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Properties of African Breadfruit (*Treculia africana*) Oil Extracted Using Petroleum Ether and N-Hexane in Two Extraction Times

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Abstract
There is high demand for plant oil because of the unsaturation of its fatty acid. African breadfruit is also a seasonal crop which is underutilized when in excess. Again, the properties of oil are affected by the solvent used for the extraction. The above problems necessitated this study to evaluate the effects of petroleum ether and n-hexane on the oil yield and other properties of African breadfruit oil. Two extracting times (4h and 6h) were used to determine their effects on the oil. The properties analyzed were the physicochemical and mineral composition of the oil. Soxhlet extraction technique was used in extracting the oil. The experimental design used was a completely randomized design (CRD). The specific gravity of the oil ranged from 0.836 to 0.901, peroxide value from 4.165 to 5.990 mg/g and the oil yield from 16.61 to 19.31 % with oil extracted using hexane for 6h having the highest ($p<0.05$) oil yield (19.31 %). The oil (hexane-6h) also had the highest ($p<0.05$) value for specific gravity, saponification value, acid value, Calcium, and vitamin A content. The Magnesium content ranged from 19.70 mg/100g to 30.75 mg/100g. Oil extracted for 6 hours using petroleum ether had the highest ($p<0.05$) peroxide value (5.99 mg/g), Magnesium (30.75 mg/100g) and Calcium composition (13.70 mg/100g). There was a significant difference ($p<0.05$) in the extracting time of the oils except for the saponification value of oil extracted using hexane and vitamin A content of oil extracted using petroleum ether. Oil extracted using n-Hexane for six hours was recommended although it will require refining.

Keywords: African breadfruit oil, solvent extraction, Treculia africana, oil yield, extraction time, n-hexane, saponification value, peroxide value

Introduction

African breadfruit (*Treculia africana*) is a monoecious evergreen fruit belonging to the *Moraceae* family and Urticales order (Nwajiobi *et al*., 2019). It is native to tropical countries such as the West Indies, Ghana, Sierra Leone, Jamaica and Nigeria (Emenonye, 2016) where it is found mostly in swampy areas growing up to 40-50 feet high. The plant flowers between October and February and produces large, usually round, greenish-yellow, compound fruits covered with pointed outgrowths with seeds submerged in the spongy pulp of the fruits. The mature tree produces up to 30 fruits yearly, which it bears on the main trunk and big lateral branches (Amujiri *et al*., 2018; Betu *et al*., 2019). Each fruit yields 5-10 kg of edible and nutritious seeds bearing about ten percent edible plant oil. Plant oils are a rich source of essential fatty acids, fat-soluble vitamins and energy yielding about nine kilocalories per gram. They are also important in the development of natural flavor and palatability of a wide range of foods. Most oilseeds are cultivated primarily for their oil and meal and most Nigerian oil crops such as the African breadfruit are not yet economically exploited and as such regarded as under-utilized. The characteristics of any oil from any plant depend on the composition of the source plant and no oil from a single source can be suitable for all purposes (Okolie *et al*., 2012). This implies that the characteristics of oil from African breadfruit differ from oil from groundnut due to difference in composition of the plants, necessitating the search for more edible plant oils. Although various methods used in extraction of oil from seeds exist such as pressing, solvent extraction and supercritical fluid extraction, solvent extraction where the fresh solvent contacts the sample frequently. Also, oil extraction from oilseeds depends on a number of factors namely, the nature of the solvent and oil, the contact time of the sample with the solvent, extraction temperature, particle size and solvent ratio (Ipeghan *et al*., 2019). The objective of this work therefore was to extract oil from the African breadfruit using two different solvents (petroleum ether and n-hexane) at different extracting time (6 h and 4 h) and to determine the properties of the oil extracts.

Materials and Methods

Material Collection

The material used in this project which is mainly the seed of African breadfruit (*Treculia qfricana*) was procured from Ubani market in Abia state. Reagents and equipment used for this study were from the Department of Food Science and Technology Laboratory, Michael Okpara University of Agriculture, Umudike, Abia State.

Preparation of Sample

The method used in the production of African breadfruit flour is the modified method as described by Emenonye and Nwabueze (2016). Mature African breadfruit seeds were sorted to remove stones, spoilt ones and other contaminants which may affect the quality of the product, then washed to remove debris and other contaminants that were not removed during sorting, and parboiled (5 min. at 100 ^{0}C) to soften the endocarp for easy removal. The seeds were then air dried (32 ^{0}C for 15 min.) to reduce the moisture content which may hinder the crushing of the fruits, and crushed (using a manual crusher Corona model GX-200) to enable the easy separation of the endocarp by winnowing. The separated endocarps were sun-dried (RH 80- 92 % for 14 days) to reduce the moisture content. The seeds were then milled into powder using a mechanical grinder (model TT-F115) to hasten the diffusion process of the solvent into solute and to ensure that all the oils present in the solute were completely extracted. The milled seeds were stored at room temperature (20 ^{0}C) until used for oil extraction.

Extraction of Oil from African breadfruit

The extraction of oil from the African breadfruit was carried out using solvent extraction described in AOAC (2010). The oil samples were extracted using two different solvents (petroleum ether and n-hexane) and two different times (4 h and 6 h). Ten grams of African breadfruit flour was weighed using an analytical weighing balance, before putting into a thimble. Two hundred millilitres of petroleum ether were pipetted and both were refluxed in a soxhlet extractor having an extractor column and oil-collecting round bottom flask and extraction was done for 4 h. At the end of the extraction, the solvent and oil mixture were heated and the oil and solvent were recovered in different chambers. The percentage of oil yield was calculated from the weight of the oil and the weight of the sample. The same method was followed for the extraction using petroleum ether for 6h and with N-hexane for another 4 h and 6 h respectively.

Experimental Design

The experimental design used for this experiment is a completely randomized factorial design. Four products were obtained when two solvents and two extracting time was used. Analysis was carried out on the products and the results of the analysis were compared using Analysis of Variance.

Physicochemical Analysis

The determination of specific gravity, melting point, cloud point, moisture content, smoke point, acid value or free fatty acid (FFA), peroxide value, saponification value (SV) and vitamin A of the oil samples was determined using the method described by Onwuka (2018).

Minerals Determination

The calcium and magnesium content of the samples was carried out by versant EDTA complexiometric titration described by AOAC (2010).

Experimental Design and Statistical Analysis

The experimental design used for this experiment was the completely randomized factorial design. Four products were obtained when two solvents and two extracting time was used. Analysis was carried out on the products and the results of the analysis were compared using Analysis of Variance.

Results and Discussion

The first table (Table 1) shows the oil yield of petroleum ether and hexane extraction with ranges of 16.60-18.14% and 17.26-19.31% respectively; their respective 4h extraction time ranking the least. The oil yield of hexane extraction was higher for both extracting times than the oil yield from petroleum ether. This compares with the result obtained by Okereke *et al*. (2014) which had oil yield for sun dried African breadfruit extracted using petroleum ether at 19.05% while that of hexane was 19.56%. This is as a result of extraction temperature which depends on the boiling point of solvent, solubility of oil on the solvent (Keneni *et al*., 2020,) and polarity of the solvent. Increase in temperature increases solubility of lipids by disrupting the cohesive and adhesive interactions between oil molecules and oil matrix molecules respectively (Efthymiopoulos *et al*., 2018). Other factors with influence on oil yield are maturity of the oilseed, extraction method, solvent type, solvent to solid ratio, extraction time, particle size and moisture content of the meal (Keneni *et al*., 2020; Ghazali and Yasin, 2016).

The physical properties of the oil is shown in Table 2 beginning with the specific gravity which ranged from 0.8355 to 0.9009 with Hexane-6h having the highest value while Petrol.ether-4h had the least. The mean value obtained in specific gravity of the Hexane-6h was lower than that of Eze (2012) which may be attributed to temperature difference. The specific gravity of Niger seed oil (0.88) according to Yonnas *et al*. (2019) was compared with the specific gravity of oil obtained from Hexane-4h. The moisture content of any food material is an index of stability and susceptibility to fungal infection and the presence of a sufficient amount of moisture favors microbial growth. Results showed that Hexane-4h had the highest moisture content while Petroleum-6h had the lowest, and the extracting time increased with decreasing moisture content. However, all the values were above the maximum allowable limit for edible oils (0.2 %) provided in the WHO/FAO guideline. Additionally, the saponification value decreased with increasing moisture content while the iodine value increased with increasing moisture content. High moisture content also causes oxidative degradation and rancidity. The smoke point of the oil ranging from 252.5 to 280.8 ^{0}C had Hexane-4h as the highest and Petroleum-6h the least. The smoke point decreased with increasing extracting time while oil samples extracted using petroleum ether had lower smoke point. The smoke point which is the temperature at which heated oil starts to give off smoke varies due to source of oil, extraction method, moisture content of the seed, whether or not the oil is refined, impurities in the oil and the fact that oils break down gradually. In general, the more refined an oil is, the higher its smoke point, because refining removes impurities and free fatty acids that can cause the oil to smoke. The melting point is the temperature at which a lipid transforms from solid to liquid state, and fats and oils, according to Amita and Khatkar (2016), show a melting point range because these are blends of different triacylglycerol (TAGs). The melting point of oil extracted in 4h using petroleum ether (i.e. petrol.ether-4h) had the same melting point as African breadfruit oil extracted using acetone (Nwabueze and Okocha, 2008). The result of the cloud point was 130.0 to 165.5 ^{0}C with Hexane-4h having the highest and Petroleum-6h having the least, and values decreasing with increasing extracting time. The significant difference also implies that the solvent affected the cloud point. The cloud point represents the temperature at which wax or paraffin begins to precipitate from a hydrocarbon solution, that is it begins to separate when chilled to a low temperature and the presence of unsaturated fatty acid reduces the cloud points of oil (Reaume and Ellis, 2013).

The chemical properties of the extracted oils including the saponification value (237 to 259 mg/g) are presented in Table 3. The result showed that the saponification value increased with increasing extracting time. Oil samples extracted using hexane had higher saponification values implying high molecular weight which requires more alkali to neutralize the free fatty acids. The hexane-6h oil extract compares with the value obtained by Eze (2012).

The Saponification Value of the oil from this study was observed to be less than the standard Codex Alimentarius (2013) values of 250-260 mgKOH/g. Saponification value gives information concerning the character of the fatty acid present in the oil, the solubility in water of the soap derived from it and the nature of fatty acid available in the triacylglycerol. Elevated saponification values suggest low fatty acid fraction of the oil (Adejumo *et al*., 2013); it also indicates high proportion of low molecular weight or short chain fatty acids appropriate for soap production. The acid value of the oil extracts decreased with decreasing extracting time. Acid value from Hexane-4h oil extract compares with the acid value obtained by Nwabueze and Okocha (2008). High acid value in hexane-6h shows that the oil will decompose easily in the presence of heat and light. The acid value contents are lower than the one obtained by Eze (2012) on African breadfruit which is 12.9030mg/KOH/g. According to Audu *et al.* (2019), acid content (which is an index of free fatty acid content) shows the degree to which the glycerides in the oil has been decomposed due to enzymatic activity such as lipase action and other physical factors such as light and heat. The high acid value of the oil suggests that the oil is susceptible to lipase activity, other hydrolytic action or oxidation and is not suitable for cooking. High amount of FFA increases oil acidity, seed up degradation and inhibit alkaline catalyzed trans esterification (Al-Hamamre *et al*., 2012). The acid value of the oil can be made fit for consumption by subjecting the oil to refining.

The peroxide value of the oil extracts ranged from 4.17 to 5.99 mg/g with the values increasing with extracting time. The result also showed that oil extracted using petroleum ether at 6h had a higher peroxide value compared to the oil extracted using hexane at 6h, conversely at 4h oil extraction, hexane had a higher peroxide value than its petroleum ether counterpart. The mean peroxide values obtained from the oil samples are lower than the peroxide value obtained by Eze (2012). Peroxide value measures the amount of peroxide formed during the initial stage of oxidative rancidity. While a low peroxide value indicates high levels of antioxidants which decreases liability to deterioration, a high peroxide value reduces the sensory quality of the oil. The result also showed that the mean vitamin A content of oils extracted using hexane is higher than that of oil extracted using petroleum ether. The result also showed that vitamin A content of the oil increased with an increase in extracting time. The mean vitamin values obtained are lower than the one obtained by Nwabueze and Okocha (2008) 27.39, the mean vitamin A values in the oil samples are lower than 20,000 I.U./kg which is the CODEX acceptable limit and therefore need to be fortified. Vitamin A is stable in oil and it helps to prevent avoidable blindness in children.

Mineral composition is presented in Table 4. Calcium was affected by extracting time as well as the solvent type. While the extracting time increased with the increasing calcium content of the oil extracts, the hexane-6h and Petrol.ether-6h oil extracts had significantly ($p<0.05$) higher calcium composition. The result also shows that the calcium content decreased with decreasing extracting time. The result also showed that extracting time and choice of solvent affected the magnesium content of the oil extracts and it increased with increasing extracting time. The average oil extracted using petroleum ether had high magnesium content than oil extracted using hexane. Magnesium is needed to activate numerous enzymes that control the metabolism of carbohydrates, fats and electrolytes.

Conclusion

The result showed that the oils were successfully extracted using petroleum ether and n-hexane at 4 and 6 hours. The oil yield, chemical properties and metal contents of the oil increased with increasing extracting time but moisture content, smoke point, melting point and cloud point decreased with increasing extracting time. All the properties were affected by the solvent used. Oil extracted using petroleum ether had high peroxide value, potassium and magnesium content then compared with that of hexane oil samples while in other parameters, oil samples extracted using hexane have higher values. The use of hexane with 6 h extracting time is recommended for African breadfruit oil extraction. This is because of high oil yield as well as high calcium and vitamin A content. Although it has high saponification and acid value they can be reduced through refining.

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Table 1: Oil yield obtained using petroleum ether and hexane for 6 and 4 hours

Solvent-Time	Oil yield (%)
Petrol.ether-6h	18.14^{b} ± 0.01
Petrol.ether-4h	16.61^{d} ± 0.01
Hexane-6h	19.31^{a} ± 0.01
Hexane-4h	17.26^{c} ± 0.01

Values presented are mean± standard deviation of replicate determination. Means with different superscript in the same column are significantly different at (p>0.005)

Table 2: Physical qualities of oil extracted using petroleum ether and hexane for 6 and 4 hours

Physical Properties	Solvent-Time			
	Petrol.ether-6h	Petrol.ether-4h	Hexane-6h	Hexane-4h
Specific gravity	0.858^{c}±0.00	0.836^{d}±0.00	0.901^{a}±0.00	0.875^{b} ±0.00
Moisture (%)	9.26^{d}±0.01	11.14^{b}±0.03	10.27^{c}±0.02	17.77^{a}±0.02
Smoke point (°C)	252.5^{d}±0.70	265.0^{c}±1.41	271.5^{b}±2.12	280.0^{a}±1.41
Melting point (°C)	23.50^{d} ±0.14	25.10^{c}±0.14	35.07^{b}±0.21	39.81^{a}±0.01
Cloud point (°C)	130.0^{d}±1.41	149.5^{b}±2.12	141.0^{c}±1.41	165.5^{a}±1.41

Values presented are mean± standard deviation of replicate determination. Means with same superscript in a column are not significantly different (p>0.005)

Table 3: Chemical qualities of extracted oil samples

Solvent-Time	Saponification value (mg/g)	Acid value (mg/KOH/g)	Peroxide value (mg/g)	Vitamin A (i/μ)
Petrol.ether-6h	243^{b}±1.41	3.27^{c}±0.01	5.990^{a}±0.04	10.05^{c}±0.10
Petrol.ether-4h	237^{c}±2.12	3.16^{d}±0.03	4.165^{d}±0.02	9.98^{c}±0.01
Hexane-6h	259^{a}±1.41	3.99^{a}±0.01	5.645^{b}±0.02	22.45^{a}±0.21
Hexane-4h	255^{a}±1.41	3.40^{b}±0.03	4.635^{c}±0.02	18.26^{b}±0.01

Values presented are mean± standard deviation of replicate determination. Means with same superscript in a column are not significantly different at (p>0.005)

Table 4: Mineral composition of the oil extracted using petroleum ether and Hexane

Solvent-Time	Calcium (mg/100g)	Magnesium (mg100g)
Petrol.ether-6h	13.70^{a} ± 0.14	30.75^{a} ± 0.07
Petrol.ether-4h	10.35^{b} ± 0.21	24.80^{c} ± 0.14
Hexane-6h	14.01^{a} ± 0.01	29.55^{b} ± 0.21
Hexane-4h	10. 63^{b} ± 0.01	19.70^{d} ± 0.14

Values presented are mean±standard deviation of replicate determination. Means with same superscript in a column are not significantly different at (p>0.005)