

Comparative Analysis of the Physico-chemical Properties of Oil from *Cucumis melo* ssp. *agrestis* (Wild melon) Seeds, its Biodiesel, Biodiesel Blend and Petro- diesel

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ABSTRACT

Oil from the powdered seeds of *Cucumis melo* ssp. *agrestis* was extracted by Soxhlet extraction method, using petroleum ether (60°C to 80°C). Methyl ester (biodiesel) of the oil was prepared by hydrochloric acid catalysed transesterification reaction between the oil and methanol. A blend of the biodiesel and petrodiesel was made in the ratio of 10:90 (B10) respectively. Physico-chemical properties such as percent oil content, relative density, total acid number, iodine value, calorific value, flash point, pour point and bond structure stability of the oil, biodiesel, biodiesel, biodiesel blend and petro-diesel were determined at 28, 50, 100, 150, 200 and 250°C. GC-MS of the fatty acid methyl ester profile of the biodiesel was also determined. The Fourier Transform Infrared (FTIR) spectra of the biodiesel which was rich in polyunsaturated fatty acid methyl ester (59.47%) with little percent of monounsaturated fatty acid methyl ester (2.02%) and appreciable percent of saturated fatty acid methyl ester (38.51%) showed no breakage of bonds at the above temperatures. The bond structure stability relationship deduced from FTIR spectra confirmed that the structures of the oil, biodiesel and biodiesel blend were similar to that of the petrodiesel. Comparative analysis of the results also showed that engine performance of both biodiesel and petro-diesel in terms of their ignition qualities, relative densities, acidities, viscosities, flash points, pour points, percent ash, aromatics, sulphur and carbon residues could be improved by blending the two fuels.

Key words: *Cucumis melo* ssp. *agrestis*, oil, biodiesel, blend, petro-diesel, comparative, analysis.

INTRODUCTION

Vegetable oils, also known as triglycerides are esters of three fatty acids and one glycerol¹. These contain substantial amounts of oxygen in their structures². The fatty acids vary in their carbon chain length and in the number of double bonds³. Different types of vegetable oils have different types of fatty acids. Fixed oils are often called the non-volatile oils, in distinction to the essential or volatile oils which are readily vapourized by heat. Fixed oils are usually classified into three groups: drying, semi-drying, and non-drying oils. Drying oils are those which on exposure to air form tough elastic film. Their iodine numbers are normally above 120mg/g. Non-drying oils are those which remain permanently greasy or sticky, becoming rancid after a time. These are oils that do not form hard, glassy or rubbery, surface when exposed to the atmosphere. They have very low iodine number. Semi-drying oils are intermediate in nature between non-drying oil and drying oils. Their iodine values range between 90 – 140mg/g⁴. The "modern" way of processing vegetable oil is by chemical extraction, using solvent extraction which produces higher yields and is quicker and less expensive. Another way is by physical extraction. It is made the "traditional" way using several different types of mechanical extraction⁵. Oils can be analyzed for their compositions, characteristics and nutritional values and the storage qualities of the oil can be assessed in terms of the physico-chemical analysis while its utilization can be determined from its

composition and physico-chemical properties⁶. The combustion of hydrocarbon fuels contributes to over half of greenhouse emissions. The nitrogen oxides, sulphur oxides, carbon oxides and particulate matter from fossil diesel have largely contributed to the pollution of environment as a result deplete ozone layer which leads to climate change. Hence, today's world is facing urgency in developing alternative fuels. Among the various alternatives is the biodiesel⁷. Biodiesel is produced by transesterification of triglycerides with alcohol and has become more attractive recently because it is renewable and environmentally friendly⁸. Most of the problems associated with the use of straight vegetable oil as fuel can be attributed to the high viscosity of vegetable oil and the reactivity of the polyunsaturated fatty acid components of the triglycerides⁹. In recent times, there are search for sustainable feedstocks for biodiesel from both cultivated and wild plant varieties. *Cucumis melo ssp. agrestis* (common name: wild or bush melon) belongs to the family of Cucurbitaceae (Pumpkin family) with vernacular name of *Egusi Ohia*. The wild form of *Cucumis. melo* is distinguished as subspecies *agrestis*, showing short hairs on the ovary, and the diameter of the stem, flowers and fruits comparatively smaller than the cultivated form (subspecies *melo*) whose ovaries have longer hairs with the diameter of the stem, flowers and larger fruits¹⁰. *Cucumis. melo* subspecie. *agrestis* is a wild plant that colonizes various habitats in West Africa¹¹. Sample of *Cucumis. melo* subspecie. *agrestis* reported in the revised version of the Flora of West Africa is described as follows¹². It is a creeping, annual herb. Leaf and tendril alternates. The leaves are broadly ovate, widely cordate at base and 5-lobed. The fruit is globose, obovoid or oblong and longitudinally striped with green. When young, the fruit is ovoid, about the size of the pigeon's egg, furry hairy, soft bristles and short. The surface is decorated with longitudinal stripes of dark green spots. Burkill¹³ also recognizes the existence of wild forms and spontaneous *Cucumis melo* in West Africa. The author argues, moreover, that the center of origin of the species could be Africa. The objective of this research was to extract oil from seeds of *Cucumis melo ssp. agrestis*, prepare methyl ester, blend the methyl ester with petrodiesel and comparatively analyse the physico-chemical properties of the oil, methyl ester, methyl ester blend and petrodiesel.

MATERIALS AND METHOD

Oil of *Cucumis melo ssp. agrestis* was extracted from the powdered seeds by Soxhlet extraction method, using petroleum ether (60°C to 80°C). The oil was used to prepare biodiesel by acid catalysed transesterification reaction with methanol. Petrodiesel was purchased from a local fuel filling station. Blend of the biodiesel and petrodiesel was made in the ratio of 10:90 respectively. Physico-chemical properties of the oil, biodiesel, biodiesel blend and petrodiesel were determined using the methods outlined by the American oil Chemists Society, Institute of Petroleum (IP) and the American Society for Testing and Materials (ASTM)^{14, 15, 16}. The bond structure stability of the oil was determined by Fourier Transform Infrared Spectrometer.

Percent oil yield

Procedure: 300g of the powdered seeds was wrapped in a white paper and fed into a dry extraction thimble. The thimble was lowered into the Soxhlet extractor. The extraction process was allowed to run continuously for 24 hours until all the oil in the sample was extracted as observed by a colour change of the oil/solvent mixture. The oil was finally separated from the solvent by simple distillation at 80°C and weighed. The process was repeated two more times. The percent oil yield was calculated as follows

$$\text{Percent oil yield} = \frac{\text{Weight of oil}}{\text{Weight of sample}} \times 100$$

Relative density

Procedure: Clean and dry 25cm³ specific gravity bottle was weighed (W_1). It was filled with distilled water and weighed again (W_2). The bottle was emptied, allowed to dry and filled with sample and weighed (W_3). All the weights were taken at room temperature (28°C). The relative density was calculated using the formula below

$$\text{Relative Density} = \frac{W_2 - W_1}{W_3 - W_1} = \frac{\text{Weight of sample}}{\text{Weight of water}}$$

Total acid number

Procedure: 1.0g of sample was weighed in a conical flask. 4cm³ of chloroform was added to dissolve the sample. 2 drops of phenolphthalein were added. The solution was titrated with 0.05M alcoholic potassium hydroxide (KOH) to a pink end point.

$$\text{Total Acid Number} = \frac{\text{cm}^3 \text{ of KOH used} \times \text{molarity of KOH} \times \text{molar mass of KOH}}{\text{mass of sample}}$$

Flash point

Procedure: A Pensky Martens tester was used to determine the flash point. Each sample was poured into the flash point cup to a level inscribed on the cup. The sample was stirred as the power was switched on to heat. The pilot flame was then ignited. With the aid of the shutter operating knob, the cup was lowered at interval to check when it had flashed. The flash point was noted as the temperature when the flame flashed inside the cup.

Iodine value (I.V)

Procedure: 1.0 g of sample was weighed into a reagent bottle and dissolved with 5cm³ of chloroform after which the solution became clear. Some quantity of glacial acetic acid was added to the clear solution. 100cm³ of iodine monochloride was added to this mixture. This clear solution was allowed to stand for two hours, shaking it rigorously at interval of 30 minutes in a dark cupboard. A spatula full of potassium iodide was added. The solution was then titrated with a standard solution of 0.1M sodium thiosulphate to a colourless end point. The iodine value was calculated using the following formula:

$$\text{I.V} = \frac{(a - b) \times \text{molarity of thiosulphate} \times \text{molarity of iodine} \times \text{molar mass of iodine}}{\text{mass of sample}}$$

Where a = cm³ of iodine monochloride added
b = cm³ of thiosulphate consumed

Calorific value

Procedure: The calorific value of oil was determined using the American Petroleum Institute (API) Table which relates the relative density and gross calorific value.

Viscosity

Procedure: A sample was poured into a viscometer to the mark A, corked and allowed to stand to attain the temperature of the bath (40⁰C), the cork was removed and the sample was allowed to flow down gently. On getting to the mark 'D' on the viscometer, the stop watch was switch on and stopped when the sample got to the mark 'B', thus turning the exact rate of flow from 'D' to 'B'. The sample was pumped back with the aid of pipette filler and the experiment repeated. The average rate of flow was calculated. The viscosity in centistokes was recorded as the time taken to flow from 'D' to 'B' multiplied by the viscometer constant i.e. V = Ct.

Pour point

Procedure: 30cm³ of sample was poured into a test jar and placed inside a cooling bath. For every subsequent 3⁰C, the test jar was removed and tilted to check for surface movement. When the sample did not flow, the test jar was held horizontally for 5 seconds. If it did not flow, 3⁰C was added to the corresponding temperature to obtain the pour point temperature.

Bond structure stability

Procedure: 10cm³ of each sample was kept at room temperature (28⁰C). 10cm³ of each sample was separately heated to 50, 100, 150, 200 and 250⁰C. Each sample was mixed with a little nujol and was placed in between two sodium chloride plates with groove. The chloride plate was then attached to a Fourier Transform Infrared spectrometer coupled to a computer with a print out system. The sample in the

plate was irradiated by infrared lamp source at one end of the spectrometer and it was excited to different energy frequencies (cm^{-1}).

Gas Chromatography (GC)/Mass Spectroscopy (MS) Analysis

Procedure: GCMS-QP2010 PLUS SHIMADZU JAPAN auto sampler and auto analyzer model was fused with HP-Inn wax fused capillary column (60 x 0.25mm i.d) and helium as the carrier gas (flow rate = 1.61ml/min). The injection temperature was 250°C and oven temperature kept at 60°C for 5 minutes, then kept constant at 140°C for another 5 minutes. This was followed by adjusting it to 280°C at a rate of 20°C/min and then kept at 280°C for 15 minutes. The end of capillary column was inserted directly into the ion source of MS and interface temperature was maintained at 250°C. The mass spectrometric data were acquired and processed with SHIDMAZU MAT 330 data system. The run button was switched on to generate the peaks which started appearing on the computer screen at different times. This profile was later printed out. The set up had an accompanying library coupled to it. The esters and fatty acids were identified using the National Institute of Standards and Technology (NIST) mass spectral library based on their similarity indices.

RESULTS

TABLE 1: PHYSICO-CHEMICAL PROPERTIES OF WILD MELON OIL, ITS BIODIESEL, BIODIESEL BLEND AND PETRO-DIESEL

Sample	Oil Content (%)	Relative Density	Total Acid Number (mg/g)	Iodine Value (mg/g)	Calorific Value (kJ/kg)	Kinematic Viscosity (mm^2/s)	Flash Point ($^{\circ}\text{C}$)	Pour Point ($^{\circ}\text{C}$)
Petro-diesel		0.86	0.23	8.60	45,124	2.57	70	9
Wild melon Oil	30 ± 2	0.94	72.93	121.82	43,519	25.43	110	-10
Biodiesel		0.92	92.57	122.46	43,938	12.16	88	-6
Biodiesel blend (B10)		0.86	5.61	118.02	45,124	3.37	78	-8

TABLE 2: FTIR BOND STRUCTURE COMPARISON FOR WILD MELON OIL, ITS BIODIESEL, BIODIESEL BLEND AND PETRO-DIESEL (cm^{-1}) AT 28°C

Petro-diesel	Wild melon Oil	Biodiesel	Biodiesel Blend (B10)	Description
n.p	3425.69	3471.02	n.p	Free O-H stretch.
2922.25	2927.08	n.p	n.p	C-H stretch for alkenes and aromatics.
n.p	2855.7	2888.50	2857.67	C-H stretch for alkanes.
			2359.98	
1601.93	1744.67	1750.46	1744.67	C=O stretch for esters.
1456.30	1460.16	1455.34	1458.23	C=C stretch for alkenes and aromatics.
1373.36	1377.22	1367.58	1374.33	C-O stretch for esters.
	1238.34	1266.31		
1161.19	1165.04	n.p	1167.94	C-O deformation bonds.
963.48	n.p	1105.25	n.p	Deformation bonds for alkyl and aryl groups.
735.87	722.37	722.37	728.15	C-H deformation for methyl groups.

n.p = not present

TABLE 3: FTIR BOND STRUCTURE COMPARISON FOR WILD MELON OIL, ITS BIODIESEL, BIODIESEL BLEND AND PETRO-DIESEL (cm^{-1}) AT 50°C

Petro-diesel	Wild melon Oil	Biodiesel	Biodiesel Blend (B10)	Description
n.p	3425.69	3464.27	3419.90	Free O-H stretch.
2923.22	2927.08	2927.08	2959.87	C-H stretch for alkenes and aromatics.
n.p	2855.7	n.p	n.p	C-H stretch for alkanes.
n.p	1744.67	1742.74	1744.67	C=O stretch for esters.
			1608.69	
1457.27	1460.16	1453.41	1456.30	C=C stretch for alkenes and aromatics.
1375.29	1377.22	1239.31	1373.36	C-O stretch for esters.
	1238.34			
n.p	1165.04	1166.97	1167.94	C-O deformation for methyl groups.
n.p	722.37	719.47	731.05	C-H deformation for methyl groups.
465.82	431.1	450.39	464.86	C-H deformation for alkyl groups.

n.p = not present

TABLE 4: FTIR BOND STRUCTURE COMPARISON FOR WILD MELON OIL, ITS BIODIESEL, BIODIESEL BLEND AND PETRO-DIESEL (cm⁻¹) AT 100°C

Petro-diesel	Wild melon Oil	Biodiesel	Biodiesel Blend (B10)	Description
n.p	3425.69	n.p	n.p	Free O-H stretch.
2930.93	2927.08	2926.11	2928.04	C-H stretch for alkenes and aromatics.
n.p	2855.7	n.p	n.p	C - H stretch for alkanes.
n.p	1744.67	1744.67	1738.87	C=O stretch for esters.
1456.30	1460.16	1454.38	1453.41	C=C stretch for alkenes and aromatics.
1373.36	1377.22	n.p	1369.50	C-O stretch for esters.
	1238.34			
n.p	1165.04	1166.97	1174.69	C-O deformation for methyl groups.
n.p	722.37	720.44	n.p	C-H deformation for methyl groups.
472.58	431.1	433.03	467.75	C-H deformation for alkyl groups.

n.p = not present

TABLE 5: FTIR BOND STRUCTURE COMPARISON FOR WILD MELON OIL, ITS BIODIESEL, BIODIESEL BLEND AND PETRO-DIESEL (cm⁻¹) AT 150°C

Petro-diesel	Wild melon Oil	Biodiesel	Biodiesel Blend (B10)	Description
n.p	3425.69	3468.13	n.p	Free O-H stretch.
2923.22	2927.08	2926.11	2956.01	C-H stretch for alkenes and aromatics.
n.p	2855.7	n.p	n.p	C-H stretch for alkanes.
n.p	1744.67	1744.67	1745.64	C=O stretch for esters.
1457.27	1460.16	1455.34	1459.20	C=C stretch for alkenes and aromatics.
1375.29	1377.22	1240.27	1375.29	C-O stretch for esters.
	1238.34			
n.p	1165.04	1166.97	1167.94	C-O deformation for methyl groups.
n.p	n.p	n.p	1026.16	C-H deformation for alkyl and aryl.
			877.64	
			811.09	
n.p	722.37	720.44	727.19	C-H deformation for methyl groups.
465.82	431.1	442.68	n.p	C-H deformation for alkyl groups.

n.p = not present

TABLE 6: FTIR BOND STRUCTURE COMPARISON FOR WILD MELON OIL, ITS BIODIESEL, BIODIESEL BLEND AND PETRO-DIESEL (cm⁻¹) AT 200°C

Petro-diesel	Wild melon Oil	Biodiesel	Biodiesel Blend (B10)	Description
n.p	3425.69	3469.09	3421.83	Free O-H stretch.
2922.25	2927.08	n.p	2924.18	C-H stretch for alkenes and aromatics.
n.p	2855.7	n.p	n.p	C-H stretch for alkanes.
1600.01	1744.67	1731.17	1745.64	C=O stretch for esters.
1457.27	1460.16	1442.80	1457.27	C=C stretch for alkenes and aromatics.
1374.33	1377.22	1372.40	1379.15	C-O stretch for esters.
	1238.34	1262.45		
n.p	1165.04	1144.79	1169.01	C-O deformation for methyl groups.
n.p	n.p	n.p	1030.99	C-H deformation for alkyl and aryl.
			814.95	
n.p	722.37	722.37	729.12	C-H deformation for methyl groups.
465.82	431.1	433.03	440.75	C - H deformation for alkyl groups.

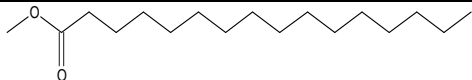
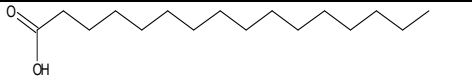
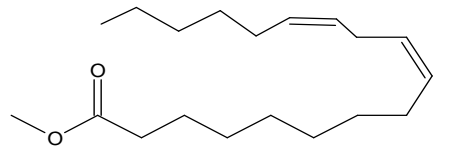
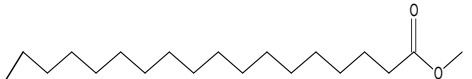
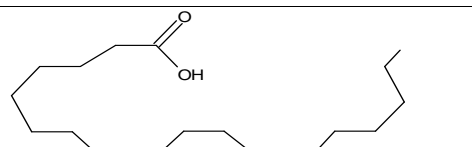
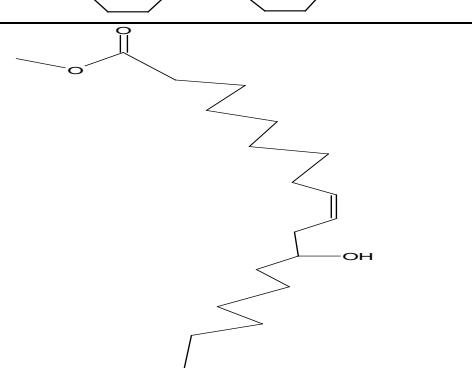
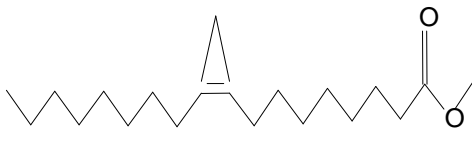
n.p = not present

TABLE 7: FTIR BOND STRUCTURE COMPARISON FOR WILD MELON OIL, ITS BIODIESEL, BIODIESEL BLEND AND PETRO-DIESEL (cm⁻¹) AT 250°C

Petro-diesel	Wild melon Oil	Biodiesel	Biodiesel Blend (B10)	Description
n.p	3425.69	3464.27	n.p	Free O-H stretch.
2927.08	2927.08	2930.93	2923.22	C-H stretch for alkenes and aromatics.
2862.46	2855.7	2861.49	n.p	C - H stretch for alkanes.
1601.93	1744.67	1739.85	1743.71	C=O stretch for esters.
1456.30	1460.16	1449.55	1456.30	C=C stretch for alkenes and aromatics.
1374.33	1377.22	n.p	1373.36	C-O stretch for esters.
	1238.34			
n.p	1165.04	1167.94	n.p	C - O deformation for methyl groups.
n.p	722.37	717.54	n.p	C - H deformation for methyl groups.
468.72	431.1	432.07	459.07	C - H deformation for alkyl groups.

n.p = not present

TABLE 8: GC-MS PROFILE OF FATTY ACIDS COMPOUNDS OF CRUDE WILD MELON METHYL ESTER

Peak No	R.A (%)	Name	Chemical Formula	Chemical Structure	M. Wt
1	19.35	Hexadecanoic acid, methyl ester	C ₁₇ H ₃₄ O ₂		270
2	4.06	n-Hexadecanoic acid	C ₁₆ H ₃₂ O ₂		256
3	59.47	9,12-Octadecadienoic acid, (Z,Z)-, methyl ester	C ₁₉ H ₃₄ O ₂		284
4	13.07	Octadecanoic acid, methyl ester	C ₁₉ H ₃₈ O ₂		298
5	2.03	Octadecanoic acid,	C ₁₈ H ₃₆ O ₂		284
6	0.56	Methyl ricinoleate	C ₁₉ H ₃₆ O ₃		312
7	1.46	Methyl -2-Octylcyclopropene-1-octanoate	C ₂₀ H ₃₆ O ₂		308

DISCUSSION

Table 1 showed the physico-chemical properties of oil of wild melon, its biodiesel, biodiesel blend and petrodiesel. The relative densities of the oil and biodiesel were higher than that of hydrocarbon diesel. This could be attributed to higher molecular weight of fatty acid components of the oil and biodiesel¹⁷. There was reduction in relative density from oil to biodiesel. This could also be attributed to the fact that some of the fatty acid components of the oil were reduced during saponification and esterification¹⁸. The biodiesel blend had relative density of 0.86 equal to that of petrodiesel. This was an indication of good ignition properties and is therefore considered to have better ignition qualities than oil and biodiesel. The total acid number of oil was much higher than that of petrodiesel. This was attributed to the fatty acids components of the oil. Results in Table 1 confirmed that acid catalysed esterification reaction greatly increased acidity of biodiesel. The acidity of the blend in Table 1 confirmed that blending could reduce acidity. Biodiesel purification by washing with sodium hydroxide and blending are therefore highly recommended.

The iodine value of wild melon oil placed it in the semi-drying group. The iodine value is a measure of the unsaturation of fats and oils. Higher iodine value indicates higher unsaturation¹⁹. The Iodine Value can be important because many biodiesel fuel standards specify an upper limit for fuel that meets the specification. For example, Europe's EN14214 specification allows a maximum of 120 for the Iodine number, Germany's DIN 51606 tops out at 115. The USA ASTM D6751 does not specify an Iodine value because the Iodine value (IV) does not necessarily make the best measurement for stability as it does not take into account the positions of the double bonds available for oxidation. In some cases this can lead to iodine values that are misleading. However, the iodine values of the biofuels were close to specifications. Results in Table 1 confirmed reduction in viscosity after transesterification but the viscosity of the biodiesel could be considered to be very high when compared with that of petro-diesel. This could be attributed to the fatty acid components of the biodiesel. However, blending the two fuels produced fuel with viscosity of 3.37mm²/s much lower than the viscosity of the biodiesel and closer to that of the petrodiesel. The flash point of the oil was higher than that of petro-diesel and could be attributed to higher number of fatty acids which were not volatile. The flash point of the biodiesel indicated that there was methanol left over. The pour points of oil, biodiesel and biodiesel blend in Table 1 confirmed that they were liquid at room temperature (28°C) and could still flow well in cold weather environments. The blend had lower pour point than biodiesel and petro-diesel. However, purification of the biodiesel would increase its flash point and further reduce its pour point which would as well improve the flash and pour points of the blend. The heating (calorific) values of the oil, biodiesel and blend in Table 1 were high when compared with the energy value of the petro-diesel. The blend had the same energy content with petro-diesel. This showed that the energy released when a known quantity of blend was burned under specific condition was similar to that released by the petrodiesel.

Tables 2, 3, 4, 5, 6 and 7 showed the FTIR spectra of wild melon oil, biodiesel, biodiesel blend and petrodiesel at 28°C (room temperature), 50, 100, 150, 200 and 250°C. The bond stability of oil, biodiesel and biodiesel blend when compared to the hydrocarbon diesel showed that they were stable at the above temperatures. The bond structure stability relationship deduced from FTIR spectra confirmed the structure of oil, ester and ester blend are similar to that of the petrodiesel and are therefore equivalent diesel fuels. Although the GC-MS profile of fatty acids composition of the crude biodiesel in Table 8 showed that the biodiesel was rich in polyunsaturated fatty acid methyl ester (59.47%) with little percent of monounsaturated fatty acid methyl ester (2.02%) and appreciable percent of saturated fatty acid methyl ester (38.51%), the FTIR spectra in the above tables showed no polymerization or breakage of bonds. Although some bonds were deformed, the bond structures of the samples were generally considered to be stable.

CONCLUSIONS AND RECOMMENDATIONS

The following conclusions could be made from this comparative analysis.

The physico-chemical properties of the biodiesel blend in terms of its relative density, total acid number, calorific value and viscosity were considered to be better than those of the oil and biodiesel and better

than those of petro-diesel in terms of its flash and pour points. Therefore, engine performances of both biodiesel and petro-diesel could be improved by blending the two fuels.

The Fourier Transform Infrared (FTIR) spectra of the crude biodiesel which was rich in polyunsaturated fatty acid methyl ester (59.47%) with little percent of monounsaturated fatty acid methyl ester (2.02%) and appreciable percent of saturated fatty acid methyl ester (38.51%) showed no breakage of bonds at maximum temperature of 250°C.

Acid catalysed esterification reaction greatly increased the acidity of biodiesel. Purification by washing with sodium hydroxide and blending are highly recommended.

Wild melon seeds had appreciable percent oil yield and the oil could be utilized for biodiesel production.

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