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Analyzing Physicochemical Properties of Wild Grapes (*Lannea Microcarpa*) Seed Oil

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ABSTRACT

The purpose of this study was to analyze physicochemical properties of wild grape (*Lannea microcarpa*) seed oil. Several characterizations were conducted, including a gas chromatography and mass spectrometry (GC-MS) and cold saponification. The analyses were also supported by measuring acid, iodine, saponification, and peroxide values. Other analyses were relative density and refractive index. Experimental results showed that the oil was dark purple with the composition of oil of 59%. The qualitative GC-MS revealed the oil contained several fatty acids, including decanoic acid, palmitic acid, stearic acid, margaric acid, 1-octadecanoic acid, oleic, and erucic acid. The soap produced from the seed oil has basic pH and relatively high foam value. When the high concentration of oil was used, the appearance of oil was very dark purple and slightly soluble in water. This is due to the fact that most of the oil compositions were non-polar structure. This result confirmed the potential use of the oil for soap and other cosmetic materials.

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1. INTRODUCTION

Lannea microcarpa known in Hausa language as “Faarruu” is found in derived savannah and drier forest mostly in Sudanian zones of West Africa (Tarhule & Hughes, 2002). The fruits of the plant are edible and traded commercially (Hermann, 2009). Ethnobotanical investigations on local oilseed species in Burkina Faso revealed that oil from *Lannea microcarpa* tree oil seed is frequently used for food, cosmetics and traditional medicine by local people (Hilou et al., 2017).

Physicochemical properties of biodiesel from African grapes (*Lannea microcarpa*) was assessed by Yunus et al. (2013). Physicochemical properties of biodiesel from wild grape seeds oil and petro-diesel blends as chemically stable, environmentally friendly, and economically viable for use in compression ignition engine as a blend to partly replace the automotive gasoline oil was reported by other researchers (Kaisan et al., 2013). Evaluation of proximate composition of seeds and main physicochemical properties and thermal stability of oil extracted from *Lannea microcarpa* seeds has been reported by Bazongo et al. (2014). However, they did not report in detail physicochemical properties of Lannea microcarpa. Here, this work focused on the first report of physicochemical properties of *Lannea microcarpa*. Several analyses were done to support this study: gas chromatography and mass spectrometry analysis and soap production from wild grapes (*Lannea microcarpa*) seed oil.

2. MATERIALS AND METHODS

2.1. Sample Collection, Identification and Preparation.

The seeds of *Lannea microcarpa* were obtained directly from fruit of the plant in the month of July, 2016 at Yauri town, Kebbi state, Nigeria. They were dried and crushed into powder using mortar and pestle and stored in a plastic container prior to oil extraction.

2.2. Oil Extraction Procedure in.

The hexane extract was obtained by complete extraction using the Soxhlet extractor (GG-17, SHUNIU). The 50 grams of each powdered sample was put into a porous thimble and placed in a Soxhlet extractor. This study used 150 cm^3 of n-hexane (Brataco, Indonesia; with boiling point of 40-60^oC) as extracting solvent for 6 hours repeatedly until required quantity was obtained. The oil was obtained after evaporation using a water bath at 70^oC to remove the excess solvent from the extracted oil. The oil was then stored in refrigerator prior to GC-MS analysis.

2.3. Yield Analysis

The oil was recovered by complete distilling of most of the solvent on a heating mantle. Then, the recovered oil was transferred to a beaker. The beaker was placed over water bath for complete evaporation of solvent for about 2 hours and volume of the oil was recorded and expressed as oil content (%) in line with literature report.

2.4. Determination of Color Intensity

The color of the oil sample was determined by observation using several analysis and compared with color charts based on literature (Okolie et al., 2012).

2.5. Determination of Relative Density

10 mL of the oil was measured in a pre-weighed measuring cylinder. The weight of the cylinder and oil was measured and compared with the weight of oil. Then, by subtracting the weight of the cylinder from the weight of the oil and cylinder, the density of oil can be obtained.

2.6. GC-MS Analysis

The analysis of the fatty acids in the *Lannea microcarpa* oil sample was done at National Institute of Chemical Technology (NARICT), Zaria, Nigeria, using a Shimadzu QP2010 plus series gas chromatography coupled with Shimadzu QP2010 plus mass spectroscopy detector (GCMS) system was used. The temperature programmed was set up from 70 to 280^oC. Helium gas was used as carrier gas. The injection volume was 2 µL with injection temperature of 250^oC and a column flow of 1.80 mL/min for the GC. For the mass spectroscopy, mode scanner with a scan range of 30-700 amu at the speed of 1478 was used. The mass spectra were compared with the NIST05 mass spectral library.

2.7. Preparation and Analysis of Lannea microcarpa seed oil Soap

Saponification Procedure: 200 grams of *sodium hydroxide* pellets was dissolved in 1,000 cm^3 of volumetric flask and the volume made to the mark with distilled water. The required quantity of *alkaline* solution was mixed with *Lannea microcarpa* seed oil. The oil was warmed gently and poured into the beaker followed by the alkali solution to form an intimate mix and then stirred frequently for 7 minutes using stirring rod until reaction reached equilibrium. The saponification mixture was then poured into mould and allowed to dry (cure) for 24 hours.

2.8. pH Determination

The pH was determined using pH meter (350 JENWAY Model). 5 grams of the soap shavings were weighed and dissolved with distilled water in a 100 mL of volumetric flask. The electrode of the pH meter was inserted into the solution of the soap and the pH reading was recorded.

2.9. Foam Ability Test

A 2 grams of the soap was added to a 500 cm^3 of measuring cylinder containing 100 cm^3 of distilled water. The mixture was shaken vigorously so as to generate foams. After shaking for some time, the cylinder was allowed to stand for 10 minutes. The height of the foam in the solution was measured and recorded.

3. RESULTS

Figure 1 shows typical GC-MS total ionic chromatogram (TIC) of hexane extract of *Lannea microcarpa* L. seed oil. Several peaks were obtained, in which this can be classified as seed oil.

It is also reported in **Figure 2** that GC-MS fragments of hexane extract of *Lannea microcarpa* L. seed oil. Figure can be classified as 6 elements. Detailed elements in the seed oil are attached in each Figure. It can be confirmed that seed oils contained these elements.

Table 1 tabulated physico-chemical properties of *Lannea microcarpa* Seed Oil. There are several analyses. The main analysis is the oil yield, in which the percentage of oil can reach near to 60%.

To confirm analysis in **Table 1**, **Table 2** shows Major fatty acids derived from oil of *Lannea microcarpa* seed. There are 7 fatty acids in the oil. Most of the fatty acid is molecule with more than 11 carbon atoms. The molecular weight of the oil can reach more than 186.

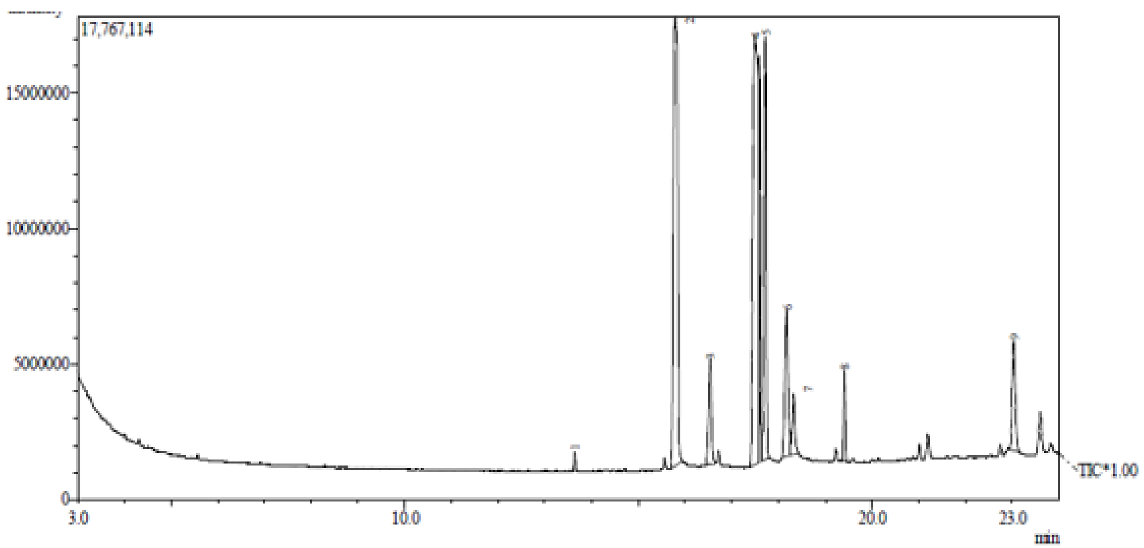

Figure 1. Typical GC-MS total ionic chromatogram (TIC) of hexane extract of *Lannea microcarpa* L. seed oil

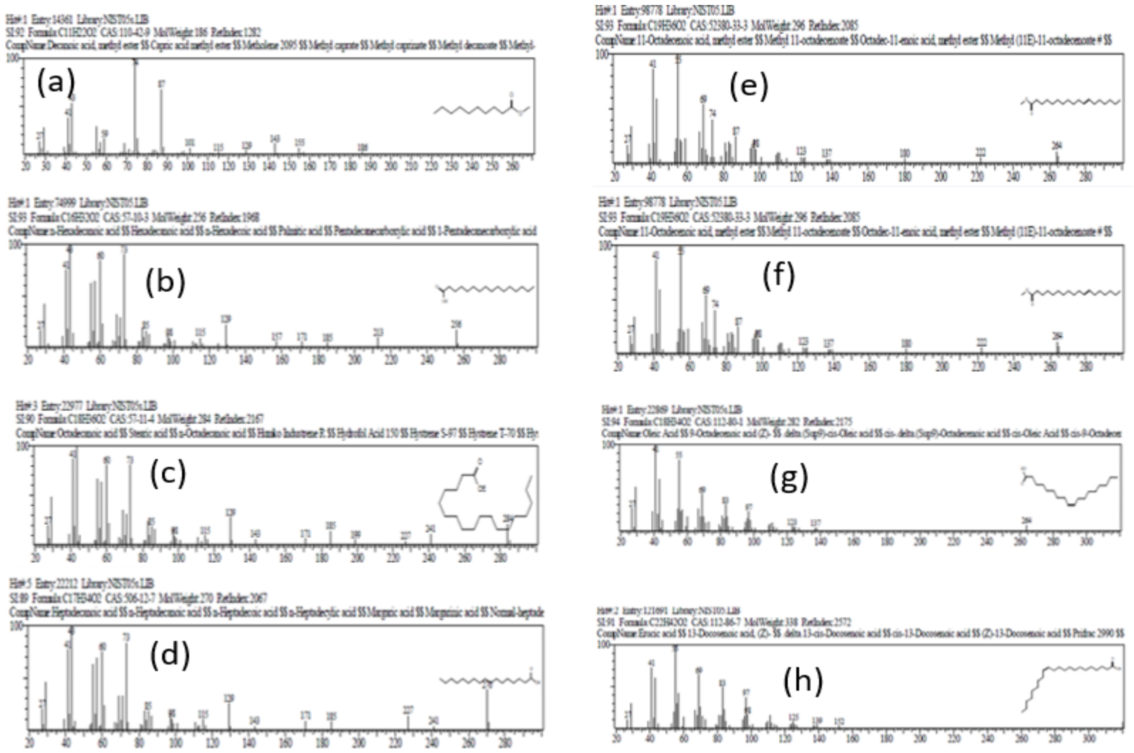

Figure 2. GC-MS fragments of hexane extract of *Lannea microcarpa* L. seed oil.

Table 1. Physicochemical properties of *Lannea microcarpa* Seed Oil*

Parameters	Values
Oil yield (%)	59.21±0.01
Colour of oil	Dark purple
Acid value mgKOH/g	016± 0.01
Iodine value gI_2/100g	121.6±0.1
Saponification value mgKOH/g	231.25±0.02
Peroxide value meq H_2O_2,	3.02±0.01
Relative density (g/cm3)	0.5983±0.0001
Refractive index	1.43±0.01
Oil yield (%)	59.21±0.01
Colour of oil	Dark purple
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Values are expressed as mean and ± standard deviation of triplicate determinations *

Table 3 shows physicochemical characteristics of *Lannea microcarpa* seed oil soap. The main parameters used are pH, foam height, solubility, and color intensity. The results showed that the analyses confirmed the oil is identical to the *Lannea microcarpa* seed oil soap.

Table 2. Major fatty acids derived from oil of *Lannea microcarpa* seed.

S/N	Name of fatty acid	MF	MW	RI	SI% to T.C.
1	Decanoic acid.	$C_{11}H_{22}O_2$	186	1282	92
2	Palmitic acid.	$C_{16}H_{32}O_2$	256	1968	93
3	Stearic acid.	$C_{18}H_{36}O_2$	284	2167	90
4	Margaric acid	$C_{17}H_{34}O_2$	270	2067	89
5	11-octadecanoic acid	$C_{19}H_{36}O_2$	296	2085	93
6	Oleic	$C_{18}H_{34}O_2$	282	2175	94
7	Erucic acid	$C_{22}H_{42}O_2$	338	2572	91

Note: S/N = Serial number, M.F.= Molecular formula, M.W. = Molecular weight, RI= Retention index SI% = Similarity index, T.C. = Target compound.

Table 3. Physicochemical characteristics of *Lannea microcarpa* seed oil soap

Parameters	Values/Observation
pH	10.18±0.01
Foam height (cm^3)	105.1±0.1
Solubility in water	Slightly soluble
Color	Very dark purple

Note: All values are expressed as mean ± standard deviation of triplicate determinations

4. DISCUSSION

Physicochemical, GC-MS, and cold saponification yielded the following results. Oil yield was 59.21±0.01%, this is higher than previous studies as reported by (Warra, 2017) (56.50 ± 0.10%) and (Warra et al., 2015) (50.28 ± 0.01%). This is in the good recommendation for cosmetic uses. The color of the oil was dark purple. It was reported that many consumers preferred the bright color, transparent, but close to its natural color of oil (Zzaman et al., 2014).

From the results of the physicochemical analysis, acid value of 016 ± 0.01 mgKOH/g was obtained, in which this is higher than other reports such as (Warra et al., 2015) (0.35 ± 0.01 mgKOH/g) and (Okolie et al., 2012) (27.09 ± 2.30 mgKOH/g). Lower acid value makes oil suitable for soap production. Saponification value that reaches 231.25 ± 0.02 mgKOH/g showed higher value than normal saponification values (mgKOH/g) of about 210 mgKOH/g (see references in (Warra et al., 2016)). This value is good for soap production. Iodine value of the sample was 121.6 ± 0.1 gI_2/100g, in which this is higher than 50.50 ± 8.023, I_2/100g reported by (Warra et al., 2012). This is recommended for cosmetics and medicinal purposes. In the case of peroxide value, the value reaches 3.02 ± 0.01 mEq H_2O_2. The peroxide value is used as an indicator of deterioration of oils. Fresh oils have values less than 10 mEq Kg-1. Values between 20 and 40 result to rancid taste. High values can be reduced by alkaline refining (Akubugwo & Ugbogu, 2007).

Relative density value was 0.5983 ± 0.0001 g/cm^3. Refractive index value was lower than previous study, such as (Mudawi et al., 2014) and (Amira et al., 2014). Increase in refractive index values in the triacylglycerols or degree of unsaturation result in increase in chain length of fatty acids (Segura-Campos et al., 2014). Qualitative GC-MS revealed the following fatty acids, including decanoic acid, palmitic acid, stearic acid, margaric acid, 1-octadecanoic acid, oleic, and erucic acid. The soap produced from the seed oil has pH and Foam height, 10.18 ± 0.01 and 105.1±0.1 cm^3, respectively. Very dark purple color and slightly soluble in water was obtained. The results showed the potential of the seed oil in soap and other cosmetic preparations.

5. CONCLUSION

The present study has successfully analyzed physicochemical properties of wild grape (*Lannea microcarpa*) seed oil. Experimental results showed that the oil contains several fatty acids, including decanoic acid, palmitic acid, stearic acid, margaric acid, 1-octadecanoic acid, oleic, and erucic acid. The existences of these chemicals in the oil give the potential use of the oil for soap and other cosmetic materials.

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