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Production and Characterization of Ackee Apple (*Blighia sapida*) Seeds and African Star Apple (*Chrysophyllum albidum*) Seeds Oil Mixtures and their Biodiesel

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Abstract*: This paper focused on the characterization of oils and biodiesels derived from Ackee apple seeds and African star apple seeds obtained from local markets. The oils from individual seeds and their mixtures at varying ratios were characterised for relative density, free fatty acid, acid, iodine, and saponification, which yielded 0.91 g/cm^3, 1.06 mg-KOH/g, 2.12 mg-KOH/g, 38.36mg-iodine/100g, and 195.74 mg-KOH/g of ackee seed oil, respectively. And 0.89 g/cm^3, 2.105 mg-KOH/g, 4.2 mg-KOH/g, 52.49 mg-iodine/100 g, and 227.7 mg-KOH/g of African star apple seed oil, respectively. The highest relative density of 0.9064 g/cm^3 and free fatty acid of 3.73 mg-KOH/g were achieved from the mixture of ackee apple and African star apple seeds' oils at 80 and 20%, respectively, while the highest saponification of 221.264 mg-KOH/g and iodine of 49.66 mg-iodine/100 g were obtained from the oil mixture of 20 and 80%, respectively. Also, the oils extracted from the seeds, were subjected to a transesterification process to produce biodiesel. 144°C flash point, 206°C fire point, and 2.8°C cloud point were obtained from the biodiesel of the oil mixture of 20 and 80%, respectively. Further analysis of the mixtures showed low volatility and high resistance to fire due to their high flash and fire points. The highest value recorded for the flash point is lower when compared with some other seed oils flash points; however this value is higher than the standard flash point for biodiesels .Highest boiling point of 64°C was attained at an oil mixture ratio of 60 and 40%, respectively. This value is too low compared to the normal boiling point range of 315-350°C for biodiesels, and the high acid values recoded for the mixtures make the oils inedible. The oils, however, have advantages over other edible seed oils as they will serve as valuable ingredients in the soap-making industries since they are not competing with food resources.*

Keywords: *Ackee Apple, African Star Apple, Biodiesels, Physicochemical Properties, Transesterification.*

1. INTRODUCTION

Growing uses and demands for biodegradable oils are one method of addressing global warming; thus, continual identification and study of oils taken from natural sources for physical and chemical properties is required. Furthermore, the present global fuel supply instability, as well as the predicted growth in demand, has encouraged the search for accessible alternative fuel sources [1, 2]. Natural sources of oil include ackee apple seed (*Blighia sapida*) and African star apple seed (*Chrysophyllum albidum*). These oils' physical properties include their colours, scents, viscosities, and densities, while their chemical properties include their fatty acid composition, iodine values, saponification values, and acidic values. The characterisation of their biodiesel included determining the pour point, cloud point, fire point, flash point, and boiling point [3 - 5].

Transesterification is the general term used to describe the important class of organic reactions where an ester is transformed into another through the interchange of the alkoxy moiety. When the original ester reacts with alcohol, the transesterification process is called alcoholysis [6, 7]. In this reaction, triacyl glycerides from a variety of feedstocks, such as non-edible oil seeds, vegetable oils, animal fats or tallow, waste cooking oil, and microbial lipids or single-cell oil (from algae, oleaginous yeast, filamentous fungi, and bacteria), are converted into fatty acid methyl esters (biodiesel) in the presence of alcohol (methanol or ethanol) [8]. The reaction is an equilibrium reaction, and the trans-formation occurs essentially by mixing the reactants. However, the presence of a catalyst (typically a strong acid or base) accelerates considerably the adjustment of the equilibrium (Equation 1). In order to achieve a high yield of the ester, the alcohol has to be used in excess [9, 10].

$$RCOOR' + R''OH \overset{Catalyst}{\leftrightarrow} RCOOR'' + R'OH \tag{1}$$

This reaction has been extensively used to reduce the viscosity of feedstock or nonedible oil and improve its compatible fuel properties to a level similar to fossil-based diesel oil. Thus, the biodiesel production method applied worldwide at the industrial level is the alkalosis (transesterification) of triglycerides, which are the main component of vegetable oils and animal fats. The most common type of esters is methyl esters, mainly because methanol is usually the cheapest alcohol [11 - 13]. Shown in Table 1 are the standard (ASTM D93) values of fuel properties of biodiesels, while Tables 2 and 3 compare the physicochemical properties of some selected seeds oils [14 - 17].

Table 1: Standard values of properties of biodiesels [14 - 17]

Properties	**Limits**	**Units**
Flash points	130min	°C
Kinematics viscosity	1.9 – 6.0	mm^2/sec
Cloud point	-3 - 12	°C
Pour point	-15 - 16	°C
Boiling point	>202	°C

Table 2: Comparative analysis of the fuel properties of some selected seed oils [14 - 17]

Properties	**Rapeseed**	**Ground Nut**	**Soyabeans**	**Ackee seeds**	**Neem Seeds**	**African star apple seeds**
Viscosity	4.5cp	8.33cp	3.15cp	-	-	-
Specific gravity	0.91	0.870	0.896	0.81	-	0.896
Flash point	286 °C	220 °C	138 °C	114 °C	68 °C	178 °C
Cloud point	1 °C	5 °C	0 °C	1 °C	-2 °C	-
Pour point	-4 °C	-5 °C	-3 °C	11 °C	10 °C	-
Fire point					58 °C	72 °C

Table 3: Comparison of physicochemical properties of some selected seed oils [14 - 17]

Parameters	**Ackee apple seed**	**Soya beans**	**Hemp seed oil**	**African star apple seeds**
Oil Yield %	23.54	21.4	32.3	14.92
Acid Value (MgNaOH/g)	1.112	2.13	2.15	3.23
Free Fatty Acid(MgNaOH/g)	0.556	2.08	1.79	2.07
Iodine Value(Mgiodine/100g oil)	66.2	135.7	163.5	47.63
Saponification Value(Mg/KOH/g oil)	589.1	185.4	198.2	231.32

Ackee fruit grows on evergreen trees and is available throughout the year in Nigeria. It grows on a tropical evergreen tree that is native to West Africa and also goes by the names ackee and ackee apple [18]. When the fruit is fully developed and ripped, the pods are bright red, and they split open naturally to expose the edible fruit inside (Figure 1). The pod opens to expose three or four cream-coloured sections of flesh called arils underneath large, glossy black seeds (Figure 2). The arils are the edible part of the fruits, while the black seeds are often discarded because they are not edible [19].

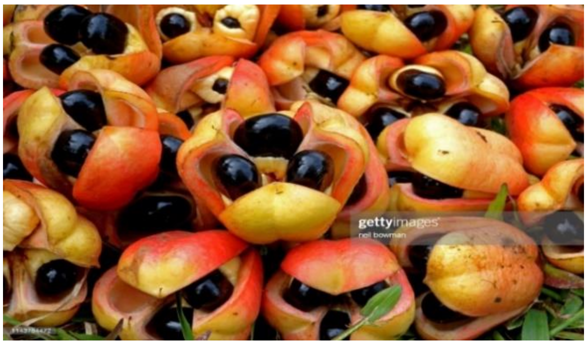

Figure 1: Ackee fruits

Figure 2: Ackee seeds

The African star apple is a fruit (Fig. 3) that comes from tropical Africa but is known and widely consumed in Nigeria. African star apple is also very productive in the Republic of Benin, Togo, and Ghana. Another name is *agbalumo* (Yoruba language) or *air* (Igbo language). *Chrysophyllum albidum* is the scientific name of the African star apple. The fruit is available from around December to April. African star apple has 4 to 6 seeds (Fig. 4) in it that have a star-like shape, which is why this fruit is named African star apple [20].

Figure 3: Apple fruits

Figure 4: Apple Seeds

The interesting thing about this fruit is that some people take it before it falls naturally to the ground. When they eat that, the taste of the fruit is very acidic. However, when an African star apple is naturally fallen to the ground, the fruit will have a very sweet taste. That means the fruit is ripe [21]. The seeds of the two crops have some oil content, which may be extracted and used for a variety of purposes dependent on the classification of the oil and biodiesel generated from them. Results from previous works on separate physicochemical characterization of these seeds oils [17, 22], had advanced their potential use in bioresin production and the industrial application of their biodiesels. Specific work on renewable Ackee seed oil by Rominiyi et al [17] established that fuel properties of Ackee seed oil biodiesel are close to that of diesel fuel and also meet the specification of ASTM standards. Also, work on African star apple seeds oil [22] and many other selected oil seeds [10] shows that these seeds have good physicochemical properties that meet ASTM standards. But, biofuel production requires cheap feedstock with high oil yield [10]. The shortcoming of low oil content may however be compensated for if these oils and their biodiesels are separately blended at optimum ratios. As a result, this study characterises the oils and biodiesels from ackee and African star apple seeds, as well as blends of the two oils at varied mixing ratios. It also compares the characterisation data obtained from pure and blended oils.

2. MATERIALS AND METHODS

Matured ackee seeds were sourced from ackee trees found in Ikole-Ekiti, Ikole Local Government Area of Ekiti State, Nigeria, and her neighbouring communities, while African star apple seeds were purchased in a local market in the same area. The seeds, after careful separation from the fleshy mesocarps, were first air-dried in the sun at an average temperature of 29°C for 5 days, and then the shell, dark brown for African star apple and black for the ackee, was cracked mechanically to de-coat the shell and subsequently reveal the inner cotyledons, which were subsequently sun-dried for 72 h. They were oven-dried further at a temperature of 100°C for 24 hours in a laboratory oven. The dried cotyledons were ground into fine particles using an electric blender and stored in airtight containers until needed for the experiments. The powders were distinctly marked for African star apple seeds and ackee seeds. The following apparatuses were used in the study: soxhlet apparatus, pipette, 500 ml beaker, 500 ml measuring cylinder, 1000 ml separating funnel, digital weighing balance, crucible, drying oven, thermometer, measuring spoon, titrating apparatus, density bottle, heating mantle, reflux condenser, and desiccator.

2.1 Extraction of Oil from Ackee and African Star Apple Seeds

The method employed by Betiku *et al.* [23] was used for the extraction. The Soxhlet extractor was the apparatus, and n-Hexane was the solvent used for the oil extraction. 100 g of the powdered Chrysophyllum albidium seeds was wrapped with Whatman filter paper and transferred into the thimble of the Soxhlet extractor. The thimble was carefully fixed on a 1-litre-capacity round-bottomed flask. 700 ml of nhexane (B.P. 40-60 °C) was poured into about two-third of the volume of the flask, then heated at 60°C on a thermostatically controlled heating mantle and allowed to reflux continuously for 6 hours. After the completion of the extraction process, oil separation from the poured chemical was achieved by heating. The solution of oil and n-hexane was heated to 60°C for the n-hexane to evaporate, leaving the oil. The same process was repeated for the extraction of oil from ackee seed powder.

2.1.1 Oil Yield Percentage

The amount of oil that may be extracted from an oil seed is referred to as its yield. It is generally expressed as a percentage of the weight of the oil seed (Equation 2 and 3):

$$\%\text{ yield from the African star apple} = \frac{Y_1 - Y_2}{Y_1} \times 100 \qquad (2)$$

where Y_1 represents the weight of African star apple seed's powder before extraction, and Y_2 represents the weight of the seed's powder after extraction. Likewise;

$$\% \text{ yield from the ackee apple} = \frac{X_1 - X_2}{X_1} \times 100 \quad (3)$$

where X_1 represents the weight of Blighia sapida seed powder before extraction, and X_2 represents the weight of Blighia sapida seed powder after extraction.

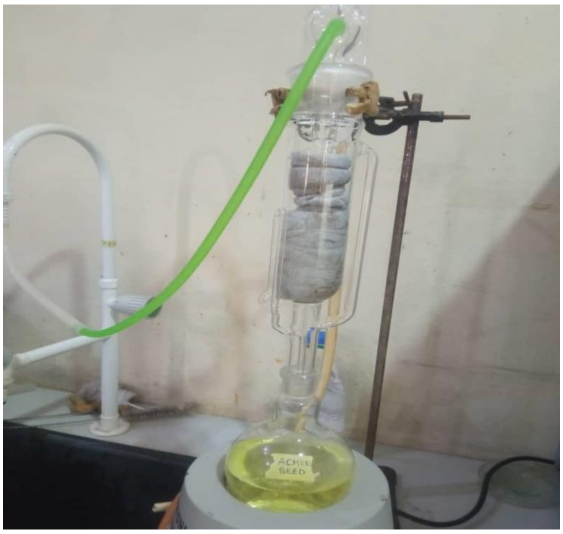

Figure 5: Oil being extracted at the laboratory

2.2 Transesterification Process

The transesterification was carried out using a 250 ml flat-bottomed flask equipped with a condenser placed on a thermostatic magnetic stirrer hot plate set at 60℃. 25 g of each oil sample was weighed into the flask and initially heated to a set temperature of 60℃ on the hot plate. A freshly prepared metabolic solution of 0.75% was added at a molar ratio of 6:1, then the heating and stirring were continuous for six hours of reaction time.

i. **Addition of metabolic solution:** Once the oil had reached the desired temperature and stabilizsd a 0.75% metabolic solution (mixture of methanol and potassium hydroxide) was carefully added to the flask. The solution was added slowly and steadily to prevent any splashing or sudden temperature fluctuations.
ii. **Reaction initiation:** As the metabolic solution comes into contact with the heated oil, the transesterification reaction begins. The base catalyst (KOH) in the solution deprotonates the alcohol molecules, making them more reactive. This allows the alcohol molecules to attack the ester bonds in the triglyceride molecules, leading to the formation of esters and glycerol (Equation 4):

$$\text{Triglyceride + Alcohol (Methanol)} \rightarrow \text{Biodiesel + Glycerol} \quad (4)$$

The actual chemical structure of a triglyceride and the biodiesel molecule can be quite complex, so the reaction is typically represented in a simplified form. For example, the reaction of a generic triglyceride with three fatty acid chains (R1, R2, and R3) is represented as (Eq. 5):

$$3\ \text{R-COOR}' + 3\ CH_3OH \rightarrow 3\ \text{R-COCH}_3 + 3\ \text{R}'\text{OH} \quad (5)$$

where R, R', R1, R2, and R3 are hydrocarbon chains (fatty acid chains); R-COOR' represents the fatty acid chains attached to a glycerol backbone in the triglyceride; R-$COCH_3$ represents the fatty acid chains attached to a methyl (CH_3) group in the biodiesel molecule; R'OH represents the alcohol chains after they have been used in the transesterification process; and CH_3OH represents Methanol.

iii. **Stirring and heating**: The magnetic stirrer maintains consistent mixing of the reaction mixture, ensuring that the reagents are thoroughly mixed and promoting contact between the oil and the alcohol. Stirring is crucial for the even distribution of the catalyst and heat, which in turn enhances the efficiency of the reaction. The heating of the mixture was maintained below the boiling point of the methanol. Methanol has a boiling point of 148°F or 64.7°C. This is because heating at a temperature above could lead to a violent eruption when the reactants are mixed.

iv. **Reaction progress**: The reaction was allowed to continue with stirring and heating for a total of six hours. This extended reaction time was necessary for the completion of the transesterification process. During this time, esters and glycerol were formed as the triglycerides were converted into biodiesel.

v. **Phase separation**: The mixture naturally separated into two distinct layers due to the differing densities of the components. The biodiesel, being lighter, floated on top, while the glycerol and other heavier components settled at the bottom.

vi. **Glycerol removal**: The glycerol and other heavier layers were carefully decanted. Glycerol is a valuable by-product that can be used in various applications, but for the purpose of biodiesel production, it needs to be separated from the biodiesel.

vii. **Washing and drying**: The separated biodiesel layer was washed with water to remove any remaining impurities, residual catalyst, and alcohol. The washed biodiesel was later dried to remove any remaining water, using anhydrous sodium sulphate as a drying agent.

viii. **Final product**: The resulting biodiesel is a mixture of fatty acid esters that can be used as a renewable fuel source in diesel engines. It has properties similar to conventional diesel fuel but with lower emissions of certain pollutants.

2.3 Physiochemical Properties of Mixed Ackee Seeds Oil and African Star Apple Seeds Oil

The physiochemical properties of the Ackee seed oil and African star apple seed oil mixes were carried out for the following mixing ratios: 80 and 20, 60 and 40, 40 and 60, and 20 and 80 % by weight of the oils, respectively. The oils were analysed for their physicochemical properties using standard methods via: specific gravity (ASTM D4052), acid value (ASTM D8045), saponification value (ISO), iodine value (ISO), and the free fatty acid value of the oil was determined using the Association of Official Analytical Chemists' method [24].

2.3.1 Specific gravity value process

Specific gravity is the ratio of the density of a substance to that of a standard or given material or substance, often a liquid. A capillary stopper relative specific gravity bottle, which is pyknometer bottle of 50 ml capacity, was used in determining the specific gravity of the oil mixes. Eq. 6 gives the specific gravity at 30°C:

$$\frac{B-E}{D-E} \tag{6}$$

where B is the weight in grams of the specific gravity bottle with the mix sample ratio at 30°C; E is the weight in grams of the empty specific gravity bottle; and D is the weight in gram of the specific gravity bottle with distilled water at 30°C.

2.3.2 Acid value

The acid value of the oil was determined using the Association of Official Analytical Chemists' method [24]. The free fatty acid value of the oil was multiplied by a constant of 2 to determine the acid value of the oil (Equation 7):

$$A_V = 2\,(\%\ FFA) \tag{7}$$

2.3.3 Iodine and SAP values

The iodine and SAP values were determined by titration using the ISO method [25].

2.3.4 Free fatty acid (FFA)

The free fatty acid value of the oil mixes was determined using the Association of Official Analytical Chemists' method as described by [24]. One gram of the oil mix was placed in a 250 cm^3 conical flask and warmed. 25 ml of methanol was added while stirring thoroughly, and 2 drops of phenolphthalein indicator and a drop of 0.14 M NaOH solution were also added. The contents were titrated with a 0.14M NaOH solution until a light pink colour that persisted for one minute appeared and the volume at this point was recorded. At the end of the titrations, the free fatty acid (FFA) was calculated from (Equation 8). The procedure was repeated twice.

$$FFA = \frac{T_v \times M \times 28.2}{W_o} \tag{8}$$

where *Tv* is the titre volume, W_o is the weight of oil in g, and M is the morality of the base used (NaOH).

2.4 Fuel Properties of Ackee Seeds Biodiesel

The characteristic analysis of the ackee seed oil biofuel involves the determination of the pour point, cloud point, fire point, flash point, and boiling point. The standard test method (ASTM D975-97) was used, and the temperature at which these properties occurred was recorded.

3. RESULTS AND DISCUSSION

3.1 Characterization Results of the Oils Mixture and their Biodiesels

The results of the assessment of the physicochemical properties of ackee apple (Blighiasapida) and African star apple (Chrysophyllum albidum) seeds' oils are shown in Table 4. The values of the assessment conducted twice for the oil mixtures from the two seeds are presented in Table 5, while their computed average values are shown in Table 6. Table 7 shows the result of fuel properties of Ackee and African star apple seeds' oils Biodiesel while the result of the mixtures of their biodiesel are presented in Table 8.

Table 4: Physicochemical properties of Ackee and African star apple seeds' oils

Sample ratio	Specific gravity	Relative index	Acidic value (mg KOH/g)	Saponification value (mg KOH/g)	Iodine value (mg/100g)	Free fatty acid (%)
Pure Ackee seed oil	0.91	1.42	2.12	195.74	38.36	1.06
Pure African Star Apple seed oil	0.89	1.56	4.2	227.68	52.49	2.105

Table 5: Physicochemical properties of the oil mixtures

Sample ratio	Specific gravity	Relative index	Acidic value (mg KOH/g)	Saponification value (mg KOH/g)	Iodine value (mg/100g)	Free fatty acid (%)
20:80	0.8968	1.54	5.06	222.240	49.54	2.53
	0.8944	1.53	4.98	220.288	49.78	2.49
40:60	0.8996	1.51	5.88	215.880	46.78	2.94
	0.8988	1.50	5.89	213.890	46.89	2.89
60:40	0.9024	1.48	6.7	209.520	44.03	3.35
	0.9032	1.47	6.58	207.488	43.99	3.29
80:20	0.9052	1.46	7.52	203.160	41.27	3.76
	0.9076	1.44	7.38	201.084	41.09	3.69

Table 6: Average values of the physicochemical properties of oil mixtures

Sample ratio	Specific gravity	Relative index	Acidic value (mg KOH/g)	Saponification value (mg KOH/g)	Iodine value (mg/100g)	Free fatty acid (%)
20:80	0.8956	1.54	5.05	221.264	49.66	2.51
40:60	0.8992	1.51	5.89	214.885	46.80	2.91
60:40	0.9028	1.48	6.64	208.504	44.00	3.30
80:20	0.9064	1.45	7.45	202.122	41.18	3.73

Table 7: Fuel properties of Ackee and African star apple seeds' oils Biodiesel

Sample Ratio	Flash Point ℃	Boiling Point ℃	Fire Point ℃	Cloud Point ℃	Pour point ℃
Pure Ackee seed oil	116.00	56.00	173.00	2.00	4.00
Pure African Star Apple seed oil	132	44	195	2.5	0.70

Table 8: Fuel properties of the mixtures Biodiesel

Sample Ratio	Flash Point ℃	Boiling Point ℃	Fire Point ℃	Cloud Point ℃	Pour point ℃
20:80	144	51	209	2.8	1
40:60	139	47	201	2.6	1.4
60:40	127	64	187	2.6	12.2
80:20	121	62	184	2.4	12.6

It can be seen from table 4 that the Free Fatty Acid and Acid values increase linearly as the percentage of Ackee seed oil in the mixture increases with decrease in the quantity of African star Apple seed oil. Both Free Fatty Acid and the Acid Value have their maximum value of 3.73% and 7.45(mg KOH/g) at mixture of Ackee apple and African star apple seeds' oils of 80 and 20% respectively. Slight increment was observed in the value of specific gravity of the mixture when the values were compared for 20/80% and 80/20% mixing ratios for the two seeds' oil respectively. The assessment however shows a decline in the values of the relative index with lowest value recorded at 80% Ackee to 20% African Star Apple. The analysis shows that the iodine value decreases with increase in the quantity of Ackee seed oil in the mixture, while the saponification values decrease with increase in the quantity of Ackee seed oil in the mixture. Though, all the saponification values recorded for the mixtures fall within the range of 172 – 199 mgKOH/g applicable for soap making [27], however, this can only be achieved with the value recorded at 20% Ackee to 80% African Star Apple due to low iodine value recorded in all the mixtures. Only the figure (49.66 mg iodine/100g oil) recorded for iodine value at 20% Ackee to 80% African Star Apple was slightly acceptable for soap production. The minimum iodine value suitable for soap making 50 mg/100g oil [17].

The pour point for the mixture of Ackee and African Star Apple biodiesel increases with increase in the quantity of Ackee biodiesel in the mixture. Sharp increase in the value of pour point from 1.4^{o}C to 12.2^{o}C was observed when the quantity of Ackee biodiesel was increased to 60%. Close analysis shows that the pour point increases with 0.4^{o}C in-between data points except between 40 to 60% and 60 to 40% of Ackee and African star Apple respectively. The pour point is the minimum temperature at which movement of the fuel sample can be obtained when the sample container is tilted. All the values obtained for the pour points fall within the limit (-15 – 16^{o}C) for petroleum diesel [14]. The cloud point declines in values with increasing value of the quantity of Ackee biodiesel in the mixture. The flash point and the fire point for the mixture of Ackee and African Star Apple biodiesels decrease with increase in the quantity of Ackee biodiesel in the mixture. Minimum flash point of 121^{o}C and fire point of 184^{o}C were recorded when the mixture of the biodiesel was 80%Ackee and 20% African Star Apple. The changes in the values of boiling point did not follow a regular pattern as the quantity of Ackee biodiesel in the mixture increases. From table 8, it can be seen that the value of boiling point declines generally except in between data point 40 ratio 60 and 60 ratio 40 where an increment was only observed in the boiling point.

4. CONCLUSION

This research work cantered on characterization the oils and biodiesels gotten from Ackee apple seeds and African star apple seeds separately and compared their values with the results of the characterization of their mixtures by varying their mixing ratio. The extraction process was carried out using the method employed by Betiku *et al*.[23]. This process explored the use of Soxhlet extractor as the apparatus and n-Hexane as the solvent. The characterization of the extracted oils mixture and their biodiesels mixture by varying their mixing ratio provided valuable insights into their chemical composition, thermal stability, and potential applications as compared to their unmixed characterization. For all the mixing ratios that were assessed, it was affirmed that the mixture of Ackee seed oil and African star apple seed oil is not useful in the food industry as the oils mixture has a high acid value above 2.0mgKOH/g which makes it unfit for consumption [26]. High acid values were also recorded for Ackee seeds oil and African star apple oil in their unmixed forms. All the mixtures have high saponification values of above 180mgKOH/mg, a favourable property that suggests the mixture hold a promise and valuable ingredient in industries such as the soap making industry.

The analysis of all the mixture of the biodiesels for Cloud Point indicated values above 2°C. Highest cloud point of 2.8^{o}C was recorded when the mixture was set to ratio 20:80 (Ackee seed biodiesel: African star apple seeds biodiesel). This indicates that they cannot withstand colder temperatures without solidifying or forming wax crystals [27]. This characteristic makes the mixture only suitable for applications in regions of the world with high temperatures. Highest fire point of 209^{o}C was recorded when the mixture was set to ratio 20:80(Ackee seed biodiesel: African star apple seeds biodiesel). The high value indicates that the mixtures has good fire resistance and can withstand high temperature at set ratio of 20% Ackee seeds biodiesel and 80% African Star apple seeds biodiesel. The flash point falls within normal range of 100^{0}C-170^{0}C for all biodiesel [28]. Highest flash point of 144^{o}C was also recorded when the mixing ratio was set at 20:80.

This study gave a wider approach that some underutilized seed can be focus on to harness their benefit for the nations, the society and even serve as alternative source of oil for renewable energy. The study characterised the two selected seeds oils and their biodiesels individually and also the mixture of their biodiesels for their chemical and fuel properties. Also of good importance is further study on the determination of the electrical properties of their biodiesels in order to harness their suitability for electrical power applications.

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