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FATTY ACID AND TRIGLYCERIDE PROFILES OF THE FRUIT EXTRACTS OF CANARIUM SCHWEINFURTHII

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ABSTRACT:

The seeds and oil of *Canarium schweinfurthii* Engl. are believed to have therapeutic properties and healthy for consumption. In Vom Plateau State, the oil is extracted from the fruits and the marc is considered to have no value. This study extracted the oil traditionally from the fruits as done locally and the marc extracted with different solvents to evaluate the nature of phytoconstituents in the oil and marc extracts. The extracts were subjected to LCMS to determine their fatty acid profiles with significant amounts of Saturated, mono unsaturated and poly unsaturated fatty acids detected in the oil and extracts of the marc. NMR analysis was used to isolate and characterize Compound UT-ME02 (Trilinolein), Compound UT- EA02 (Triolein) and Compound UT- PE01 (Trielaidin) from the Methanol, Ethyl acetate and Petroleum ether extracts respectively after Column chromatography over silica gel (60-120 mesh) as the stationary phase was eluted with a mobile phase mixture of n-Hexane; n-Hexane-Ethyl acetate; Ethyl acetate; Ethyl acetate-Methanol and Methanol with gradient increasing polarity. The 1H NMR spectra for UT- ME02, UT-EA02 and UT-PE01 all showed nine signals; signals A - I and the 13C NMR for same extracts all showed recognizable signals for the carbonyl ester of triacylglycerols at different chemical shifts. These assignments of Proton and carbon signals in the 1H NMR and 13C NMR were made from reports of other studies and the results showed that important phytoconstituents were present

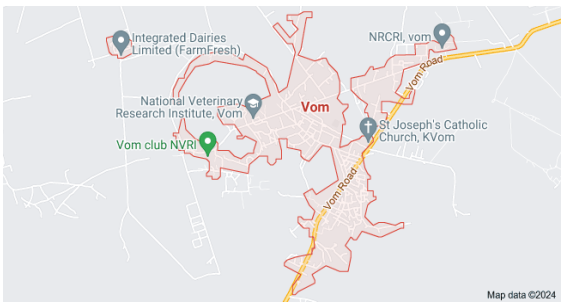
in both the oil and solvent extracts of the marc such that we can boldly claim that the marc isn't really useless as perceived in the community. The marc can be valuable in animal feed formulations as a valuable source of lipids.

KEYWORDS: *Canarium schweinfurthii, Fatty acids, Triglycerides, Spectroscopy, Marc Extracts, Oils.*

INTRODUCTION:

Canarium schweinfurthii produces an edible fruit that can be eaten raw or softened in warm water. In Nigeria, the plant is referred to as Oda in Idoma, Opa in Igede, Atili in Hausa/Berom, and *Dacryodes edalis* in Tiv. African canarium and white mahogany are two of its common names. It is used as a food ingredient or cooked and processed into a fruity butter that serves as a shea butter substitute. It is traditionally used as an insecticide or as an emollient, stimulant, diuretic, for skin infections, eczema, leprosy, ulcers, diabetes, colic, stomach pains, post-natal pains, gale, fever, constipation, malaria, sexually transmitted diseases, and rheumatism. [1] [2] In the traditional process of extracting the oil from the fruits of *Canarium schweinfurthii*, the flaky fibrous husks (marc) left after the extraction have little or no value. Therefore, this study investigates the useful potential of the marc by using phytochemical methods to isolate, characterize, and identify any compounds in the traditionally extracted oil and in the extracts of the marc to ascertain its usefulness. Since there is presently a demand for alternative food supplements, this could unveil untold phytoconstituents in the in the oil and marc, which could serve as a source of fatty acids and other phytochemicals. Also, since the method of extraction affects the quality of the oil, [3] the study will reveal if the traditionally extracted oil has more phytoconstituents than the other oils extracted from the fruits using other methods of extraction.

MAP OF STUDY AREA:



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MATERIALS AND METHODS:

Plant collection and authentication:

Fresh fruits of *Canarium schweinfurthii* were collected from Vom market, Vwang District of Jos-South LGA Plateau State, Nigeria; GPS coordinates: 9° 43' 44.8" North, 8° 47' 29" East. The fruits and plant were authenticated by Mrs. Jepson Gyelkur of the College of Animal Health and Production Technology, Vom, Plateau State, Nigeria where a voucher specimen was deposited under the reference Number 021529/CAH/Vom.

Extraction

Traditional method:

500 g of the fruits were weighed and boiled for one hour. The boiled fruits were then mashed with a mortar and pestle until the fruit pulp completely separates from the seeds. The mashed fruit pulp was then transferred into boiling water and heated until the oil from the fruits begins to seep into the water and it was continuously scooped off. This continued until there was no further oil on the surface of the boiling mixture. The oil collected was then boiled with *Digitaria exilis* locally known as Acha, to absorb the water in the oil. The oil is then decanted and bottled.

Solvent extraction:

The flaky fibrous marc of the fruits after its oil has been extracted were air dried at room temperature for 72 hours. The dried marc was ground into powder using a mortar and pestle. The powdered material, 100 g, was macerated successively in 1000 cm3 of AnalaR grade solvents, petroleum ether, ethyl acetate and then methanol for 48 hours each. The extracts were filtered and left to dry under ambient conditions. The solvent free extracts were transferred into sample bottles and placed in a desiccator containing anhydrous sodium sulfate to remove any traces of moisture.

LC-MS analysis:

The traditionally made oil, petroleum ether and ethyl acetate extracts of the marc were subjected to LCMS. A Thermo Electron LTQ-Orbitrap XL mass spectrometer equipped with a nano electrospray ion source (ThermoFisher Scientific, Bremen, Germany) and operated under Xcalibur 2.1 version software, was used in positive ionization mode for the MS analysis using data-dependent automatic switching between MS and MS/MSn acquisition modes.

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Identification of phytoconstituents:

The identification of the compounds was based on comparison of their mass spectra with NIST Ver. 2.0 Year 2008 library WILEY8, FAME.

NMR analysis

1H and 13C NMR analysis were carried out on a Bruker DRX-400 (Bruker, Rheinstetten, Germany) spectrophotometer (400 MHz for proton and 125 MHz for carbon-13 in CDCl3).

RESULTS AND DISCUSSION:

Table 1: Yield of the Traditionally made oil and Solvent Extraction of the marc of Canarium schweinfurthii fruits

Method	Weight of fruits/marc (g)		Yield (g)	Percentage (%)
	Before extraction	After extraction		
Traditional	500.00*	287.00	210.40	42.08
Solvent				
Petroleum ether	287.00^	281.60	5.40	1.88
Ethyl acetate	281.60^	277.10	4.50	1.60
Methanol	279.10^	272.80	6.30	2.26

Key: * fruits, ^ Marc

LC-MS analysis of phytoconstituents of the traditionally made oil of Canarium schweinfurthii Fruit:

LC-MS analysis of the traditionally made oil showed twenty-eight peaks indicating different phytochemical constituents (Figure 1). On comparison of the mass spectra of the constituents with Mass database, the compounds were identified in Table 2a and 2b.

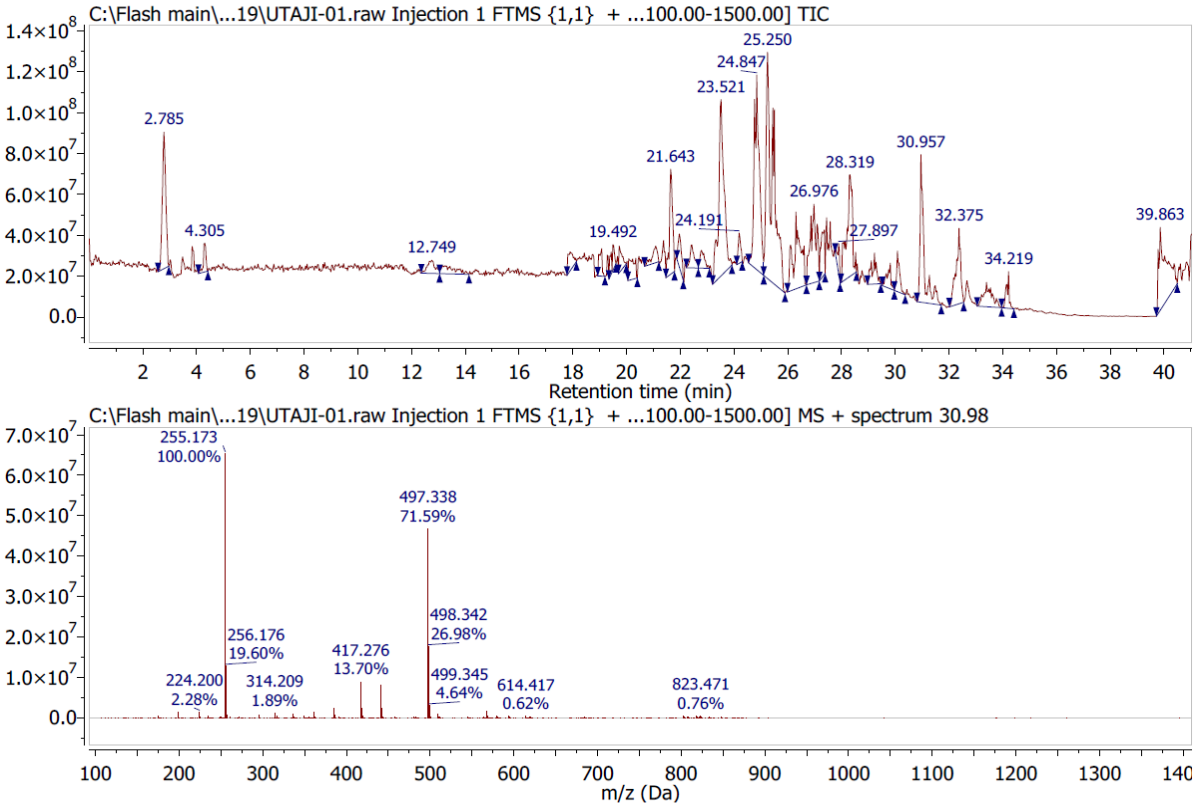


Figure 1: LC-MS Chromatogram of the traditionally made oil of *Canarium schweinfurthii* Fruit

LC-MS analysis of phytoconstituents of the Petroleum ether extract of Canarium schweinfurthii Fruit:

LC-MS chromatogram of the Petroleum ether extract of *Canarium schweinfurthii* fruits showed twenty-eight peaks which indicates the presence of different phytochemical constituents (Figure 2). On comparison of the mass spectra of the constituents with Mass database, the compounds were identified (Table 2a and 2b).

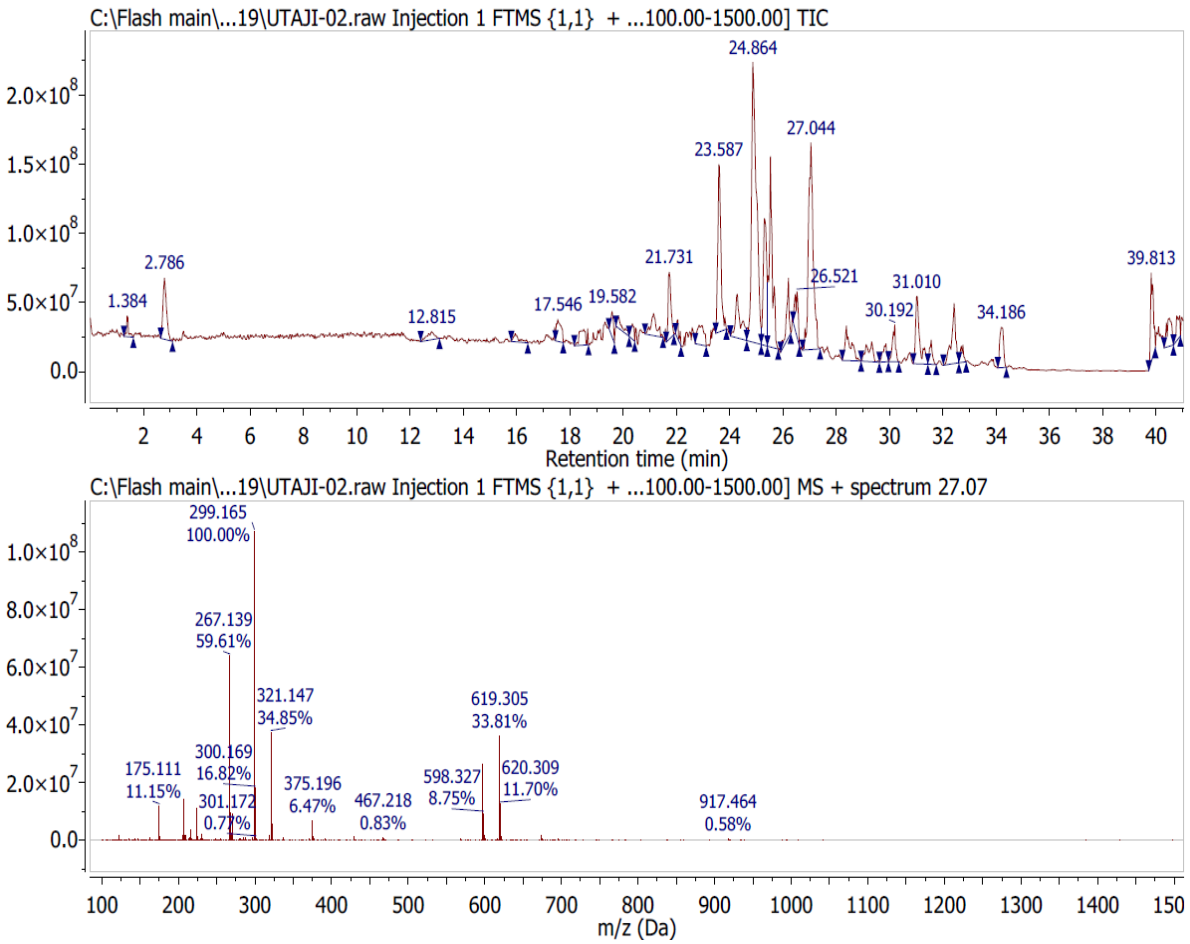


Figure 2: LC-MS Chromatogram of the Petroleum ether extract of *Canarium schweinfurthii* Fruit Marc

LC-MS of the Ethyl acetate extract of Canarium schweinfurthii Fruit Marc:

LC-MS analysis of the ethyl acetate extract showed nine peaks indicating different phytochemical constituents (Figure 3). On comparison of the mass spectra of the constituents with Mass database, the compounds were identified (Table 2a and 2b).

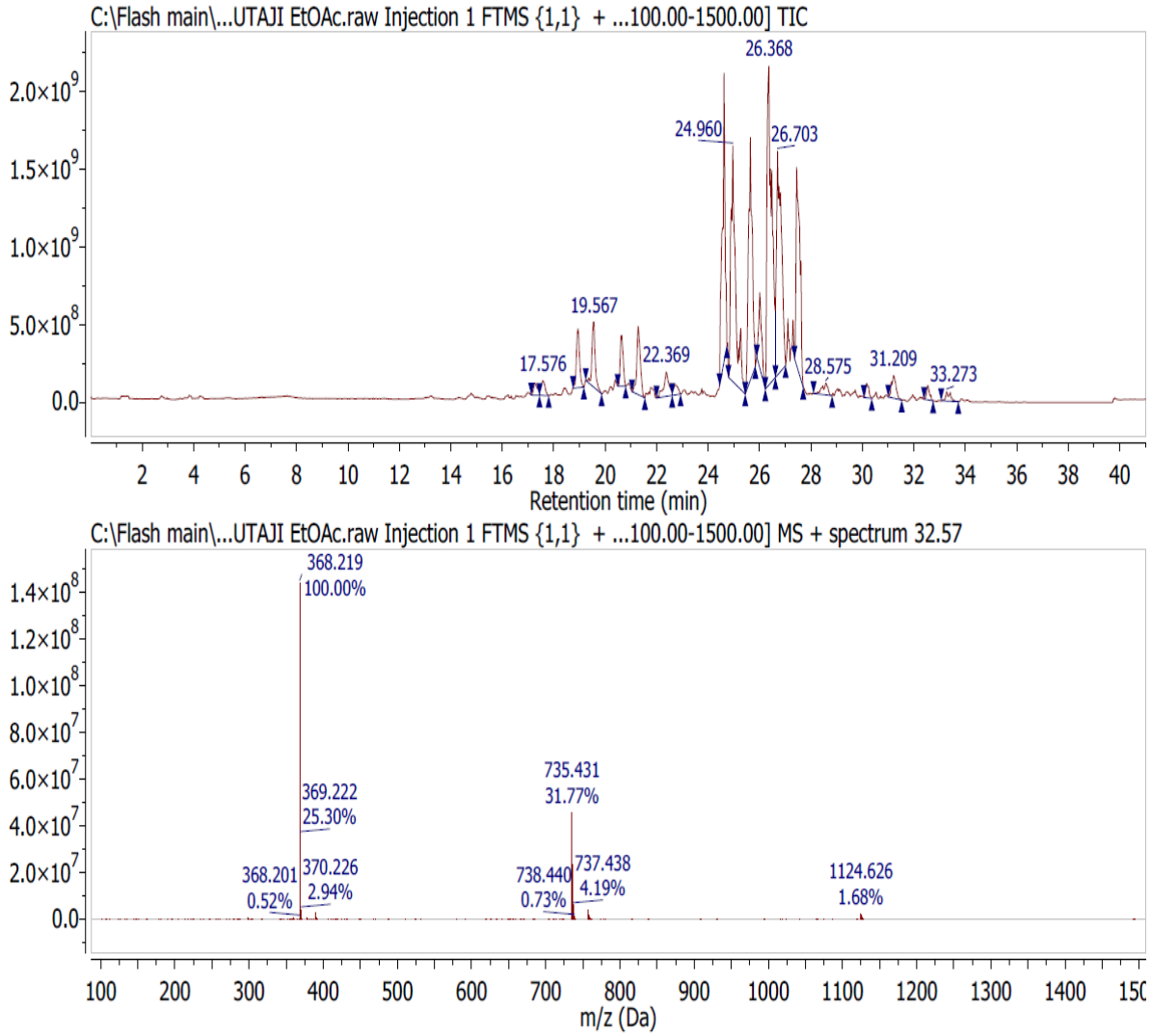


Figure 3: LC/MS chromatogram of the Ethyl acetate extract of Canarium schweinfurthii fruit marc

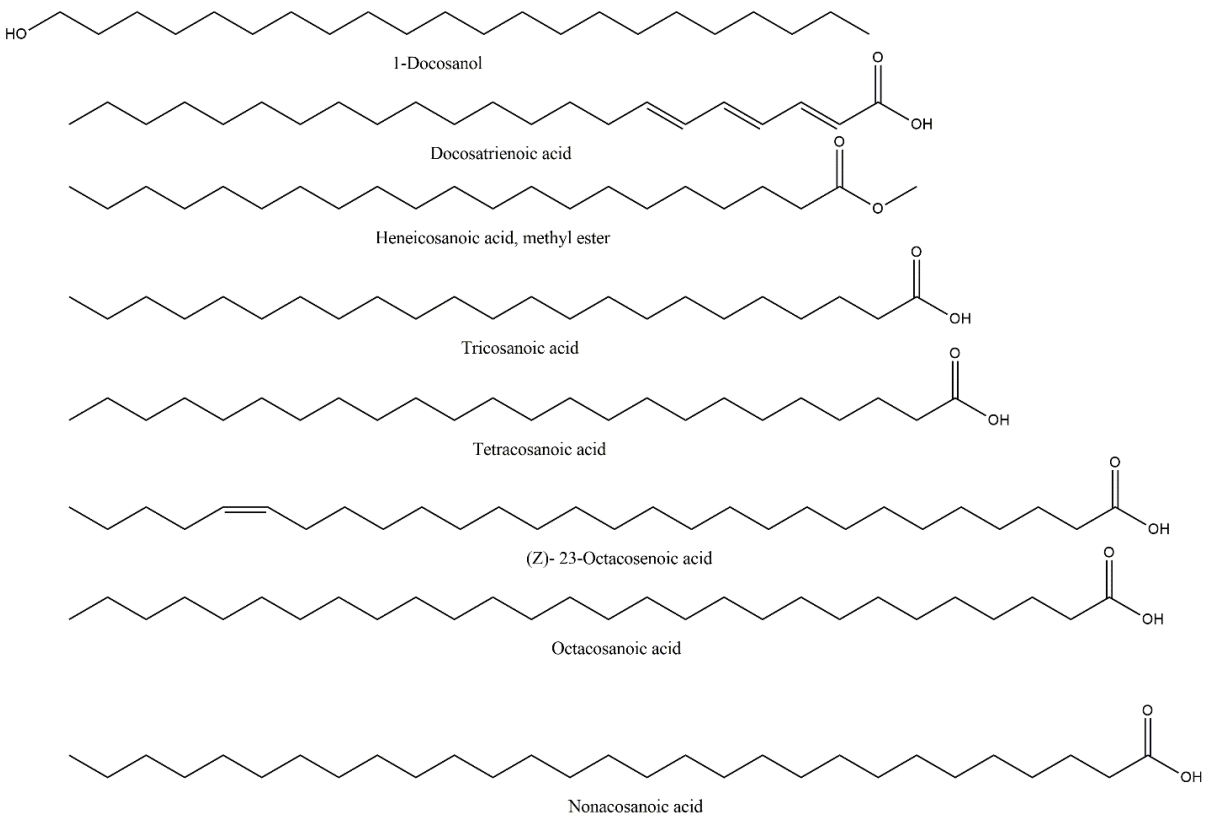


Figure 4: Structures of some of the long chain phytocomponents detected in the traditionally made oil and marc extracts of Canarium schweinfurthii fruits

Table 2a: Phytocomponents in the extracts of the fruits of *Canarium schweinfurthii*

Phytocomponent	Molecular formula	Molecular weight (g/mol)	Extract (Oil/Marc)		
			Traditionally made oil	Petroleum ether	Ethyl acetate
Octenoic acid	$C_8H_{14}O_2$	142.060	+	+	-
Nonenoic acid	$C_9H_{16}O_2$	156.067	-	+	-
α.-Methylcinnamic acid	$C_{10}H_{10}O_2$	162.111	+	-	-
2-Cyclopentene-1-pentanoic acid	$C_{10}H_{16}O_2$	168.012	+	+	-
Decenoic acid	$C_{10}H_{18}O_2$	170.086	+	+	
Undecenoic acid	$C_{11}H_{20}O_2$	184.088	+	+	
2-Cyclopentene-1-heptanoic acid	$C_{12}H_{20}O_2$	196.040	+	+	

Nonadecanol	$C_{19}H_{40}O$	198.039	-	+	-
4-Dodecenoic acid	$C_{12}H_{22}O_2$	198.114	-	+	-
Dodecanoic acid (Lauric acid)	$C_{12}H_{24}O_2$	200.114	-	+	-
Decanedioic acid	$C_{10}H_{18}O_4$	202.109	-	+	-
Tetradecadienoic acid	$C_{14}H_{24}O_2$	224.155	-	-	+
Pentadecanol	$C_{15}H_{32}O$	228.109	+	-	-
10-Pentadecenoic acid	$C_{15}H_{28}O_2$	240.998	+	+	-
2-hexyldecanol	$C_{16}H_{34}O$	242.124	+	+	-
9,12-Hexadecadienoic acid	$C_{16}H_{28}O_2$	252.124	-	+	-
2-Octylcyclopropanepentanoic acid	$C_{16}H_{30}O_2$	254.140	+	-	-
9-Hexadecenoic acid	$C_{16}H_{30}O_2$	254.172	-	+	-
Hexadecenoic acid	$C_{16}H_{30}O_2$	254.173	+	-	-
Heptadecadienoic acid	$C_{17}H_{30}O_2$	266.139	+	+	-
Stearaldehyde	$C_{18}H_{36}O$	268.155	+	-	-
9-Heptadecenoic acid	$C_{17}H_{32}O_2$	268.155	-	+	-
Hexadecanoic acid, methyl ester	$C_{17}H_{34}O_2$	270.134	+	+	-
15-Methylhexadecanoic acid	$C_{17}H_{34}O_2$	270.135	-	+	-
6,9,12-Octadecatrienoic acid	$C_{18}H_{30}O_2$	278.157	-	+	-
9-Octadecenoic acid, (9E)-	$C_{18}H_{34}O_2$	282.171	+	-	-
n-Octadecanoic acid	$C_{18}H_{36}O_2$	284.150	-	+	-
Nonadecanol	$C_{19}H_{40}O$	284.171	+	-	-
1,2-octadecanediol	$C_{18}H_{38}O_2$	286.150	-	+	-

Key:

- = Absent in extract

+ = Present in extract

Table 2b: Phytocomponents in the extracts of the fruits of *Canarium schweinfurthii*

Phytocomponent	Molecular formula	Molecular weight (g/mol)	Extract (Oil/Marc)		
			Traditionally made oil	Petroleum ether	Ethyl acetate
9,11,13-Octadecatrienoic acid, 4-oxo-,	$C_{18}H_{28}O_3$	292.116	+	+	-
9-Octadecenoic acid, 12-hydroxy-, (9Z,12R)	$C_{18}H_{34}O_3$	298.165	-	+	-
9-Octadecenoic acid	$C_{18}H_{34}O_3$	298.199	+	-	-
Octadecanoic acid, 9-hydroxy-	$C_{18}H_{36}O_3$	300.178	+	+	-
5,11,14-Eicosatrienoic Acid	$C_{20}H_{30}O_2$	302.181	+	-	-
Nonadecanoic acid, 18-methyl-	$C_{20}H_{40}O_2$	312.181	-	+	-
9,10-dihydroxy-octadecanoic acid	$C_{18}H_{36}O_4$	316.155	-	-	+
1-Docosanol	$C_{22}H_{46}O$	326.173	-	-	+
11-Eicosenoic acid, 14-hydroxy-	$C_{20}H_{38}O_3$	326.176	-	-	+
Docosatrienoic acid	$C_{22}H_{38}O_2$	334.163	-	+	-
11-Docosenoic acid	$C_{22}H_{42}O_2$	338.230	+	-	-
Heneicosanoic acid, methyl ester	$C_{22}H_{44}O_2$	340.191	-	-	+
Docosanoic acid	$C_{22}H_{44}O_2$	340.191	-	-	+
Tricosanoic acid	$C_{23}H_{46}O_2$	354.224	-	+	+
Tetracosanoic acid	$C_{24}H_{48}O_2$	368.183	-	+	+
(Z)- 23-Octacosenoic acid	$C_{28}H_{54}O_2$	422.211	-	+	-
Octacosanoic acid	$C_{28}H_{56}O_2$	424.227	+	+	-
Nonacosanoic acid	$C_{29}H_{58}O_2$	438.242	-	+	-

Key:

- = Absent in extract

+ = Present in extract

Characterization of isolated compounds:

The pure fractions obtained from column chromatography of the extracts of the marc were subjected to NMR analysis to identify their constituent compounds.

^{1}H-NMR spectrum of UT-ME02 and its characterisations:

The proton NMR spectrum of the methanol extract of the marc of Canarium schweinfurthii fruits (Fig. 5) and subsequent characterization (Table 3) are given below.

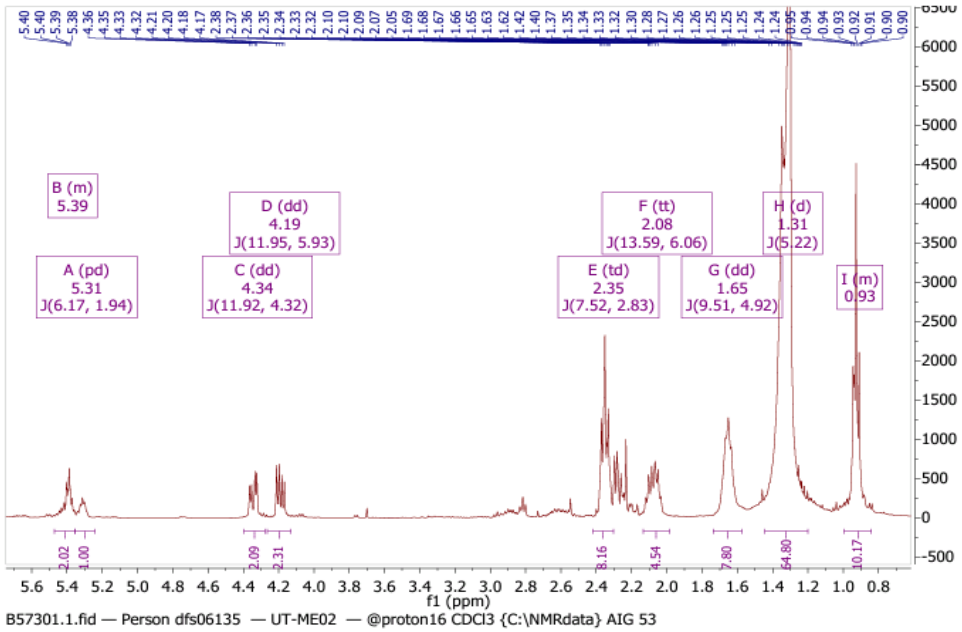


Figure 5: ^{1}H NMR spectrum of compound UT-ME02 isolated from the marc of *Canarium schweinfurthii* fruits

Table 3: ^{1}H-NMR assignment for compounds UT-ME02 isolated from the marc of *Canarium schweinfurthii* fruits

Signal	Position	Chemical shift (ppm)	Reference Chemical shifts (ppm)	Proton	Assignment
A	1"	5.31	5.343[a]	-C**H**OCOR	Glycerol (triglycerides)
B	9,10,12,13	5.39 (m)	5.38[b]	-C**H**=C**H**-	Olefinic (all unsaturated fatty acids)
C	1'	4.34 (dd)	4.340[a]	-C**H$_2$**OCOR-	Glycerol (triglycerides)
D	1	4.19 (dd)	4.095[a]	-C**H$_2$**OCOR-	Glycerol (triglycerides)
E	2	2.35 (td)	2.34[b]	-O-CO-CH$_2$-C**H$_2$**-	All acyl chains

F	8, 11,14	2.08 (tt)	2.08[b]	- C**H**$_2$-CH=CH-	All unsaturated fatty acids
G	3	1.65 (dd)	1.65[b]	-O-CO- C**H**$_2$- CH$_2$-	All acyl chains
H	4,5,6,7,15,16,17	1.31 (d)	1.19-1.42[c]	-(C**H**$_2$)$_n$ -	Methylene
I	18	0.93 (m)	0.88[b]	-C**H**$_3$	Terminal methyl

Key:

dd: double of doublets

m: multiplet

td: triplet of doublets

t: triplet

tt: triplet of triplets

a: Xi et al., 2016.

b: Dos Santos et al., 2017.

c: Nieva-Echevarría et al., 2014

^{13}C-NMR spectrum of UT-ME02 and its characterisations:

The carbon-13 NMR spectrum of the methanol extract of the marc of Canarium schweinfurthii fruits (Fig. 6) and subsequent characterization (Table 4) are given below.

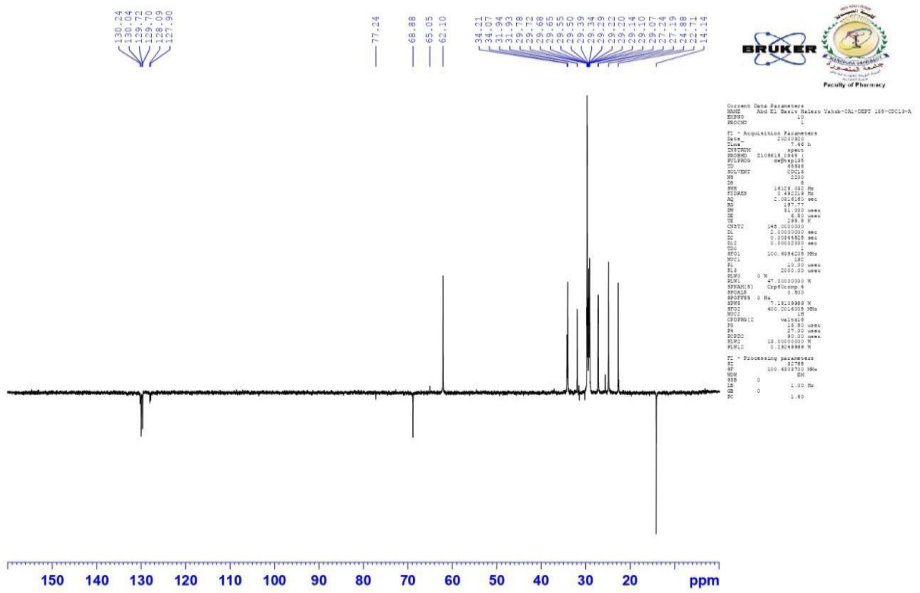
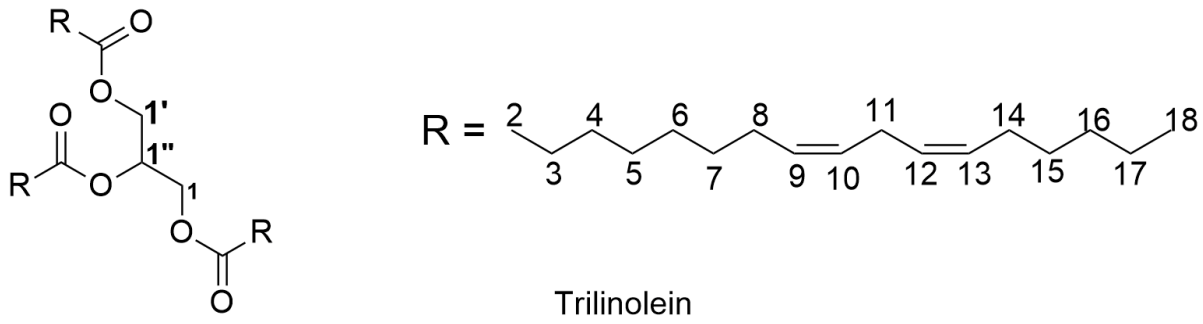


Figure 6: ^{13}C-NMR spectrum of compound UT-ME02 from the marc extract of Canarium schweinfurthii fruits

Table 4: ^{13}C-NMR experimental values and assignment for compound UT-ME02 ME02 from the marc extract *of Canarium schweinfurthii* fruits

Position	UT-ME02 (δ_C, ppm)	Reference δ_C (ppm)		Assignment
		Alemeny, 2002	Thoss *et al.*, 2012	
C-1	173.32	173.23	173.10	Glycerol (triacylglycerols)
C-2	34.06	34.03	34.06	All acyl chains
C-3	24.88	24.85	24.77	All acyl chains
C-4	29.10	29.09	28.95–29.67	All acyl chains
C-5	29.29	29.19	28.95–29.67	All acyl chains
C-6	29.14	29.13	28.95–29.67	All acyl chains
C-7	29.68	29.62	28.95–29.67	All acyl chains
C-8	27.23	27.20	27.11	Allylic
C-9	130.03	129.99	129.56	Oleyl
C-10	128.08	128.08	129.88	Oleyl
C-11	129.71	125.64	129.68	Gondoyl
C-12	127.90	127.91	129.78	Gondoyl
C-13	130.24	130.20	129.72	Erucyl
C-14	27.19	27.21	27.11	Allylic (linoleyl)
C-15	29.38	29.36	29.67	All acyl chains
C-16	31.94	31.54	31.50	Linoleoyl
C-17	22.70	22.59	22.55	All acyl chains
C-18	14.13	14.08	13.96	All acyl chains
Glycerol H-C-O- (1")	68.88	62.91	68.80	Glycerol (triacylglycerols)
Glycerol CH_2O (1,1')	62.10	62.11	61.97	Glycerol (triacylglycerols)

The proposed structure of UT-ME02 (Trilinolein) in the methanol extract of the marc is shown in Fig. 7 below



Trilinolein

Figure 7: Parent chain of the proposed structure of UT-ME02 showing its carbons carrying each proton in their various positions

[1]H-NMR spectrum of UT-EA02 and its characterisations:

The proton NMR spectrum of the Ethyl acetate extract of the marc of *Canarium schweinfurthii* fruits (Fig. 8) and subsequent characterization (Table 5) are given below.

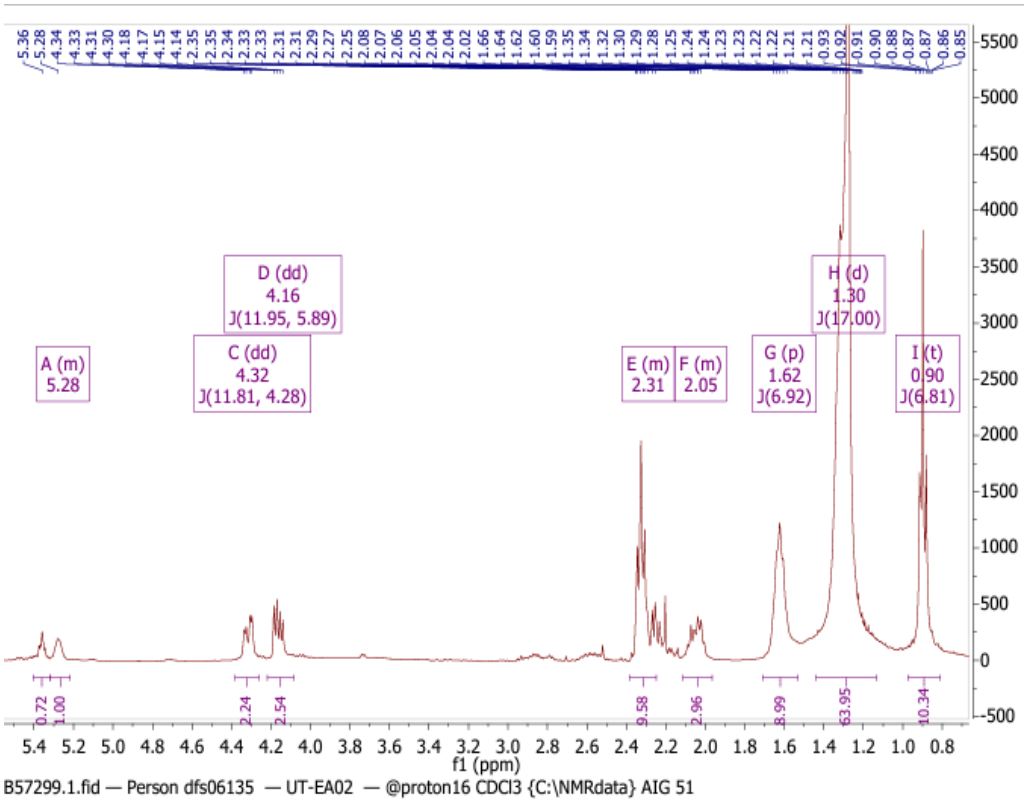


Figure 8: [1]H NMR spectrum of compound UT-EA02 from the marc of *Canarium schweinfurthii* fruits

Table 5: [1H]-NMR assignment for compound UT-EA02 in the ethyl acetate extract of the marc of *Canarium schweinfurthii* fruits

Signal	Position	UT-EA02 Chemical shift (ppm)	Reference	Proton	Assignment
A	1"	5.28 (m)	5.32[a]	-CHOCOR	Glycerol (triglycerides)
B	9,10	5.25(m)	5.25 [a]	-CH=CH-	Olefinic (all unsaturated fatty acids)
C	1'	4.32 (dd)	4.14-4.27 [a]	-CH_2 OCOR	Glycerol (triglycerides)
D	1	4.16(dd)	4.14-4.27 [a]	-CH_2OCOR	Glycerol (triglycerides)
E	2	2.31 (m)	2.27 [a]	-O-CO-CH_2-CH_2	All acyl chains
F	8, 11,12,13,14	2.05 (m)	2.00 [a]	- CH_2-CH=CH	All unsaturated fatty acids

G	3	1.62 (dd)	1.60 [a]	-O-CO- CH_2-CH_2	All acyl chains
H	4,5,6,7,15,16,17	1.30 (d)	1.25[a]	-(CH_2) n-	Methylene
I	18	0.90 (t)	0.86[a]	-CH_3	Terminal methyl

Key:

dd: double of doublets

M: multiplet

t: triplet

d: doublet

a: Thoss *et al.,* 2012.

^{13}C-NMR spectrum of UT-EA02 and its characterisations:

The 13C- NMR spectrum of the Ethyl acetate extract of the marc of Canarium schweinfurthii fruits (Fig. 9) and subsequent characterization (Table 6) are given below.

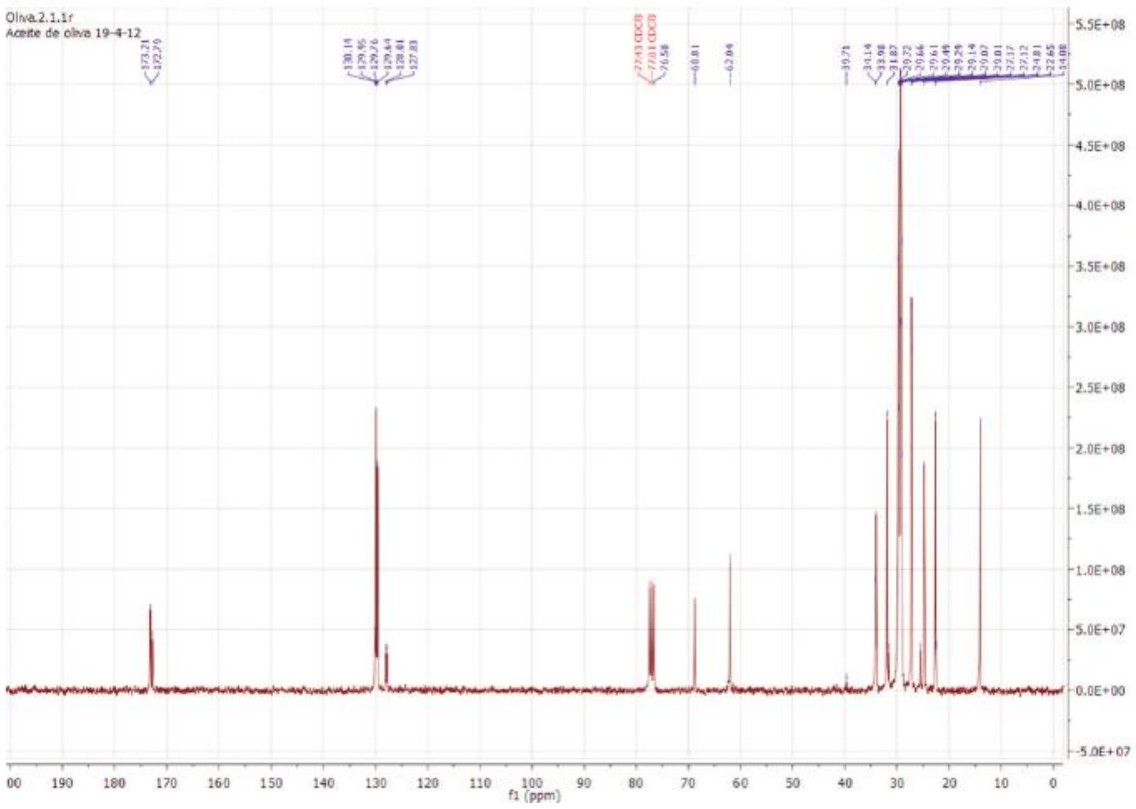


Figure 9: ^{13}C NMR spectrum of compound UT-EA02 in the ethyl acetate extract of the marc of *Canarium schweinfurthii* fruits

Table 6:^{13}C-NMR experimental values and assignment for compound UT-EA02 in the ethyl acetate extract of the marc of *Canarium schweinfurthii* fruits

Position	UT-EA02 Chemical shift (δ_C, ppm)	Reference δ_C (ppm)		Assignment
		Alemeny, 2002	Thoss *et al.*, 2012	
C-1	173.35	173.23	173.10	Glycerol (triacylglycerols)
C-2	34.09	34.03	34.06	All acyl chains
C-3	24.90	24.85	24.77	All acyl chains
C-4	29.50	29.09	28.95–29.67	All acyl chains
C-5	29.29	29.19	28.95–29.67	All acyl chains
C-6	29.66	29.13	28.95–29.67	All acyl chains
C-7	29.68	29.62	28.95–29.67	All acyl chains
C-8	27.23	27.20	27.11	Allylic
C-9	130.03	129.99	129.56	Oleyl
C-10	128.08	128.08	129.88	Oleyl
C-11	129.71	125.64	129.68	Gondoyl
C-12	127.95	127.91	129.78	Gondoyl
C-13	130.24	130.20	129.72	Erucyl
C-14	27.19	27.21	27.11	Allylic (linoleyl)
C-15	29.38	29.36	29.67	All acyl chains
C-16	31.94	31.54	31.50	Linoleoyl
C-17	22.70	22.59	22.55	All acyl chains
C-18	14.10	14.08	13.96	All acyl chains
Glycerol H-C-O-(1")	68.88	62.91	68.80	Glycerol (triacylglycerols
Glycerol CH_2O 1, 1'	62.10	62.11	61.97	Glycerol (triacylglycerols)

The proposed structure of Compound UT-EA02 (Triolein) is shown in Fig. 10 with carbons of its parent chain carrying its protons.

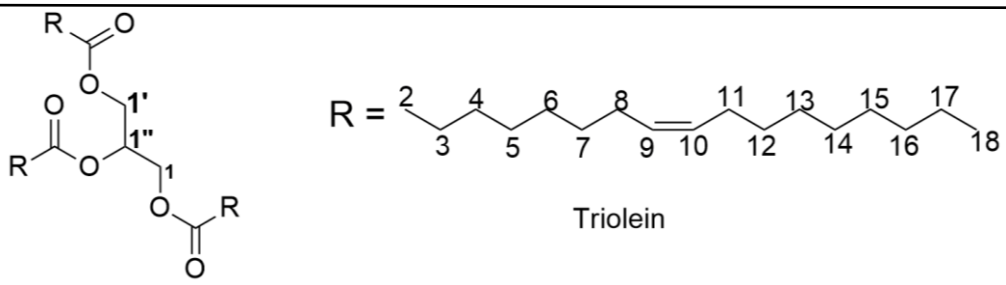


Figure 10: Proposed structure of Compound UT-EA02 (Triolein) in the ethyl acetate extract of the marc *of Canarium schweinfurthii* fruits

^{1}H-NMR spectrum of UT-PE01 and its characterisations:

The proton NMR spectrum of the pet. Ether extract of the marc of *Canarium schweinfurthii* fruits (Fig.11) and subsequent characterization (Table 7) are given below.

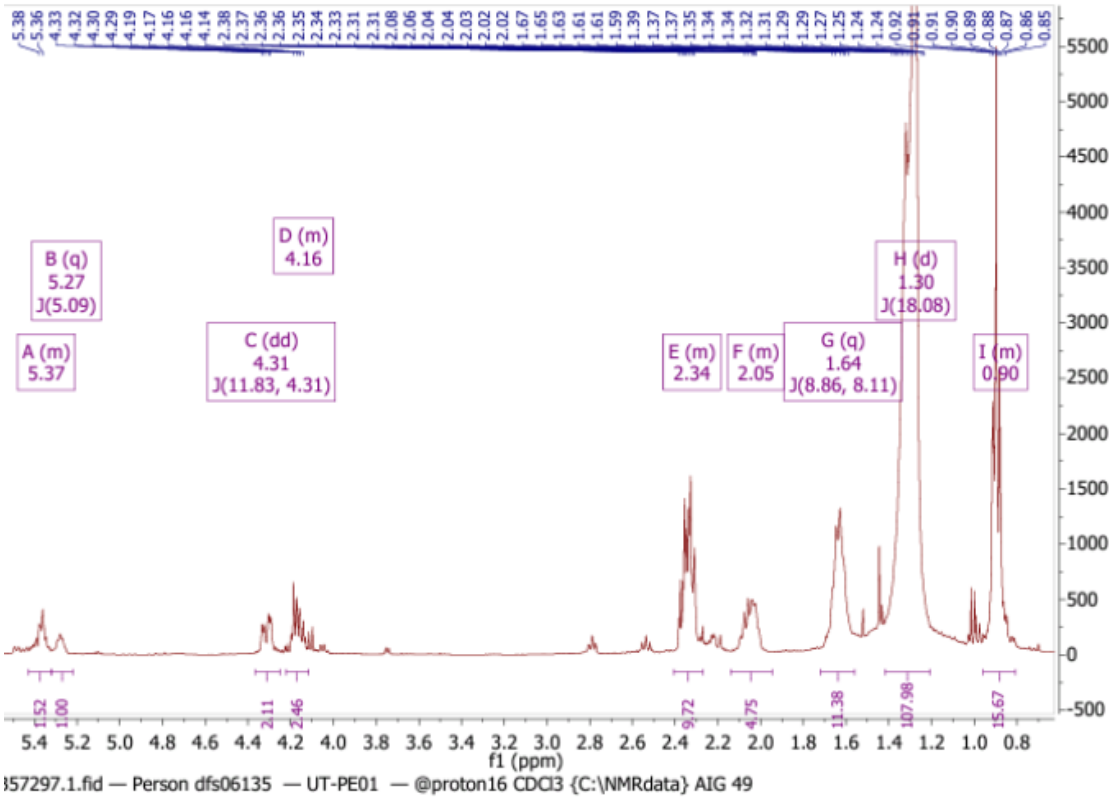


Figure 11 : ^{1}H NMR spectrum of compound UT-PE01 in the Pet. ether extract of the marc of *Canarium schweinfurthii* fruits

Table 7: ^{1}H-NMR assignment for compound UT-PE01 in the petroleum ether extract of the marc of *Canarium schweinfurthii* fruits

Signal	Position	UT-PE01 Chemical shift (ppm)	Literature[a]	Proton	Assignment
A	1"	5.37 (m)	5.32	-C**H**OCOR	Glycerol (triglycerides)
B	9,10	5.27(q)	5.25	-C**H**=C**H**-	Olefinic (all unsaturated fatty acids)
C	1'	4.31 (dd)	4.14-4.27	-C**H$_2$** OCOR	Glycerol (triglycerides)
D	1	4.16(m)	4.14-4.27	-C**H$_2$**OCOR	Glycerol (triglycerides)
E	2	2.41 (m)	2.27	-O-CO-C**H$_2$**-CH$_2$	All acyl chains
F	8, 11,12,13,14	2.05 (m)	2.00	- C**H$_2$**-CH=CH	All unsaturated fatty acids
G	3	1.64 (q)	1.60	-O-CO- CH$_2$-C**H$_2$**	All acyl chains
H	4,5,6,7,15,16,17	1.30 (d)	1.25	-(C**H$_2$**)n-	Methylene
I	18	0.90 (t)	0.86	-C**H$_3$**	Terminal methyl

Key: a: Thoss et al., 2012.

^{13}C-NMR spectrum of UT-PE01 and its characterisations:

The proton NMR spectrum of the pet. ether extract of the marc of Canarium schweinfurthii fruits (Fig.12) and subsequent characterization (Table 8) are given below.

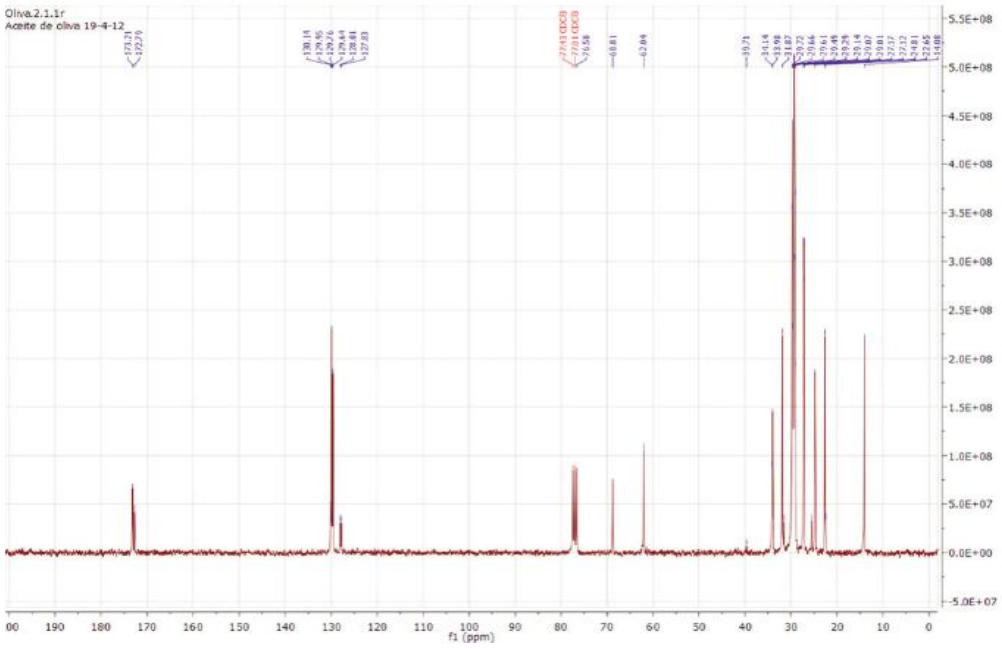
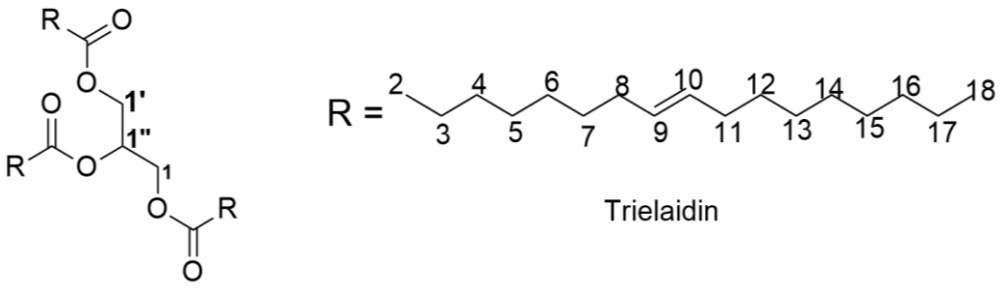


Figure 12: ^{13}C NMR spectrum of the isolated compound UT-PE01 in the Pet. ether extract of the marc of *Canarium schweinfurthii* fruits

Table 8: ^{13}C-NMR experimental values and assignment for compound UT-PE01 in the Pet. ether extract of the marc of *Canarium schweinfurthii* fruits

Position	UT-ME02 (δ_C, ppm)	Literature δ_C (ppm)		Assignment
		Alemeny, 2002	Thoss *et al.*, 2012	
C1	173.35	173.23	173.10	Glycerol (triacylglycerols)
C2	34.09	34.03	34.06	All acyl chains
C3	24.90	24.85	24.77	All acyl chains
C4	29.50	29.09	28.95–29.67	All acyl chains
C5	29.29	29.19	28.95–29.67	All acyl chains
C6	29.66	29.13	28.95–29.67	All acyl chains
C7	29.68	29.62	28.95–29.67	All acyl chains
C8	27.23	27.20	27.11	Allylic
C9	130.03	129.99	129.56	Oleyl
C10	128.08	128.08	129.88	Oleyl
C11	29.71	25.64	129.68	Gondoyl
C12	127.95	127.91	129.78	Gondoyl
C13	130.24	130.20	129.72	Erucyl
C14	27.19	27.21	27.11	Allylic (linoleyl)
C15	29.38	29.36	29.67	All acyl chains
C16	31.94	31.54	31.50	Linoleoyl
C17	22.70	22.59	22.55	All acyl chains
C18	14.10	14.08	13.96	All acyl chains
Glycerol H-C-O-(1")	68.88	62.91	68.80	Glycerol (triacylglycerols
Glycerol CH_2O 1, 1'	62.10	62.11	61.97	Glycerol (triacylglycerols)

The proposed sstructure of Compound UT-PE01 (Trielaidin) in the petroleum ether extract of the marc (Fig. 13) with carbons of its parent chain carrying each proton.



Trielaidin

Figure 13: Compound UT-PE01(Trielaidin) with carbons of its parent chain carrying each proton

DISCUSSION:

Yield of oils and extracts:

The traditionally made oil of *Canarium Schweinfurthii* gave a yield of 42.08%. This is higher than the 35.29% reported by Olawale, 2012 [4] using Soxhlet extraction and 34.04% reported by Mohammed et al., 2015. [5] using solvent extraction for the pulp. The higher yield of the oil using the traditional method makes it a preferred method if high yield is desired. Also, the much lower yield of 1.88%, 1.60% and 2.26% of the solvent extracts of the marc using petroleum ether, ethyl acetate and methanol attests to the fact that almost all of the oil constituents of the fruit pulp may have been extracted during the traditional method hence, the low yield of the solvent extracts.

Chemical composition of the traditionally made oil of Canarium schweinfurthii fruits

Fatty acid components identified by LC-MS were 17 in the oil and the results showed that 17.7% of the fatty acids were saturated, 58.8% were monounsaturated and 23.5% were polyunsaturated. This is in contrast to the 12 fatty acids (50% saturated and 50% unsaturated) reported by Kiin-Kabari et al., 2020 [3] when they employed a GC-MS technique. This variation in the percentage could be attributed to the analytical method employed in the study. Ijeoma and Tochukwu, (2020) [6] reported only six fatty acids with 36.51% saturated and 18.26% unsaturated fatty acids. Fatty acid methyl esters made up 45.2% in a study conducted using GC-MS as the analytical technique. The samples were found to be rich in fatty acids and the major ones identified in the traditionally made oil had in order of abundance, palmitoleic acid < 9-hydroxy linoleic acid < cetoleic acid < α-methylcinnamic acid < caprylic acid and this is in contrast to the order caprylic acid < stearic acid < palmitioleic acid < linoleic acid < oleic acid < palmitic acid and caprylic acid < palmitic acid < stearic acid < oleic acid < linoleic acid (C18:2) < linolenic acid reported by Anyalogbu et al., 2017 [8] for Canarium schweinfurthii fruit pulp.

Chemical composition of the petroleum ether extract of the marc Canarium schweinfurthii fruit:

The phytoconstituents in the petroleum ether extract of the flaky husk identified by LC-MS revealed a total of 27 fatty acids with 48.1% monounsaturated, 22.2% polyunsaturated and 29.6% saturated. The petroleum ether extract of the marc was found to be rich in fatty acids and the major ones identified in the traditionally extracted oil were, in order of abundance, 9-hydroxystearic acid < palmitoleic acid < pentadecylic

acid < linderic acid < capric acid < caprylic acid in contrast to the order of abundance reported by Anyalogbu et al., 2017 [8].

Chemical composition of the ethyl acetate extract of the marc Canarium schweinfurthii fruit:

The phytochemicals in the ethyl acetate extract of the flaky husk identified by LC-MS analysis revealed a total of six fatty acids which were majorly saturated (66.67%) while monounsaturated and polyunsaturated fatty acids accounted for 16.67%. The fatty acids, megatomic acid, 9, 10-dihydroxystearic acid, lesquerolic acid, behenic acid, tricosylic acid, lignoceric acid were in the same amount of abundance.

^{1}H NMR spectra of compounds UT- ME02, UT-EA02 and UT-PE01 from the marc Canarium schweinfurthii fruit

The ^{1}H NMR spectra for UT- ME02, UT-EA02 and UT-PE01 all showed nine signals; signals A - I (Fig. 5, 8 and 11). Band A showed a signal for glycerol (-CHOCOR-) at 5.3 ppm (1H; H-1'') while Band B showed a signal of an olefinic proton (-CH=CH-) on the first α-C chain at 5.4, 5.2 and 5.3 respectively (2H, H-9, H-10, H-12, and H-13). Band C showed a glycerol moiety (-CH2OCOR-) on the third α′-C at were all at 4.3 ppm (1H; H-1′) while a peak also for glycerol (-CH2OCOR-) at 4.2 respectively (1H; H-1) was observed in band D. A peak of methylene group α to the acyl group (-O-CO-CH2-CH2-) at 2.4, 2.3 and 2.4 respectively (1H, H-2) is observed on band E while band F showed signals for that of an allylic methylene group (-CH2-CH=CH-) all at 2.1 respectively (2H, H-8, H-11 and H-14). Band G signal was identified as a methylene group βeta to the acyl group (-O-CO-CH2-CH2-) at 1.7, 1.6 and 1.6 respectively (1H; H-3) while band H gave a signal peak of a methylene group – (CH2)n – all at 1.3 (1H; H-4, H-5, H-6, H-7, H-15, H-16, and H-17) and band I signal was a terminal methyl group (-CH3) all at 0.9 (1H; H-18). These chemical shifts correspond to that of Trilinolein, Triolein and Trielaidin respectively. This finding in compound UT-ME02 is in agreement with the report of Abdubasit et al., 2021 [16] who reported similar proton shifts from methanol extract of the roots of Cyphostemma adenocaule and Nieva-Echevarría et al., 2014 [17] who reported a method based on 1H NMR spectral data useful to evaluate the hydrolysis level in complex lipid mixtures while results in compound UT-EA02 and UT-PE01 is in agreement with a report by Thoss et al., 2012 [20] who reported similar chemical shifts for triglyceride in British Bluebell seed oil.

^{13}C NMR spectra of compound UT- ME02, UT-EA02 and UT-PE01 from the marc Canarium schweinfurthii fruit:

The ^{13}C NMR for UT- ME02, UT-EA02 and UT-PE01 (Fig. 6, 9 and 12) all showed recognizable signals for the carbonyl ester of triacyglycerols at C 173.3 ppm assigned to C-1. Acyl moiety signals at 34.06 - 34.1 and 24.88 - 24.9 ppm were assigned to C-2 and C-3 respectively while signals of acyl chains were detected at chemical shifts between 29.1 – 29.7 assigned to C-4, C-5, C-6 and C-7. Allylic signal at 27.2 was assigned to C-8 while signals at 130.0 and 128.1 were assigned to C-9 and C-10 indicating oleyl moieties. Signals at 129.7 and 127.9 were assigned to C-11 and C-12 indicating gondoyl moieties while erucyl moiety signal at 130.2 was assigned to C-13 and allylic (linoleyl) moiety at 27.2 was assigned to C14. Signal at 29.4 ppm was assigned to C-15 showing an acyl chain while linoleyl moiety at 31.9 was assigned to C-16. Signals at 22.7 and 14.1 were assigned to C-17 and C-18 both of acyl chains. Signals at 68.9 was assigned to H-C-O- (1") and 62.1 was assigned to CH2O (1, 1') both of which are glycerol moieties. These assignments of carbon signals in the 13C NMR are in agreement with those reported by Alemany, 2002 [19] and Thoss et al., 2012 [20].

CONCLUSION:

A study of the traditionally made oil and solvent extracts of the marc using LC - MS found high amounts of unsaturated fatty acids (82.4 %). Similarly, extracts of the marc had 70.4 % unsaturated fatty acids which is suitable for use as a drying oil but the ethyl acetate extract had 66.67 % of saturated fatty acids and so may be good for use as a semi drying oil. The research future prospects may include the use of the marc as supplement in animal feed formulations to further establish the possibility of using it as a valuable source of lipids which could be investigated in the growth performance of the animals, feed conversion ratio and cost effectiveness of its use as animal feed supplement.

CONFLICT OF INTEREST:

The authors declare no conflict of interest.

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