Extraction Characterization of Ackee Seed Oil and its Biodiesel Value.

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Received: 29-FEB-2024; Reviewed: 20-MARCH-2024; Accepted: 24-MARCH-2024
https://dx.doi.org/10.4314/fuoyejet.v9i1.17

ORIGINAL RESEARCH

Abstract— The research work is focused on the extraction and characterization of the oil and biodiesel obtained from Achee (*Blighia sapida*) seeds. N-hexane was used as solvent in the extraction of oil from ackee apple seed. The oil was characterized for yield, relative density, free fatty acid value, acid value, iodine value, and saponification value. The values obtained were: oil yield 19.51 %, relative density 0.91 g/cm^3, free fatty acid value 1.06 mgKOH/g oil, acid value 2.12 mgKOH/g oil, iodine value 38.36mg iodine/100 g oil and saponification value 195.74 mg KOH/g oil. The results obtained for characterization for fuel properties of ackee seeds oil biodiesel are: boiling point 56°C, flash point 116°C, cloud point 2°C, pour point 4°C. Though the oil yield was low, the figure obtained for acid value affirmed that the ackee seed oil is not useful in the food industry as the extracted oil has a high acid value which makes it unfit for consumption. The oil however, has advantages over other edible seed oils as it is a waste material that is readily available and sustainable; thus, its use in bioresin production will not compete for its use as edible oil. Furthermore, the values obtained for iodine value and saponification value shows that ackee apple seed oil is suitable for bio-resin production. The study has contributed to the broader understanding of utilizing natural resources efficiently and underscores the need for continued exploration into the diverse applications of agricultural by-products.

Keywords— Bio-resin, characterization, transesterification
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1 INTRODUCTION

Transesterification is the general term used to describe the important class of organic reactions where an ester is transformed into another through interchange of the alkoxymoiety. When the original ester is reacted with an alcohol, the transesterification process is called alcoholysis (Otera, 1993). In this reaction, triacyl glycerides from a variety of feedstock 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). The reaction is an equilibrium reaction and the transformation 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. In order to achieve a high yield of the ester, the alcohol has to be used in excess (Schuchardt *et al.*, 1998).

$$RCOOR' + R''OH \xleftrightarrow{catalyst} RCOOR'' + R'OH \quad (1)$$

This reaction has been extensively used to reduce the viscosity of feedstock or nonedible oil and improve its

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Section C- MECHANICAL/ MECHATRONICS ENGINEERING
Can be cited as:
Olumoroti I.A., Oyelaran A.O.., and Bolaji B.O. (2024).
Extraction Characterization of Achee Seed Oil and its Biodiesel Value., FUOYE Journal of Engineering and Technology (FUOYE**JET), 9(1), 111-116.**
https://dx.doi.org/10.4314/fuoyejet.v9i1.17

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 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 are methyl esters, mainly because methanol is usually the cheapest alcohol (Schuchardt *et al.*, 1998).

The characteristics of oils from different sources depend mainly on their compositions; no oil from a single source can be suitable for all purposes thus the study of their constituents is important. It has been established that fuel properties of Ackee seed oil biodiesel are close to that of diesel fuel and also meet the specification of ASTM standards (Rominiyi et al., 2024). Also, work on many other selected oil seeds (Khan et al., 2024) shows that these seeds have good physicochemical properties that meet ASTM standards. But, biofuel production requires cheap and sustainable feedstock with high oil content (Abel et al, 2020). 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 Akee, and Ackee apple. When the fruit is fully developed and ripped, the pods are bright red and they split open naturally to expose the edible fruit inside. The pod opens to expose three or four cream-colored sections of flesh called arils underneath large, glossy black seeds.

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Fig.1 Ackee Seed

The arils are generally consumed as food in Nigeria while the black seeds are often discarded.

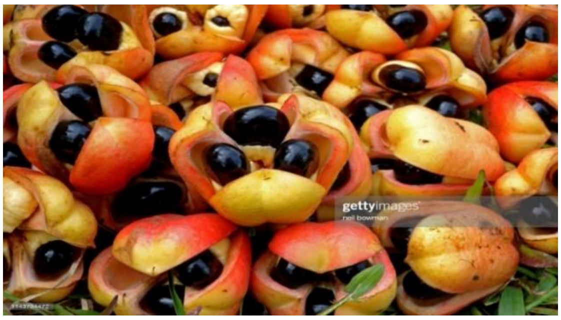

Fig.2 Ackee Fruit

Arils of the immature fruit are not consumed as they contain the toxic compound hypoglycin A (HGA) (Jackson-Malate *et al.,* 2015).

This work focussed on the extraction and characterization of the oil and biodiesel obtained from Ackee (*Blighia sapida*) seeds. This characterization involves the analysis of various physicochemical properties, including density, Flash point, viscosity, acid value, peroxide value, and iodine value (Sayyed Siraj *et al.,* 2013). Therefore, this paper provides basically information about physio— chemical properties of the oil and biodiesel obtained from Ackee seeds.

2. Materials and Methods

2.1 Materials

Matured seeds of Ackee were collected from Ackee trees found in the Ootunja area in Ikole Local Government Area of Ekiti State. The town has abundant Ackee trees as the arils of the fruit were eaten by the locals. The fruits were collected, and the seeds were removed from the pods manually. The extracted seeds were sun-dried for four weeks in order to remove the moisture content. Finally, the dried seeds were crushed into powder using a grinding machine, after which they were blended into powder to allow for easy solvent contact and better oil extraction. 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.

2.2 Oil Extraction

Soxhlet extraction process was used for the oil extraction. Small quantity of dry Ackee seed powder sample was loaded into a thimble. The thimble was then positioned within a distillation flask that holds the specific solvent chosen for the extraction process (N-Hexane). As the solvent approached an overflow level, the solution contained within the thimble holder was drawn up using a siphon mechanism. This action returns the solution to the main distillation flask. This solution effectively transports the extracted components from the thimble-contained sample into the larger solvent volume within the distillation flask. While the extracted components remain in the distillation flask, the solvent is cycled back to saturate the Ackee seed powder sample within the thimble. This cyclic procedure is repeated iteratively until the extraction process achieves completeness.

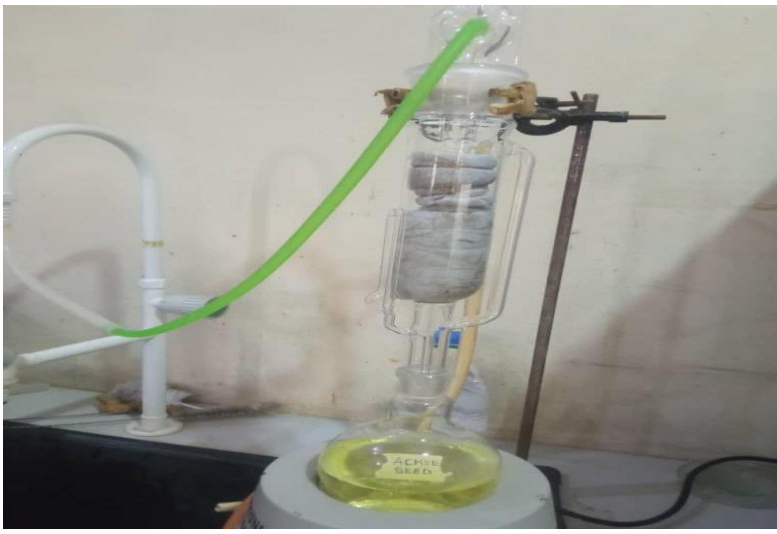

Fig 3 Oil being extracted at the laboratory (Federal university, Oye-Ekiti).

2.2.1 Oil Yield Percentage

$$\frac{(y_1 - y_2)}{y_1} X\, 100 \quad (2)$$

Where, y_1 = weight of Ackee Seed powder before extraction

y_2 = weight of Ackee Seed powder after extraction.

2.3. Transesterification Process

The transesterification process was carried out using a 250ml flat-bottomed flask equipped with a condenser placed on a thermostatic, magnetic stirrer hot plate set at 60°C. 25g of the oil was weighed into the flask and initially heated to the set temperature of 60°C on the hot plate. After the initial heating of the oil to the set temperature of 60°C on the hot plate, the next step was the addition of the freshly prepared metabolic solution at the molar ratio of 6:1. This solution contains a base catalyst, potassium hydroxide (KOH), which helps

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facilitate the transesterification reaction. The molar ratio of 6:1 indicates that for every 6 moles of alcohol (in this case, Methanol), there is 1 mole of the oil (triglyceride).

2.3.1 Addition of Metabolic Solution:

Once the oil had reached the desired temperature and stabilized, 0.75% metabolic solution was carefully added to the flask. The solution was added slowly and steadily to prevent any splashing or sudden temperature fluctuations.

2.3.2 Reaction Initiation:

As the metabolic solution comes into contact with the heated oil, the transesterification reaction begins. The base catalyst 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;

Triglyceride + Alcohol (Methanol) → Biodiesel + Glycerol

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, if we consider a generic triglyceride with three fatty acid chains (R, R'), the reaction could be represented as:

$$3\ R\text{-}COOR' + 3\ CH_3OH \rightarrow 3\ R\text{-}COCH_3 + 3\ R'OH \quad (3)$$

(a) R and R' are hydrocarbon chains (fatty acid chains).
(b) R-COOR' represents the fatty acid chains attached to a glycerol backbone in the triglyceride.
(c) $R\text{-}COCH_3$ represents the fatty acid chains attached to a methyl (CH_3) group in the biodiesel molecule.
(d) R'OH represents the alcohol chains after they have been used in the transesterification process.
(e) CH_3OH represents Methanol.

2.3.3 Stirring and Heating:

The magnetic stirrer maintains consistent mixing of the reaction mixture, ensuring that the reagents are thoroughly mixed and promoting the contact between the oil and the alcohol. Stirring is crucial for 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.

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.

2.3.5 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.

2.3.6 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

2.3.7 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 drying agent like.

2.3.8. 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.

2.4 Determination Physiochemical Properties of the Oil

The oil was analysed for its 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 (AOAC, 1998)'s method.

2.4.1 Acid Value

The Acid value of the oil was determined using the Association of Official Analytical Chemists (AOAC, 1998)'s method as described by (Reyes et al, 1988). The free fatty acid value of the oil was multiplied by a constant (2) to determine the acid value of the oil (Equation 4)

$$A_V = \%\ FFA \times 2 \quad (4)$$

2.4.2 Iodine Value

The iodine value was determined by titration using ISO method as reported Gunstone (Gunstone, 2009).

2.4.3 Saponification Value

The SAP value was also determined by titration using ISO method as reported Gunstone (Gunstone, 2009).

2.4.4 Free Fatty Acid- FFA

The free fatty acid value of the oil was determined using the Association of Official Analytical Chemists (AOAC, 1998)'s method as described by (Reyes *et al.*, 1988).1g of the oil was placed in 250 cm^3 conical flask and warmed. 25ml of methanol was added while stirring thoroughly; 2 drops of Phenolphthalein indicator and a drop of 0.14M NaOH solution were also added. The contents were titrated with 0.14M NaOH solution until a light pink colour which persisted for one minute appeared. Volume at this point was recorded at the end. At the end of the

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titrations, the free fatty acid (FFA) was calculated from (Equation 5). The procedure was repeated twice.

$$FFA = \frac{T_v \, X \, M \, X \, 28.2}{W_o} \quad (5)$$

Where: Tv = Titre volume,
Wo = Weight of oil, g
M = morality of the base (NaOH) used.

2.5 Fuel Properties of Ackee Seeds Biodiesel

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

3.0 RESULTS AND DISCUSSION

3.1. Results

The results of the assessment of physicochemical properties of ackee apple (Blighiasapida) seed oil and its biodiesel were presented in Table 1-3. Table 1 shows the values obtained when the process of characterisation was repeated (doubled) for each property while the averages values of the properties obtained in table 1 were computed and recorded in table 2. Table 3 compared the results of characterisation of Ackee seed oil with other three selected seeds oils while Table 4 shows the fuel properties of ackee seed oil biodiesel.

Table 1: physicochemical characteristics of oil samples extracted from Ackee seeds

SAMPLE	YIELD (%)	SPECIFIC GRAVITY	REFRACTIVE INDEX	ACID VALUE	SAP VALUE (mgKOH/g)	IODINE VALUE (mg/100g)	FFA %
ACKEE	18.92	0.908	1.43	2.15	196.80	38.52	1.07
	20.10	0.912	1.41	2.09	194.68	38.20	1.05

Table 2: physicochemical characteristics of oil samples extracted from Ackee seeds (Average Values)

Properties	Value
Yield (%)	19.51
Specific Gravity	0.91
Refractive Index	1.42
Acid Value (mgKOH/g)	2.12
Sap Value(mgKOH/g)	195.74
Iodine Value	38.36
Free Fat (mgKOH/g oil)	1.06

Table 3: Comparison of physicochemical properties of some selected seed oils with Ackee seed oil (Adeyemi et al., 2021), (Rominiyi et al., 2024)

Parameters	Ackee apple seed	Soya beans	Hemp seed oil	African star apple seeds
Oil Yield %	19.51	21.4	32.3	14.92
Acid Value (MgNaOH/g)	2.12	2.13	2.15	3.23
Free Fatty Acid(MgNaOH/g)	1.06	2.08	1.79	2.07
Iodine Value(Mgiodine/100g oil)	38.36	135.7	163.5	47.63
Saponification Value(Mg/KOH/g oil)	195.74	185.4	198.2	231.32

Table 4: Fuel properties of Ackee Bio-diesel

Parameters	Value (°C)
Flash Point	116.00
Fire Point	173.00
Cloud Point	2.00
Pour Point	4.00
Boiling Point	56.00

3.2 Discussion

The oil yield of 19.51% (Table 2) was achieved through Soxhlet extraction process. Mechanical screw pressing was initially employed for the extraction but with no appreciable result. it is evident that the solvent extraction technique yielded better than the mechanical pressing. Though the oil yield value is low but, considering the fact that the source of the oil is a waste material discarded by consumers and being poisonous, the low yield value may be tolerated since the seeds are abundantly available for use with virtually no cost.

The specific gravity of ackee apple seed oil obtained in this study is 0.91. This value of 0.91 implies that the oil is less dense than water due to the absence of heavy element or hydroxyl groups in it. The value of relative density of any oil gives an idea about its dense nature when compared with that of distilled water (Kakani et al., 2004). Generally, resins should be light in weight so as to contribute to the overall light weight of the composite product and this has to start from the raw material (oil) itself (Sadiq *et al*, 2017). The acid value of (2.12mgKOH/g oil) was obtained in this study. This implies that 2.15 milligrams of potassium hydroxide are needed to neutralize the free acids contained within one gram of the oil. A higher acid value directly corresponds to an elevated concentration of free acids, thereby indicating greater acidity within the oil. In general, a low acid value is considered to be below 0.5 mgKOH/g, while a high acid value is considered to be above 2 mgKOH/g. This shows that Ackee oil is unfit for consumption. The determination iodine value was carried out accordance to Gunstone (Gunstone, 2009). The iodine value of oil extracted from Ackee was found to be 38.52mg/100g. This value indicates that the oil contains a moderate amount of unsaturated fatty acids.The high SAP value of 196.80

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mgKOH/g obtained in this indicates that the oil is suitable for soap making (Gunstone, 2009).

The flash point of a volatile liquid is the lowest temperature at which it can vaporize to form an ignitable mixture in air or the flash point is the temperature at which the fuel becomes a mixture that will ignite when exposed to a spark or flame (Tariq et al., 2012). A low flash point is considered to be below 130 degrees Fahrenheit (54 degrees Celsius), while a high flash point is considered to be above 150 degrees Fahrenheit (66 degrees Celsius).The biodiesel extracted from Ackee has a flash point of 116°C, this indicates that it has a relatively low risk of igniting at normal temperatures and pressures (Anggono et al., 2024). The flash point was determined by the ASTM D93 (Hughes *et al.,* 1996). The cloud point of biodiesel is the temperature at which waxes and other components in the fuel start to crystallize and form a cloud. It is the highest temperature at which the oil begins to solidify (Bertrand et al', 2004). The cloud Point of 2°C recorded in this study indicates that Ackee seeds biodiesel can withstand colder temperatures without solidifying or forming wax crystals, therefore can be used in colder climates without any issues

4. Conclusion

This research successfully explored the extraction and characterization of oil and biodiesel obtained from ackee seeds (*Blighia sapida*). The extraction was done using (N-Hexane) as a solvent for the extraction. This extraction technique was discovered to be more efficient than Mechanical screw pressing which was earlier tried.

The subsequent characterization of the extracted oil provided valuable insights into its chemical composition, thermal stability, and potential applications. It was affirmed that the Ackee seed oil is not useful in the food industry as the extracted oil has a high acid value which makes it unfit for consumption. The presence of bioactive compounds and favourable properties suggests that ackee seed oil holds promise as a valuable ingredient in industries such as the soap making industry.

Ackee biodiesel has a Cloud Point of 2°C which indicates that it can moderately withstand colder temperatures without solidifying or forming wax crystals.The use of ackee apple oil for bioresin production will not only give room for alternative resin that is renewable and sustainable, but will also help to ameliorate some of the challenges associated with total dependence on petrochemical resins thereby hindering the adverse effects of synthetic resins on health, environment and economy.

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