

Characterization of *Neocarya Macrophylla* Seed Oil using Gas Chromatography-Mass Spectrometry (GC-MS) and Fourier Transform Infra-Red (FT-IR) Techniques

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Authors' contributions

This first report research was carried out in collaboration among all authors. Author AAW initiated the concept of the research, conducted the literature search, collected the plant materials, performed the laboratory work, formulated recommendations and prepared the write-up for publication. Authors LGH, LJB, AAO, RUU and GAB identified some issues, formulated recommendations and reviewed the paper. All authors read and approved the final manuscript.

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ABSTRACT

Aim: Modern researches described incorporating gingerbread plum (*Neocarya macrophylla*) kernel oils into food, cosmetics and pharmaceutical products by exploiting its physicochemical properties.

Study Design: Experimental and instrumental study was done to determine the general and the saturated fatty acids present in the seed oil and its suitability for industrial applications. This study examined the fatty acids by qualitative determination from hexane extracts of *Neocarya macrophylla* seed using GC-MS.

Materials and Methods: Indigenous *Neocarya macrophylla* seeds obtained from Gingerbread plum tree were collected in the Month of August from Birnin Kebbi, Kebbi State, Nigeria. Soxhlet extraction method was used for the oil extraction. The GC-MS analysis was by coupling system of Shimadzu QP2010 series gas chromatography with Shimadzu QP2010 plus mass spectroscopy detector (GCMS). For the FT-IR analysis, software of OMNIC operating system (Version 7.0 Thermo Nicolet) was connected to Fourier Transform Infrared Spectrometer Nicolet 8400S equipped with a detector of deuterated triglycine sulphate (DTGS).

Results: The results of the GC-MS obtained from the analysis showed that the oil contain the following fatty acids; myristic acid, palmitic acid, stearic acid, palmitoleic acid, elaidic acid, oleic acid, erucic acid, behenic acid, heneicosanoic, icosatetraenoate and eicosatrienoic acid. The results of the FT-IR analysis showed 3475.84 cm^{-1} Which corresponds to O-H of carboxylic acid 2934.79 cm^{-1} , 2869.21 cm^{-1} and 2037.86 cm^{-1} The asymmetrical and symmetrical modes of vibration is at the region of unsaturated fatty acid. =O Corresponds to ester carbonyl group which showed a very strong and sharp band at 1740.81 cm^{-1} . The bending vibrations of the CH_2 and CH_3 aliphatic groups, and the in-plane bending vibration of CH cis-olefinic groups are seen at around 1358.9 cm^{-1} . At 721.4 cm^{-1} and 442.68 cm^{-1} , showed the frequencies of the in- and out-of plane rocking of the cis-olefinic CH_2 group and C – O stretching vibration (e.g in triacylglycerols).

Conclusion: These results showed the potential of this oil in cosmetic industry.

Keywords: *Neocarya macrophylla*; seed oil; GC-MS; FT-IR; fatty acids; cosmetics.

1. INTRODUCTION

Gingerbread plum (*Neocarya macrophylla*) belongs to *chrysobalanaceae* family. The tree grow in arid and semiarid parts of West Africa and Central America [1]. It is known in Hausa Language as 'Gawasa' [2]. It is semi-cultivated [3]. Soaps are made from seed oils of such plants [4]. Physical and chemical properties as well as cold saponification of gingerbread plum (*Neocarya macrophylla*) seed oil was reported [5]. Gingerbread plum kernel oils can be incorporated into food, cosmetics and

pharmaceutical products [6]. Quality characteristics and antioxidant properties of gingerbread plum (*Neocarya macrophylla*) seed oils was also reported [7]. To provide another source of greener energy $\text{MgO/Al}_2\text{O}_3$ catalyst was used to produce biodiesel from gingerbread plum (*Parinari macrophylla*) seed oil [8]. This research work is aimed at fatty acid analysis of *Neocarya macrophylla* seed oil by qualitative method and characterization by Gas Chromatography-Mass Spectrometry (GC-MS) Analysis and Fourier Transform Infra-Red (FT-IR).



Fig. 1. *Neocarya macrophylla* seeds



Fig. 2. *Neocarya macrophylla* seed oil

2. MATERIALS AND METHODS

2.1 Seed Material

Indigenous *Neocarya macrophylla* seeds obtained from Gingerbread plum tree were collected in the Month of August from Birnin Kebbi, Kebbi State, Nigeria. The other parts of the plant collected were the leaves, the fruits and the flowers for the purpose of identification. The plant was identified and authenticated by a Botanist at the Biological Sciences Department, Bayero University, and Kano (BUK) Nigeria. The plant was identified and confirmed by taxonomist and use of documented literature [9]. Comparison with voucher specimen (voucher No. 175) kept at the Herbarium of the Department of Biological Sciences was done. The seeds were selected by cleaning and discarding of the damaged ones. The seeds were de-shelled, dried and pulverized using laboratory plastic pestle and Mortar prior to extraction.

2.2 Oil Extraction

Extraction of 35 g of pulverized seeds of *Neocarya macrophylla* was carried out using soxhlet extraction method boiling with n-hexane (between 40–60°C) for six hours repeatedly until required amount was obtained. Reduced temperature and pressure and refluxing at 70°C was done to remove excess solvent from the oil and was placed on water bath for more solvent expulsion. Complete distilling was done on a heating mantle to recover most of the solvent. The oil extract was stored in freezer at -2°C for subsequent physicochemical analysis.

2.3 Oil Yield

The oil was transferred into measuring cylinder. The measuring cylinder was then placed over water bath for complete evaporation of solvent for about 2-3 hours in accordance with the method reported [10]. The weight of the oil was determined and expressed in percentage.

$$\text{Oil content (\%)} = \frac{\text{Oil weight}}{\text{Sample weight}} \times 100$$

2.4 FT-IR Analysis

Software of OMNIC operating system (Version 7.0 Thermo Nicolet) was connected to Fourier Transform Infrared Spectrometer Nicolet 8400S equipped with a detector of deuterated triglycine

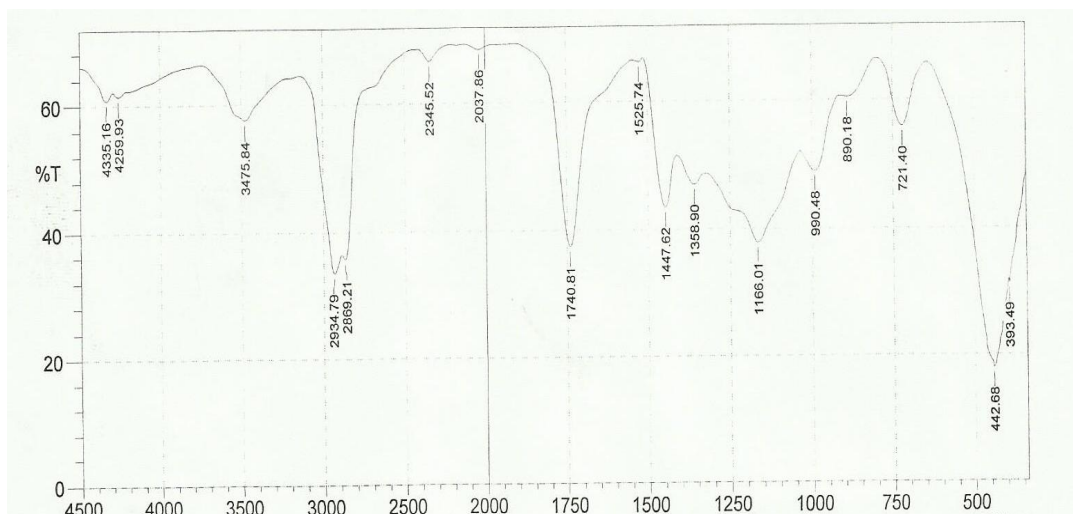
sulphate (DTGS). FT-IR spectra of samples was obtained using this system. KBr disc was made to contact with the sample to collect the FT-IR spectra in frequency 4500-400 cm^{-1} by coadding 32 scans and at resolution of 4 cm^{-1} . The spectrum was rationed against a background spectrum. A new reference background spectrum was detected in each scan. Triplicate record of the spectra was presented as absorbance values at each data point [11].

2.5 GC-MS Analysis

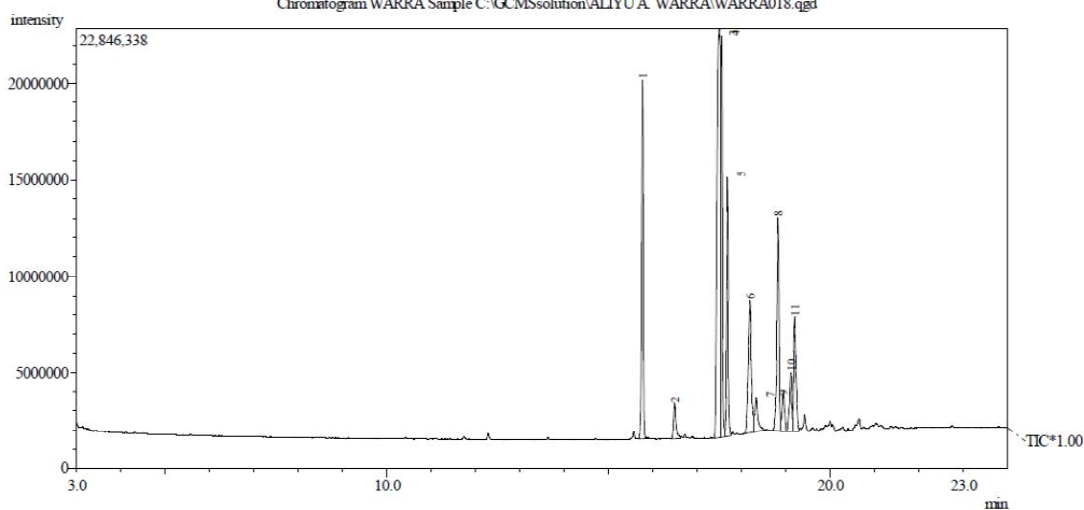
Characterization of *Neocarya macrophylla* oil sample was performed using a coupling system of Shimadzu QP2010 series gas chromatography with Shimadzu QP2010 plus mass spectroscopy detector (GCMS). Temperature programmed was ranged between 70°C to 280°C using Helium as a carrier gas. For the mass spectroscopy the injection volume was 2 μL with injection temperature of 250°C and a column flow of 1.80 mL/min for the GC, ACQ mode scanner with scan range of 30-700 amu at the speed of 1478rp was used. The mass spectra were compared with the NIST05 mass spectral library [11].

3. RESULTS AND DISCUSSION

Band at 4259.93 is aliphatic C-H stretching, Absorption band at 3475 cm^{-1} indicated the presence of O-H of carboxylic acids at 3475.84 cm^{-1} . A strong and sharp spectra band was exhibited at the region of unsaturated fatty acid 2934.79 cm^{-1} , 2869.21 cm^{-1} and 2037.86 cm^{-1} which is the asymmetrical and symmetrical modes of vibration, this include CH Stretching vibration of Cis double bond within unsaturated fatty acyl ester asymmetric $-\text{CH}_2-$, symmetric $-\text{CH}_3$ and $-\text{CH}_2-$ stretching vibrations. very strong and sharp band due to ester carbonyl functional group i.e C=O stretching vibrations is seen as at 1740.81 cm^{-1} [12] C-O vibrations (e.g. in the $-\text{COOR}$ /aromatic $-\text{C}=\text{C}$ stretching vibrations) are seen 1447.62 cm^{-1} (Rsc,1992) and (O- CH_2 -C) The bending vibrations of the CH_2 and CH_3 aliphatic groups, C-CO-O-, and the in-plane bending vibration of CH cis-olefinic groups are seen at around 1358.9 cm^{-1} , respectively. The vibration frequency at, 1166.01 cm^{-1} is finger print of the stretching vibration of the C-O ester group. The band around 990.48 cm^{-1} shows trans out of plane CH=CH. The frequencies of the in- and out-of plane rocking of the cis- olefinic CH_2 group and C - O stretching Vibration (e.g. in triacylglycerols), are at 721.4 cm^{-1} and 442.68 cm^{-1} .

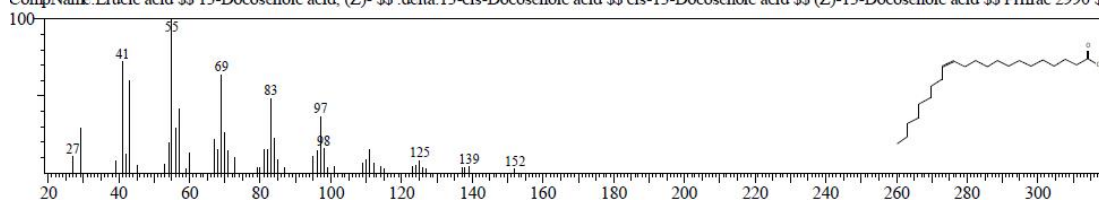


Chromatogram WARRA Sample C:\GCMSolution\ALIYU A WARRA\WARRA018.qgd

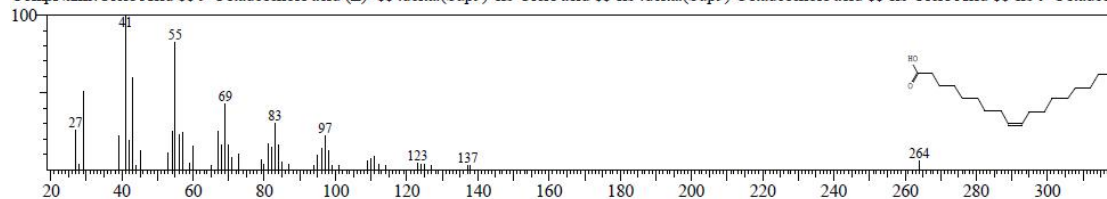


SI:92 Formula:C22H42O2 CAS:112-86-7 MolWeight:338 RetIndex:2572

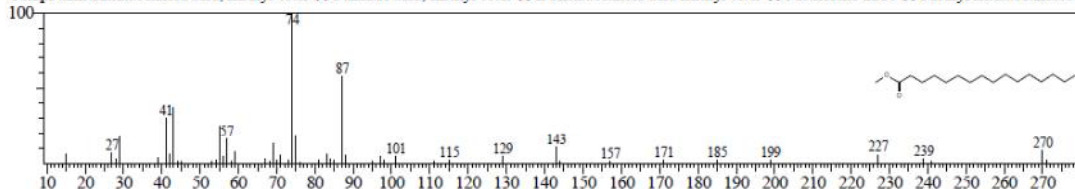
CompName:Erucic acid \$\$ 13-Docosenoic acid, (Z)- \$\$.delta.13-cis-Docosenoic acid \$\$ cis-13-Docosenoic acid \$\$ (Z)-13-Docosenoic acid \$\$ Prifrac 2990 \$\$



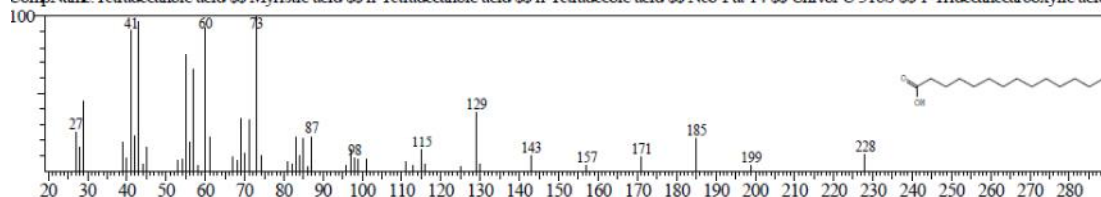
CompName:Oleic Acid \$\$ 9-Octadecenoic acid (Z)- \$\$.delta.(Sup9)-cis-Oleic acid \$\$ cis-.delta.(Sup9)-Octadecenoic acid \$\$ cis-Oleic Acid \$\$ cis-9-Octadecenoic acid



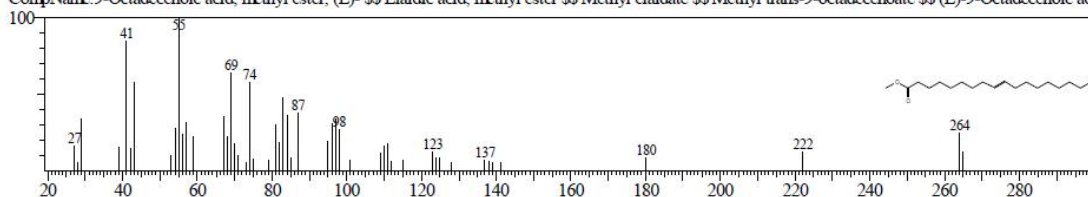
SI:92 Formula:C17H34O2 CAS:112-39-0 MolWeight:270 RetIndex:1878
 CompName:Hexadecanoic acid, methyl ester \$\$ Palmitic acid, methyl ester \$\$ n-Hexadecanoic acid methyl ester \$\$ Metholene 2216 \$\$ Methyl hexadecanoate \$



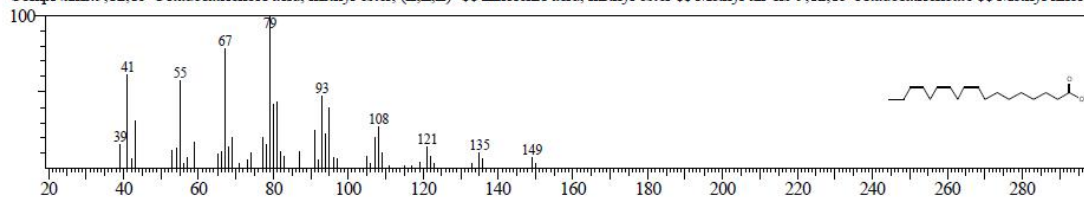
SI:90 Formula:C14H28O2 CAS:544-63-8 MolWeight:228 RetIndex:1769
 CompName:Tetradecanoic acid \$\$ Myristic acid \$\$ n-Tetradecanoic acid \$\$ n-Tetradecoic acid \$\$ Neo-Fat 14 \$\$ Univol U 316S \$\$ 1-Tridecanecarboxylic acid



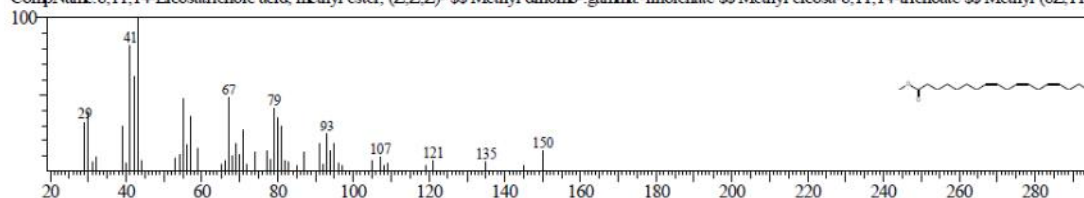
SI:86 Formula:C19H36O2 CAS:1937-62-8 MolWeight:296 RetIndex:2085
 CompName:9-Octadecenoic acid, methyl ester, (E)- \$\$ Elaidic acid, methyl ester \$\$ Methyl elaidate \$\$ Methyl trans-9-octadecenoate \$\$ (E)-9-Octadecenoic aci



SI:89 Formula:C19H32O2 CAS:301-00-8 MolWeight:292 RetIndex:2101
 CompName:9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)- \$\$ Linolenic acid, methyl ester \$\$ Methyl all-cis-9,12,15-octadecatrienoate \$\$ Methyl linolen



SI:71 Formula:C21H36O2 CAS:21061-10-9 MolWeight:320 RetIndex:2300
 CompName:8,11,14-Eicosatrienoic acid, methyl ester, (Z,Z,Z)- \$\$ Methyl dihomo-gamma-linolenate \$\$ Methyl eicosa-8,11,14-trienoate \$\$ Methyl (8Z,11Z,14



Hit#:1 Entry:22977 Library:NIST05s.LIB
 SI:89 Formula:C18H36O2 CAS:57-11-4 MolWeight:284 RetIndex:2167
 CompName:Octadecanoic acid \$\$ Stearic acid \$\$ n-Octadecanoic acid \$\$ Humko Industriene R \$\$ Hydrofol Acid 150 \$\$ Hystrene S-97 \$\$ Hystrene T-70 \$\$ Hys

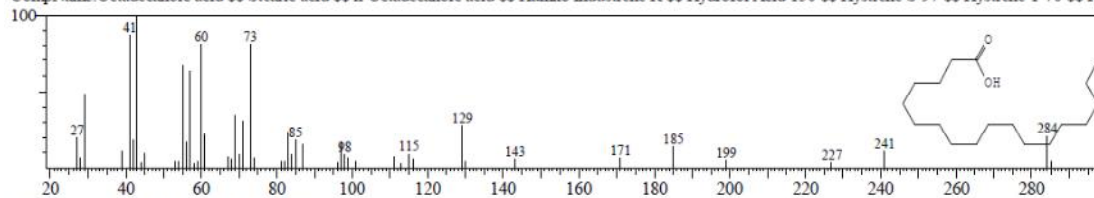


Table 1. FTIR results of oil extract from *Neocarya macrophylla* seed

Characteristics wave number (cm ⁻¹)	Intensity	Remark
4259.93	M	Aliphatic C-H Stretch
3475.84	S	Stretching vibration for –OH groups.
2934.79	M	CH Stretching Vibration of Cis double bond within unsaturated fatty acyl ester
2869.21	M	Asymmetric –CH ₂ –, symmetric –CH ₃
2345.52	S	CH Stretching Vibration of Cis double bond
1740.81	M	C=O stretching vibrations
1521.74	S	C–O vibrations
1166.01	M	–CH ₂ –wagging and twisting vibrations
990.48	M	Trans out of plane CH=CH
721.4	S	C – O stretching Vibration(e.g. in triacylglycerols),
442.68	W	

W= Weak band,S= Strong band and M= moderate band

Table 2. Major fatty acids derived from oil of *Neocarya macrophylla* seed

S/N	Name of fatty acid
	Saturated (SFA)
1	Palmitic acid
2	Stearic acid
3	Behenic acid
4	Heneicosanoic
5	Myristic acid
	Monounsaturated (MUFA)
6	Palmitoleic
7	Oleic acid
8	Erucic acid
	Polyunsaturated (PUFA)
9	Eicosatrienoic acid
10	Icosatetraenoate
11	Elaidic acid

The major fatty acids derived from oil of *Neocarya macrophylla* seed are; Saturated fatty acids (SFA); Palmitic acid, Stearic acid, Behenic acid, Heneicosanoic, Myristic acid, monounsaturated (MUFA); Palmitoleic, Oleic acid, Erucic acid, Polyunsaturated (PUFA) Eicosatrienoic acid, Icosatetraenoate, Elaidic acid all suitable for applications use in the preparation of detergents, moisturizers, hair conditioners, shampoos and shaving cream products [13,14,12,15,16].

4. CONCLUSION

The results of FT-IR and fatty acid composition of oil from *Neocarya macrophylla* through GC-MS analysis indicated the suitability of the seed oil for cosmetic production.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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