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Research Article

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Examination on Characterization of Oil Extracts from *Luffa Cylinderica* and *Hura Creptian* Seeds

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Abstract This research work was carried to examine the composition and the characterization of oil extracted from *Hura crepitan* and *Luffa cylinderica* seeds using n-hexane as solvent. The two varieties of the seed was subjected into Soxhlet extractor mechanism with the aim to extract oils from the two varieties of seeds study. The extraction was carried out at different particle sizes of the given rang 0.425mm,1.00mm, 1.80mm and 2.80 mm and the process was conducted at various times interval of 20min, 30min, 40min, 50min, 60min and 80 min; at various operating condition of temperature ranging from 30°C to 60 °C with an incremental step of 10°C. The separation process was then carried out to separate the oil from the solvent (n-hexane) in the oil-solvent mixture, obtained from the extraction process. The production of the oils were examined and characterized to determine the physicochemical properties and their suitability for industrial applications.

Keywords Examination, characterization, oil extracts, *luffa cylinderica, hura creptian,* seeds

Introduction

The need for diversification of the economy and calls by Nigerian citizens for the National Assembly to pass into law, the local content bill came at the right time, as the Nation's economy continued to dwindle. This desire has necessitated the Federal Government to place a ban on the importation of most foreign products, including vegetable oil. Nigeria is blessed with sources of vegetable oil, which include amongst others, palm, coconut, groundnut, cotton, olive, soya bean seed and conophor seeds [1]. Vegetable oil can be extracted from varieties of crops and animal fats. However, the quantity of yield is an important factor for extractability of oil from its raw materials. The yields of oil from several raw materials have been investigated [2–5].

Vegetable oil is triglycerides of different chain lengths with different degree of saturations. There are many types of triglyceride, with the main division being between saturated and unsaturated. The saturated triglyceride has all its carbon atoms attached to hydrogen atoms. They have a high melting point and are likely to be solid at room temperature while unsaturated triglyceride has double bonds between some of the carbon atoms which reduce the number of places where hydrogen atoms could have been bonded to carbon atoms. This phenomenon makes it have a lower melting point and is likely to be liquid at room temperature [4].

The use of vegetable oil dates back 6000 BC and 4500 BC when oil was extracted from olives in the present day Israel and Palestine [6]. However, the vast applicability of vegetable oil has increased its demand and importance. Thus, further research conducted revealed that the annual production and consumption of oil and fats to 119 million tons and predicted a steady increase of 2-6 million tons per year. This rise, according to him is caused by the increase in population and income derived from vegetable oil, especially in the developing countries [7].

Vegetable oil can be edible or nonedible; consumed directly or indirectly as food ingredients. About 14% of vegetable oil produced was used as raw materials in the industry, 6% as animal feed while the remaining 80% was used domestically for food processing for man ranging from frying oil, salad oil and cooking oil amongst others [8]. The uses of vegetable oil depend on the source of the raw material. In summary, vegetable oil is used in food processing, soap and cream production, margarine, biofuels, especially in biodiesel production, paint for coating and drug production.

It is important to note that oil used for food via heating must have a high flash point and they include soybean, rapeseed, canola, sunflower, safflower, peanut, cottonseed, avocado oil etc. Tropical oils, such as coconut, palm, and rice bran oils, are particularly notable for their high-temperature and thus, suitable for cooking because of their very high flash points [9]. And other research conducted revealed that fats and oils from seeds serve as sources of energy to the body in the absence of carbohydrates, while flavour from most of the vegetable oils have attractive impart to soap [10]. Vegetable oils are extracted chemically by solvent extraction or mechanically by pressing from whole fruits, seeds, kernels and nuts. The solvent extraction, which is the most commonly applied method, uses solvent such as hexane, carbon dioxide and diethyl ether to extract the oil from the crushed solid. In the pressing method, an expeller press or cold press (pressing at low temperatures to prevent oil heating) is used to extract the oil from the raw material. Other auxiliary processes could be carried out to refine the extracted oil such as distillation, degumming, neutralization, or de-acidification, bleaching, filtration dewaxing, deodorizing, and preservative [11].

This research therefore, investigated the yield of vegetable oil from locally sourced plants in the Niger Delta areas of Nigeria, using the solvent extraction method as well as the kinetics and thermodynamics of the extraction process. The domestic and industrial application of vegetable oil will inevitably continue to increase. Thus, there is need to search for raw material locally, to reduce the over dependence on foreign manufactured oils. Nigeria as one of the developing countries, will certainly improve on her economy if the utilization of her abundance potential raw materials for vegetable oil extraction. However, to achieve the required standard set for oil extracted from plants, suitable for domestic and industrial application, certain properties must be ascertained. The increase in demand for oil with reduced health effect, high import duty and call for alternative to fuels that have become problematic to the environment, invokes attention for continuous search for availability of sources of local materials for vegetable oil extraction. Therefore if more raw materials with high oil yields are discovered and exploited, local production of vegetable oil will increase hence, saving our local industries of high cost associated with import duty, foreign exchange and transportation thereby improving the nation's economy [12–14].

The aim of the study is to extract and characterized oil from *Luffa cylindrica* (looffah gourd seeds) and *Hura crepitan (*sand box seeds) obtained wealthy in Niger Delta region using in-hexane as solvent. The extraction kinetics of the oils is also studied.

To achieve the above aim, the following objectives were taken: determination of physiochemical properties of the extracted oils (such as free fatty acid, density, viscosity, iodine value, refractive index etc). This study is limited to extraction of oil from *Luffa Cylindrica* and *Hura Crepitan*, characterization of the oils.

Vegetable oils are ester of glycerol with three fatty acids. Fatty acids are carboxylic acid derived from hydrolysis of animal fats, vegetable oils, or membrane phospholipids with even number of carbon atoms, mostly between 12 and 20, in an unbranched chain.

The three most abundant fatty acids are palmitic (16:0), stearic (18:0), and oleic acid (18:1), where the first number is the number of carbons and the second is the number of double bonds in the hydrocarbon chain [1]. Unsaturated fatty acids have lower melting points than their saturated counterparts; the greater the degree of un-saturation, the lower the melting point.

The general structural formula of vegetable oil is shown below.

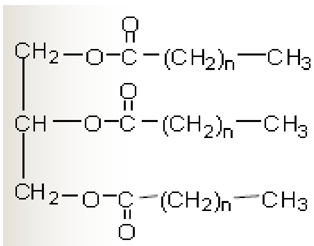

Figure 1: Typical Structure of Vegetable Oil [15].

The various sources of vegetable oil, extraction process and solvents used are reviewed in this chapter. Also, the physicochemical properties, extraction kinetics and thermodynamic parameters of vegetable oil were reviewed.

Materials and Methods

The materials used in the experiment include: *luffa cylindrica* fruits, *Hura crepitan* seeds, soxhlet extractor, reflux condenser, heater, water bath, G-clamp, flasks, beakers, pipette, burette, sample bottle, specific gravity bottle, weighing balance, Abbe refractometer, Pensky Martens flash point tester, crusher, Agilent 7890A GC system coupled with 5975 Mass Spectroscopy detector (GC-MS), HANNA pH meter (HI 8424), n-hexane, acetic acid, hydrochloric acid, chloroform, potassium iodide, carbon tetra chloride (CCl_4), sodium thiosulphate, phenolphthalein, sodium hydroxide and potassium hydroxide.

Sample Collection and Preparation

The *luffa cylindrica* fruits were collected from swampy area in Ikot Akpa Inyang Village, while the *Hura crepitan* seeds were obtained from its fruit, plucked from sandbox tree along Mbiakot road, Mbiakot village, all of Oruk Anam Local Government Area, Akwa Ibom, Nigeria. The fruits were cut open to expose the seeds and sun dried for two days. Thereafter, they were crushed and siev`ed to particle sizes of 0.425, 1.00, 1.80 and 2.80 mm. The 0.425 mm particle size was divided into 20 equal portions with constant weight of 40.00g. This was done to study the effect of time and temperature on the yield of *Luffa cylindrical* and *Hura crepitan* oil.

Oil Extraction

The oil extraction process was carried out in the soxhlet apparatus using n-hexane as solvent. Each of the prepared particle size was placed in the thimble of the extractor and heated via mantle to temperatures of 30, 40, 50 and 60 ºC at extraction times of 20, 40, 60 and 80 min respectively.

Yield

The effect of various parameters like time, particle size and temperatures on oil yield was estimated during the extraction process. The yield of oil extracted was calculated using the formula.

$$Yield = \frac{weight\ of\ \ pure\ oil\ extracted\ (g)}{weight\ of\ \ particle\ (g)} \qquad (1)$$

Characterization of the Extracted Seed Oil

The oils were characterized after extraction and distillation using standard methods. Iodine value, acid value, saponification value and peroxides value were determined using AOAC, (1990) method, while refractive index, flash point, viscosity and specific gravity were determined by AOAC, (1990) method.

Saponification Value

The Saponification Value (SV) was calculated using the expression

$$SV = \frac{(B-S)\times N\times M}{w_o} \qquad (2)$$

Where: B = blank titre value
S = sample titre value
N = normality of KOH (0.5M)
M = molar mass of KOH (56.1)
w_o = weight of oil sample.

Acid Value

The Acid Value (AV) was calculated using the expression

$$AV = \frac{TV\times N\times M}{w_o} \qquad (3)$$

Where TV = titre value
N = normality of KOH (0.1M)
M = molar mass of KOH (56.1)
w_o = weight of oil sample.

Iodine Value
The Iodine Value (IV) was calculated from the equation

$$IV = \frac{(B-S)\times N\times 12.69}{w_o} \quad (4)$$

Where B = blank titre value
S = sample titre value
N = normality of sodium thiosulphate
w_o = weight of oil sample.

Peroxide Value
The peroxide value (PV) was calculated from the equation

$$Peroxide\ Value\ (PV) = \frac{(S-B)\times N\times (1000)}{w_o} \quad (5)$$

Where B = blank titre value
S = sample titre value
N = normality of sodium thiosulphate
w_o = weight of oil sample

Refractive Index
The refractive index was determined using Abbe Refractometer.
Flash Point
The flash point of oil was obtained using the Pensky Martens flash point tester.

Specific Gravity
The specific gravity of the oil was obtained using the expression

$$Specific\ gravity = \frac{Weight\ of\ Oil}{Weight\ of\ equal\ Volume\ of\ water} \quad (6)$$

Results and Discussion
The results of the experiment at varying extraction times, particle sizes and temperatures were presented and discussed in this chapter. Also presented are results obtained from the characterization, extraction kinetics and thermodynamic parameters. Furthermore, predictive models for the extraction of *Luffa cylindrica* and *Hura crepitan* oils were investigated.

Experimental Results of Oil Extraction
The yield of oil investigated in this research was obtained from equation (1). The mass and yield of *Luffa cylindrica* and *Hura crepitan* obtained at constant particle size of 0.425mm and varying temperature of 30 – 60 ºC are presented in Tables 1 to 4 while the study of particle size effect on oil yield was presented in Table 5. The investigation of temperature effect on oil yield was performed at 0.425mm because the maximum yield of oil was observed at this particle size than other sizes investigated in this work, which agreed with reported works in literature that the yield of vegetable oil was obtained at smaller particle sizes [16 – 21]

Table 1: Mass and Yield of Oil at 0.425mm and 30 ºC

Time (min)	*Luffa Cylindrica*		*Hura Crepitan*	
	Mass(g)	Yield (g/g)	Mass(g)	Yield (g/g)
20	1.99	0.4980	2.54	0.6340
40	3.81	0.9530	3.78	0.9450
60	5.07	0.1267	6.32	0.1579
80	6.25	0.1562	7.32	0.1831

Table 2: Mass and Yield of Oil at 0.425mm and 40 ºC

Time (min)	*Luffa Cylindrica*		*Hura Crepitan*	
	Mass(g)	**Yield (g/g)**	**Mass(g)**	**Yield (g/g)**
20	3.66	0.915	4.1	0.1025
40	6.71	0.1678	7.26	0.1815
60	9.42	0.2356	10.4	0.2601
80	10.88	0.2721	12.23	0.3058

Table 3: Mass and Yield of Oil at 0.425mm and 50 ºC

Time (min)	*Luffa Cylindrica*		*Hura Crepitan*	
	Mass(g)	**Yield (g/g)**	**Mass(g)**	**Yield (g/g)**
20	3.98	0.996	4.75	0.1187
40	7.46	0.1865	9.01	0.2253
60	10.46	0.2615	12.63	0.3157
80	13.08	0.3269	15.66	0.3915

Table 4: Mass and Yield of Oil at 0.425mm and 60 ºC

Time (min)	*Luffa Cylindrica*		*Hura Crepitan*	
	Mass(g)	**Yield (g/g)**	**Mass(g)**	**Yield (g/g)**
20	4.56	0.1140	5.49	0.1372
40	7.72	0.1930	10.39	0.2598
60	11.48	0.2869	13.35	0.3338
80	14.36	0.3590	16.80	0.4201
100	14.364	0.3591	16.808	0.4202

Table 5: Mass and Yield of Oil at 80 min and 60 ºC

Particle Size (mm)	*Luffa Cylindrica*		*Hura Crepitan*	
	Mass(g)	**Yield (g/g)**	**Mass(g)**	**Yield (g/g)**
0.425	14.36	0.3590	16.8	0.4201
1.00	13.24	0.3311	14.46	0.3614
1.80	8.49	0.2123	10.54	0.2634
2.80	7.46	0.1865	9.07	0.2268

Some selected yields of vegetable oil were compared with the oils investigated in this work as summarized in Table 6 below. These results showed that the percentage of vegetable oil yield depend on its source and conditions of the extraction.

Physicochemical Properties of Extracted Oil

The results of the physicochemical characteristics of *Luffa cylindrica* and *Hura crepitan* oils obtained are presented in Tables 6 while the free fatty acid component of the extracted oils obtained from the GC-MS are shown in Tables 7 and 8. Two free fatty acids (linoleic acid and palmitic acid) were identified from the GC-MS analysis and results showed that the percentage of linoleic acid was higher than palmitic acid for both *Hura crepitan* and *Luffa cylindrica* oil.

Table 6: Physicochemical Characteristics of Extracted Oils

Parameter	*Luffa cylindrica*	*Hura crepitan*
Saponification Value (mgKOH/g)	132.45	290.32
Acid value (mgKOH/g)	12.62	27.21
Iodine value (g/100g)	31.33	176.89
Peroxide value (mg eq./kg sample)	10.50	3.75
Viscosity at 15 ºC (cp)	2.43	3.40
Refractive index (-)	1.42	1.43
Flash point (ºC)	128	130
Specific gravity (-)	0.93	0.94
Moisture content (%)	6.61	8.11
pH	5.89	5.10
Colour	Dark brown	Golden yellow

Table 7: Summary of GC-MS Chart Peaks of *Hura crepitan* Seed Oil

Retention Time (min)	Compound	Formula	M.W	Weight (%)	Peak
5.970	Palmitic acid	$C_6H_{32}O_2$	256	10.01	I
20.372	Linoleic acid	$C_{18}H_{32}O_2$	280	5.25	II
20.658	Linoleic acid	$C_{18}H_{32}O_2$	280	66.62	III

Table 8: Summary of GC-MS Chart Peaks of *Luffa cylindrica* Seed Oil

Retention Time (min)	Fatty Acid	Formula	M.W	Weight (%)	Peak
18.701	Palmitic acid	$C_6H_{32}O_2$	256	3.15	I
20.560	Linoleic acid	$C_{18}H_{32}O_2$	280	96.85	II

Where M.W = Molecular weight

Effect of Extraction Time on Oil Yield

Effect of extraction time on the yield of oil obtained from *Luffa cylindrica* and *Hura crepitan* was studied at constant temperature of 60 ºC and particle size of 0.425 mm. The extraction time was varied from 20 to 80 minutes. The experimental results as presented in Figure 2 showed that the yield of oil from both raw materials investigated, increases as the extraction time is increased. Thus, from 20 to 80min of extraction, the yield of oil increases from 0.114 to 0.359 in *Luffa cylindrical* and 0.1372 to 0.4201 in *Hura crepitan*. This showed that higher oil yield was obtained from *Hura crepitan* than in *Luffa cylindrica*. The mixture of n-hexane and the seed oil (miscella) obtained from the extraction was further distilled to separate the oil from the entrained solvent. At the end of the distillation process, the weight of the oil was recorded.

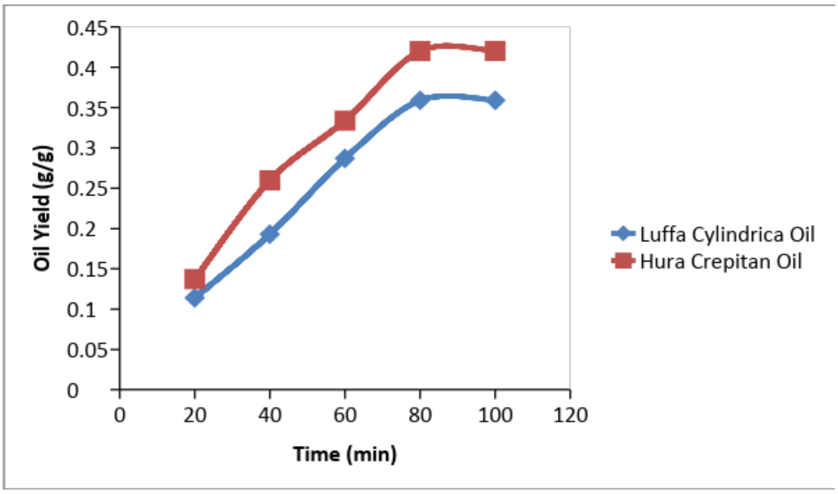

Figure 2: Comparison of Luffa Cylindrica and Hura Crepitan Oil Yield with Time

Conclusion

The extraction of vegetable oil from *Luffa cylidrica* and *Hura crepitan* oil using n-hexane was performed to investigate the effect of extraction time. The physicochemical analysis of both oils showed high saponification and iodine value, which is an indication that the oils extracted are suitable for domestic and industrial applications, such as soap production.

However, the percentage yield of oil increases as extraction time and temperature are increased but high oil yields are obtained at smaller particle size. The highest yield of oil for both *Luffa cylindrica and Hura crepitan* seeds was observed at particle size of 0.425 mm, temperature of 60 ºC and extraction time of 80 min.

Hura crepitan fruit is most used as playing toy for children, while *Luffa cylindrica* is used as sponge in local communities with the seeds discarded as waste. However, with this study, it has been known that both plants are useful raw materials for vegetable oil. The oils obtained from *Luffa Cylindrica* and *Hura Crepitan* seeds have excellent physiochemical properties suitable for industrial applications (manufacture of cosmetics, drying agent in paints, food processing as well as for the production of transportation fuel (biodiesel).

Recommendations

The following recommendations are made following observations from this study:

- To obtain high yield of vegetable oil, the extraction temperature should not be operated below 60 ºC and the particles size must be reduced to increases the surface area of contact.
- *Hura crepitan* and *Luffa cylindrica* seed oil should be massively produced for use in the industry.

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