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Characterization and fatty acid profile analysis of oil extracted from unexploited seed of African star apple (*Udara*)

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Abstract – This study sought to characterize the phyto-oil extracted from an unexploited seed of African star apple (*Udara*) using soxhlet extraction method, normal hexane was used as the solvent at 67 °C for 4 h. The percentage oil yield was 23.8%. The extracted oil was liquid at room temperature, pleasant sweet smell with honey-like colour. The oil physicochemical properties such as acid value, peroxide value and saponification value were 17.41 ± 0.43 mg/KOH/g, 57.74 ± 2.77 meq/kg⁻¹ and 236.341 ± 6.80 mg/KOH/g, respectively. Also, free fatty acid of 8.75% and iodine value of 29 ± 0.16 mg/100g were obtained. The identified fatty acids present included n-hexadecanoic acid (7.55%), 13-hexyloxacyclic-dec-10-en-2-one (1.19%), oleic acids (30.21%), octadecanoic acid (5.28%), hexadecanoic acid (2.37%), undecylenic acid (40.33%), 9-octadecanal (7.09%), and 9, 17-octadecadienal (5.98%). The properties of oil extracted revealed that the seed is a good source of oil which could be employed for industrial purposes.

Keywords: African star apple / fatty acid analysis / GC-MS / phyto-oil / unexploited seed

Résumé – Caractérisation et analyse du profil en acides gras de l'huile extraite des graines non exploitées de l'African star apple (*Udara*). Cette étude visait à caractériser l'huile extraite d'une graine non exploitée d'un arbre appelé *African star apple* (*Udara*), par la méthode d'extraction au Soxhlet ; de l'hexane normal a été utilisé comme solvant à 67 °C pendant 4 h. Le pourcentage de rendement en huile était de 23,8 %. L'huile extraite était liquide à température ambiante, de couleur miel et dégageait une agréable odeur. Les propriétés physicochimiques de l'huile telles que l'indice d'acide, l'indice de peroxyde et l'indice de saponification étaient respectivement de $17,41 \pm 0,43$ mg/KOH/g, de $57,74 \pm 2,77$ meq/kg⁻¹ et de $236,341 \pm 6,80$ mg/KOH/g. Des acides gras libres à hauteur de 8,75 % et un indice d'iode de $29 \pm 0,16$ mg/100g ont également été mesurés. Les acides gras identifiés présents comprenaient l'acide n-hexadécanoïque (7,55 %), le 13-hexyloxacyclic-déc-10-ène-2-one (1,19 %), les acides oléiques (30,21 %), l'acide octadécanoïque (5,28 %), l'acide hexadécanoïque (2,37 %), acide undécylénique (40,33 %), le 9-octadécanal (7,09 %) et le 9,17-octadécadiénal (5,98 %). Les propriétés de l'huile extraite ont révélé que la graine est une bonne source d'huile pouvant être utilisée à des fins industrielles.

Mots clés : African star apple / analyse des acides gras / GC-MS / huile / semences inexploitées

1 Introduction

Fats and oils (lipids) are one of the important macromolecules of the living organisms. Its importance spans from energy generation, through membrane formation and maintenance to the biosynthesis of other essential compounds in the body. Any kind of changes in lipid metabolism

can result in modification of membrane composition and subsequently in changes in its permeability (Orsavova et al., 2015). They consist of mixtures of organic molecules, which are mainly triacylglycerols, diacylglycerols, monoacylglycerols, free fatty acids and other minor components such as phospholipids, phytosterols, tocopherols and tocotrienols and hydrocarbons (Hamm et al., 2013). They could be classified as saturated (no double bonds), monounsaturated (one double bond), and polyunsaturated fatty acids (multiple double bonds) (Enechi, 2001).

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Plant seeds have been used since antiquity as sources of vegetable oil (Adebayo *et al.*, 2012). Vegetable fats and oils are lipid materials derived from plants, which are solids and liquids at room temperature, respectively (Adebayo *et al.*, 2012). Some major oilseeds as enumerated by Ononogbu (2002) are soybeans, groundnuts, cottonseeds, sunflower, rapeseeds, oil palm, and coconut. These plants have been developed to maximize their oil production capacity, as they are traditional and economic products of most tropical and subtropical countries (Ononogbu, 2002).

They are some unexploited source of phyto-oil, which could be alternative to the conventional plants. One of such plants is African star apple (*Chrysophyllum albidum*). There is dearth of information to any usefulness of the seed of African star apple (*Chrysophyllum albidum*).

African star apple (*Udara* in Igbo) belongs to the family of Sapotaceae which comprises of about 800 species (Ehiagbonare *et al.*, 2008). It is an evergreen tree and can grow up to 40 m high and about 2 m in girth. It has a straight and long fluted bole with small buttress at the base (Adebayo *et al.*, 2012). The fruit when ripe is ovoid to sub-globose, and contain three to five seeds which are not eaten. The seeds are dark brown or blackish, obliquely ellipsoid to obovoid, up to 2.8 cm long and 1.2 cm wide; its coat are hard, bony, shiny and dark brown and when broken reveals white coloured cotyledons (Adebayo *et al.*, 2012).

African star apple (*Chrysophyllum albidum*) is one fruit of great economic value in tropical Africa due to its diverse medicinal and food uses (Adebayo *et al.*, 2012). In recent times, the plant has become a crop of commercial value in Nigeria (Oboh *et al.*, 2009). The seeds are been discarded in indiscriminately after the consumption of the succulent fruit. In this research, oil was extracted from the unexploited seed of African star apple (*Chrysophyllum albidum*), characterized and the fatty acid profiled using gas chromatography-mass spectroscopy. The extraction of oil from this agro waste would lead to the eradication of the waste from the environment, thereby creating a clean environment. Also, the oil produced could be applied in various aspect of industries. Being a non-edible material, it is a good oil feed stock since there would not be any debate as regards the creation of food scarcity due to its usage in oil production. Similarly, the oil could be used in the production of biodiesel because it is rich in fatty acids.

2 Materials and methods

2.1 Apparatus

Gas chromatography-mass spectroscopy (GC-MS-QP2010 plus Shimadzu, Japan), n-hexane and other chemicals were products of Sigma-Aldrich, USA.

2.2 Plant sample collection and preparation

Seed of African star apple (*Chrysophyllum albidum*) were picked from its natural habitat in Ikem village of Nguru community of Nsukka LGA, Enugu state, Nigeria. The seeds were de-shelled and cotyledons were sun dried for twelve days. The dried cotyledon was ground using electric blender.

2.3 Seed oil determination using soxhlet extraction

Milled sample weighing 20.86 g was placed in a thimble before adding the solvent (hexane: chloroform) in a ratio 50:50 ml in the flat bottom flask. The set-up was heated at 67 °C for 4 h. After the extraction processes, the oil residue was exposed to the atmosphere and the solvent allowed to evaporate and oil extracted was quantified.

2.4 Gas chromatography-mass spectrometry analysis

Gas chromatography-mass spectroscopy (GC-MS-QP2010 plus Shimadzu, Japan) system, is a very efficient technique commonly used for the identification and quantification of fatty acids in substances (Fig. 1) The unknown organic compounds in the complex mixture found in the oil were matched with the National Institute of Standards and Technology (NIST) library.

3 Results and discussion

Oil was extracted using normal hexane and chloroform in the ratio 50:50. The percentage oil yield from the seed of African star cherry was 23.80%. The properties of the oil extracted are shown in Table 1. Figures 2 and 3 show the picture of African star apple seed and the ground seed respectively. The oil had a pleasant sweet smell, with honey-like colour and was semi solid at room temperature. Acid value of the oil was 17.41 ± 0.43 mg/KOH/g, peroxide value was 57.74 ± 2.77 meq/kg⁻¹ and 8.75% free fatty acid. Also, iodine value of 29.00 ± 0.16 mg/100g and saponification value of 236.34 ± 6.80 mg/KOH/g were obtained for the oil. The percentage oil yield was low when compared to those reported earlier by researchers on oil seed such as *B. parkii*, (34.0%), *L. lanceolata* (40.0%), *S. setegera* (33.0%) and *S. birrea* (42%) and in agreement with the value *B. sapida* (26.0%) but high when compared to the value of *D. microcarpum* (7.42%) (Kyari, 2008), African star fruit (10.71%) (Adebayo *et al.*, 2012).

The acid value (17.41 ± 0.43 mg/KOH/g) of African star apple (ASA) was high when compared to the value of seed oil extracted from soursop (1.82 mg/KOH/g) (Adepoju *et al.*, 2014). Acid values less than 1% were reported by Kyari (2008). Subsequently, Adebayo *et al.* (2012), reported acid value of 4.50 mg/KOH/g for African star fruit seed oil. Acid value is the measure of percentage content of free fatty acids in a substance, and degree of rancidity (Ononogbu, 2002). It is used as a parameter in determining freshness (Ononogbu, 2002). This implied high content of free fatty acid which could lead to high lipolytic activities, and a reduced shelf life.

Peroxide value (PV) of 57.74 ± 2.77 meq/kg⁻¹ was obtained. Peroxide value is an index of rancidity (Adebayo *et al.*, 2012). The PV was high when compared to that (1.57 meq/kg⁻¹) obtained by Adebayo *et al.* (2012). In the report of Kyari (2008) on six oil seeds, high PV of 77.5, 95.0, 150.0, 135.0 were obtained for *B. parkii*, *L. lanceolata*, *D. microcarpum* and *B. sapida*. Also, a low PV was reported by Adepoju *et al.* (2014) on their work on soursop seed oil. Free fatty acids FFA (8.75%) was obtained in the study. The concentration of FFA in an oil is an important quality

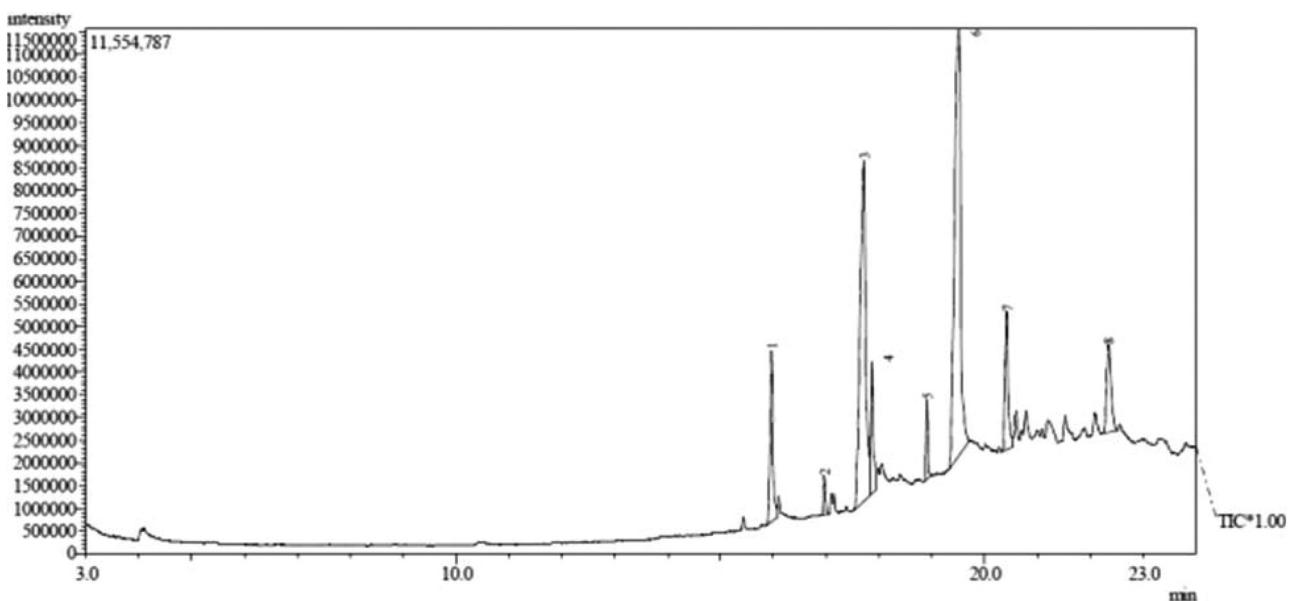


Fig. 1. Chromatogram of African star fruit seed oil.

Table 1. Characterization table of the extracted oil.

Parameters	Properties
Odour	Pleasant
Colour	Honey-like
State at room temp.	Liquid
Acid value	17.41 ± 0.43 mg/KOH/g ⁻¹
Peroxide value	57.74 ± 2.77 meq/kg ⁻¹
% Free fatty acid	8.75%
Iodine value	29.00 ± 0.16 g/100g
Saponification value	236.341 ± 6.80 mg/KOH/g



Fig. 2. African star apple seed (*Chrysophyllum albidum*).



Fig. 3. The ground seed of African star apple.

parameter. Frega *et al.* (1999) reported that FFA added to refined oil shortened their induction time. Similarly, Scarpellini *et al.* (2005) also reported the prooxidant activity of FFA. Hence, it could be opined that high concentration of FFA could lead to short shelf life of oil. Adepoju *et al.* (2014) obtained FFA value of 0.91 for soursop seed oil. Also, *Jatropha* seed oil gave free fatty acid content 2.24%. The high concentration of unsaturated free fatty acids showed that the oil could be prone to autoxidation; and high peroxide value suggest the seed oil

Table 2. Fatty acids profile of the extracted African star fruit seed oil.

Peaks	Name	Formulae	RT	MW	Area (%)
1	n-hexadecanoic acid	C ₁₆ H ₃₂ O ₂	15.974	256	7.55
2	13-hexyloxacyctri-dec 10-en-2-one	C ₁₈ H ₃₂ O ₂	16.966	280	1.19
3	Oleic acid	C ₁₈ H ₃₄ O ₂	17.718	282	30.21
4	Octadecanoic acid	C ₁₈ H ₃₆ O ₂	17.868	284	5.28
5	Hexadecanoic acid	C ₁₉ H ₃₈ O ₂	18.907	330	2.37
6	Undecylenic acid	C ₁₁ H ₂₀ O ₂	19.512	184	40.33
7	9-octadecanal	C ₁₈ H ₃₄ O	20.412	266	7.09
8	9, 17-octadecadienal	C ₁₈ H ₃₂ O	22.345	264	5.98

had high concentration of peroxide and hydro-peroxide which are primary products of autoxidation. The seed oil been rich in unsaturated fatty acids may be beneficial in the formulation of animal feeds and production of biodiesel.

Iodine value (IV) 29 ± 0.16 g/100g was obtained in the work. It is the number of grams of iodine that combines with 100 g of lipids, which shows the degree of unsaturation of the fat or oil (Ononogbu, 2002), the oil is classified as a non-drying oil, and since its iodine value is less than 100. This value is low when compared to 35 mg/100g obtained by Adebayo *et al.* (2012). Also, the value is lower than all the six values obtained by Kyari (2008). The oil of soursop seed oil gave iodine value of 115.30 g/100g oil as reported by Adepoju *et al.* (2014). Also, the seed oil of *C. albidum* was reported to have iodine value of 31.06 g/100g by Osamudiamen and Afolabi (2014). The value was higher when compared to 29 ± 0.16 g/100g obtained in this study.

Saponification value was another physicochemical analysis conducted on African star fruit seed oil. This is the number of milligram of KOH required to neutralize the fatty acids resulting from complex hydrolysis of 1 g of oil or fat (Ononogbu, 2002). Saponification value of 236.341 ± 6.80 mg/KOH/gram was obtained. This value conforms to the values of some vegetable oils used in soap making such as groundnut and coconut. Similarly, it is in agreement with the work of Kyari (2008) on *B. sapidia*, *L. lanceolata*, *S. setegera* and *B. sapidia*, but higher than the values of *D. microcarpum* and *S. birrea* which were 123.3 and 199.3 mg/KOH/g, respectively. Also, the result obtained in this work is higher when compared to the value 199.50 obtained by Adebayo *et al.* (2012) on ASC seed oil. Similarly, the saponification value is in agreement with that reported for soursop seed oil of 235.46 mg/KOH/g (Adepoju *et al.*, 2014).

Gas chromatography-mass spectrometry analysis (GC-MS) has been reported as an important tool for the identification and quantification of fatty acids (Shibula and Velavan, 2015; Okereke *et al.*, 2017). The spectra of the study revealed eight peaks that corresponded to eight fatty acids and other organic substances present in the oil extracted from the seed of African star fruit. The compounds were confirmed by their retention time, percentage area, molecular weight and formulae, respectively. The major fatty acids revealed were undecylenic acid (40.33%), oleic acid (30.21%) and 9-octadecanal (7.09%). Other identified fatty acids present included n-hexadecanoic acid (7.55%), 13-hexyloxacyctri-dec-10-en-2-one (1.19%), octadecanoic acid (5.28%), hexadecanoic acid (2.37%), undecylenic

acid (40.33%), 9-octadecanal (7.09%), and 9, 17-octadecadienal (5.98%) as shown in Table 2. Similarly, Figure 1 showed eight peaks representing different fatty acids, with the concentration of undecylenic acid being highest as depicted by peak 6. The concentration of undecylenic acid detected was higher when compared to the quantity (6.31%) detected in *P. americana* seed oil as reported by Omeje *et al.* (2018). Avram *et al.* (2014) reported the presence oleic acid, linoleic acid and linolenic acid in the seed oil extracted from rapeseed using GC-MS. Gas chromatography analysis of fatty acids present in soursop seed oil revealed 73.42% of unsaturated fatty acid and 26.68% of saturated fatty acid (Adepoju *et al.*, 2014). The physicochemical properties of the African star apple seed oil indicate it possess useful features of industrial oil which could be employed in food and chemical industries.

4 Conclusion

The percentage oil yield was 23.8%. The extracted oil was liquid at room temperature, pleasant sweet smell with honey-like colour. African star fruit seed oil comprised of 85.74% fatty acids. Some of its physicochemical properties were good for industrial processes. African star fruit seed is non edible and a serious environmental waste which could be a source of oil which has a potential properties for applications in industries and other sectors of human endeavours.

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