



## Extraction and characterization of biomass oils from non-edible sources

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### Abstract

Studies have revealed that the used of edible oils for industrial purposes creates food crises, thus, the need to search for oil from non-edible sources. In this study, oils were extracted via soxhlet extractor using ethanol as solvent from non-edible sources (Bush Mango seed (BMS), African Native Pear Seeds (ANPS) and Orange Peels (OP) and analyzed to ascertain their fatty acid profile using GC-MS and also physicochemical properties (free fatty acid content, kinematic viscosity, specific gravity, iodine value, saponification value and acid value) in order to attest whether there are suitable for industrial applications like biodiesel production. The yields of oils obtained from the biomass were 43.78%, 3.5% and 41.04 for BMS, OP and ANPS respectively. The physicochemical properties of the oils indicate free fatty acid content of 5.54, 3.21 and 5.44 for BMSO, ANPO and OPO respectively, density, viscosity, iodine values, saponification number and specific gravity were comparable to available oils. Fatty acid profile of the oils shows 27.26% and 18.21% of oleic acid in ANPO and BMO respectively and 25.56% of limonene acid in OPO as the fatty acid's components with the highest percentage in the oils. All the oils have good percentage of monounsaturated, polyunsaturated and saturated fatty acid, thus there are suitable for biodiesel, skincare, soap production etc.

**Keywords:** Biomass oil, Solvent extraction, Physicochemical property, Wild plant species, Fatty acid

### Introduction

Oil in broad sense is any non-polar chemical substance that is composed primarily of hydrocarbons which carry's amphiphilic properties. Oils can be flammable, surface active and viscous liquid derived from either mineral or biological sources. When oil is extracted from biological sources (such as plant and animal) it is called lipid, composed of long chain fatty acids with either glycerol or phospholipid backbone (Ahmed, 2023). Oils obtained from biomass have wide applications in industries, namely in food processing, cosmetics, pharmaceuticals, tannery or leather processing, plastics and coatings, friction modifying and transmission fluids for automobiles, in cultural and religious applications etc., implying that the usage of oils are inexhaustible and evolving due to research dynamics.

Due to food application of vegetable oils, edible oils supplies become insufficient or not enough for the process industries, hence, it has become imperative to search for non-edible source in order to reduce food insecurity. Therefore, this report focuses on extracting oils from non-edible biological materials such as Orange peels, Bush mango seeds and Africa native pear seeds. For instance, the orange peel is obtained from the orange fruit as part of its waste, yet essential for the production of peel oil which found uses in food, cosmetics, medicine and energy production (Onwualu, 2015 and Adebisi, 2014) <sup>[1]</sup>.

The bush mango tree bears a wild fruit (it is also called *Irvingia gabonensis*) which grows in the forest of sub-saharan Africa such as Gabon, Ghana, Nigeria and the others. The fruit is fibrous, juicing and bitter taste, and mostly ellipsoidal in shape, greenish in colour when unripe and yellowish when ripe. It encapsulates a stony nut which contains the oil rich seed, often used in the preparation of varieties of soups or thickeners (Ekpe *et al*, 2007) <sup>[12]</sup>. The fibrous pulp of the ripe fruit is eaten raw without being cooked (Kengni *et al*, 2011) <sup>[11]</sup>, while the defatted seed flour of the wild mango is useful as raw material for food

products development and additions (Ainge and Brown, 2001) <sup>[13]</sup>. In some sub-Saharan countries, it is been used to mitigate deforestation problems because of its massive tree size (Leakey *et al*, 2005) <sup>[2]</sup>.

African native pear tree (also called *Dacryodes edulis*), belongs to the family of Burseraceae. It is also found in Sub-saharan Africa like Liberia, Cameroun, Nigeria etc. The tree stands up to 40 m tall with pale grey rough bark that drop resin when cut. The leaves are arranged in pair. The dark blue ellipsoidal fruit is about 4-12cm long with well arrange seeds wrapped in endocarp (Aponjosun *et al*, 2022 and Orwa *et al*, 2009) <sup>[15]</sup>. The pulp can be eaten as raw fruit or can be eaten when roasted or boiled into buttery and creamy texture. The fruit and seed have high nutrient profile for amino acid, lipid, vitamins, minerals and antioxidants which can serve medicinal purposes (Ifeoma, 2012) <sup>[16]</sup>.

Empirical studies on Africa native pear revealed that the pulp contains 48 % oil and that a plantation of it can give 7 - 8 tonnes of oil per hectare (Awono *et al*, 2002) <sup>[17]</sup>. Also, a physicochemical property study of the African native pear seed has revealed as follows: 51 % lipid, 21 % protein, 20 % carbohydrate and 12% crude fiber (Mustapha, 2021) <sup>[6]</sup>. Show more of empirical studies. This research seeks to evaluate and compare the non-edible oil contents of selected fruits of wild origin. The oils obtained from these fruits would also be characterized for physicochemical properties.

### Materials and Method

#### 1. Materials

The main materials used for this study include orange peels, bush mango seeds and Africa native pear seeds (Figures 1 – 3). These materials were collected in polyethylene bags from the local farmers around the University of Port Harcourt, Port Harcourt, Nigeria. Solvent used for the extraction of oils from the fruits and seeds is pure grade ethanol (99 %+ w/w, Geochemical shop). These materials were washed thoroughly with distilled water, sun dried and

further dried in an oven. Thereafter were crushed and sieved into fine particles (or powder) of 500 – 800  $\mu\text{m}$ . They were

subsequently weighed and stored in labeled airtight containers.



Fig 1 Orange Peels



Fig 2: Bush Mango Seed



Fig 3: African Native Pear Fruit with Seed

## 2. Extraction of Oils

For ease of extraction, three batches of extraction were conducted for each biomass material that amounted to 746 g of the biomass powder. They were each wrapped with 0.45  $\mu\text{m}$  Whatman filter paper and inserted into the thimble of a Soxhlet extractor. The thimble was properly fixed onto a 500 ml capacity round-bottom flask. Pure grade ethanol (99%+ w/w) concentration was used for the extraction on a solvent to biomass solid ratio of 4:1. The solvent (ethanol) was poured into the round bottom flask and heated to 60°C using heating mantle (with thermostat) and allowed to reflux continuously for 3 hours. Thereafter, the mixture of the oil and ethanol was poured into a rotary evaporator, and heated to 80°C for 2 mins to recover the oil. The percentage yield of oil from each biomass solid was calculated using the Equation 1

$$\% \text{ Yield of oil} = \frac{W_o}{W_s} \times 100 \quad (1)$$

Where,  $W_o$  = weight of the oil obtained,  $W_s$  = weight of the biomass solid.

## 3. Characterization of Oils

Basic analytical components and physicochemical properties (such as fatty acid profile, iodine value, saponification value, acid number, free fatty acid content, and specific gravity) of the biomass oils were measured as follows:

### 3.1 GC-MS analysis

A GC-MS analyzer (model GC-7890A/MSD5975C) is a double function analyzer which was used for determining the range of fatty acid content in the biomass oil samples. In the GC section, the helium carrier gas carried the atomized oil sample through the column to cause interaction in the analyzer, resulting in the separation or elution of components in the oil samples. The eluted oil components from the GC were ionized in the MS section, and separated on the basis of mass-charge ratio. An ion detector was used to identify the different range of fatty acids present in the samples.

### 3.2 Iodine Value

0.2 g of a biomass oil sample was dissolved in 15 ml of carbon tetrachloride in a 100 ml stoppered conical flask. Thereafter, 25ml of iodine solution (or Wiji's solution) was then added into the flask amid vigorous agitation, and was then allowed to rest for 2h in a dark room temperature condition. Furthermore, 20ml of potassium iodide solution, KI (10%w/w) was added, while the mixture was titrated with 0.2N sodium thiosulphate,  $\text{Na}_2\text{S}_2\text{O}_3$  using starch as indicator. A blank sample determination was carried out using 10 ml of carbon tetrachloride,  $\text{CCl}_4$  and the iodine value was calculated as:

$$\text{Iodine value} = \frac{12.69 * N * (V_2 - V_1)}{W_{\text{sample}}} \quad (2)$$

Where,  $W_{\text{sample}}$ , weight of the biomass oil sample,  $V_1$ , volume of sodium thiosulphate used in test,  $V_2$ , volume of thiosulphate used in the blank, and  $N$  is normality of sodium thiosulphate.

### 3.3 Saponification Value

1g of oil sample was dissolved in 20 ml of ethanolic potassium hydroxide solution contained in a conical flask. The mixture was then heated in boiling water for two minutes amid frequent agitation. 1ml of Phenolphthalein indicator was added and the resulting hot soap solution was titrated with 0.5N  $H_2SO_4$ . A blank sample determination was carried out under the same condition, and while the saponification value was calculated as follows,

$$\text{Saponification value} = \frac{(V_{\text{blank}} - V_{\text{sample}}) * N * 56.1}{W_{\text{sample}}} \quad (3)$$

Where  $V_{\text{blank}}$ , volume of  $H_2SO_4$  used for the blank,  $V_{\text{sample}}$ , volume of  $H_2SO_4$  used for biomass oil sample,  $W_{\text{sample}}$ , weight of sample,  $N$  is normality of  $H_2SO_4$  solution.

### Acid number and Free fatty acid content

5 g of biomass oil sample was mixed with 50 ml of ethanol solvent in a 250 ml conical flask, while 2 drops of phenolphthalein was added to the mix. The mixture was titrated against 0.1N Potassium hydroxide, KOH solution, amidst frequent agitation, resulting in a pink coloration of mixture which persisted for fifteen seconds. The acid value was the calculated using Equation 4 as,

$$\text{Acid number (mg KOH / g)} = \frac{[\text{Titre value} \times \text{Normality (KOH)} \times 56.11]}{\text{Wt of biomass oil sample}}$$

The free fatty acid value can be obtained as follows using equation 5:

$$\text{Free fatty acid (mg KOH / g)} = \frac{\text{Acid number (mg KOH / g)}}{1.99}$$

### 3.4 Specific gravity of oil

A clean 50 ml specific gravity bottle was weighed ( $W_0$ ). The bottle was filled to the brim with water and was then stoppered. The bottle was carefully wiped off and reweighed ( $W_1$ ). The same process was repeated for the biomass oil samples which weighed ( $W_2$ ). The specific gravity of the biomass oil samples was then calculated using Equation 6 as,

$$\text{Specific gravity of the sample} = \frac{W_2 - W_0}{W_1 - W_0} \quad (6)$$

### Dynamic and Kinematic Viscosity

Samples of biomass oils obtained from ethanol extraction process were subjected to viscosity measurement using Ostwald glass viscometer at 40 °C. The efflux times for the biomass oil samples and that of distilled water were measured using the Ostwald viscometer, while the actual dynamic viscosity ( $\mu$ ) was calculated using Equation 6,

$$\mu(\text{oil}) = \mu(\text{water}) \times \frac{t(\text{oil})}{t(\text{water})} \times \frac{\rho(\text{oil})}{\rho(\text{water})} \quad (6)$$

The kinematic viscosity ( $\nu$ ) for the biomass oil samples were also calculated at the same temperature condition using Equation 7,

$$\nu(\text{oil}) = \frac{\mu(\text{oil})}{\rho(\text{oil})} \quad (7)$$

## Discussion of Results

### 1. Yield of the biomass oils

The yield of the biomass oil is a measure of the amount of actual quantity of oil separated from the biomass solid. For this study, method of soxhlet extraction was used, and the quantitative values of yield as obtained are presented in Table 1. Therefore, the average value of yield for the different biomass oil samples are as follows: orange peel, 3.5 %; bush mango seed, 43.78 % and African native pear seed, 41.01 %. These results are strong indications that the bush mango seed and African native pear seed are richer in non-edible oils compared to that of orange peels. However, the studies of Olorunshola *et al*, (2023) [18] and Rezzoug and Louka, (2009) [4] on the yield of orange peel oil extraction using steam distillation showed 1.27 % and 2.01 % respectively, while the study Adebisi (2014) [1] who used soxhlet extraction as well for the same orange peels showed 1.09 %. Although the orange peel is characteristically lean in essential non-edible oils content, but this present study as shown that soxhlet extraction using ethanol as solvent is a more suitable method for separating oil from orange peels.

The yield of oil from African native pear seed of 41.04% from this present study is seen to be lower compared to the study of Ogunsuyi (2015) [5] which had 59 % oil yield, and higher than that of Mustapha (2021) [6] which had 13.34 %. The reason for this variation in oil yield can be adduced to the nature of solvent used. N-hexane is non-polar solvent, and tend extract more oil than the ethanol which is polar solvent. However, the drawbacks on the suitability of n-hexane for oil extraction lies in the nature of n-hexane in terms of its high values of flammability, volatility, toxicity and negative environmental impact (Santos *et al*, 2021 and Stevanato *et al*, 2020) [7, 8].

From this present study, the bush mango seed oil extraction yielded 43.78% using soxhlet-ethanol extraction. This result is observed to be lower than the oil yield of 64.28% obtained from the study of Orhevba *et al*, (2013) [19] which used the soxhlet-hexane method. In the same vein, Womeni *et al*, (2008) [20] reported oil yield of 34.55% using mechanical press. Therefore, is a solvent extraction is a more efficient method than mechanical press, yet, not undermining the fact that ethanol is a more appropriate solvent for bush mango seed extraction.

**Table 1:** Oil yields from the biomass sources used

S/N	Source	Average oil yield (%)
1	Bush Mango seed	43.78
2	Orange peel	3.5
3	African Native Pear seed	41.04

### 2. Characteristics of the Oils

#### a. Fatty acid composition

Basic physicochemical characteristics considered for the extracted biomass oils include fatty acid profile of oil, specific gravity, kinematic viscosity, saponification value, iodine value, acid number and free fatty acid content. The results obtained are presented Tables 2 and 3. The results of the fatty acid profiles for the biomass oil samples are presented in Table 2. In it, three oil samples were characterized for fatty acid profile namely orange-peel oil

(OPO), bush mango seed oil (BMSO), and Africa native pear seed oil (ANPSO). The GC-MS analyzer was used for the analysis, by indicating the type and percentages of the different fatty acids present in the samples.

The results showed that African native pear seed oil presented peak areas for major fatty acid acids to include 27.26% Oleic acid, 9.94% Pentadecyclic acid, 7.84% Margaric acid, 5.74% Tridecyclic acid, 5.63% Myristolenic acid and 5.43 5 Stearic acid. These results deviated slightly from the studies of Ogunsuyi, (2015) <sup>[5]</sup> and Isong *et al* (2020) <sup>[21]</sup>. This inconsistency in chemical composition may be driven by the type of specie of the African native pear used for the study. Typical species of the Africa native pear include *Dacryodes edulis*, *Dacryodes Rostrata*, *Decryode Bucttner*, *Dacryode Klaneara*, *Dacryode Hexandra* etc.

Peak areas reported for the orange peel oil in the GC-MS computer integrator include 25.56% limonene acid, 6.63 % Capric acid, 6.51 % Arachidonic acid, 5.41 % Oleic acid, 4.89 % Arachidic acid, 4.17 % Pelargonic acid etc. While that for bush mango seed oil showed the following peak areas for fatty acids: 18.21 % of Oleic acid, 9.53 % Pelargonic acid, 9.46 % Arachidic acid, 9.49 % Myristic acid, 7.89 % Lauric acid etc. Thus, results obtained for BMSO corroborated with the studies of Leakey *et al.* (2005) <sup>[2]</sup> and Ogunsina *et al.* (2012) <sup>[3]</sup> because, similar compositions of fatty acids were reported with the exception of Lauric and Myristic acids. Evident deviations in fatty acid compositions in the oil samples may be due to differences in environmental or solvent conditions.

**Table 2:** Fatty acid composition of oils

Fatty acid	Peak area (%)		
	ANPSO	BMSO	OPO
limonene	-	-	25.56
Caprylic acid	1.75	3.67	2.54
Pelargonic acid	0.49	9.53	4.17
Capric acid	4.52	7.28	6.63
Undecylic	2.84	6.44	1.73
Undecylenic acid	1.30	4.63	2.86
Lauric acid	2.51	7.89	0.42
Tridecyclic acid	5.74	4.34	3.19
Tridecylenic acid	2.41	3.26	1.53
Myristic acid	3.11	9.49	2.74
Myristolenic acid	5.63	5.41	3.53
Pentadecyclic acid	9.94	3.75	2.81
Palmitic acid	0.21	0.64	0.64
Palmiticoleic acid	0.38	0.72	1.33
Margaric acid	7.84	3.48	0.92
Margarenic acid	0.72	2.51	2.60
Stearic acid	5.43	4.34	1.98
Oleic acid	27.26	18.21	5.41
Nonadecylic acid	2.84	0.89	1.52
Arachidic acid	4.43	9.46	4.89
Paullinic acid	2.38	2.52	2.62
Eicosadienoic acid	1.22	1.85	3.37
Arachidonic acid	3.37	2.42	6.51
Benhenic acid	4.28	1.89	1.85
Erucic acid	1.79	0.51	1.57
Adrenic acid	1.36	0.48	0.93
Eicosapentaenoic acid	2.51	2.17	0.77
Docosahexaenoic acid	1.68	3.53	1.82
Tricosylic acid	1.42	2.74	0.61
Lignoceric acid	0.50	5.42	1.92
Nervonic acid	0.34	3.71	2.42

ANPSO = African native pear seed oil, BMSO = Bush mango seed oil and OPO = orange peel oil

### b. Physicochemical properties of the Oils

Physicochemical properties of interest for the oils include iodine value, saponification value, acid number, specific gravity, FFA and kinematic viscosity, where Iodine value determines the degree of unsaturation in the oils. Saponification value indicates the number of saponifiable unit per weight of oil. A high saponification value shows that the oil contains high proportion of low molecular weight fatty acid and good percentage of monounsaturated triglyceride which is good for biodiesel production. It also helps in determination of average molecular weight of the oil and to check if the oil is adulterated. Acid value is the number of milligrams of KOH required to neutralize the free fatty acid present in one gram of lipid. It helps to determine the quality of oil. Free fatty acid value is directly related to acid number. Low free fatty acid value indicates good quality of oil, while density and viscosity of the oil determines the float and flow properties of the oils.

Table 3 showed the properties of orange peel oil samples as follows: specific gravity, 0.834, which is close to 0.88 value reported by Etta-Francis *et al.*, (2022) <sup>[22]</sup>, the little deviation maybe due to environmental factors like soil components of where it was cultivated. Iodine value of 81.41g-I<sub>2</sub>/100g for orange peel oil result is relatively close to the 88.4gI<sub>2</sub>/100g reported by Seleim, (2008). Saponification value of 152.86mgKOH/g for orange peel oil is also in line with 145 mgKOH/g presented by Etta-Francis *et al.*, (2022) <sup>[22]</sup>. Acid value of 10.87 mg KOH/g for orange peel oil sample was slightly higher than 6.96 mg KOH/g reported by Etta-Francis *et al.*, (2022) <sup>[22]</sup>. Kinematic viscosity of 41.73 mm/s<sup>2</sup> at 40°C for orange peel oil is also in line with many reported values. Thus, orange peel oil has been used by Olorunshola *et al.*, (2023) <sup>[18]</sup> to produce biodiesel with NaOH as catalyst. African native Pear oil showed iodine value of 73.68gI/100g, saponification value of 174.97 mgKOH/g, specific gravity of 0.929, and kinematic viscosity of 46.21mm /s<sup>2</sup> at 400C, iodine value of 86.53gI/100 g, acid value of 6.42mgKOH/g and free fatty acid value of 3.21mgKOH/g. These results are are similar to the results reported by Mustapha, (2021) <sup>[6]</sup> which also opined that it can be used for biodiesel production.

Also, bush mango seed oil showed kinematic viscosity of 58.14mm/s<sup>2</sup> at 40°C, specific gravity of 0.947, iodine value of 86.53, saponification value of 186.17mgKOH/g, acid value of 11.74mgKOH/g and free fatty acid value of 5.83mgKOH/g. Results reported by Bello *et al* (2011) <sup>[9]</sup> showed corroborations with the present study as shown: specific gravity of 0.93 and kinematic viscosity of 45mm/s<sup>2</sup> at 60°C, while Ogunsina *et al.*, (2012) <sup>[3]</sup> reported free fatty acid value of 2.72 mgKOH/g and saponification value of 256 mgKOH/g. however, evident deviations from literature results are not significant. Therefore, bush mango seed oil is suitable for biodiesel production.

**Table 3:** Physicochemical properties of the extracted oils

Sample	Bush Mango Seeds Extract	African Native Pear Seeds Extract	Orange Peels Extract
Specific gravity	0.97	0.929	0.834
Kinematic Viscosity @ 40°C (mm/s <sup>2</sup> )	58.14	46.21	41.73
Iodine Value (g I <sub>2</sub> /100g)	86.53	73.68	81.42

Saponification Value (mg KOH/g)	186.17	174.97	152.86
Acid Value (mg KOH/g)	11.74	6.42	10.87
FFA (mgKOH/g)	5.54	3.21	5.44

## Conclusion

From the composition of the oils, there are evident that the fatty acids profiles, specific gravity, saponification value, iodine value are adequate and comparable to oils used for biodiesel production. Also, the kinematic viscosities are high enough to encourage flow. The free fatty acids are relatively permissible which in turn will minimize the cost of pretreatment. The composition of the three oils samples shows that there are suitable for biodiesel production.

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