Blend Characteristics of Biodiesel Obtained from Four Virgin Tropical Seed Oils

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Abstract

Four feedstocks comprising two non-edible oils (*Telfairia occidentals Hook F*, (TVO) and *Hura crepitians L* (HVO)) and two edible oils *Cucumeropsis manii* Naud (CSVO) and *Canarium schweinfurthii Engl.* (CVO) were studied for production of methyl esters (biodiesel). Base catalysis was used for the transesterification reaction with methanol at different oil/alcohol molar ratios (4:1, 6:1 and 9:1). Two catalyst types; KOH and NaOH were used. Reaction times of 5 and 30 mins; temperatures of 38 and 55 $^{\circ}$C and a constant catalyst concentration of 1% wt/wt were employed. These process variables were used to achieve optimum biodiesel yields of 97%, (TVO and HVO); 94% and 96% (CSVO) and (CVO) respectively. Their viscosities ranged from 3.60–4.80 mm^2/s and specific gravities from 0.87–0.88g/cm^3. The specific gravities and viscosities of the blended biodiesel; B5, B10, B15 ranged from 0.960–0.930g/cm^3 and B20, 1.620–1.650 g/cm^3, the pour points ranged from -18 to -21. Water and sediments for all the blends were ≤0.05.

Keywords: biodiesel, transesterification, base catalysis, viscosity, specific gravity, pour point

1. Introduction

Vegetable oils and esters are receiving increasing attention as non-toxic, biodegradable and renewable alternative diesel fuels. Limited energy resources and increasingly strict emission regulations have motivated an intense search for alternative transportation fuels over the last three decades. Biodiesel is an oxygenated, sulphur-free, biodegradable, non-toxic and environmentally friendly alternative diesel fuel. Biodiesel is defined as the alkyl monoesters of fatty acids from renewable resources such as vegetable oils, animal fats and waste restaurant greases. One of the alternative characteristics of biodiesel is that its use does not require any significant modifications of the diesel engines, so the engines do not have to be dedicated for biodiesel alone. However, similar to alcohol fuels, biodiesels have lower engine content and different physical properties than diesel fuels (Tat & Gerpen, 1999, 2009; Tat et al., 2006).

Biodiesel is miscible in petroleum diesel. This means that the two can be mixed in any proportion and poured into the fuel tank. Common language for a biodiesel/diesel mix is "B" followed by the percentage of biodiesel; so 20% biodiesel and 80% diesel is called B20. Pure biodiesel is called B100. The miscibility of biodiesel and petroleum diesel has advantage in the sense that users can choose to increase the proportion of petroleum diesel in the blend if they are discouraged by the slightly higher cost of biodiesel or cold weather operation problems encountered using high proportion of biodiesel. In very cold conditions, biodiesel begins to crystallize, becomes thicker and may be difficult to use in an engine in that way one may use additives in the fuel, install any of a number of heating systems or use the biodiesel in a blend with petroleum diesel (which crystallizes) at a lower temperature. (University of Colorado, 2004; Mccormick, 2006).

Biodiesel can also be used in its pure form (B100), but may require certain engine modifications to avoid maintenance and performance problems. Due to it different properties, biodiesel will cause some changes in the engine's performance and emissions including lower power and emission of higher oxides of nitrogen. It can be blended in any proportion with petroleum based diesel fuel and the impact of the changes is usually proportional to the fraction of biodiesel being used. These changes can be eliminated by the engines electronic control system by either injecting more fuel or adjusting the fuel injection timing if the proportion of the blend is known. This

will require advanced knowledge of biodiesel fuel properties, their impact on engine performance and emissions, blend characteristics of biodiesel with No.1 (winter blend) and No.2 diesel fuel, and blend detection (Tat & Gerpen, 2002).

The American Society for Testing and Materials International (ASTM) specification for biodiesel (B100) is ASTMD6751-03 (Table 1). This specification is intended to insure the quality of biodiesel to be used as a blend stock at 20% and blend levels. Any biodiesel used for blending, should meet ASTM D6751 prior to blending (Mccormick, 2006).

Table 1. Requirement for biodiesel (B100) blend stock as listed in ASTM D6751-03

Property	ASTM Method	Limits	Units
Flash point	D93	130.0 min	^{o}C
Water and sediment	D2709	0.050 max	% vol
Kinematic viscosity, 40 ^{o}C	D445	1.9-6.0	Mm^2/s
Sulphated ash	D874	0.020 max	% mass
Sulphur	D5453	0.0015 max (S15) 0.05 max (S500)	% mass
Copper strip corrosion	D130	No.3 max	
Cetane number	D613	47 min	
Cloud point	D2500	Report to customer	^{o}C
Carbon residue	D4530	0.050 max	% mass
Acid number	D664	0.80 max	mgKOH/g
Free glycerine	D6584	0.020 max	% mass
Total glycerine	D6584	0.240 max	% mass
Phosphorus content	D4951	0.001 max	% mass
Distillation temperature 90% recovered (790)	D1160	360 max	^{o}C

The performance properties of B100 can be significantly different from conventional diesel. Blending biodiesel into petroleum diesel is a way to minimize these property differences while still retaining some of the benefits of B100.

Canarium schweinfurthii is a large forest tree which often grows as high as 50 m tall in the Savanna and sub-Savanna belts of Nigeria. They are often cultivated for its fruits which are edible, purplish, ellipsoid but slightly three-angled. The seeds were obtained from Jos, Plateau State during the dry season.

Hura crepitans is a large forest tree often found in the tropical rain forest and Savanna regions of Nigeria. The seeds are enclosed in hard protective coat which usually and suddenly splash open and scatters when the seeds are well dried. The tree has broad leaves with thorns all over its trunk.

Telfaria occidentalis belongs to the family *Cucurbitacae spp*. It is known as "Ugu" among the Igede, Idoma peoples of Benue State and the Igbos of Nigeria. There is tremendous genetic diversity within the family, and a range of adaptation for Cucurbits species includes tropical and subtropical regions and deserts. The genetic diversity in *Cucurbitacae* extends to both vegetative and reproductive characteristics.

Cucumeropsis manii is a species of melon native to tropical Africa west of the area Rift valley, where it is grown for food and as a source of oil. Its common names include egusi in Yoruba and aguishi in Hausa. In English, it is known as white seed melon. It produces climbing vines up to 4m long which are covered in stiff hairs. The heart-shape or roughly palmate leaves are up to 12 cm long and 14 wide. It bears small yellow male and female flowers with petals under a centimetre in length. The fruit is egg-shape or an elongated ovate shape up to about 19 cm long and 8 wide and cream in colour with green streaks. The fruits and white seed are edible. The plant is grown more often for the seed oil than for the fruit.

This work aims at optimising trans-esterification variables to obtain yields from extracts of the four tropical seed oils mentioned above, blend the biodiesel obtained with petroleum diesel (AGO) and characterize the blends using ASTM Standards methods of analysis.

2. Materials and Methods

2.1 Test Materials/Sample Collection

Canarium schweinfurthii The seeds were obtained from Jos, Plateau State during the dry season. The seeds were sun dried for about two weeks to remove moisture after which they were ground into coarse powder and ready for extraction. While *Hura crepitans, Telfaria occidentalis* and *Cucumeropsis manii* seeds were collected from Makurdi metropolis, Benue State between December and January. All the seeds were extracted from the bulb and dried to a constant weight after which the mesocarps were removed by dehulling. The dried seeds were pounded into coarse powder and ready for extraction. The seeds were identified by a botanist in the Department of Biological Science of Benue State University, Makurdi.

2.2 Degumming of Crude Oils

The oils were degummed to remove phospholipids, calcium and magnesium salts of phosphatidic and lysophasidic acids which are strong emulsifiers that inhibit the separation of the glycerol which lowers the yields of neutral oil. In degumming, the crude oil was mixed with about 3% of warm water and the mixture was agitated mechanically for 30 min at 70 $^{\circ}$C. This hydrates the phospholipids and gums thus making them insoluble in the oil. They were thereafter separated by settling.

2.3 Production of Biodiesel Fuels

The 100 mL of pretreated oil was measured and poured into a large beaker and heated to a temperature of 110 $^{\circ}$C using bunsen burner to remove the remains of solvent or moisture content. The heated oil was cooled to 38/55 $^{\circ}$C and poured into a blender fabricated for use as a reactor, With the reactor still switch off, methoxide was prepared by weighing 1 g of KOH or NaOH in a pet bottle and a calculated volume of methanol was added based on the desired alcohol/oil molar ratio (4:1, 6:1 and 9:1) and shaken to dissolve the catalyst completely. The prepared methoxide was emptied into the oil and properly covered to prevent air from entering into the mixture. The mixture was reacted for the required time of mixing (5 or 30 mins) after which the blender was switched off and the mixture immediately transferred into a separatory funnel and closed tightly. The mixture was allowed to settle for 24 h after which a dark color glycerin settled at the bottom while a pale liquid layer which is the methyl ester separated at the top (Wu et al., 1998).

Figure 1. Biodiesel and glycerin after separation

2.4 Biodiesel Separation

Upon completion of reaction, two major products were formed, glycerin and biodiesel. The clear liquid, methyl ester (biodiesel) found at the top layer was decanted into a graduated beaker (NBB).

2.5 Biodiesel Washing

The biodiesel was turned into a separating funnel after which an equal amount of distilled water was added. The separating funnel was gently swirled severally and allowed to stand for some minutes and the water drained off from the bottom of the funnel by turning on the tap of the separating funnel. The tap was turned off when it reached the methyl ester. This procedure was repeated twice to ensure complete washing. After washing, the biodiesel was dried by heating (Tat et al., 2006).

2.6 Blend Preparation (Derived Biodiesel with Petrol Diesel)

Blending of biodiesel was done using an in-tank method of petroleum blending, the biodiesel obtained after thermo- physical screening was blended with AGO obtained from fuel station at different proportions. They include, B5, B10, B20 and unblended biodiesel (B100). The blends were centrifuged for homogeneity before proceeding with fuel analysis using ASTM standards.

Figure 2. Samples of blends of petro-diesel/biodiesel

2.7 ASTM Quality Evaluation/Physicochemical Analysis

The ASTM quality evaluation/Physicochemical analysis of the biodiesel derived were carried out following procedures adopted from other works (Sim, 1982; Ayhan, 2005; ASTM, 1985, 2003; Horwitz & Latimer, 2005;

Kirk, 1965; Pockington, 1990; IPMAN, 1983; Chevron, 2007; Lewkowitsch, 1921, 1923; Paquot, 1979; Tat & Gerpen, 2000).

2.7.1 Determination of Viscosities of the Neat Oils before Transesterification

Determination of kinematic viscosity(ASTM D445); The time was measured for a 60ml of the oil sample to flow under gravity through a capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled and a temperature of 40 °C. The kinematic viscosity is the product of the measured flow time and calibration constant of the viscometer in centistokes (cSt). Apparatus used include; viscometer tubes, viscometer holders, Cannon–Fenske routine viscometer, thermostatic baths at 40°C and 100 °C, ASTM 120 °C thermometer, stop watch and 10ml syringe.

This can be calculated from the formula:

$$v = c \times t \tag{1}$$

v = kinematic viscosity;

c = tube calibration constant;

t = average flow time.

2.7.2 Determination of Density/Specific Gravity (ASTM D4052/ISO-AOAC Methods)

Weight of given volume of sample at desired temperature (20 °C) was determined using a pycnometer previously calibrated at same temperature. A 50ml capacity, Pycnometer; equipped with cap and thermometer graduated in 0.1 °C (Kimble Glass Inc. No. 15123-50 or equivalent) was carefully cleaned and filled with chromic acid it was allowed to stand for several hours, emptied and rinsed with H_2O. Thereafter it was placed in a thermostatic water bath at 20 °C (T) followed by addition of 0.1mg test sample. Kirk (1965) developed a formula for calculating density using fatty indices. Density (d_{15}) = 0.8467+0.003 (saponification value)+0.0014 (iodine value) where d_{15} = density of oil at 15 °C. The weight per volume (D_T) of sample at temperature T (20 °C) in g/ml was calculated as follows:

$$D_T \ (g/ml) \ = \ W\text{-}W_1/V_T \tag{2}$$

Where W and W_1 = weight (g) of pycnometer empty and filled with test sample;

V_T = volume of pycnometer (mL) at temperature, T (20 °C).

Specific gravity of sample at 20 °C was calculated from,

$$\text{Specific gravity at 20 °C} \ = \ D_{20}/dH_2O_T \tag{3}$$

where D_T = wt per unit vol. (g/mL) of test sample at T and dH_2O_T = density of H_2O at T.

2.7.3 Determination of Flash Point

Semi automatic cleveland close or open cup apparatus also called flash point analyzer was used for determining the flash or fire points of biodiesel produced.

The equipment has the following components; a stirrer, flame controller, sample compartment or test cup, heater, temperature, heating plate support and thermometer as components. Precaution was taken to prevent fire incidence for the determination of fuel oil, diesel, ASTM D93 was used. Approximately 70 ml of test specimen was filled into the test cup. The temperature of the test specimen increased rapidly at first and then at a slower content rate as the flash point was approached. At specified intervals, a test flame was passed across the cup. To determine the fire point, the test was continued until the application of the test flame caused the test specimen to ignite and sustain burning for a minimum of 5 s.

2.7.4 Determination of Pour Point (ASTM D97 Method)

A special instrument (cryogenic in nature) called Pour Point Analyzer (PPA) was used. It has the following components: two PPA tubes (one for sample and the other for control), thermometer, cotton wool for lagging and refrigeration chamber. The PPA used in this work has temperature ranged of 0 °C to 57 °C. An Oil sample was poured into a 50 ml PPA tubes, inserted into the lowest temperature range column of the PPA, lagged and powered. The PPA control was used to monitor the point at which the oil started to pour.

2.7.5 Determination of Cloud Point

The procedure for the cloud point was the same as in the case of pour point. Here, the temperature at which the oil started to cloud was noted.

2.7.6 ASTM D 2709 Standard Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge (Biodiesel Limit–0.05 Volume %)

A 100 mL sample of undiluted fuel was centrifuged in a tube readable to 0.005 mL. After centrifugation, the volume of water and sediment which had settled into the tip of the centrifuge tube was read to the nearest 0.005 mL and recorded as the volumetric percent water and sediment by centrifuge. The Centrifuge used in this work as one which can be controlled to give a relative centrifugal force (rcf) of 800±60 at the tip of the tubes. A 100 mL of a well-shaken sample was poured into a centrifuge tube and spun at 800rcf for 10 min.

The volume of the combined water and sediment was read from the tube as a percentage of the total sample. Results lower than 0.005% was reported as either 0 or 0.005% volume.

2.7.7 Determination of Carbon Residue (ASTM D189 Method)

The carbonator also called Carbshits used for carbonating the oil included a furnace/ oven, coil, regulator, lagging materials and chimney (exhaust exit). Using crucible with higher surface area, the oil samples were weighed and inserted into the carbonator which regulated at the temperature of $550^{\circ}C$ (ASTM recommendation for carbonating oils) for 2 h and thereafter reweighed.

The result was calculated from the formula;

$$\% \text{ carbon content } = \frac{W_3 - W_1}{W_2 - W_1} \times 100\% \qquad (4)$$

W_1 = weight of crucible;

W_2 = weight of crucible+oil;

W_3 = weight of crucible+residue.

2.7.8 ASTM D-874: Standard Test Method for Sulphated Ash from Lubricating Oils and Additives (Biodiesel Limit–0.02 Mass %)

The sample was ignited and burned until only ash and carbon remained. After cooling, the residue was treated with sulphuric acid and heated at 775 °C until oxidation of carbon was complete. The ash was then cooled, re-treated with the sulphuric acid, and heated at 775 °C to constant weight.

Sample dish was prepared by heating it at 775 $^{\circ}$C for at least 10 min, cooled, and weighed; the sample to be determined was weighed and heated until the contents sustained a flame. Thereafter, the sample was burned until there was no further smoke or fumes. Moisture contents of all samples were sufficiently reduced to avoid foaming before proceeding to the next step. The sample was then cooled, water and sulphuric acid were added then heated until no further fumes evolved; The dish was placed in a furnace and heated at 775 $^{\circ}$C until oxidation of the carbon was complete. Cooling, acidification and heating process were continued until successive weighing differed by not more than 1.0 mg.

Sulphated ash was calculated as a percentage of the original sample using the formula;

$$\text{Sulphated Ash (mass in }\%) = w/W*100 \qquad (5)$$

w = mass of sulphated ash (g);

W = mass of sample used in test (g).

The result was reported to the nearest 0.001% mass for samples below 0.02% mass and to the nearest 0.01% mass for higher levels.

2.7.9 ASTM D–7039: Standard Test Method for Determination of Sulphur in Gasoline, And Diesel Fuel by MWDFXS (Biodiesel Limit–0.05 Mass %)

A 5 ml of the sample was poured in the disposable sample cell/holder and inserted into a Horiba Sulphur-in-oil Analyser, Model SLFA-20 and analysed. The results were read in percentages.

2.7.10 Refractive Index (N)

Refractive index was determined using Abbe Refractometer at 20 $^{\circ}$C, 25 $^{\circ}$C or 40 $^{\circ}$C. The instrument was placed so that diffused light or some form of artificial light such as Na vapour lamp can be used for illumination. Stream of constant temperature (±0.2 $^{\circ}$C) H_2O was circulated through prisms. Approximate corrections of butyrofractometer reading were made using the formulae:

$$R = R^1 + K(T^1 - T) \tag{6}$$

Where R = reading obtained at temperature T^1;

T = standard temperature;

K = 0.58 for oils.

3. Results

The results presented in tables 2 to 5 show the Yields, Viscosity and Specific gravity of biodiesel from the four oils while tables 6 to 14 show the blend characteristics of the biodiesel under different conditions of time, temperature, catalyst and ratio of blends.

Table 2. Yields, viscosity and specific gravity of biodiesel from *Telfairia Occidentalis* seed oil

S/No.	Feedstock	Catalyst	Catalyst Conc. (%)	Alcohol/ Oil Ratio	Retention Temp. (^{o}C)	Rxn Time (mins)	% Yield	Viscosity mm^2/s	Specific Gravity g/cm^3
1	TVO-ME	KOH	1	4:1	38	5	94	6.40	0.90
2	TVO-ME	KOH	1	4:1	55	5	94.5	5.50	0.90
3	TVO-ME	KOH	1	4:1	38	30	94	6.21	0.90
4	TVO-ME	KOH	1	4:1	55	30	93	5.22	0.89
5	TVO-ME	NaOH	1	4:1	38	5	8	8.30	0.99
6	TVO-ME	NaOH	1	4:1	55	5	8	7.80	0.99
7	TVO-ME	NaOH	1	4:1	38	30	9	8.01	0.99
8	TVO-ME	NaOH	1	4:1	55	30	9	7.40	0.99
9	TVO-ME	KOH	1	6:1	38	5	92	4.01	0.87
10	TVO-ME	KOH	1	6:1	55	5	94	6.20	0.88
11	TVO-ME	KOH	1	6:1	38	30	93	4.80	0.89
12	TVO-ME	KOH	1	6:1	55	30	93	4.30	0.87
13	TVO-ME	NaOH	1	6:1	38	5	97	3.91	0.87
14	TVO-ME	NaOH	1	6:1	55	5	97	4.12	0.87
15	TVO-ME	NaOH	1	6:1	38	30	85	4.02	0.87
16	TVO-ME	NaOH	1	6:1	55	30	87	4.20	0.87
17	AGO							1.62	0.96

Table 3. Yields, viscosity and specific gravity of biodiesel from *Hura crepitans* seed oil

S/No.	Feedstock	Catalyst	Catalyst Conc. (%)	Alcohol /Oil Ratio	Retention Temp. (^{o}C)	Rxn Time (mins)	% Yield	Viscosity mm^2/s	Specific Gravity g/cm^3
1	HVO-ME	KOH	1	4:1	38	5	80	19.50	0.91
2	HVO-ME	KOH	1	4:1	55	5	92	22.0	0.96
3	HVO-ME	KOH	1	4:1	38	30	90	17.32	0.90
4	HVO-ME	KOH	1	4:1	55	30	90	17.30	0.90
5	HVO-ME	NaOH	1	4:1	38	5	59	4.60	0.88
6	HVO-ME	NaOH	1	4:1	55	5	9	4.20	0.87
7	HVO-ME	NaOH	1	4:1	38	30	58	4.23	0.87
8	HVO-ME	NaOH	1	4:1	55	30	62	4.00	0.87
9	HVO-ME	KOH	1	6:1	38	5	88	6.40	0.90
10	HVO-ME	KOH	1	6:1	55	5	70	17.90	0.91
11	HVO-ME	KOH	1	6:1	38	30	92	18.51	0.91
12	HVO-ME	KOH	1	6:1	55	30	83	16.61	0.90
13	HVO-ME	NaOH	1	6:1	38	5	97	5.02	0.88
14	HVO-ME	NaOH	1	6:1	55	5	90	4.20	0.87
15	HVO-ME	NaOH	1	6:1	38	30	72	3.60	0.87
16	HVO-ME	NaOH	1	6:1	55	30	85	4.02	0.87
17	AGO							1.62	0.96

Table 4. Yields, viscosity and specific gravity of biodiesel from *Cucumeropsis manii* seed oil

S/No.	Feedstock	Catalyst	Catalsyt Conc. (%)	Alcohol /Oil Ratio	Retention Temp. (^{o}C)	Rxn Time (mins)	% Yield	Viscosity mm^2/s	Specific Gravity g/cm^3
1	CVO-ME	KOH	1	4:1	38	5	T G	H V	-
2	CVO-ME	KOH	1	4:1	55	5	T G	H V	-
3	CVO-ME	KOH	1	4:1	38	30	T G	H V	-
4	CVO-ME	KOH	1	4:1	55	30	T G	H V	-
5	CVO-ME	NaOH	1	4:1	38	5	T G	H V	-
6	CVO-ME	NaOH	1	4:1	55	5	T G	H V	-
7	CVO-ME	NaOH	1	4:1	38	30	T G	H V	-
8	CVO-ME	NaOH	1	4:1	55	30	T G	H V	-
9	CVO-ME	KOH	1	6:1	38	5	T G	H V	1.26
10	CVO-ME	KOH	1	6:1	55	5	T G	H V	1.26
11	CVO-ME	KOH	1	6:1	38	30	T G	H V	1.24
12	CVO-ME	KOH	1	6:1	55	30	T G	H V	1.20
13	CVO-ME	NaOH	1	6:1	38	5	T G	HV	1.12
14	CVO-ME	NaOH	1	6:1	55	5	T G	H V	1.12
15	CVO-ME	NaOH	1	6:1	38	30	T G	H V	1.14
16	CVO-ME	NaOH	1	6:1	55	30	T G	H V	1.12
17	AGO							1.62	0.96
18	CVO-ME	KOH	1	9:1	38	5	92	4.33	0.87
19	CVO-ME	KOH	1	9:1	55	5	95	4.36	0.87
20	CVO-ME	KOH	1	9:1	38	30	94	4.02	0.87
21	CVO-ME	KOH	1	9:1	55	30	96	4.21	0.87
22	CVO-ME	NaOH	1	9:1	38	5	86	4.60	0.88
23	CVO-ME	NaOH	1	9:1	55	5	89	4.21	0.87
24	CVO-ME	NaOH	1	9:1	38	30	83	4.40	0.87
25	CVO-ME	NaOH	1	9:1	55	30	88	4.20	0.87

Table 5. Yields, viscosity and specific gravity of biodiesel from C*anarium schweinfurthii* seed oil

S/No.	Feedstock	Catalyst	Catalyst Conc. (%)	Alcohol /Oil Ratio	Retention Temp. (^{o}C)	Rxn Time (mins)	% Yield	Viscosity mm^2/s	Specific Gravity g/cm^3
1	CSVO-ME	KOH	1	4:1	38	5	74	5.82	0.90
2	CSVO-ME	KOH	1	4:1	55	5	82	3.60	0.87
3	CSVO-ME	KOH	1	4:1	38	30	83	3.83	0.87
4	CSVO-ME	KOH	1	4:1	55	30	85	4.02	0.87
5	CSVO-ME	NaOH	1	4:1	38	5	9	4.20	0.87
6	CSVO-ME	NaOH	1	4:1	55	5	9	4.10	0.87
7	CSVO-ME	NaOH	1	4:1	38	30	9	4.60	0.88
8	CSVO-ME	NaOH	1	4:1	55	30	9	4.11	0.87
9	CSVO-ME	KOH	1	6:1	38	5	94	4.51	0.88
10	CSVO-ME	KOH	1	6:1	55	5	93	4.22	0.88
11	CSVO-ME	KOH	1	6:1	38	30	83	4.40	0.88
12	CSVO-ME	KOH	1	6:1	55	30	82	4.91	0.89
13	CSVO-ME	NaOH	1	6:1	38	5	7	4.82	0.88
14	CSVO-ME	NaOH	1	6:1	55	5	8	4.60	0.87
15	CSVO-ME	NaOH	1	6:1	38	30	8	4.41	0.87
16	CSVO-ME	NaOH	1	6:1	55	30	7	4.60	0.87
17	AGO							1.62	0.96

Table 6. Blend characteristics of TVO-ME (TVO, KOH, 1%, 6:1, 38 ^{o}C, 30 minutes)

Blends	S.G	K.V	F.P	C.P	P.P	W & S	T.S	R.I	C.R	S.A
AGO	0.960	1.620	128	Nil	$\leq$-23	0.05	0.031	1.471	0.035	0.006
B5	0.960	1.620	129	-6	-18	<0.05	Nil	1.470	0.026	0.005
B10	0.940	1.630	130	-6	-18	<0.05	Nil	1.467	0.020	0.010
B15	0.935	1.640	131	-3	-18	<0.05	Nil	1.467	0.020	0.014
B20	0.930	1.650	136	-3	-18	<0.05	Nil	1.465	0.016	0.006
B100	0.87	4.80	132	+8	+3	0.05	Nil	1.457	0.022	0.007

Table 7. Blend characteristics of TVO-ME (TVO, NaOH, 1%, 6:1, 38 ^{o}C, 5 minutes)

Blends	S.G	K.V	F.P	C.P	P.P	W & S	T.S	R.I	C.R	S.A
AGO	0.960	1.620	128	Nil	$\leq$-23	0.05	0.031	1.471	0.035	0.006
B5	0.960	1.620	128	-4	-17	<0.05	Nil	1.470	0.033	0.013
B10	0.940	1.630	130	-4	-17	<0.05	Nil	1.468	0.033	0.010
B15	0.935	1.640	130	-3	-17	<0.05	Nil	1.466	0.029	0.006
B20	0.930	1.650	131	-3	-17	<0.05	Nil	1.465	0.031	0.011
B100	0.87	3.91	134	+11	+4	<0.05	Nil	1.456	0.021	0.006

Table 8. Blend characteristics of HVO-ME (HVO, NaOH, 1%, 4:1, 55 ^{o}C, 30 minutes)

Blends	S.G	K.V	F.P	C.P	P.P	W & S	T.S	R.I	C.R	S.A
AGO	0.960	1.620	128	Nil	$\leq$-23	0.05	0.0310	1.471	0.035	0.006
B5	0.960	1.620	130	-6	<-17	0.05	Nil	1.471	0.034	0.006
B10	0.940	1.630	131	-5	<-17	0.05	Nil	1.468	0.026	0.006
B15	0.935	1.640	133	-2	<-17	0.05	Nil	1.467	0.029	0.005
B20	0.930	1.650	135	-2	<-17	0.05	Nil	1.467	0.025	0.006
B100	0.870	4.00	146	+1	-4	0.25	Nil	1.467	0.014	0.004

Table 9. Blend characteristics of HVO-ME (HVO, NaOH, 1%, 6:1, 38 ^{o}C, 5 minutes)

Blends	S.G	K.V	F.P	C.P	P.P	W & S	T.S	R.I	C.R	S.A
AGO	0.960	1.620	128	Nil	$\leq$-23	0.05	0.0310	1.471	0.035	0.006
B5	0.960	1.620	129	-5	<-21	0.05	Nil	1.471	0.032	0.013
B10	0.940	1.630	131	-4	<-21	0.05	Nil	1.468	0.030	0.012
B15	0.935	1.640	132	-2	<-21	0.05	Nil	1.467	0.028	0.007
B20	0.930	1.650	133	-2	<-21	0.05	Nil	1.467	0.028	0.012
B100	0.880	5.020	136	0	-9	0.15	Nil	1.467	0.019	0.010

Table 10. Blend characteristics of CSVO-ME (CSVO, KOH, 1%, 6:1, 38 °C, 30 minutes)

Blends	S.G	K.V	F.P	C.P	P.P	W & S	T.S	R.I	C.R	S.A
AGO	0.960	1.620	128	Nil	≤-23	0.05	0.0310	1.471	0.035	0.006
B5	0.960	1.620	130	-3	<-18	<0.05	Nil	1.470	0.036	0.007
B10	0.940	1.630	132	-3	<-18	<0.05	Nil	1.468	0.034	0.005
B15	0.935	1.640	133	-1	<-18	<0.05	Nil	1.466	0.035	0.007
B20	0.930	1.650	135	-1	<-18	<0.05	Nil	1.465	0.036	0.006
B100	0.880	4.40	139	+11	+3	0.05	Nil	1.453	0.035	0.007

Table 11. Blend characteristics of CSVO-ME (CSVO, KOH, 1%, 6:1, 55 °C, 5 minutes)

Blends	S.G	K.V	F.P	C.P	P.P	W & S	T.S	R.I	C.R	S.A
AGO	0.960	1.620	128	Nil	≤-23	0.05	0.0310	1.471	0.035	0.006
B5	0.960	1.620	129	-4	<-18	<0.05	Nil	1.470	0.034	0.006
B10	0.940	1.630	130	-3	<-18	<0.05	Nil	1.468	0.030	0.010
B15	0.935	1.640	130	-2	<-18	<0.05	Nil	1.466	0.031	0.012
B20	0.930	1.650	135	-1	<-18	<0.05	Nil	1.465	0.033	0.010
B100	0.870	4.91	141	+11	+3	<0.05	Nil	1.453	0.029	0.009

Table 12. Blend characteristics of CVO-ME (CVO, KOH, 1%, 9:1, 55 °C, 30 minutes)

Blends	S.G	K.V	F.P	C.P	P.P	W & S	T.S	R.I	C.R	S.A
AGO	0.960	1.620	128	Nil	≤-23	0.05	0.0310	1.471	0.035	0.006
B5	0.960	1.620	128	-2	<-21	<0.05	Nil	1.471	0.029	0.014
B10	0.940	1.630	129	-2	<-21	<0.05	Nil	1.466	0.025	0.013
B15	0.935	1.640	129	-2	<-21	<0.05	Nil	1.465	0.026	0.005
B20	0.930	1.650	130	-2	<-21	<0.05	Nil	1.463	0.025	0.007
B100	0.870	4.21	136	+11	+4	<0.05	Nil	1.453	0.021	0.020

Table 13. Blend characteristics of CVO-ME (CVO, NaOH, 1%, 9:1, 38 °C, 5 minutes)

Blends	S.G	K.V	F.P	C.P	P.P	W & S	T.S	R.I	C.R	S.A
AGO	0.960	1.620	128	Nil	≤-23	0.05	0.0310	1.471	0.035	0.006
B5	0.960	1.620	127	-2	<-21	<0.05	Nil	1.471	0.032	0.013
B10	0.940	1.630	125	-2	<-21	<0.05	Nil	1.467	0.036	0.012
B15	0.935	1.640	125	-2	<-18	<0.05	Nil	1.465	0.035	0.017
B20	0.930	1.650	124	-2	<-18	<0.05	Nil	1.463	0.039	0.015
B100	0.880	4.600	121	+8	+4	<0.05	Nil	1.456	0.044	0.020

Key:

S.G–Specific Gravity

K.V–Kinematic Viscosity

F.P–Flash Point

C.P–Cloud Point

P.P–Pour Point

W&S–Water and Sediments

T.S–Total Sulphur

R.I–Refractive Index

C.R–Carbon Residue

S.A–Sulphated Ash

TVO–ME *Telfairia occidentals* methyl esters

HVO–ME - *Hura crepitians* methyl esters

CVO–ME - *Cucumeropsis manii* methyl esters

CSVO–ME - *Canarium schweinfurthii* methyl ester

AGO–Automotive Gas Oil (petrol diesel)

4. Discussion

One of the most important parameters affecting the yield of ester is the molar ratio of alcohol to vegetable oil. The stoichiometry of the transesterification reaction requires 3:1 molar ratio to yield 3 mol of ester and 1 mol of glycerol, but most researchers found that excess alcohol was required to drive the reaction close to completion. In this work, methanol was used. The effect of methanol in the ratio of 4:1and 6:1 was investigated, keeping catalyst concentration constant. Catalyst type, reaction time (duration of mixing) and temperature were varied in each reaction one or two variables were constant at a time. The results are shown in Tables 2 to 5. Ester yield increased with oil/alcohol molar ratio of 6:1 with 94%, 97%, and 97% for CSVO-ME, TVO-ME, and HVO-ME respectively. However CVO-ME gave 0% yield at 4:1 and 6:1 ratio, but gave 96% yield at the ratio of 9:1.

High yields were obtained when 1% wt/wt concentration of catalyst was used for the transesterification reaction with 97% from TVO and HVO when NaOH was used, whereas 94% and 96% yields from CSVO and CVO respectively were obtained when KOH was used. Best results of viscosities (3.91–4.80 g/cm^3) were obtained from TVO-ME 6:1 molar ratio when NaOH was used. Viscosities (3.60–5.02 g/cm^3) were obtained at both molar ratios. HVO-ME CSVO-ME showed a range of values of viscosities from 3.60–5.82 g/cm^3 at both molar ratios and CVO-ME (4.0–4.60 g/cm^3) at 9:1, when KOH and NaOH were used respectively. The values of specific gravities of all the biodiesel at these viscosities mentioned ranged from 0.87–0.88 mm^2/s for all the samples. Biodiesel with the best properties was obtained using KOH as catalyst in many studies (Encinar et al., 2006; Jeong & Park, 1996; Darnoko & Cheryman, 2000; Ugheoke et al., 2007; El Mashad et al., 2006). Besides, many other studies achieved best results using NaOH (Felizardo et al., 2006; Vicente et al., 2004; Cheng et al., 2004; de Oliveira et al., 2005). Methanolysis with 1% wt of either NaoH or KOH catalyst resulted in successful conversion giving the best yields and viscosities of the esters in most of the literature reviewed. It was observed that the product volume steadily increased from 0.5% w/v concentration of the catalyst until it reaches its peak at 0.9% wt/ v catalyst concentration. Thereafter, a decrease was observed. Catalyst concentration levels greater than 1% may favour backward reaction (Ugheoke et al., 2007). For maximum conversion to the ester, a molar ratio of 6:1 was mostly used (Vicente et al., 2004; Encinar et al., 2005; Darnoko & Cheryman, 2000; Meka et al., 2007; Ugheoke et al., 2007). In other studies, the optimum ratio was 10:1 (Jeong & Park, 1996; Cheng et al., 2004; Karmee & Chadha, 2005).

The mixing intensity appears to be of particular importance for the transesterification process. It increases the impact area between oils and catalyst-methanol solution. Thorough mixing facilitates the initiation of the reaction. Without mixing, the reaction occurred only at the interface of the two layers and is considered too slow to be feasible. In this study, a stirring rate of 800rpm was used. The yield, viscosity and specific gravity of biodiesel are shown in Tables 2–5. It was observed that the reaction of methanolysis was practically complete within 5 min of mixing due to the speed of the rotor for all cases because improvement in the yields, viscosities and specific gravity was obtained when the duration of mixing was increased (Ma et al., 1999; Peplinski, 2008). The effect of agitation on the transesterification of vegetable oil was studied and concluded that higher agitation promoted the homogenization of the reactant and thus lead to higher yields. Also, it has been noted elsewhere that the yield of biodiesel at 360 rpm and 600 rpm mixing was same i.e., 96% after 2 h of reaction (Umar & Anwar, 2008).

Ester content increased with reaction time at the beginning, reached a maximum at a reaction time of 15 min at 70 °C, and then remained relatively constant with increasing reaction time. Increasing reaction time from 15 minutes to 30 minutes had no significant effect on the conversion of triglycerides, but led to a reduction in the product yield from 87.5% to 85.3%. This is because longer reaction enhanced the hydrolysis of esters (reverse reaction of transesterification), resulting in a loss of esters as well as causing more fatty acid to form soap (Leung et al., 2006).

This shows that if the rate of agitation is high, the time of mixing can be reduced to as low as 5 minutes as deduced from Tables 2 to 5. This is because at 5 min, high yields were equally obtained for TVO-ME, HVO-ME, CSVO-ME and CVO-ME. Specific gravity and viscosities within specification were also obtained when the duration of mixing was 5 min. A decrease in yield was also noticeable in TVO-ME at 30minutes (e.g 97% to 85%) as shown in Table 2.

Duration of mixing and reaction temperature affected the yields, specific gravity and viscosities. Longer duration of mixing generated an increase in temperature which is believed to enhance the hydrolysis of esters' (reverse reaction of transesterification) resulting in a loss of esters as well as causing more fatty acid to form soap.

Trends observed in Tables 2 to 5 have been reported in a previous publication (Igbum et al., 2012). The biodiesel (B100) obtained from Tables 2 to 5 whose properties are within ASTM specification for biodiesel were thereby used to prepare the blends.

Blends prepared from the biodiesel obtained were: B5, B10, B15, and B20. They were characterized and their properties compared with the corresponding B100 and AGO. It was generally observed that all the results are basically the same as shown in Tables 6 to 13 with slight differences as the blends possess properties that are close to the properties of AGO than the corresponding B100.

Specific gravity of the AGO used was 0.960 g/cm^3. Specific gravity of all the blends irrespective of the feedstock (TVO-ME, HVO-ME, CSVO-ME and CVO-ME) are similar as it decreases from B5 (0.960 g/cm^3) to B20 (0.930 g/cm^3) as shown in Tables 6 to 13. The values are higher than those for B100 (0.87–0.89 g/cm^3) as shown in Tables 2–5.

Kinematic viscosity increases from AGO (1.620 mm^2/s), to B20 (1.650 mm^2/s) for all the blends prepared from the four feedstocks as shown in Tables 6 to 13 which is lower than the viscosities of B100 shown in Tables 2 to 5.

A key property determining the flammability of a fuel is the flash point. The flash point is the lowest temperature at which an applied ignition source will cause the vapors of a sample to ignite. The flash point for the AGO used was 128 ^oC. Flash points for B100 blend stock are expected to be >130 ^oC as shown in Table 1, this is because blending can bring about a reduction in flash point since the flash point of AGO is usually lower than B100. This argument is depicted in Tables 6, 7, 8, 9, 10, 11, and 12 as the flash points of the blends decrease from B5 to B2. For blend stock that have flash point >130 ^oC the results show that flash points increase slightly in B5 to B20 (Table 13).

Cloud points are shown to be much lower in the blends than their corresponding B100. AGO has no cloud point and therefore, the cloud points for all blends were greatly enhanced. All TVO-ME (B100) had cloud points ranging from +8 to +12 while their blends (B5 to B20) had cloud points ranging from 0 to -6 as shown in Table 6 to 7. The cloud point for HVO-ME and it blends as depicted in Tables 8 to 9 ranged from -1 to +4 and -2 to -9 respectively, whereas CSVO-ME and it blends ranged from +11 and -2 to -6 respectively as shown in Table 10 to 11. CVO-ME and its blends have cloud points ranging from +8 to +11 and -2 to -4 respectively as shown in Tables 12 to 13. (All the values are in degree Celsius).

Pour points for all TVO-ME, HVO-ME, CSVO-ME and CVO-ME (B100) range from +3 to 4, -4 to -9, +3 to +4 and +4 respectively. All the blends had pour points ranged from -15 to -21 as shown in Tables 6 to 13 (All the values are in degree Celsius).

The Free fatty Acid composition of the oils used has shown that all the oils contains mainly saturated and polyunsaturated fatty acids (Bello et al., 2011; Essien et al., 2012; Oyeleke et al., 2012; Bello et al., 2005), which compared favourably with common feedstocks like cottonseeds oil composed primarily of unsaturated linolenic and the saturated palmitic acids which gave 98.6% optimum yield (Georgios et al., 2011). This is evident in the values of cloud points and pour points obtained for all the B100.

All the values for Water and Sediment were 0.05 and below. Total sulphur was not detected in all the blends and B100. Values of carbon residue, sulphated ash and refractive index showed slight variation from AGO. All B100 were clear and transparent biodiesel as shown in Fig. 1. The indemnity of the AGO was slightly improved giving rise to this results as tabulated in Tables 6 to 13. All the properties are shown in Figures 3 to 7 for TVO-ME, HVO-ME, CSVO-ME and CVO-ME.

5. Conclusion

High oil yields were obtained from the four oil seeds used in this study and therefore a sustainable biodiesel industry can be achieved based on these feedstocks. Variation of parameters in the processing of the biodiesel

has tremendous effects on yields and optimum properties of biodiesel. Blending these biodiesel with petroleum diesel (AGO) showed improvement in the characteristics of the blends as compared to the AGO and the corresponding biodiesel used while still retaining some of the benefits of the biodiesel. Cloud and pour points which are of utmost importance were greatly enhanced in the blends as AGO does not pour.

References

Addison, K. (1999). *Make your own biodiesel*. Retrieved from http://journeytoforever.org/market/home/default.asp

ASTM (American Society for Testing Materials). (1985). *ASTM Standards Methods*, ASTM pub; Philadelphia, (pp. 31-36, 48-50, 497-500).

ASTM (American Society for Testing Materials). (2003). *ASTM Standards Methods*, ASTM pub; Philadelphia.

Ayhan, D. (2005). Biodiesel production from vegetable oils via catalytic and non-catalytic supercritical methanol transesterification methods. *Progress on Energy and Combustion Science, 31*, 466-487. http://dx.doi.org/10.1016/j.pecs.2005.09.001

Bello, E. I., Anjorin, S. A., & Agge, M. (2005). Production of Biodiesel from fluted pumpkin (Telfairia occidentalis Hook. F.) seed oil. *Int. Journal of Mechanical Engineering, 2*(1), 22-31.

Bello, M. O., Akindele, T. A., & Adeoye, D. O. (2011). Pysicochemical properties and fatty acids profile of seed oil of Telfairia occidentalis Hook. F. *International Journal of Basic & Applied Sciences, 11*(6), 9-14.

Cheng, S. F., Choo, Y. M., Ma, A. N., & Chuah, C. H. (2004). Kinetics study on transesterification of palm oil. *J. Oil Palm Res*., *16*(2), 19-29.

Chevron Corporation. (2007). *Diesel fuel technical review*. Retrieved from www.chevron.com/products/prodserv/fuels/bulletin/diesel

Darnoko, D., & Cheryman, M. (2000). Kinetics of palm oil transesterification in a batch reactor. *J. Am. Oil Chem. Soc., 77*(12), 1263-1267. http://dx.doi.org/10.1007/s11746-000-0198-y

De Oliveira, D., Di Luccio, M., Faccio, C., Dalla-Rosa, C., Bender, J. P., Lipke, N., ... De Oliveira, J. V. (2005). Optimization of alkaline transesterification of soybean oil and castor oil for biodiesel production, *Appl. Biochem. Biotech., 122*(1-3), 553-560. http://dx.doi.org/10.1385/ABAB:122:1-3:0553

El Mashad, H. M., Zhang, R., & Roberto, J. (2006). *Biodiesel production from fish oil*, American Society of Agricultural Engineers Annual Meeting, 066144.

Encinar, J. M., Juan, F., Gonzalez, J. F., & Rodriguez-Reinares, A. (2005). Biodiesel from used frying oil: variables affecting the yields and characteristics of the biodiesel. *Ind. Eng. Chem. Res., 44*(15), 5491-5499. http://dx.doi.org/10.1021/ie040214f

EPACT. (1992). *Energy Policy Act of 1992*. Retrieved from http://www.eere.gov/vehiclesandfuels/deployment/fcvtepact.shtml

Essien, E. A., Umoren, S. A., Essien, E. E., & Udoh, P. P. (2012). Preparation and evaluation of Cumeropsis manii Naud seed oil metallic soaps as driers in gloss paint. *J. Mater. Environ. Sci., 3*(3), 477-484.

Felizardo, P., Correia, M. J., Raposo, I., Mendes, J. F., Berkemeier, R., & Bordado, J. M. (2006). Production of biodiesel from waste frying oils. *Waste Management, 26*(5), 487-494. http://dx.doi.org/10.1016/j.wasman.2005.02.025

Georgios, K., Georgios, A., & Stamos, S. (2011). Tetramethylguanidine as an efficient catalyst for transesterification of waste frying oils. *Applied Energy, 88*, 3645-3650. http://dx.doi.org/10.1016/j.apenergy.2011.03.050

Horwitz, W., & Latimer, G. W. (2005). *Official Methods of Analysis of AOAC International*. AOAC International, Maryland, USA.

Igbum, O. G., Eloka-Eboka, A. C., & Nwadinigwe, C. A. (2012). Effects of Transesterification Variables on Yields and Properties of Biodiesel Fuels Produced from Four Virgin Tropical Seeds Oils. *International Journal of Environment and Bioenergy, 1*(2), 119-130.

Institute of Petroleum (IPMAN). (1983). *Standard for Petroleum and its Products* (42nd ed., p. 15, 71, 160, 242, and 742). London Institute of Petroleum Pub.

Jeong, G. T., & Park, D. H. (1996). Batch (one- and two-stage) production of biodiesel fuel from rapeseed oil.

Biotechn. Appl. Bioc., 131(1-3), 668-679. http://dx.doi.org/10.1385/ABAB:131:1:668

Karmee, S. K., & Chadha, A. (2005). Preparation of biodiesel from crude oil of Pongamia pinnata. *Bioresourec Tech., 96*(13), 1325-1429. http://dx.doi.org/10.1016/j.biortech.2004.12.011

Kirk, O. (Ed.) (1965). *Encyclopedia of Chemical Technology Vol. 2*. Oxford: Longman University Press.

Leung, D. Y. C., & Guo, Y. (2006). Transesterification of neat and used frying oil: optimization for biodiesel production. *Fuel Processing Technology, 87*, 883-890. http://dx.doi.org/10.1016/j.fuproc.2006.06.003

Lewkowitsch, P. (1921). *Chemical technology and analysis of oils*, *Fats and waxes* (6th ed., Vol. 1, p. 325).

Lewkowitsch, P. (1923). *Chemical technology and analysis of oils*, *Fats and waxes* (6th ed., Vol. 3, p. 31-54).

Ma F., Clements, L. D., & Hanna, M. A. (1999). The effect of mixing on transesterification of beef fallow. *Bioresource Technology, 69*, 289-93. http://dx.doi.org/10.1016/S0960-8524(98)00184-9

Mccormick, R. L. (2006). *Biodiesel handling and use guidelines. US Department of Energy. DOK\GO-102006-2258* (3rd ed.). Retrieved from http://www.nrel.gov/vehiclesandfuels/npbf/pdfs/40555.pdf

Meka, P. K., Tripathi, V., & Singh, R. P. (2007). Synthesis of biodiesel fuel from safflower oil using various reaction parameters. *J. Oleo Sci., 56*(1), 9-12. http://dx.doi.org/10.5650/jos.56.9

Oyeleke, G. O., Olayiwola, O. A., & Latona, D. F. (2012). Chemical Examination of Sandbox (Hura crepitans) seed: Proximate elemental and fatty acid profile. *Journal of Applied Chemistry (IOSRJAC), 1*(2), 10-13.

Paquot, C. (1979). *Standard Methods for the Analysis of oils, fats and derivatives* (6th ed.). New York: Pergamon Press. Retrieved from http://www.iupac.org/publications/pac/1979/pdf/5112x2503.pdf

Peplinski, B. (2008). *Free Fatty Acids–What are they and why should I care?* Retrieved from http://www.eziwearrticles.com/biodiesel-free

Pockington, W. D. (1990). Determination of the iodine value of oils and fats: results of collaborative study. *Pure and Applied Chem., 62*(12), 2339-2343. http://dx.doi.org/10.1351/pac199062122339

Sims, R. (1982). *Yield, cost and availability of natural oils/fats as diesel fuel substitutes*, report no. LF 2021 for the Liquid Fuel Trust Board, Wellinton (NZ).

Tat, M. E., & Van Gerpen, J. H. (1999). The kinematic viscosity of biodiesel and its blends with diesel fuel. *Journal of America oil Chemists Society, 76*(12), 1511-1513. http://dx.doi.org/10.1007/s11746-999-0194-0

Tat, M. E., & Van Gerpen, J. H. (2002). *Physical properties and composition detection of biodiesel-diesel fuel blends*. ASAE-CIGR Meeting paper No 026084. St Joseph, Mich: ASAE, P2.

Tat, M. E., & Van Gerpen, J. H. (2009). The specific gravity of biodiesel and its blends with diesel fuel. *Journal of America Oil Chemist Society, 77*(2), 115-119. http://dx.doi.org/10.1007/s11746-000-0019-3

Tat, M. E., Van Gerpen, J. H., Soylu, S., Canakci, M., Monyem, A., & Wormley, S. (2006). The speed of sound and isentropic bulk modulus of biodiesel at 21oC from atmospheric pressure to 35MPa. *Journal of America Oil Chemists Society, 77*(3), 285-289. http://dx.doi.org/10.1007/s11746-000-0047-z

Ugheoke, B. I., Patrick, D. O., Kefas, H. M., & Onche, E. O. (2007). Determination of optimal catalyst concentration for maximum biodiesel yield from tiger nut (Cyperus Esculentus) oil. *Journal of Science, 10*, 131-136.

Umar, R., & Anwar, F. (2008). Production of biodiesel through optimized alkaline-catalyzed transesterification of rapeseed oil. *Fuel, 87*, 265-273. http://dx.doi.org/10.1016/j.fuel.2007.05.003

University of Colorado. (2004). *Biofuels for Sustainable Transport–the lack of UK Government Policy* (pp. 1-23). Retrieved from http://www.biofuels.fsnet.co.uk

Van Gerpen, J. H, Shanks, B., Pruszko, R., Clements, D., & Knothe, G. (2002–2004). *Biodiesel Production Technology* (pp. 1-105). National Renewable Energy Laboratory (NREL/SR-510-36244).

Vicente, G., Martinez, M., & Aracil, J. (2004). Integrated biodiesel production: a comparison of different homogeneous catalysts systems. *Bioresource Tech., 92*(3), 297-305. http://dx.doi.org/10.1016/j.biortech.2003.08.014

Wu, W. H., Foglia, T. A., Marmer, W. N., & Philips, J. G. (1998). Optimizing production of ethyl esters of grease using 95% ethanol by response surface methodology. *Journal of America Oil Chemists Society, 76*, 4-10, 58.