Bima Journal of Science and Technology, Vol. 7 (2) June, 2023 ISSN: 2536-6041

DOI: 10.56892/bima.v7i2.432

PHYSICOCHEMICAL AND NUTRITIONAL CHARACTERIZATION OF SEED OIL FROM *Dacryodes edulis* (AFRICAN PEAR)

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ABSTRACT

Dacryodes Edulis is a plant with multiple applications ranging from its leaves, bark, flowers, root and fruits. In this study, oil from bush African pear *(Dacryodes Edulis)* seed was extracted and characterized. The proximate nutritional composition, physicochemical properties and GCMS analysis of the oil were carried out. The acid value, saponification value, peroxide value and iodine value were found to be 7.59 mgKOH/g, 185.1 mgKOH/g, 2.8 mgKOH/g and 3.96 mgKOH/g respectively. The oil yield was 50.6% while moisture content was 29.0%. The ash content, crude protein, fat content, crude fibre and carbohydrate determined were 2%, 11.9%, 19.6%, 25.5% and 12% respectively. The gas chromatography-mass spectrometry (GCMS) conducted on the oil revealed predominantly the presence of 9, 12-octadecadienoic and linoelaidic acids. Compounds like n-hexadecenoic acid and cyclopentane undecanoic acid were in moderate amount. Ethyl ester, oleic acid and pentadecanoic acid were in minor quantities while prop-1-en-2-yltridecyl ester was in trace amount. The result from the investigation revealed that the oil from bush pear has potential of being used as an industrial raw material and as food additives.

Keywords: Oil, characterization, extraction, solvents, properties

INTRODUCTION

Edible Fats and oils are the third most important macro nutrient required by the body after carbohydrates and proteins (Youdim, 2019). They are rich sources of vitamins and contain two and a half times the energy provided by carbohydrates. In addition, fats and oils also contain essential fatty acids which are not manufactured by the body and as such must be obtained from diets containing fats and oils (Aremu et al. 2015*)*. Oil extraction from oil bearing seeds and mesocarps can be done with the use of non-polar solvents such as hexane, diethyl ether and carbon tetrachloride. The expressed oils usually are composed of fatty acids (triglycerides), some mucilaginous, proteinaceous, pigments, resins and other fat oxidation substances which when left in oil will result in the production of flavours, odours and colours and may reduce the shelf life of the oil. These substances are usually removed during refining processes (degumming, bleaching and deodourization). Vegetable oils are important for both food and non-food industry. They are found in plant tissues such as seeds and pulps (Mariana et al., 2013). They are used in frying, baking and production of mayonnaises, shortenings, margarines, and other valuable products (Falade et al., 2017).

Oil can be grouped into edible and non-edible oil depending on the amount of unsaponified matters and impurities contained therein.

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Edible oil extracted from African Pear seed, bread fruits, cashew nuts, peanuts etc are examples of vegetable oil which are naturally occurring esters of higher fatty acids, and glycerol, and are predominantly triglycerides with traces of mono and diglycerides, sterples, antioxidants, vitamins, saturated and unsaturated free fatty acids, and other minor constituents. They are widely distributed in nature and were first consumed as food. Later, oil was discovered to be used as renewable raw materials for variety of non-food production e.g., soaps, creams, disinfectants, paints, enamels, inks etc. (Zang et al. 2018).

Oil can be extracted from various seeds; however, this work focuses on one seed (African pear seeds). Globally, an estimated 40 million tons of fats and oils are consumed by man annually and the demand is on the increase with the increasing population (Dhiman et al. 2009). As a result, there is shortage in the availability of oils with inflated cost as supplies cannot meet the demands. Therefore, over the years concerted efforts have been made to find alternative sources of oils to augment the existing and as much as possible from non-edible oil sources for non-edible industrial uses and vice versa to reduce the food- non-food clashes of oils ones (Aremu et al., 2015; Ikhuoria and Maliki, 2007).

Over -dependency on conventional vegetable oils such as groundnut oil and soybean oil, as well as their increasing scarcity and processing cost has led to improve interest in extracting oil from non-conventional cheap and available crop seeds like *Dacryodes Edulis.* Apart from this, *Dacryodes Edulis* seeds experience a lot of wastage in its on-season because of poor knowledge of processing. There are no commercially available products made from the seed, its industrial potential has not been harnessed. The fruits are only consumed locally, sold as produce in the local markets and the seeds thrown away after consumption of the mesocarp (Onuegbu et al., 2011). There are two types of *Dacryodes edulis* in Nigeria, they are *D.e.var. edulis* and *D.e.var. parvicarpa.* The two varieties are cultivated in large quantity in South- Eastern Nigeria and other African countries like Cameroon, Sierra Leone, Uganda, Liberia, and Zaire. There are two varieties of *Dacryodes edulis* in Nigeria are the (Isaac *et al.* 2014). This study is focused on the extraction of oil from *Dacryodes Edulis* seed and investigating it proximate nutritional composition, physiochemical characteristics and GCMS composition as a baseline for it quality and application.

MATERIALS AND METHODS

Sample Collection and Preparation

The bush pear seeds were collected from Itam market, Akwa-Ibom, Uyo. South-South, Nigeria. The seeds were washed with water to remove dirts and fungus. The seeds were sun dried for 48h and then dried in an oven at 110°C for 24h. One kilogram (1kg) of dried seeds sample was pulverized into fine particle and sieved with a sieved size of 75µm.

Oil Extraction

The Soxhlet extraction method employed in the extraction African pear pulp oil was adopted from Musa *et al*. (2012). Two hundred and fifty grams (250g) of pulverized seed sample was poured into five different conical flasks of 50g each. One hundred mililitre (100ml) of n-hexane was introduced into each of the five conical flasks containing the ground sample. The mixture of the sample and n-hexane was placed at the centre of the Soxhlet extractor. The extractor was heated and maintain at 79°C for 5 h. The oil was recovered by evaporation and heated in an

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oven at 80°C for 10 minutes to remove any residual n-hexane. The oil obtained was collected in an air-tight container and stored in a refrigerator prior to analysis.

Proximate Nutritional Analysis of *Dacryodes Edulis*

Proximate analysis was carried out according to the procedure of Association of Official Analytical Chemist (A.O.A.C., 1990) for moisture, ash content, crude fibre, fat content, crude protein content and carbohydrate content.

Moisture content

One gram (1.0g) of sample was placed into a petri dish, dried and weighed as W_1. The drying continued until a constant weight was achieved which was taken to be W_2

$W_1 - W_2$ = moisture content 1

Ash content

The ash content is the percentage of inorganic residue remaining after ignition of the filtered and non-filtered oil. An empty crucible was washed, dried and the weight was determined. One gram (1.0g) of sample was transferred into the crucible, placed in a furnace, and allowed to burn for 1 h until it turned to ash. It was then cooled and weighed. The ash content was determined using equation 2.

$$\text{Ash content} = \frac{w1-w2}{1g} \times \frac{100}{1} \quad 2$$

Where W_1 is weight of empty crucible + Sample before drying and W_2 is weight of crucible + Sample after drying.

Fat content

Five grams (5.0g) of sample was wrapped in a filter paper and added into a Soxhlet extractor. The system was swirled eight times to achieve maximum yield of oil. The extractor was disconnected after recycling and a distillation apparatus was set up to separate the solvent from the oil. An empty beaker was weighed and the sample containing oil and traces of the solvent after distillation was transferred into the weighed beaker and placed in an oven for drying. The oil was removed from the oven after three hours and was allowed to cool in a desiccator and weighed with the beaker again.

The fat content of the sample was calculated using equation 3

$$\text{Fat content} = \frac{\text{weight of oil}}{\text{weight of sample}} \times \frac{100}{1} \quad 3$$

Crude fibre

Fifty grams (50g) of Sample was soaked in 200ml of 1.25% H_2SO_4, 100ml of H_2O and heated for 30 minutes in a furnace. The residue was then filtered and washed with hot water. The residue was re-soaked with 200ml of 1.25% NaOH and heated again for another 30 minutes, filtered, dried, and weighed after drying. An empty crucible was then weighed; the residue was transferred into the crucible and burnt to ash, cooled in a desiccator and weighed. The crude fibre was obtained by equation 4.

$$\text{Crude fibre} = \frac{\text{weight of fibre}}{\text{weight of sample}} \times \frac{100}{1} \quad 4$$

Crude protein

One gram (1.0g) of $CuSO_4$ was added into a crucible and heated until the solution digested completely, it was then allowed to solidify for 24 h. Two hundred mililitre (200ml) of distilled water was added to dissolve the solidified sample and allowed to cool in a refrigerator. Sixty mililitre (60ml) of 40% NaOH and two pieces of zinc metal were added to the digested sample. The absorber was titrated with 0.1ml H_2SO_4 using methyl orange indicator. The crude protein was determined by equation 5.

$$\text{Crude protein} = \frac{100 \times Tv \times 0.0014 \times 6.25}{\text{Weight of sample}} \quad 5$$

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Where: 0.0014 is a constant, liberated by 1ml of 0.1N H_2SO_4, 6.25 is protein constant according to Kjedahyl method and Tv = titre value

Carbohydrate content

The percentage carbohydrate content of the oil was obtained as the difference from 100 and the % values of the moisture content, protein, crude fibre, and crude protein.

Physicochemical Analysis of the Oil

The seed oil physicochemical properties such as acid value, iodine value, saponification value, free fatty acid and peroxide value were determined using standard methods (ASTM D 1959-85; ASTM D 1980-85).

Acid value

One gram (1.0g) of oil was poured into a conical flask and 20ml of the lipid solvent was added and shaken well to dissolve the oil. Few drops of the phenolphthalein indicator was added and titrated against KOH. The acid value was calculated using equation 6.

$$\text{Acid value} = \frac{\text{Strength of KOH} \times \text{Equivalent weight of KOH} \times 100}{2\text{Weight of oil} \times 100} \quad 6$$

Iodine Value

Twenty six grams (26g) of iodine and 30g mercuric chloride in 250ml of ethanol each were mixed and made to 1 L. The Hubi's iodine was filled in a 50ml burette, and the initial reading was noted. Five mililitre (5ml) of chloroform was taken in a dry porcelain dish and three drops of Hubi's iodine after adding, and then served as a control for colour composition. Five mililitre (5ml) of chloroform was taken in another dry porcelain dish**.** Zero point five mililtre (0.5ml) of oil was added and dissolved by gently swirling. Hubi's iodine was added slowly from the burette until the colour of iodine matching with the control appeared in the solution. The burette reading was noted. The experiment was repeated and iodine value was determined by equation 7.

$$\text{Iodine value} = \frac{26x}{46\text{mg of iodine}} \quad 7$$

Peroxide value of the oil

Zero point five gram (0.5g) of sample was measured and 25ml of acetic acid and chloroform was added in the ratio of 2:1 and shaken rigorously. The mixture was covered and kept in the dark for one minute. Thirty five mililitre (35m) of distilled water drops of iodine solution and 5ml of starch indicator were added. The colour changes to purple on the addition of starch indicator. It was titrated with 0.02N sodium thiosulphate and peroxide value was evaluated using equation 8.

$$\text{Peroxide value} = \frac{1000(V1-V2)\times N}{\text{Weight of sample}} \quad 8$$

Where: V_1 is the volume of Thiosulphate used to titrate blank. V_2 is the volume of Thiosulphate used to titrate sample.

Saponification value

Zero point five gram (0.5g) of the sample was transferred into a conical flask and 50ml of 0.5N ethanolic petroleum hydroxide was then added. The content was allowed to boil gently for 30 minutes. A reflux condenser was placed on the flask containing the mixture, few drops of phenolphthalein indicator was added to the warm solution and then titrated with 0.5ml HCl to the end point. Until the pink colour of the indicator just disappeared. The same procedure was used for other samples and blank. The saponification (SV) value was calculated by equation 9.

$$Sv = 56.1N\ \frac{(Vo-V1)}{M} \quad 9$$

Where V_o is the volume of the solution used for blank test, V_1 is the volume of the solution used for determination, N is actual normality

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of the HCl used and M is the mass of the sample.

Oil pH

The pH was measured using a pH meter.

Gas Chromatography- Mass Spectrometry

Gas Chromatography/Mass Spectrometry is an analytical method that combines the features of gas chromatography and mass spectrometry to identify different substances within a sample component matrix. GCMS Analysis of the sample was done using gas chromatography (Perkin-Elmer 8500) according to the manufacturer instruction.

RESULTS AND DISCUSSION

Physicochemical Characterization of African Pear Seed Oil

The physical and chemical properties of the seed oil are summarized in Table 1.

Table 1: Physicochemical properties of African pear seed oil

Parameters	Value
Oil yield (%)	50.6
Moisture content (%)	29
Acid value (mgKOH/g)	7.59
Saponification value (mgKOH/g)	166.98
Peroxide value ((mgKOH/g)	2.8
Iodine value ((mgKOH/g)	3.9
pH	6.28

The oil content of the African pear seed was 50.6% and indicating the fraction of the fruit seed that is made up of oil. The oil content is relatively and of economic value. The high oil yield is close to the result obtained for groundnut oil with 50% and cashew nut oil, 49.1% (Akpabio et al. 2011).

The moisture content of the seed was 29%, showing that the seed of African pear has high moisture content and may be prone to spoilage.

The Acid value of the oil was 7.59 mgKOH/g and it indicates the lipid indices of the seed oil. The acid value obtained is in agreement with that of native pear oil, 7.1mgKOH/g (Akpabio et al., 2011). However, the low acid value of African pear seed oil suggests that the oil may be of advantage for paint making and that the oil is edible. The lower the acid value of oil, the few fatty acid it contains which makes it less exposed to the phenomenon of rancidity (Rogers et al . 2010).

Saponification values of the oil was 166.98 mgKOH/g was slightly lower than the FAO/WHO standard of 181.4 mgKOH/g (Adegbe et al. 2016). This shows that the oil is of lower molecular weight and if it is too high, the formation of soap will be more prevalent instead of the desired biodiesel (Folaranmi, 2013). Saponification value of oil serves as important parameter in determining the suitability of oil in soap making.

The peroxide value obtained as presented in Table 1 was 2.8 $mgKOH/g$. The peroxide value indicates whether the oil will be easily susceptible to oxidative rancidity. The lower peroxide value obtained shows that African pear seed oil will not be easily susceptible to rancidity.

The iodine value of the African seed oil determined was 3.9 mgKOH/g). The iodine value of the oil agreed with the standard and hence the oil could be classified as non-dying oil, since their iodine value are less than 100 mgKOH/g (Asuquo, 2008). The low iodine value also shows that the oil contained few unsaturated bonds.

The oil pH was 6.3 and indicating that it is slightly acidic and it is in agreement with the low acid value and the corresponding low free fatty acid contents obtained as presented in Tables 1 and 2 respectively.

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Proximate nutritional parameters

The results of proximate nutritional composition of the oil sample are presented in Table 2.

Table 2: Proximate nutritional composition of African pear seed oil

Parameters	Value (%)
Fat content	19.6
Crude fibre	25.5
Ash content	2.0
Crude protein	11.9
Carbohydrate	12.0
Free fatty acid	7.95

The ash content, crude protein, fat content, crude fibre, carbohydrate and free fatty acid determined were 2%, 11.9%, 19.6%, 25.5%, 12% and 7.95% respectively. The fat content and crude fibre are relatively high indicating that the oil can be used as food. Similarly, the low ash content, crude protein and free fatty acids implies that the oil is suitable for industrial purposes mainly in soap and paint production. The toxicity and the odor of seed oil could be due to high protein content and the ash content of seed oil indicates presence of abrasive solids, soluble metallic soaps, and silica residue in the seed (Nayak and Patel, 2010).

Table 3 summarized the various compounds in the oil sample. 9,12-octadecadienoic acid and linoelaidic acid are predominant in the oil with compositions of 29.71% and 24.99%, respectively. n-hexadecenoic acid is in moderate amount 8.838%. Ethyl ester (2.773%), Undecanoic acid (2.505%), Oleic acid (1.813%), Undec-10-ynoic acid (1.773%), 8-Dodecan-1-ol (1.587%), Pentadecanoic acid (1.504%) and 2-hydrocy-1-(hydroxy methyl) ethyl ester (1.235%) were in minor quantity while 4-ethenyl-7-octenoic acid (0.975%) and Prop-1-en-2-yltridecyl ester (0.215%) were in trace amount. Generally, the GCMS of the oil revealed the presence of ethanol, ethanoic, ester, alkanes, and carboxylic acid.

Table 3: GCMS composition of African pear seed oil

Compounds	Composition (%)
1-ethyl-3,5-dimethyl-o-cymene	0.953
Prop-1-en-2-yltridecyl ester	0.215
Ethyl ester	2.77
n-hexadecenoic acid	8.84
Undecanoic acid	2.51
Tridecanoic acid	5.40
Pentadecanoic acid	1.50
Linoelaidic acid	24.99
9,12-octadecadienoic acid	29.71
Cyclopentane undecanoic acid	4.03
Oleic acid	1.81
Undec-10-ynoic acid	1.77
8-Dodecan-1-ol	1.59
11-(2-cyclopenten-1-yl undecanoic acid	1.28
9-octadecanoic acid	1.24
2-hydrocy-1-(hydroxy methyl) ethyl ester	1.24
Cis-13,16-decasadienoc acid	1.10
4-ethenyl-7-octenoic acid	0.98
1-ethyl-3,5-dimethyl-o-cymene	0.95
Prop-1-en-2-yltridecyl ester	0.215

CONCLUSION

The oil from *Dacryodes Edulis* seed was successfully extracted and characterized for proximate nutritional composition and physicochemical properties. The oil yield (50.6%) was relatively high and the results obtained from the proximate nutritional composition revealed high content of fat content, crude fibre and protein content. The GCMS also depicted the presence alcohols, ethanoic, ester, alkanes, and carboxylic acid. The African pear has potential to improve nutrition, boost food security, foster rural development and support sustainable land care. Generally, the results show that the oil can be used for human consumption as well as industrial purposes.

DOI: 10.56892/bima.v7i2.432

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