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Comparative analysis of the fuel properties of methylesters of *Pentaclethra microphylla* and *Dacryodes edulis* seed oils

[1]Ozioma J. Anekwe-Nwekeaku, [1]Tope O. Afere, [1]Chidinma M. Muobike, [1]Ifeyinwa B. Tabugbo, [1]Njideka V. Nwankwo, [1]Vincent I. E. Ajiwe

*Corresponding Author: Ozioma J. Anekwe-Nwekeaku, Department of Pure and Industrial Chemistry, Nnamdi Azikiwe University P.M.B 5025 Awka, Anambra State, Nigeria. oj.anekwe@unizik.edu.ng, +2348039582487

ABSTRACT

The oils of Pentaclethra microphylla and Dacryodes edulis were extracted by soxhlet method. They were transesterified and different dilution ratios (10:90, 30:70, 50:50, 70:30, 90:10) of the esters were made with fossil diesel. Properties such as density, viscosity, flash point, total acid, iodine value etc were determined. The results also showed that blending biofuel with diesel improved the efficiency of the fuels in terms of increase in calorific value and reduction in the acid value of the fuels. Furthermore, based on viscosity, flash point and iodine values, blends of the esters can be used as a substitute for diesel. Although, the viscosity of blends of the esters was found within 5.12mm^2/s-6.92mm^2/s higher than that of ester/diesel blends (2.73mm^2/s-4.56mmVs), this could be probably due to possible condensation reaction between the esters which eliminate alcohol molecule. According to American Society for Testing and Materials (ASTM) standard of grading fuel in term of viscosity, ester/diesel blends fell within the grade 2-D while ester/ester blends fell into 4-D grade diesel fuel. These results demonstrate that the vegetable oils, esters and ester blends could be utilized as a substitute or complement for diesel oil in diesel engine being. They are renewable materials and environmentally friendly.

Key words: Biodiesel, blend, Comparative analysis, Dacryodes edulis, Pentaclethra microphylla

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INTRODUCTION

Energy is a fundamental requirement for human existence. Many countries in the world are resorting to biofuel technology to solve the problem of the gradual increasing rate of fuel and energy prices resulting from the depletion of the world's non-renewable fossil fuel(1). As the diesel engine became more widely adopted in subsequent years, however, petroleum-based diesel fuel proved to be less expensive and became the fuel of choice. Unfortunately the combustion of hydrocarbon fuels contributes to over half of green-house gases emission. 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 adverse climate change. Hence, today's world is facing urgency in developing alternatives fuels. Among the various alternative is the biodiesel(2). Biodiesel is produced by transesterification of triglycerides with alcohol (3). Biofuels give off mainly carbon(iv)oxide which is often absorbed by the plants during photosynthesis. Biodiesel considered to be environmentally friendly and are renewable. Triglycerides are esters of three fatty acids and one glycerol. These contain substantial amounts of oxygen in its structure(4). The fatty acids vary in their carbon chain length and in the number of double bond(5).

MATERIALS AND METHOD

COLLECTION AND PREPARATION OF SAMPLES

Matured *Dacryodes edulis* seed (African pear seed) samples were collected from African pear trees in Awka, Anambra state while *Pentaclethra microphylla* (Oil been seed) samples were collected from Oji River Local Government, Enugu state. The seeds were cut into pieces and sundried for three months after which they were ground to powder, and stored in a screw-cap plastic container before extraction of their oils. Oils from the ground sample were thoroughly extracted with petroleum ether using a soxhlet extractor and separated using rotary evaporator apparatus.

BIODIESEL SYNTHESIS

The extracted oil samples were transesterified into ethyl esters by method described in(6), using methanol via acid catalysed esterification reaction. For the esterification step, 250-mL flask equipped with a reflux condenser was used for the reaction. Digital magnetic heater with temperature controller was used to maintain the reaction temperature at 70 "C. Alcohol/FFAs ratio of 1:6 and 3 wt% of catalyst (concentrated H_2SO_4) were used. The sulfuric acid was first diluted with distilled water in the ratio of 1 :2 and mixed with the FFAs and methanol in the reactor. The reaction mixture was heated to the required temperature while stirring at 200 rpm for 3 h. The products were separated using separating funnel and washed severally with warm distilled water.

Different dilutions of each biodiesel were made with petroleum diesel in the ratio of 20:80, 50:50 and 100:0 to obtain, B_2o, B_{50} and B_{100} respectively.

CHARACTERIZATION OF THE OILS, METHYL ESTERS AND ESTERS/DIESEL BLENDS

Physicochemical properties of the oils were analysed using AOAC official methods. Add value was determined according to AOAC Official Method Cd 3a- 63. Iodine value was determined following AOAC Official Method 993.20 (7). Saponification value was analysed using AOAC Official Method 920.160. The methyl esters and esters/diesel blends were analysed for parameters such as flash point, pour point, kinematic viscosity (at 40 °C, 70 "C and 100 °C) and relative density using American Society for Testing and Materials (ASTM). Flash point was determined using Pensky Martin apparatus (ASTM D 93). Redwood Viscometer India was used to determine kinematic viscosity (ASTM D 445). Pour point was determined following ASTM D 97. The relative density of the samples was measured at 25 ^{0}C using relative density bottle (ASTM D1298). Percentage moisture and ash was determined according to ASTM methods: D 6304 D and D 482 respectively [(8)].

Results and Discussion

Table 1: Physicochemical properties of the extracted oils

Parameter	*Dacryodes edulis*	*Pentaclethra microphylla*
Acid (mg KOH/g)	1.47	1.10
Relative Density (@28^{0}C) g/cm^3	0.96	0.98
Ash Content (%)	0.11	0.12
Moisture Content (%)	0.08	0.09
Saponification value (mg KOH/g)	140.01	161.10
Iodine value (g/100g of oil)	92.0	94.12

The results of the physicochemical properties of the extracted oils are shown on table 1. The acid values of the two oils were high indicating that the oils cannot be used directly in diesel engines without transesterification, hence the oils needed purification before use as fuels so as to reduce corrosion effects in diesel engines. Iodine value is a measure of the unsaturation of fats and oils. The iodine values of the oils fitted them into the non-drying oil group (<120). The moisture content of the oils was low, High moisture content in oils could make the oil go rancid and alter the chemical structure of the oil. The saponification values of the two oils were almost the same.

Comparative Analysis of Oils, Esters, Ester/Ester and Esters/Diesel Blends with Diesel (AGO).

Table 2: Results for *Dacryodes edulis* (Pear Seed) Oil, Its Methyl Ester/Diesel Blends and Diesel (AGO)

sample	Density (g/cm^3)	Viscosity (mm^2/s)	Flash Point(^{0}C)	Acid value (mg/KOH)	Iodine (mg/l)	Saponification mg/g KOH)	% moisture	% mineral ash	Calorific value (kJ/Kg)
Diesel D	0.86	2.70	104	0.23	8.60	129.00	0.02	0.013	45,126
seed oil	0.96	7.12	241	1.47	92.0	140.01	0.08	0.11	43,101
10PE:90D	0.87	3.51	228	0.45	96.44	132.12	0.02	0.03	44,938
30PE:70D	0.88	3.89	219	0.58	93.91	136.10	0.02	0,04	44,752
50PE:50	0.89	4.24	239	0.71	78.17	139.41	0.03	0.04	44,543
70PE:30D	0.91	3.93	214	0.68	75.34	141.14	0.04	0.05	44,147
90PE:10	0.93	4.56	204	1.36	62.94	145.11	0.05	0.06	43,729

Table 3: Comparative Result for *Pentaclethra microphylla* (Oil Bean) Oil, Its Methyl Ester/Diesel Blends and Diesel (AGO)

sample	Density (g/cm^3)	Viscosity (mm^2/s)	Flash Point (°C)	Acid value (mg/g KOH)	Iodine value mg/L	Saponification value (mg/g KOH)	% moisture	% mineral ash	Calorific value (kJ/Kg)
Diesel(D)	0.86	2.70	104	0.23	8.60	129.00	0.02	0.013	45,126
Seed oil	0.98	4.53	161	1.10	94.12	161.10	0.09	0.12	42.682
10BE:90D	0.85	2.73	148	0.48	100.44	141.15	0.03	0.04	45,126
30BE:70D	0.88	2.94	165	0.66	93.53	145.11	0.04	0.06	44,752
50BE.50D	0.89	3.20	132	0.57	86.93	149.26	0.04	0.06	44,543
70BE.30D	0.92	3.04	124	0.83	65.30	151.15	0.05	0.07	43,938
90BE.10D	0.95	3.22	104	1.03	53.30	153.14	0.05	0.07	43,310

The variations of samples with respect to viscosities were investigated. Viscosity is the most important property of biodiesels since it affects the operation of fuel injection equipment, particularly at low temperatures when an increase in viscosity affects the fluidity of the fuel. The results showed that the viscosities of the two oils and their methyl esters were higher than the viscosity of diesel and this may be due to glycerin content in the oil and fatty acid composition of the esters. The viscosity of the methyl esters were expected to be lower than that of the oils because the fatty acid compositions of the oils which were major contributor to the viscosity of the oils were reduced during saponification and esterification. Viscosity is also an indication of complete formation of ester, the conversion of triglycerides into methyl or ethyl esters through the transesterification process reduces the molecular weight to one third that of the triglyceride and reduces the viscosity by a factor of about Biodiesels have a viscosity close to that of diesel fuels. As the oil temperature increases its viscosity decreases (9). This confirmed one of the commonly cited benefits of blending biodiesel with petroleum diesel, that blending petrodiesel with biodiesel improves the viscosity of the blend thereby ensuring their compatibility with modern diesel engines which have fuel injection systems and are sensitive to viscosity change(10).

Table 3 showed that *Dacryodes edulis* seed oil ester/diesel had better flash point than pentaclethra micropylla. Their flash points conformed to ASTM standard flash point for biodiesel of 130°C and above. The flash points of the esters/diesel blends and the ester blends were generally higher than that of diesel. This could be attributed to the higher number of fatty acid methyl esters (FAME) which were not volatile. Thus, the biodiesel samples were safer to handle at higher temperatures and the storage of these fuels would not constitute a fire risk. Also it was observed that the addition of ester to diesel generally reduced the flash point of the biofuels. Liquid fuel with a higher flash point can prevent auto ignition and fire hazard at high temperature during transportation and storage periods. Hence, once the higher the flash point, for instance, the higher is the safety during handling, transportation, and storage.

The relative densities of the oils were higher than that of hydrocarbon diesel. This may be probably due to higher molecular weight fatty acid constituents of the oils. The result also showed that when ester was blended with hydrocarbon diesel, their respective density values were lower than that of the oils. This could be attributed to the fact that some of the fatty acid components of the oils were reduced during saponification and esterification. It was also an indication of the complete formation of esters because complete formation of ester is shown by decrease in relative density and viscosity of the formed ester blends. The relative densities of virtually all the blends samples exceeded that of the hydrocarbon diesel (Table 2-3) but within the ASTM recommended values of 0.87-0.90 g/cm^3. This was an indication of good ignition properties and as a result of this, it will take a shorter time for biodiesel compared to petroleum diesel to travel from injection pump to injector in the motor engine. Some works had shown that a low relative density indicated a predominantly paraffin fuel with good ignition properties in diesel engine while high relative density indicated mainly aromatic or asphaltic fuel with poor combustion properties [(11)], [(6)].

Tables 2 and 3 showed that *Dacryodes edulis* and *pentaclethra microphylla* oils, methylesters and their corresponding diesel blends oils had lower energy values (calorific value) than hydrocarbon diesel. This could be attributable to the high oxygen content of the oils that produce more complete combustion of the fuel and soot. Generally petroleum diesel have been reported to have higher heating value or energy value than biodiesel because petroleum diesel fuel is mainly made up of hydrogen and carbon and little oxygen (less than 0.3%) while biodiesel contain significant amount of oxygen (9%). Hence, this makes combustion efficiency of biofuel to be higher due to higher oxygen content. It was observed that calorific values of *Dacryodes edulis* and *pentaclethra microphylla* ester/diesel blends decreased as the ester portion of the ratios increased except in 70BE:30PE blends which showed a sudden increased and thereafter the decreased trends continued

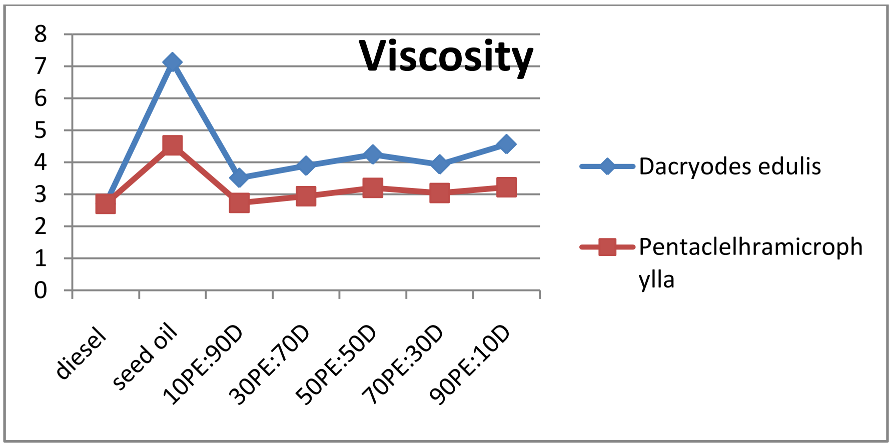

Fig 1: Variation of Viscosity of Dacryodes edulis and pentaclethra microphylla methylesters and their corresponding diesel blends.

From the plot in Fig 1, *dacryodes edulis* methylester had the highest viscosity on dilution with diesel when compared to *pentaclethra microphylla* methylesters.. Furthermore, there was a progressive decrease in viscosity as the methylesters were diluted with petroleum diesel.

The viscosities of pear oil ester/diesel and oil bean ester/diesel blends increased slightly as the ester portion of the ratios increased with exception of 70PE:30D blend which decreased slightly and thereafter the trends continued. Also, Table 4.3showed that viscosities of pear oil ester/oil bean ester blends decreased steadily from 10BE:90PE blend o 90BE:10PE blend.

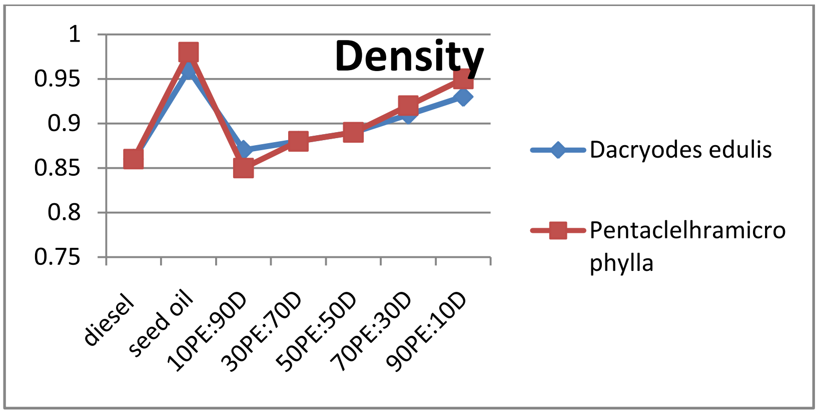

Fig 2: Variation in the relative density of the Dacryodes edulis and pentaclethra microphylla methylesters and their corresponding diesel blends.

From the plot in Fig 2 it was observed that the relative densities of the oils were higher than that of hydrocarbon diesel and the ester blends. it was also observed that both oils had similar densities which was slightly above ASTM limit of 0.80-0.9 [(12)].The density of *Dacryodes edulis* and *pentaclethra microphylla* ester/diesel blends respectively increased steadily as ester portion of the ratios increased.

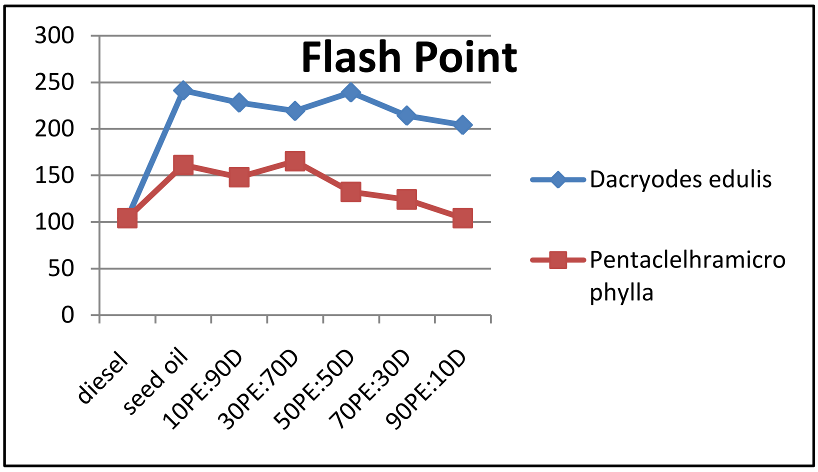

Fig 3: Variation of flash points of *Dacryodes edulis* and *pentaclethra microphylla* methylesters and their corresponding diesel blends.

Fig 3 showed the variation of flash points of *Dacryodes edulis* and *pentaclethra microphylla* methylesters and their corresponding diesel blends. From the plot it was observed that *Dacryodes edulis* methylesters and its diesel blends had the highest value of flash points than *pentaclethra microphylla*. From the plot it was also observed that the flash points of *Dacryodes edulis* oil/diesel blends were decreasing with increase in ester ratios but a sharp increase was observed at 50PE:50D blend, thereafter there was a decrease in the trend. Also, the flash point of *pentaclethra microphlla* ester/diesel blends decreased from 10BE:90D to 90BE:10D blends except in 30BE:70D blend where there was a sharp increase, and thereafter the trends continued. It is worthy to know here that IOBE:90D, 20BE:80D and 50BE:50D.

Table 4: Grading of esters/diesel blends *Dacryodes edulis* ester *Pentaclethra microphylla* ester/blends based on their viscosities and according to standard

D. ester/Diesel			**P. ester/Diesel**		
ID	2D	4D	ID	2D	4D
-	10:90	-	-	10:90	-
-	30:70	-	-	30:70	-
-	50:50	-	-	50:50	-
-	70:30	-	-	70:30	-
-	90:10	-	-	90:10	-

Note: D. ester = *Dacryodes edulis ester*, P. ester = *Pentaclethra microphylla* ester

Based on their viscosities, the esters/diesel blends and D. ester/P. ester blends fell within ID (0.5-1.9 mm^2/s), 2D (2.0-4.9 mm^2/s) and 4D (5.0-24.0 mm^2/s) .diesel grades According to ASTM standard at room temperature [(8)] as showed in Table 2 & 3.The viscosities of pear seed oil/diesel and oil bean/diesel blends fell within 2-D grades diesel fuels with 2.0-4.56 mm^2/s, 90PE:10D blend being the highest viscosity value. This Table 4.4 also proved that blending of esters in appropriate ratios can serve as diesel fuel, because here they competed with ASTM standard. The viscosities of *Dacryodes edulis* ester *Pentaclethra microphylla* ester blends all fell within the 2D grades diesel with 5.12-6,92 mm^2/s.

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