosity of biodiesel and the viscosity measurement of the obtained biodiesel was conducted at the room temperature of the laboratory which is 25°C while the ASTM D6751 standard for the measurement of biodiesel viscosity is at 40°C. Cost and time limitations of incubating biodiesel samples led to the viscosity being measured at the laboratory room temperature of 25°C and this likely accounts for the high differences in viscosity and high deviation from ASTM standard.

The moisture contents of the biodiesel samples made from the esterified waste frying oils reflects the quality of the obtained biodiesel. The maximum amount of allowable water content in biodiesel as specified in ASTM standard D6751 is 0.05%. High moisture content in biodiesel may cause many problems such as water accumulation and microbial growth in fuel handling, storage and transportation equipment (Gerpen *et al.,.,*[17a]. It is often caused by improper treatment after processing or by absorption of atmospheric moisture during storage Gerpen *et al.,.,*[17b] EWFO-B1, EWFO-B6, EWFO-B7 and EWFO-B10 had the lowest moisture contents of 0.249%, 0.246%, 0.397% and 0.246% but these exceeded the standard of 0.05% for biodiesel.

The iodine value (IV) is the measure of degree of unsaturation in oil. It is constant for a particular oil or fat. The IV is a useful parameter in studying oxidative rancidity and chemical stability properties of different oil and biodiesel fuels. Oils with iodine value above 125 are classified as drying oils; those with iodine value of 110 – 140 are classified as semi - drying oils and those with iodine value less than 110 as considered as non-drying oil. Oils that are susceptible to drying are also susceptible to becoming rancid and should not be preserved for a long period of time. The iodine values of all the biodiesel samples of this study are less than 110 (range; 0.381 – 9.644$gI_2/100$ g). These values are significantly lower than the maximum value of 120 g $I_2/100$ g approved by ASTM D6751. According to Drapcho *et al.,*.[18], the lower the IV, the better the fuel will be as a biodiesel. IVs greater than

50 may result in decreased engine life but give better viscosity characteristics in cooler conditions.

The saponification value (SV) indicates the amount of saponifiable units (acyl groups) per unit weight of oil. A high SV indicates a higher proportion of low molecular weight fatty acids in the oil or vice versa Diwakar *et al.,*.[19]. Saponification values (mg KOH/ g) of the EWFO derived biodiesels varied from 175.31 to 221.60 mg KOH/g. these were higher than the ASTM recommended minimum, 100 mg KOH/g. According to ASTM D664[20] the acid number is expressed in milligram (mg) KOH required to neutralize 1g of fatty acid methyl ester (FAME) or biodiesel. Determination of acid value (AV) is an important test to assess the quality of a particular biodiesel. The acid value of a starting material can play an important role on the % FAME of the final product (Charoenchaitrakool and Thienmethangkoon[21]. The maximum level of AV of pure biodiesel as specified in ASTMD6751 standard is 0.8 mg KOH/g. The AV of EWFO-Bs 1, 2, 4, 6, 7 and 10 were 0.137, 0.738, 0.547, 0.356, 0.465 and 0.273 respectively which are below the ASTM standard while the acid values of EWFO-Bs 3, 8 and 9 were 1.641, 1.285 and 1.367 respectively which are above the limit. It can be observed that the acid values of the biodiesel produced correspond with their various FFA contents of the EWFOs (Table 1). FFA contents for EWFOs 3, 8 and 9 were high (>1% w/w) which will cause soap formation and the separation of the products will be exceedingly difficult leading to a low yield of biodiesel Crabbe *et al.,*.[22].

Flash Point, Cloud Point and Pour Point of the Biodiesel

Flash point is the measure of flammability of the fuel and the values of flash point obtained in this study (range; 131 – 207^{o}C) showed conformance to the ASTM D6751 standard (130^{o}C minimum). The flash point of biodiesel corresponds to both the percentage amount of hydrocarbon saturated compounds and the residual of alcohol. The high sensitivity of the flash point with respect to the residual alcohol content of the biodiesel is very clear Jorge *et al.,*.[23]. In terms of safety, the lower the flash point, the higher the cost for the storage system. In this study, the flash points of the obtained biodiesel were above the ASTM D6751 minimum limit of 130^{o}C and therefore pose no risk of fire outbreaks in case of accidents.

A diesel fuel's cold-weather characteristics are measured by the cloud point (CP), the cold filter plugging point (CFPP), and the pour point (PP). The cloud point is the temperature of the fuel at which small, solid crystals can be observed as the fuel cools. The cold filter plugging point is the temperature at which a fuel filter plugs due to fuel components that have crystallized or gelled. The pour point refers to the lowest temperature at which there is movement of the fuel when the container is tipped. Compared to petro-diesel, biodiesel tends to have a much narrower range of temperature between the cloud point and the pour point. While there may be a 20-degree difference between the cloud point and the pour point of petroleum diesel, biodiesel may have a difference of only a few degrees Farm Energy[24a]. The cloud points of EWFO-Bs 1, 2 and 7 was 11^{o}C while those of EWFO-Bs 5 and 6 was 17^{o}C. But the cloud points of EWFO-Bs 3, 4, 8, 9 and 10 were 26, 7, 9, 13 and 18^{o}C respectively. The tests for cloud point and pour point are relatively quick and easy, so they are used to estimate the cold filter plugging point, which is the actual temperature above which a fuel can be used. It is possible to test a fuel for its cold filter plugging point, but the test is expensive and tedious. Therefore, most people use the cloud point and pour point to bracket the temperature at which the fuel will start to fail. A fuel may still work in an engine even if the temperature is below the cloud point. However, the fuel will definitely not work below the pour point (after it has gelled) Farm Energy [24b]. ASTM D6751 does not specify the required cloud point for biodiesel, but requires that the cloud point be reported to the customer. The results of the pour point of biodiesel varied from 5 – 21^{o}C.

Biodiesel Storage

The biodiesel samples were stored in airtight containers for 11 months. At the end of the storage period, it was found that EWFO-Bs 1 and 10 were still clear, transparent and with no deposits, EWFO-Bs 2 and 5 were reddish in colour, EWFO-B 4 had a slight dark brownish deposit at one side of the bottle, while EWFO-Bs 3 and 9 were slightly congealed, EWFO-B 8 was cloudy while EWFO-Bs 6 and 7 had golden colour.

This phenomenon relates to contact with air (oxidative stability) and water (hydrolytic stability) which are the major factors affecting the storage stability of biodiesel. Oxidation is usually accompanied by an increase in the acid value and viscosity of the fuel. Often, these changes are accompanied by the darkening of the biodiesel colour from yellow to brown and the development of a paint smell. In the presence of water, the esters can hydrolyze to long chain FFA, which also cause the acid value to increase Gerpen and Knothe[25].

CONCLUSION

Biodiesel cannot economically compete with petroleum-based diesel fuel because of its high cost problem. This problem may be solved with the use of low cost feedstock in biodiesel production. Esterified waste frying oil (EWFO) is one of such low cost feedstock. However, the feedstock properties to be processed must be controlled in detail prior to transesterification reaction, since the physical and chemical properties of the feedstock significantly influence biodiesel production reaction as well as fuel properties. This study showed that for the ten EWFO derived biodiesel (EWFO-B) samples produced, the results of the kinematic viscosity and moisture content do not conform to the ASTM D6751 standard. However, the results of the specific gravity, iodine value, saponification value and the flash points of all the biodiesels produced conformed with the ASTM D6751 standard. Biodiesel yield of was highest if the FFA content of the EWFO is ≤ 0.70.

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