Fuel and Physiochemical properties of Cashew (*Anarcardium Occidentale*) Nut oil, its Methyl Esters and Blends

Abstract

Cashew seed oil has several industrial applications but it physiochemical properties are not fully available in literature. The aim of this study is to measure the fuel and physiochemical properties of cashew nut oil. The oil was extracted by soxhlet extraction method and transesterified with methanol using sodium hydroxide as catalyst. The resulting methyl ester was washed, dried and blended 20% (B20) and 10% (B10) with diesel. The characterization of the oil, its methyl ester, and the blends was undertaken following the ASTM and EN protocols. The fatty acid profile was determined by chromatography analyzer. The results obtained shows that the properties of the methyl ester are close to those of diesel and can thus be used as feed stock for the production of biodiesel.

Key words: Cashew seed oil, physiochemical properties, biodiesel, blends.

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1. Introduction

Cashew nut is one of the possible feed stocks that can be commercially exploited in Nigeria as it has been ascertained by the Food and Agricultural Organisation of United Nations – Economic and Social Department [1], that Nigeria is the second largest cashew nut (with shell) producers in the world, producing 660 000 tonnes per year. It's extensively cultivation South America, India and Africa helped by the fact that cashew tree will tolerate a wide range of conditions including drought and poor soil. It cannot however, withstand cold frost but would quickly recover after a bush fire.

Cashew nut (Figure 1.) yields two "oils", one obtained, between the seed coat (or pericarp) and the nuts and called the Cashew Nut Shell Liquid (CNSL). CNSL is not a triglyceride hence cannot be converted to biodiesel but contains a high proportion of phenolic compound and can be polymerized in a variety of way which makes it a versatile industrial raw material with diverse use in friction linings, paints and varnishes, laminating and epoxy resins, foundry

chemicals and as an intermediary of chemicals. It is however, toxic and corrosive to the skin. Cashew nut oil obtained from the nut contains triglyceride and therefore a potentials abundant source of feedstock for the production of the biodiesel. It is also used for manufacturing wood preservatives, cardinals, coatings and friction materials [2].

Cashew tree is high yielding in Nigeria and a five years old cashew tree can produce up to 500 seeds per annum. However, the full benefit of the tree is not exploited as on typical plantation, the fruit are allowed to fall and decay to release the nut that are then picked up. Sometimes they are eaten fruit and nut raw by cows and other animals. Some studies have been done on the use a cashew nut oil [3-11] but not much work has been done on the measurement of the physiochemical properties of Nigeria's cashew seed oil hence this

Fig. 1 Cashew nuts

study is a characterization of the physiochemical and fuel properties of cashew seed oil, its biodiesel and B10 and B20 blends.

Biodiesel has been defined as the mono alkyl ester of vegetable oils and animal fats [12]. It is biodegradable, nontoxic, environment friendly and has agricultural origin hence renewable [13],[14],[15],[16] and [17].

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The fuel properties of biodiesels are to a large extent dependent on feed stocks, which vary not only with species but also where grown and method of production [18]. The fuel standards for biodiesel, which evolved from fossil fuel standards, ensures quality assurance by limiting the amount of impurities in the fuel such as triglycerides, monoglycerides, diglycerides, water, free fatty acid, soap and residual alcohol. These materials need to be limited in biodiesel because their presence can lead to fuel deterioration during storage can cause significant operational problems.

2.0 Materials and Methods

2.1 Oil extraction

Cashew nut was planted in the backyard garden in the compound in 2010 and it yielded fruits after 15 months in the month. Two kg of fruit were harvested in march 2012 and were dried in the sun for five days at 35°C to reduce moisture content to 7% [19]. They were decorticated manually to remove the kernel from the shell. The seeds which measured 5 mm diameter on the average were milled to reduce diameter to 1.16 mm and expose more area for higher oil extraction [20] and[21]. Analytical grader methanol, n-hexane and sodium hydroxide were procured from Finlab laboratory in Akure Nigeria. The oil was extracted in a soxhlet extractor with the n-hexane as the solvent. The oil was leached for 8 hours at 60°C and the hexane in the mixture was removed by using a vacuum rotary evaporator operated at 75°C. The oil obtained was weighed and the percentage oil yield was calculated by equation 1.

$$\text{Oil yield } \% = \frac{\text{weight of oil extracted}}{\text{weight of seed kernels used}} \text{x}100 \qquad 1.$$

2.2 Characterization of the Oil.

The properties of the oil were first measured to determine if pretreatment was necessary or not before alkaline transesterification. It was found that the free fatty acid value (FFA) of the oil was 23.68 mg KOH/gm (oleic)which is too high for alkaline transesterification as it can react with the catalyst to form soap which can inhibit methyl ester yield [22].

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2.3 Tranesterification Procedure

Transesterification was carried out using a laboratory scale biodiesel processor in chemistry laboratory of the Federal University of Technology, Akure, Nigeria. Methanol was used as the reagent because it is a low cost and short-chain alcohol that reacts fast. Alkaline transesterification was also done using anhydrous methanol at a molar ratio of 6 to 1 and 3g/litre of sodium hydroxide as catalyst. The processor was stirred at 600 rev/min. at a temp of 60 ^{o}C for 2 hours, after which the mixture was poured into a decanter and allowed to settle for 12 hours so that the reaction can be driven to completion and for the mixture to separate into two layers of methyl ester and glycerol which was at the bottom. The glycerol was drained off by gravity. The excess methanol in the ester was removed in a flash evaporator. To remove any impurity, the methyl ester was washed in distilled and deionized water of volume ratio 3 to 1 three times. Finally, the washed methyl ester was dried by passing it through anhydrous sodium sulphate (Na_2SO_4)[23].

2.4 Fatty Acid Profile

The fatty acid composition of the cashew seed oil, the methyl ester and blends with diesel (B10 and B20) and the standard sample of free fatty acid were determined by chromatography analyzer following the modified AOAC 965.49 and AOAC 996.06 official methods. The fatty acid methyl esters were separated using HP 6890 Gas Chromatography analyzer powered by HP ChemStation Rev A 09.11 [1206] software and equipped with a flame Ionization Detector (FID) and HP INNOwax column (30 m x 0.25 cm x 0.20 μm film thickness) The carrier gas was nitrogen and the oven initial temperature was at 60 ^{o}C. The first ramping was at 10 ^{o}C/min for 20 min and maintained for 4 minutes. The second ramping was at 15 ^{o}C/min for 4 minutes and maintained for 10 minutes. The detector temperature was 320 ^{o}C while hydrogen and compressed air pressures were 22 and 35 psi respectively. The proportion of the various fatty acids is shown in Table 1.

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2.5. Determination of Fuel and Physiochemical Properties

The fuel and physiochemical properties of the cashew seed oil, the methyl ester and blends were determined following ASTM, EN and AOCS methods and undertaken in McDonald laboratory in Warri, Delta state and Chemistry laboratory of the Federal University of Technology, Akure. Nigeria.

The methyl esters and diesel were blended using a rotary blender in the ratio10% and 20% (vol/vol). For Metal analysis, 50 ml of samples were prepared by diluting 1:10 with kerosine. The dilution was to eliminate possible viscosity effect that may affect accuracy. The analyzer was calibrated with standards prepared by diluting plasma pure biodiesel stock standards. The standard concentrations were 0.00, 10.00, 20.00, 30.00, 40.00 ppm of Na, K, Ca, mg, P and S.

For the glycerine Content, the wet Chemical AOCS method for determining glycerol, methanol, AOCS Official method Ca 14-56 entitled "Total, free and combined glycerol Iodometric-periodic Acid method as well as methanol Content were used. During the vacuum Distillation test, the system pressure was set at between 1 kpa and 83 kPa. Distillation temperatures were made to correspond to 5,10,15,20,25,30,35,40,45,50,55,60,65,70,75,80,85,90,95 volume % of the liquid fuel distilled and condensed. For the copper strip corrosion test a polished copper strip was immersed in 50 ml sample of the oil and methyl ester for 3 hours at 50°C. At the expiration of the duration each strip was washed in a standard solvent and the result were compared with standard description of tarnished and corrosion.

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3.0 Results and Discussion

The oil content was 40.4% which means that about 266 640 tonnes of biodiesel can be produced from cashew nut oil annually if all the nuts are used for the purpose . The oil is 63% unsaturated consisting mainly of 20.97% linolenic acid with triple bond , 34.47% linoleic acid with double bond and 34.47% palmitic saturated fatty acid. Many properties vary with chain length, number of bonds and degree of unsaturation [24]. It was reported that methyl esters of saturated acids have a higher cloud point, cetane number and better stability [25]. The results obtained are shown in Tables 2 to 5.

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3.1 Density: The density of biodiesels is generally higher than those of fossil diesel fuel and the values depend on their fatty acid composition as well as on their purity. CNO density of 902 kg/m^3 reduced to 874, 856 and 854 for the B100, B20 and B10 respectively. The relative density of CNOME is within the limits for biodiesel and higher than diesel except for B20.

3.2. Cloud Point and Pour Point: Cold temperature behaviour of biodiesel is an important quality criterion, as frozen fuel may cause blockage of the fuel lines and filters and starve the engine of fuel. The two most important criteria are the cloud and pour points. The cloud, pour and cold filter plugging points are all above zero which limits their application in cold regions.

3.3. Flash point: The flash point of pure biodiesels is usually higher than the ASTM limits, but fall rapidly with increasing amount of methanol. CNO has a flash point of 167 ^{o}C, the value for CNOME is 136°C while the values for B10 and B20 are 82 ^{o}C and 89 ^{o}C respectively and which although are below the minimum for biodiesel, are higher than that for diesel fuel.

3.4. Viscosity: Kinematic velocity is the dynamic viscosity/relative density. The dynamic and kinematic viscosities of the CSO are 49.62 cts and 54.92mm^2/s respectively. Those of CNOME and the blends are within the limits for biodiesel. Viscosity is an important property of biodiesel since it affects the operation of fuel injection equipment, particularly at low temperatures when the increase in viscosity affects the fluidity of the fuel or leakage at high temperature when too thin. Dynamic viscosity is of interest in relation to the settling rates of contaminants, and kinematic velocity in respect of pumping and atomization.

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3.5. Heating values: The higher and lower heating values for CNO are 40 MJ/kg and 37.30 MJ/kg, 44.00 MJ/kg and 37.20 MJ/kg for CNOME respectively and are slightly less than that of diesel fuel which is consistent with the trend for biodiesel. The heating values increases after transesterification and the amount of diesel in the blend because diesel has higher heating values.

3.6. Cetane Number Biodiesel has a higher cetane number than diesel fuel because of its oxygen content [25] and also because the fatty acids present in the fuel have very high octane number. Cetane number increases with increasing length of both fatty acid chain and ester groups, while it is inversely related to the number of double bonds. The calculated cetane number of the CNO of 49.28 increased to 60.83 for the CNOME 47.75 and 46.22 for B20 and B10 respectively. That of CNOME is above the minimum limit for biodiesel but reduces with blending because diesel has lower cetane number.

3.7. Water content and sediment:

CNO contains 10% water by volume. This falls drastically after transesterification and the values depends on the drying process. The moisture content is 3420 and reduced to 221 after transesterification. Free water promotes microbial growth, so that sludge and slime formation thus induced may cause blockage of fuel filters and fuel lines. Moreover, high water contents are also associated with hydrolysis reactions, partly converting biodiesel to free fatty acids, also linked to fuel filter blocking.

3.8. Cold soak filtration: The cold soak filtration is 310 for CNO and falls to 230, 77 and 95 for the CSNME and the B10 and B20 blends respectively.

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3.9. Iodine values:

Iodine number is a measure of the total unsaturation within a mixture of fatty acids, and is expressed in grams of iodine which react with 100 grams of biodiesel. Unsaturation can lead to deposit formation and storage stability problems with biodiesel. Fuels with higher iodine number tend to polymerize and form deposits on injector nozzles and piston when heated. The iodine values for the CNO, CNOME, B10 and B20 are below the maximum for biodiesel. The value for CNOME 1s 85.28 and 82.75 for CNOME The iodine values are all below the maximum for biodiesel and reduces further with blending. Iodine value is an indication of the drying quality of the oil.

3.10. Peroxide value: This is an indicator of oil autoignition and high value is an indication of high degree of rancidity A peroxide value of 21.70 for the biodiesel is high compared to common vegetable oils.

3.11. Oxidation stability:

FAME ages because of their nature origin and aging properties vary with method of production, raw materials used, exposure to sunlight high temperature and oxygen. This can lead to the formation of insoluble sediments and gums, which are associated with fuel filter plugging and deposits within the injection system and the combustion chamber. It leads to increased viscosity and acid value. The oxidative stability of CNO at 110 ^{o}C was 19 hr, 9 hours for CNOME, 27 and 18 for B10 and B20 respectively. It reduces after transesterification but increases with blending and in all case higher than diesel.

3.12. Acid number and Free Fatty Acid: This is the quantity of base required to titrate a sample to a specified end point. It is a measure of free fatty acid in biodiesel. Excessive free fatty acid in the fuel can be corrosive and may be a symptom of water in the fuel or poor production or subjected to oxidative degradation. Excessive free fatty acid in the fuel can inhibit the process and lead to soap formation. The free fatty acid and acid values of the oil are 2.29 mgKOH/gm and 4.56 mgKOH/gm respectively. The fatty acid is not too high for direct alkaline transesterification and would not inhibit the separation of ester and glycerol. The acid value reduced after transesterification but increased with increasing amount of diesel on the blends.

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3.14. Saponification values: The Saponification value of the oil is 117.11 for the oil is within the range for most vegetable oils [26] and [27]. It would be suitable for the production of soap and shampoo.

3.15. Soap content: The soap content for CNO was zero but increased to 4 for CNOME as a result of transesterification. It reduced further with blending.

3.16. pH value: The ph value is just above 7 indicating that they are alkaline and the alkalinity increases with blending.

3.17. Refractive index:

The refractive index was 1.466 and reduced to 1.334 after transesterification but increases to 1.481 after blending. Reflective index value is a measure of purity of the fuel.

3.18. Carbon Residue:

The carbon residue test gives the remaining part when a sample of biodiesel has been subjected to thermal decomposition and hence an indication of coking tendency of the fuel. The most common cause of excessive carbon residue in biodiesel is the excessive level of total glycerin soaps and other organic impurities. The carbon residue is 0.07 (mol/mol) CNOME and below the maximum ASTM limits for biodiesel.

3.19. sulfated Ash:

It is a measure of the mineral ash residue when a fuel is burned. It is an important test for biodiesel because it is an indicator of the quantity of residue metals in the fuel that came from the catalyst used in the transesterification process. The sulfated ashes for B100 is 0.03 % (mol/mol) and lower than the 0.05 ASTM maximum limits.

3.20. Total Glycerol:

Total glycerol is the sum of the concentrations of free glycerol and glycerol bound in the form of mono-, di- and triglycerides. The concentration depends on the production process. This can affect a wide range of fuel properties which can affect operational efficiency and engine durability. Table 3. Shows all the glyceride contents.

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3.21. Distillation temperature, 90% recovered, maximum:

This parameter is related with the boiling point of a liquid fuel and affects the combustion characteristics of diesel engines and commonly used to determine the cetane number. The vacuum distillation end point is below the 360°C maximum at 90% distillation of EN D1160. The distillation temperature increases with the amount of methyl ester in the blend this result was also noted by [28]. The result is shown in Figure 1.

3.22. Alkaline and Metal components:

Metal components constitute impurities in the fuel which are deposited on engine components after combustion. They also react with metallic components of fuel systems and should be limited to recommended values to reduce deposits on engine components, injectors and catalytic system. All the metals levels are high for the oil but below the limit for biodiesel for B100. Sulfur however increases with blending since the diesel used contains sulfur. The result is shown in Table 3.

3.23. Phosphorous content:

Phosphorus in biodiesel originates from phospholipids (animal and vegetable material) and inorganic salts contained in the feedstock. Phosphorus has adverse effect on long term activity of exhaust emission catalytic systems and therefore limited by specification. The value for the oil is less than the maximum of 10 mg/kg specified by EN standard

4.0. Conclusions and Recommendations

Rubber seed oil methyl ester contains 23.74% oleic, 37.9% linoleic, 15.160% linolenic, 19.32 palmitic and 3.876 stearic acids. It has 76.802% total unsaturation.

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Table 1: Properties of cashew nut oil measured and equipment used

Property	**Equipment**
Density	Hydrometer IP160/ASTM-1298
Cloud Point	Cloud point meter
Pour Point	Hergoz HCP 852
Cold filter plug	Analyzer Model MC840
Dynamic viscosity	Brookfield Model LV-DV11
Kinematic viscosity	Hergoz GmbH MP-480
Flash point	ASTM D6751 using Kehler Model K-16270 (Pensky-Martens Closed Flash Tester).
Heating values	Oxygen bomb calorimeter (Parr Instrument Corp.USA)
Cetane number	Analytically
Iodine number	Titration(AOCS cd8, 1980
Peroxide number	Titration(AOCS cd8, 1980
Acid value	Titration
Oxidative stability	Glassware Apparatus ASTM D 2274
Saponification value	Titration
Soap content	Titration AOCS method cc 17-79
Cold soak filtration	Neslab RTE -740 digital plus refrigerated bath. Thermo Scientific (NH USA)
Moisture value	Karl-Fisher Method (ASTM D 6304) 831 KF Coulometer(Metrohm company, Switzerland)
Water and sediment	centrifuge model HNS II by Thermo Electron corp, US
Reflective Index	Abbe Refract meter
pH value	Ph meter

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Carbon residue	Instemp Muffle furnace
Sulfated ash	Instemp Muffle furnace
Metal analysis	Teledyne Fuelpro biodiesel metal analyzer.
Free glycerine	
Copper Strip Corrosion Test	**Copper Strip Corrosion Test**
Vacuum distillation	ASTM D1160. Reduced pressure advanced Distillation Curve (DAC) apparatus
monoglycerides, diglycerides and triglycerides	

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366 **Table 2. Fatty Acid Profile of cashew**

367 **seed oil and its methyl Ester and**

Acid	**Form**	**Oil**	**B100**
Capric	C10:0	1.367	2.225
Lauric	C12:0	1.832	2.984
Myristic	C14:0	0.587	0.957
Palmitic	C16:0	28.867	23.134
Palmitoleic	C16:1	3.162	5.155
Stearic	C18.0	4.061	5.573
Oleic	C18:1	34.479	31.648
Linoleic	C18:2	4.673	7.616
Linolenic	C18:3	20.971	20.709
Total Saturation		36.715	34.871
Total unsaturation		63.285	65.128

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369 **Table 3. Properties of Cashew Seed Oil, its Ester and Blends to ASTM D6751-02 and EN**
370 **14214**

Property	CSO	B100	B10	B20	ASTM Limits	EN 14214 Standards
Density kg/m^3 at 15 ^oC	902	874.2	854.9	856.4		860-900
Relative density kg/m^3 at 15 ^oC	0.904	0.875	0.855	0.857		
Cloud point ^oC	20	6	7	9		
Pour point ^oC	13	1	5	5		
Cold filter Plug Point ^oC	15	4	6	7		
Flash Point ^oC	167	136	82	89		120 min
Dynamic Viscosity cts	49.62	4.21	2.46	2.67		
Kinematic Viscosity mm^2/s	54.92	4.81	3.12	2.88	1.9-6.0	3.5-5.0
Lower Heating Value KJ/kg	37.30	37.20	42.80	42.20		
Higher Heating Value KJ/kg	40	40.40	45.30	44.85		
Calculated Cetane number (ASTM D4737)	49.28	60.83	46.22	47.75	47 min	51 min
Free Fatty acid %	2.29	0.188	0.188	0.225		
Acid Value mgKOH/g	4.56	0.374	0.374	0.448	0.80 max	0.05 max
Iodine Value gI_2/100g	85.28	82.74	23.35	16.24		120 max
Peroxide Value gI_2/100g	21.70	27.00	17.30	16.30		
Oxidation stability (hours) 110^oC	19	9	27	18		6 min
pH value	7.10	7.40	8.50	8.67		

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Saponification Value mgKOH/g	117.11	187.94	1165.5	162.69		
Soap content %	0	4	0.5	1.0		
Cold soak filtration ^{o}C	310	230	77	95		
Water and sediments %vol	10.00	0.02	0.004	0.002		500 max
Moisture content ppm	3420.	221.0	48	24		
Refractive index at 15 ^{o}C	1.466	1.334	1.481	1.480		
Sulfated ash % (mol/mol)	0.60	0.03	0.01	0.01	0.0020 max	0.02 max
Carbon residue % (mol/mol)	0.17	0.07	0.03	0.2	0.005 max	0.30 max
Copper Strip Corrosion Test (3h, 50°C)	4	2	1	1	No.3 max	1
Distillation temperature 90% ^{o}C	355	350	320	322	360 max	

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382 **Table 4. Glycerine contents**

Property	CSO	B100	B10	B20	ASTM Limits	EN 14214 Standards
Methanol Content % (mol/mol)	0.003	0.096	0.011	0.021		0.20 max
Free Glycerol % (mol/mol)	0.960	0.011	0.002	0.003	0.020	0.02
Monoglycerine % (mol/mol)	0.002	0.150	0.018	0.032		0.80
Diglycerine % (mol/mol)	0.001	0.009	0.002	0.003		0.20
Triglyceride % (mol/mol)	98.752	0.005	0.003	0.005		0.20
Total Bounded glycerol	98.755	0.164	0.023	0.040		
Total Glycerol % (mol/mol)	9.715	0.175	0.025	0.043	0.240	0.25

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392 **Table 5. Metal content.**

Metal	Detection Limit (ppm)	Spike recovery	CSO	B100	B20	B10	ASTM	EN
Sodium Na 589.592r	0.019380	100.9	4.50	2.50	0.03	0.02		5.0 max
Potassium K766.491r	0.07040	101.1	2.80	1.00	0.08	0.04		5.0 max
Calcium Ca317.933	0.00292	101.6	3.40	0.50	0.01	0.06		5.0 max
Magnesium Mg285.213r	0.000920	99.1	28.50	0.70	0.15	0.08		5.0 max
Phosphorous P212.61	0.02275	99.1	40.6	5.20	7.50	3.60	0.001	10.0 max
Sulphur S180.731r	0.11415	98.7	21.0	9.20	27.50	26.9	0.05 max	10.0 max

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394 **Table 6. State of cashew nut oil, Methyl ester and Blends.**

S/N	Fuel	State at room temperature (25^oC)	Colour
1	Cashew nut oil	Liquid	Dark brown
2	B100	Liquid	Golden yellow
3	B20	Liquid	Dark brown
4	B10	Liquid	Dark brown
5	Diesel	Liquid	Dark brown

395

396

UNDER PEER REVIEW

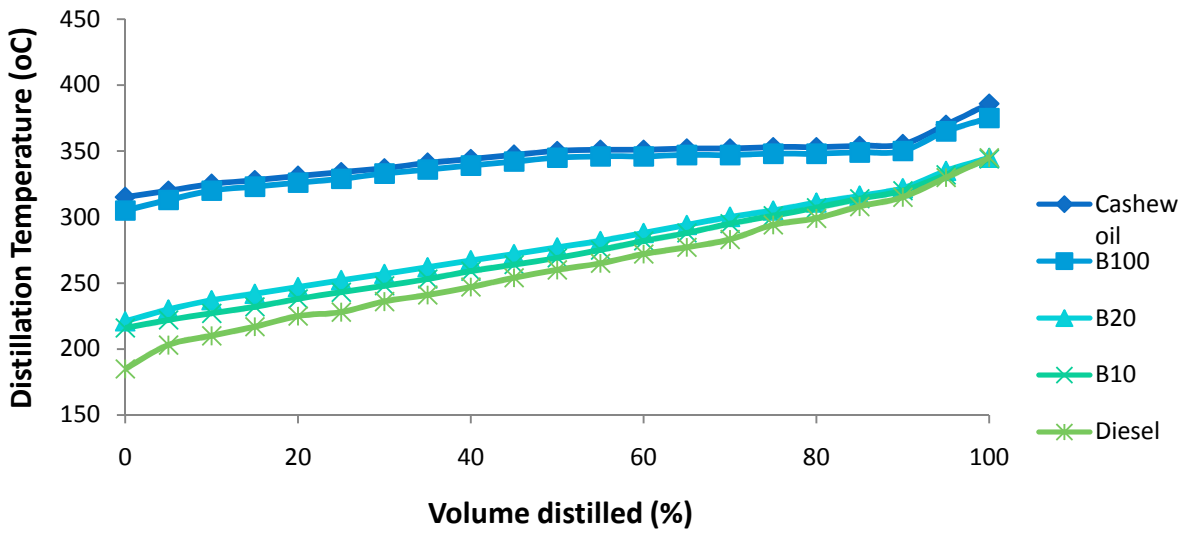

Fig.1. Distillation curves for cashew nut oil, Methyl ester and Blends.

UNDER PEER REVIEW