



Physico-chemical, Gas Chromatography-Mass Spectrometry (GC-MS) Analysis and Cold Saponification of Wild Grapes (*Lannea microcarpa* Engl & K. Krause) Seed Oil

**Aliyu Ahmad Warra^{1*}, Hadiza Ibrahim Mohammed²
and Adejuwon Olorunsola Morayo³**

¹Centre for Entrepreneurial Development, Federal University, Gusau, Nigeria.

²Department of Pharmacognosy and Drug Development, Ahmadu Bello University, Zaria, Nigeria.

³Centre for Entrepreneurship Development and Vocational Studies, Federal Polytechnic, Ado-Ekiti, Nigeria.

Authors' contributions

This work was carried out in collaboration among all authors. Author AAW designed the study, performed the analysis, wrote the protocol and wrote the first draft of the manuscript and did literature searches. Author HIM managed the review, provided scholarly contributions and some literature searches and author AOM contributed in review. All authors read and approved the final manuscript.

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ABSTRACT

Aims: Physico-chemical, Gas Chromatography-Mass Spectrometry (GC-MS) and Cold saponification was carried out on *Lannea microcarpa* (Wild grape) seed oil with the aim of identifying the quality and quantity of the oil and its suitability in soap production.

Study Design: Experimental and instrumental study was done to determine the physicochemical characteristics, fatty acids present in the seed oil and its suitability for soap production.

*Corresponding author: E-mail: aliyuwarra@yahoo.com;

Place and Duration of Study: The study was conducted at the Biochemistry Laboratory, Kebbi State University of Science and Technology, Aliero, Nigeria from May to June, 2014.

Methodology: The hexane extract of the sample was obtained by complete extraction using Soxhlet extractor, physicochemical analysis was carried out. A gas chromatography coupled with mass spectroscopy detector (GC-MS) system was used for the qualitative fatty acid determination. Simple cold method saponification was used in producing the soap.

Results: The powdered seed (50 g) yielded $59.21 \pm 0.01\%$ of the oil. Results from the physicochemical analysis showed the seed oil to be dark purple in colour and partially soluble in water with the acid, iodine, saponification and peroxide values at 16 ± 0.01 mgKOH/g, 121.6 ± 0.1 gI₂/100 g, 231.25 ± 0.02 mgKOH/g, 3.02 ± 0.01 meq H₂O₂ respectively. The relative density and refractive index of the oil are at 0.5983 ± 0.0001 (g/cm³) and 1.43 ± 0.01 respectively. Qualitative GC-MS revealed the following fatty acids; 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 (cm³) respectively.

Conclusion: The present study on seeds of wild grape revealed that the oil is a viable natural product that can be utilized in the food and cosmetic industry.

Keywords: Lannea microcarpa; seed oil; physico-chemical; GC-MS; saponification.

1. INTRODUCTION

Lannea microcarpa Engl & K. Krause called Wild grape and "Fààrúú" in English and Hausa Language, is a deciduous tree with dense, hemispherical crown, found in derived savannah and drier forest mostly in Sudanian zones of West Africa [1,2]. The plant grows to about 16m tall, bearing a bole that can be 60cm in diameter, producing green fruits that turns purplish black when ripe. The fruits and leaves are consumed as food and traded commercially for production of juice, while all parts of the plant are used in traditional medicine to treat disease such as hypertension, dysentery, rheumatism, cough and inflammation fruits of the plant are edible and traded commercially [2,3,4,5].

Ethnobotanical investigations on the locally produced oil seed plants in Burkina Faso revealed that oil from *Lannea microcarpa* seeds are frequently used for food, cosmetics and traditional medicine by the local people [6]. The physico-chemical properties of bio-diesel from wild grape seeds oil and petro-diesel blends aschemically stable, environ-mentally friendly and economically viable for use in compression ignition engine as a blend to partly replace the automotive gasoline oil was reported [7,8]. Also, the evaluation of the proximate composition of the seeds and the thermal stability of oil extracted from seeds were reported [9]. This work appears to be the first report of the physico-chemical evaluation, Gas Chromatography-Mass Spectrometry (GC-MS) analysis and soap production of the wild grapes (Fig. 1c.) seed oil growing in Northern Nigeria.

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 May, 2014 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

The hexane extract was obtained by complete extraction by using the Soxhlet extractor (GG-17, SHUNIU). The 50 g of each powdered sample was put into a porous thimble and placed in a Soxhlet extractor, using 150 cm³ of n-hexane (with boiling point of 40- 60°C) as extracting solvent for 6 hours repeatedly until required quantity was obtained. The oil was obtained after evaporation using Water bath at 70°C to remove the excess solvent from the extracted oil. The oil was then stored in refrigerator prior to further analysis by GC-MS technique.

2.3 Percentage Yield

The oil which was recovered by complete distilling of most of the solvent on a heating mantle was transferred to a beaker. The beaker was then 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.

$$\text{Oil content (\%)} = \frac{\text{Weight of the oil}}{\text{Weight of sample}} \times 100$$

2.4 Determination of Colour

The colour of the oil sample was determined by observation using several independent competent individuals. Oil colour was correlated using colour charts [10].

2.5 Determination of Relative Density

This was performed according to literature report [11]. The 10 ml of the oil was measured in a pre-weighed measuring cylinder. The weight of the cylinder and oil was measured; the weight of the oil was then obtained by subtracting the weight of the cylinder from the weight of the oil and cylinder. The density of the oil was obtained using equation below.

$$\text{Density of oil} = \frac{W_1 - W_0}{V_0}$$

where,

W1 = weight of empty measuring cylinder + oil.

W0 = weight of measuring cylinder, V0 = volume of oil used.

2.6 Physico-Chemical Analysis

The physico- chemical analysis of the *Lannea microcarpa* L. Seed oil was carried out using the methods reported [12,13,14].

2.7 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, 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°C to 280°C. Helium gas was used as carrier gas. The injection volume was 2 µL with injection temperature of 250°C and a column flow of 1.80 mL/min for the GC. For the mass spectroscopy ACQ mode scanner with scan range of 30-700 amu at the speed of 1478 was used. The mass spectra were

compared with the NIST05 mass spectral library [15].

2.8 Preparation and Analysis *Lannea microcarpa* Seed Oil Soap

2.8.1 Saponification procedure

As reported in literature [16]. 200 grams of sodium hydroxide pellets was dissolved in 1000cm³ 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 (ratio 1:1 v/v). 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.

2.8.2 pH determination

The pH was determined using pH meter (350 JENWAY Model). A 5 g of the soap shavings were weighed and dissolved with distilled water in a 100 ml 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 g of the soap was added to a 500 cm³ measuring cylinder containing 100 cm³ 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. DISCUSSION

Physico-chemical, Gas Chromatography-Mass Spectrometry (GC-MS) and Cold saponification was carried out on Wild grape (*Lannea microcarpa*) Seed Oil and have yielded the following results; oil yield was 59.21±0.01%, higher than 56.50 ± 0.10 (%), reported for the vetia seed oil [17]. and 50.28±0.01% reported for onion seed oil [18]recommended for cosmetic uses. The colour 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 [19]. From the results of the physicochemical analysis, acid value of 016± 0.01 mgKOH/g was obtained, higher than

0.35±0.01 reported for canary melon seed oil [20]. Lower than 22.37 ± 1.168 reported for neemseed oil [21]. Lower acid value makes oil suitable for soap production. Saponification value, 231.25±0.02 mgKOH/g showed higher value than saponification values (mgKOH/g) 203.00±0.00 and 218.52± 0.01 reported for two varieties of sesame seed oils [22] lower than 246.60 mg KOH/g reported for *Elaeis guineensis* seed oil [23] range of recommended values suitable for soap making. Iodine value of 121.6±0.1 gI₂/100 g obtained is higher than 50.50 ± 8.023, I₂/100 g reported for *Jatropha curcas* L. seed oil [24] lower value than iodine value (mg/100g) of 152.3, reported for wild *Corchorus olitorius* seed oil [25] recommended for cosmetics and medicinal purposes. Peroxide value of 3.02±0.01 meq H₂O₂ was obtained. The peroxide value is used as an indicator of deterioration of oils. Lower value is an indicator of freshness and purity. Relative density (g/cm³) value was 0.5983±0.0001. Refractive index value was 1.43±0.01 lower than 1.4750 reported for Corn oil [26]. Higher than 1.412 reported for Palm Kernel Oil [27] Olaniyi et al. 2014. Increase in refractive

index values in the triacylglycerols or degree of unsaturation result in increase in chain length of fatty acids [28]. Qualitative GC-MS revealed the following fatty acids; 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(cm³) respectively, Very dark purple colour and slightly soluble in water. The results showed the potential of the seed oil in soap and other cosmetic preparations.

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 ₂ O ₂ ,	3.02±0.01
Relative density (g/cm ³)	0.5983±0.0001
Refractive index	1.43±0.01



(a)



(b)

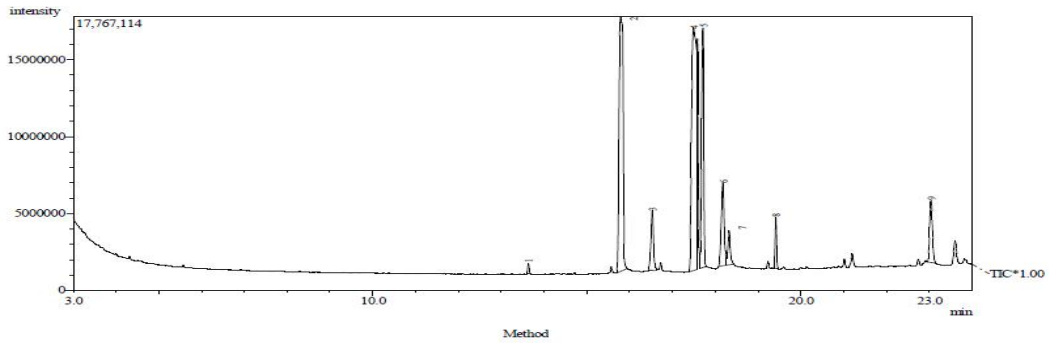


(c)



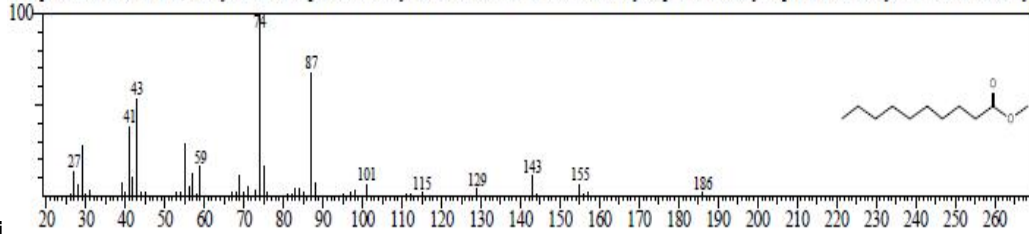
(d)

Fig. 1.0.(a) *Lannea microcarpa* fruits (b) *Lannea microcarpa* deshelled seeds (c) *Lannea microcarpa* seed oil (d) *Lannea microcarpa* oil fresh soap



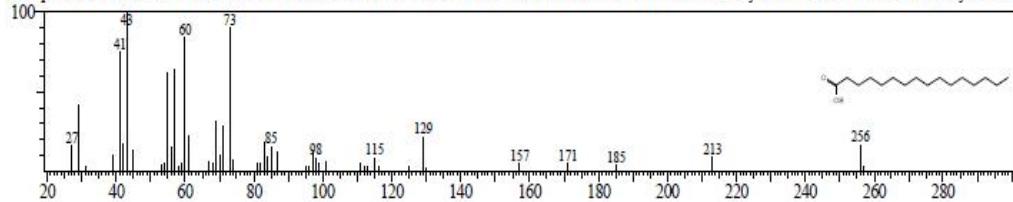
(a)

Hit#1 Entry:14361 Library:NIST05s.LIB
 SI:92 Formula:C11H22O2 CAS:110-42-9 MolWeight:186 RetIndex:1282
 CompName:Decanoic acid, methyl ester \$\$ Capric acid methyl ester \$\$ Metholene 2095 \$\$ Methyl caprate \$\$ Methyl caprinate \$\$ Methyl decanoate \$\$ Methyl-



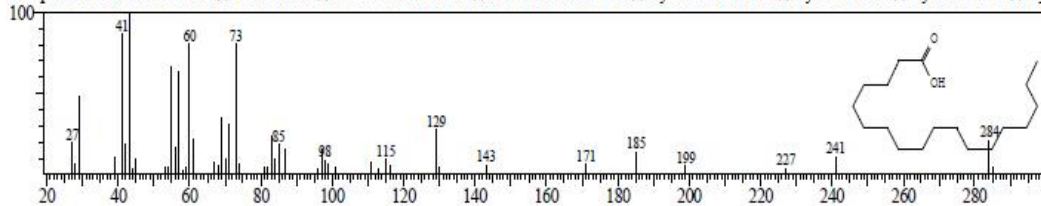
(b) i

Hit#1 Entry:74999 Library:NIST05.LIB
 SI:93 Formula:C16H32O2 CAS:57-10-3 MolWeight:256 RetIndex:1968
 CompName:n-Hexadecanoic acid \$\$ Hexadecanoic acid \$\$ Palmitic acid \$\$ Pentadecanecarboxylic acid \$\$ 1-Pentadecanecarboxylic acid



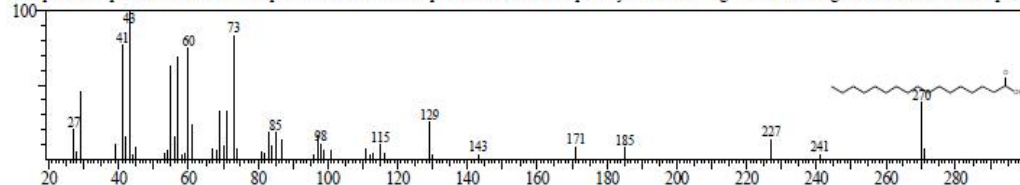
(b) ii

Hit#3 Entry:22977 Library:NIST05s.LIB
 SI:90 Formula:C18H36O2 CAS:57-11-4 MolWeight:284 RetIndex:2167
 CompName:Octadecanoic acid \$\$ Stearic acid \$\$ n-Octadecanoic acid \$\$ Himko Industrene R \$\$ Hydrofol Acid 150 \$\$ Hystrene S-97 \$\$ Hystrene T-70 \$\$ Hys-



(b) iii

Hit#5 Entry:22212 Library:NIST05s.LIB
 SI:89 Formula:C17H34O2 CAS:506-12-7 MolWeight:270 RetIndex:2067
 CompName:Heptadecanoic acid \$\$ n-Heptadecanoic acid \$\$ n-Heptadecic acid \$\$ n-Heptadecylic acid \$\$ Marganic acid \$\$ Margannic acid \$\$ Normal-heptade



(b) iv

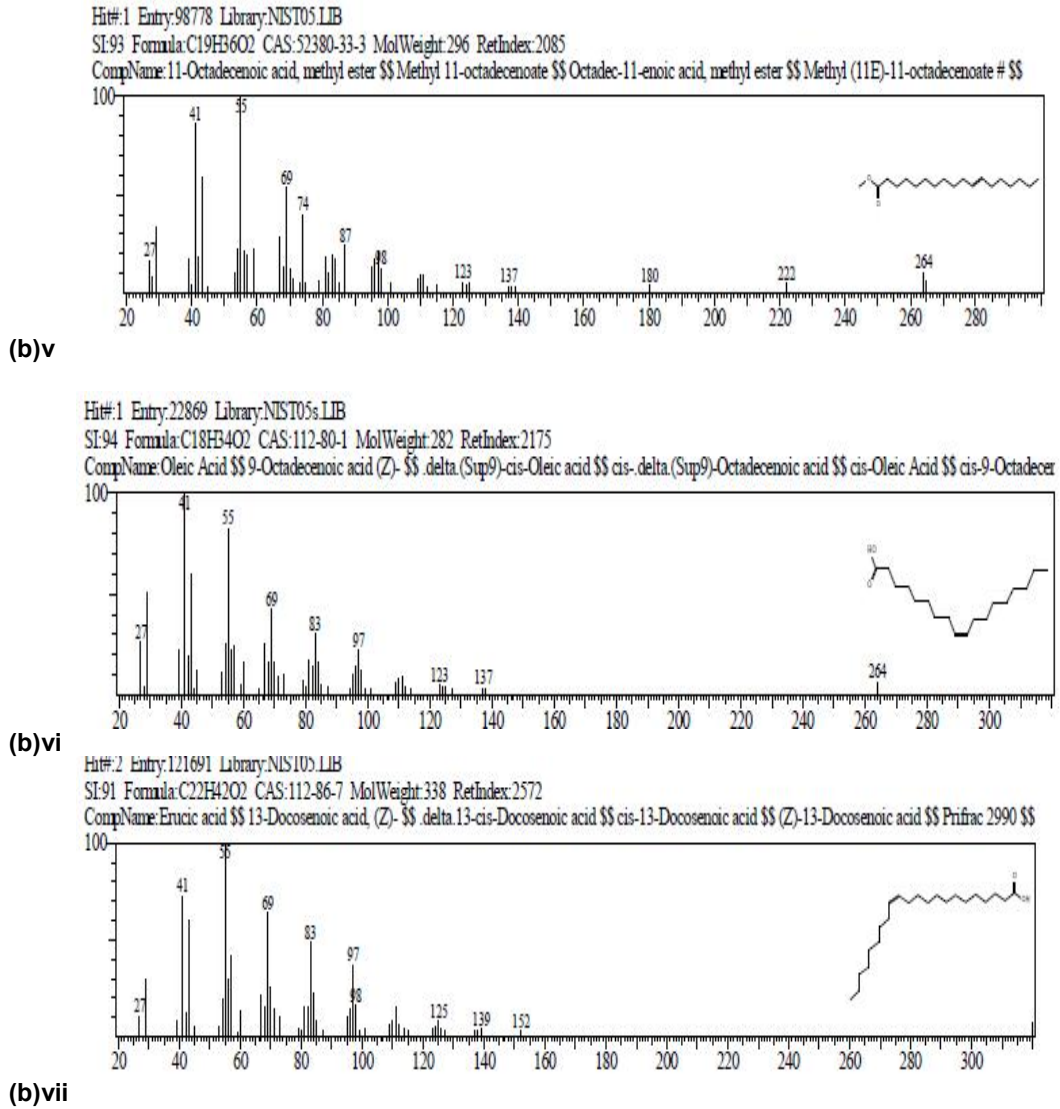


Fig. 1.1 (a)Typical GC-MS total ionic chromatogram (TIC) of hexane extract of *Lanena microcarpa* L. seed oil.(b) i- vii GC-MS fragments of hexane extract of *Lanena microcarpa* L. seed oil

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

S/N	Name of fatty acid	MF	MM	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.M. = Molecular mass, 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

* Values are expressed as mean ± standard deviation of triplicate determinations

4. CONCLUSION

It is concluded that the physico-chemical, GC-MS analysis and the soaps produced from the hexane extract of *Lannea microcarpa* seed oil indicated its potential for soap and other cosmetic utilization.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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