



Characterization of Oil Extracted from Two Varieties of Tiger Nut (*Cyperus esculentus* L.) Tubers

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Abstract: The qualitative determination of fatty acids from hexane extract of two varieties of *Cyperus esculentus* L. tuber oil using Gas Chromatography-Mass Spectrometry (GC-MS) analysis revealed the following fatty acids; palmitic acid, steric acid, Margaric acid, elaidic acid, oleic acid, erucic acid, behenic acid. and arachidic acid. For the Fourier Transform Infra-Red (FT-IR) analysis, bands of 3469.09 cm^{-1} , 3463.3 cm^{-1} , sharp bands at 2867.28 cm^{-1} , 2037.86 cm^{-1} and 2866.32 cm^{-1} asymmetrical and symmetrical modes of vibration of $-\text{CH}_2-$, strong band 1742.74 cm^{-1} and 1743.71 cm^{-1} due to ester carbonyl functional group of the triacylglycerols, bending vibrations of the CH_2 and CH_3 aliphatic groups, (C-CO-O- and O- CH_2 -C) and the in-plane bending vibration of CH cis-olefinic groups seen at 1449.55 cm^{-1} , 1364 cm^{-1} and 1450.52 cm^{-1} , 1365.65 , and 1450.52 cm^{-1} , 1365.65 cm^{-1} , vibration frequency at, 1166.97 cm^{-1} finger print of the stretching vibration of the C-O ester group, frequencies of the in-and out-of plane rocking of the cis-olefinic CH_2 group at 718.51 cm^{-1} were observed for the brown and yellow tiger nut tubers oil respectively. The results were in favour of the utilization of the two varieties of Tiger nut (*Cyperus esculentus* L.) tubers oil in cosmetics, polymer and food industries.

Keywords: Tiger Nut Tuber Oil, GC-MS, FT-IR, Fatty Acids, Cosmetics, Polymers, Food

1. Introduction

Tiger nut is an underutilized crop of the family *Cyperaceae*, which produces rhizomes from the base and tubers that are somewhat spherical. Pollination is by wind. Young tubers are white, while older tubers are covered by a yellow outer membrane; they are usually found within six inches of the ground surface. Vegetative colonies of its plants are often produced from the tubers and their rhizomes. The derivatives and benefits of *Cyperus esculentus Lativum* as a plant was reported [1]. It has many other names like; Chufa, Zulu nuts, yellow nut grass, and ground almond, edible rush and rush nuts [2]. In Nigeria, some of its native names include 'Aya' in Hausa, 'Imumu' in Yoruba, 'Ofio' or 'aki-hausa' in Igbo [3]. Oil is a One of the major source of essential mineral content in the diets of common people in

Africa. Edible and Non-edible Oil Potentials of Tiger Nut (*Cyperus esculentus*) Grown in Nigeria was investigated [4]. Quality Characteristics of oil from two varieties of *Cyperus esculentus* L. tubers was physicochemical determined [5]. Despite its high nutritional value, tiger nut oil is hardly used in food industries compared to other vegetable oils such as olive and peanut oil. However, its benefits are increasingly being recognized, including its stability and similarity to olive oil in particular. Based on the available data, tiger nut oil has been established as an oil of good nutritional value which may be exploited to the great benefit of growers, processors and dealers of the tuber [6]. Evaluation of the physicochemical properties and fatty acids composition of tiger nut (*Cyperus. esculentus*) tuber oil in comparison with olive, maize, sunflower and soybean oils was reported. It showed that Tiger nut tuber oil can replace imported olive,

maize, sunflower and / or soy bean oils in foods to face the high consumption of edible oils in Egypt [7]. Safety profile and antioxidant activity of fatty hydroxamic acid from underutilized seed oil of *Cyperus esculentus* was evaluated [8]. Physico-Chemical Characterization of Vegetable Oil and Defatted Meal from Two Varieties of *Cyperus esculentus* from Benin was reported [9]. This research is aimed at GC-MS and FT-IR analysis of oil from Brown and Yellow *Cyperus esculentus* tubers and justify their industrial potential for cosmetics, polymer and food preparations.

2. Experimental

2.1. Sample Collection, Identification and Preparation

The dried tubers of the two varieties of *Cyperus esculentus* L. were crushed into powder using mortar and pestle and were stored in a plastic container prior to oil extraction.

2.2. Oil Extraction Procedure

The hexane extract was obtained by complete extraction using the Soxhlet extractor (GG-17, SHUNI). 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 GC-MS analysis.

2.3. FT-IR Analysis

The analysis of the fatty acids in the *Cyperus esculentus* L. tuber oil sample was done at National Institute of Chemical Technology (NARICT), Zaria, Nigeria. Shimadzu 8400s Fourier Transformed Infra-red spectrophotometer was used.



Figure 1. brown variety tiger nut tubers.

2.4. GC-MS Analysis

The analysis of the fatty acids in the *Cyperus esculentus* L. tuber oil sample was done at National Institute of Chemical

Technology (NARICT), Zaria, Nigeria, a Shimadzu QP2010 plus series gas chromatography coupled with Shimadzu QP 2010 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 μ L 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 NIST 05 mass spectral library [10].



Figure 2. Yellow variety tiger nut tubers.



Figure 3. Brown variety tiger nut tuber oil.



Figure 4. Yellow variety tiger nut tuber oil.

3. Results and Discussions

Table 1. Frequencies, Functional group, Vibrational mode and Intensities of the FT- IR Spectra.

Brown variety	Yellow variety	Wave no (cm ⁻¹)	Wave no (cm ⁻¹)	Functional group	Mode of vibration	Intensity
3463.3	3469.09			unsaturated fatty acid / C=O (ester)		Overtone S
2867.28	2866.32			C-H (CH ₂)		Stretching (assym) M
2037.86	-----			-CNO		Stretching (Symm) S
1742.74	1743.71			C=O (ester)		Stretching M
1450.52	1449.55			C-H (CH ₃)		Bending (wagging) S
1364.68	1365.65			-CH ₂ -C		Bending (twisting) M
718.51	718.51			cis- olefinic CH ₂		Bending (Rocking) S
430.14	439.78			C-O-C		Bending (Rocking) W

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

Table 2. Major fatty acids derived from oil of yellow variety of *Cyperus esculentus L. tubers*.

S/N	Name of fatty acid	MF	MM	RI	SI%	T. C.
	Palmitic acid	C ₁₇ H ₃₄ O ₂	270	1878		93
	Stearic acid	C ₁₈ H ₃₆ O ₂	284	2167		91
	Margaric acid	C ₁₈ H ₃₆ O ₂	270	2067		90
	Elaidic acid	C ₁₈ H ₃₆ O ₂	296	2085		91
	Oleic acid	C ₁₈ H ₃₄ O ₂	282	2178		94
	Erucic acid	C ₂₂ H ₄₂ O ₂	338	2572		92
	Behenic acid	C ₂₃ H ₄₆ O ₂	354	2475		91

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

Table 3. Major fatty acids derived from oil of brown variety of *Cyperus esculentus L. tubers*.

S/N	Name of fatty acid	MF	MM	RI	SI%	T. C.
	Palmitic acid	C ₁₇ H ₃₄ O ₂	270	1878		92
	Stearic acid	C ₁₈ H ₃₆ O ₂	284	2167		90
	Margaric	C ₁₈ H ₃₆ O ₂	270	2067		89
	Behenic acid	C ₂₃ H ₄₆ O ₂	354	2475		93
	Oleic acid	C ₁₈ H ₃₄ O ₂	282	2175		94
	Erucic acid	C ₂₂ H ₄₂ O ₂	338	2572		93
	Arachidic acid	C ₂₀ H ₄₀ O ₂	312	2366		88

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

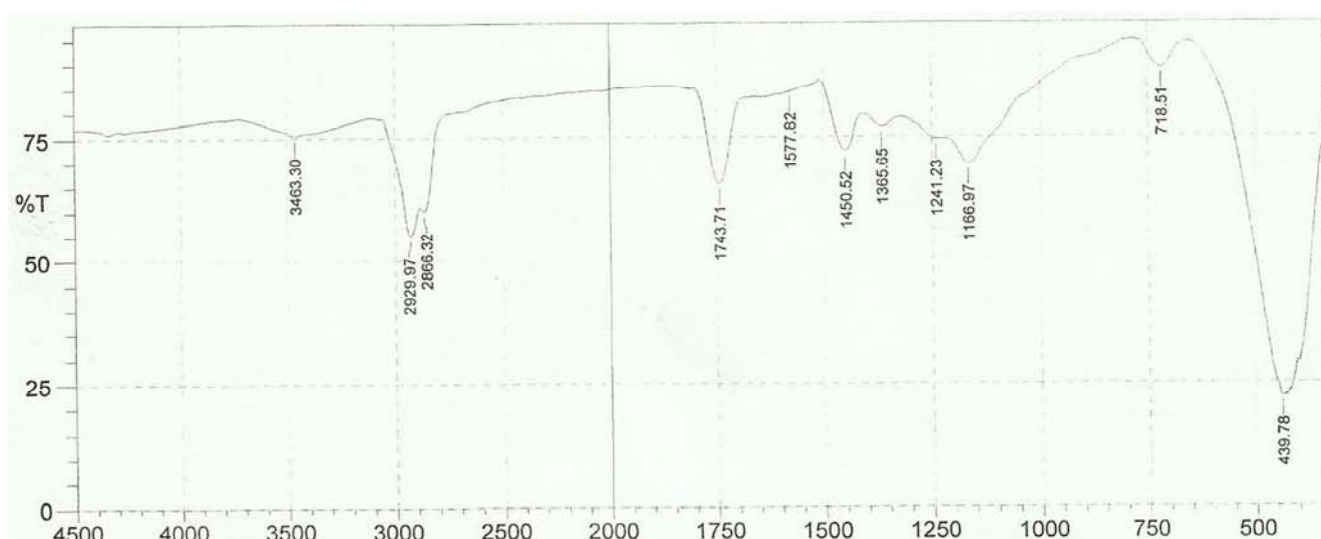


Figure 5. %T against wave no. plot of typical Infra-Red Spectrum of Brown variety of *Cyperus esculentus L. tuber oil*.

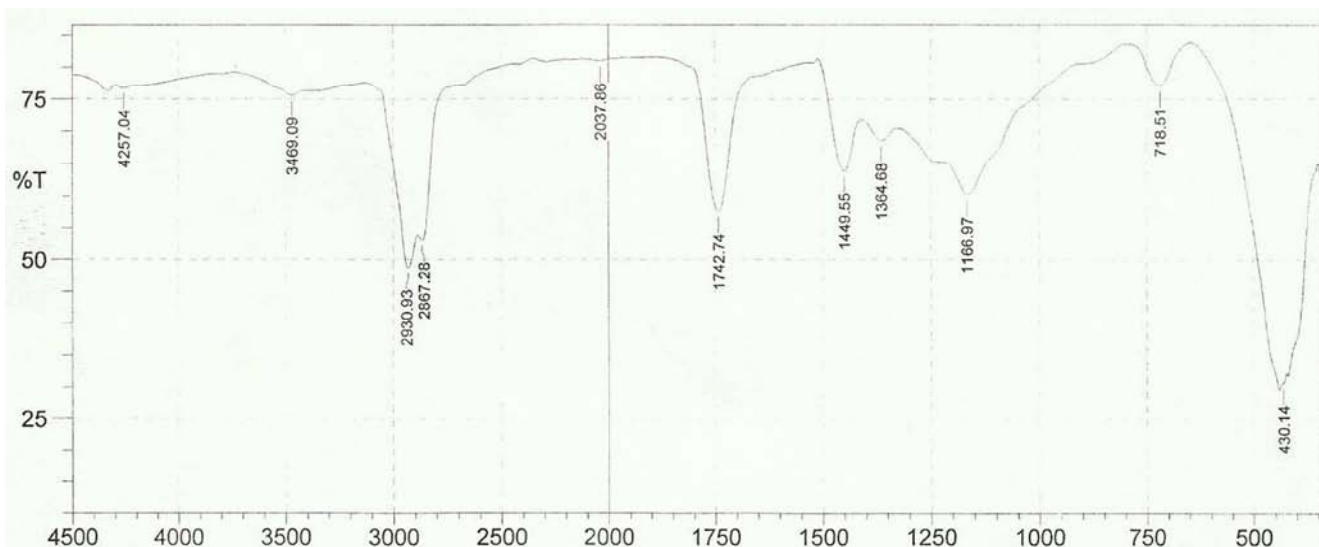


Figure 6. %T against wave no. plot of typical Infra-Red Spectrum of Yellow variety of *Cyperus esculentus* L. tuber oil.

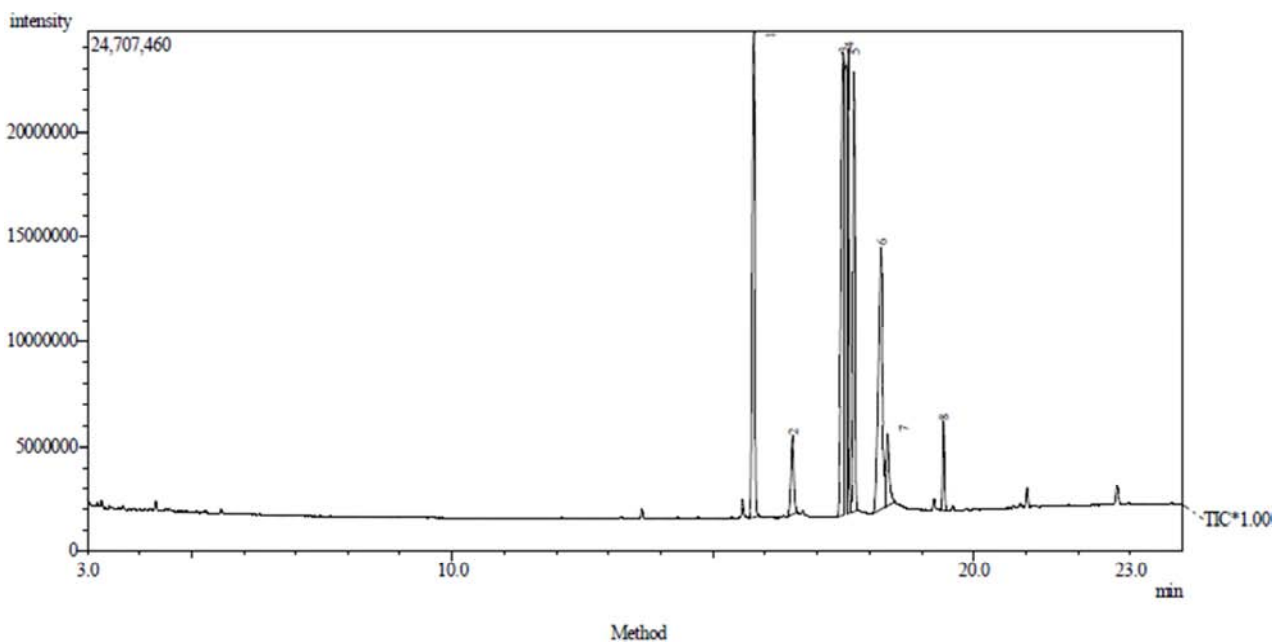
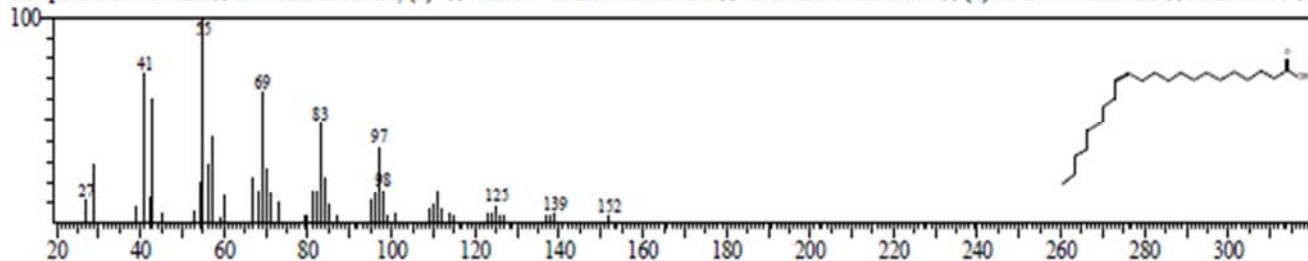


Figure 7. Typical GC-MS total ionic chromatogram (TIC) of hexane extract of brown variety of *Cyperus esculentus* L. tuber oil.

Hit#:2 Entry:121691 Library:NIST05.LIB

SI:93 Formula:C22H42O2 CAS:112-86-7 MolWeight:338 RetIndex:2572

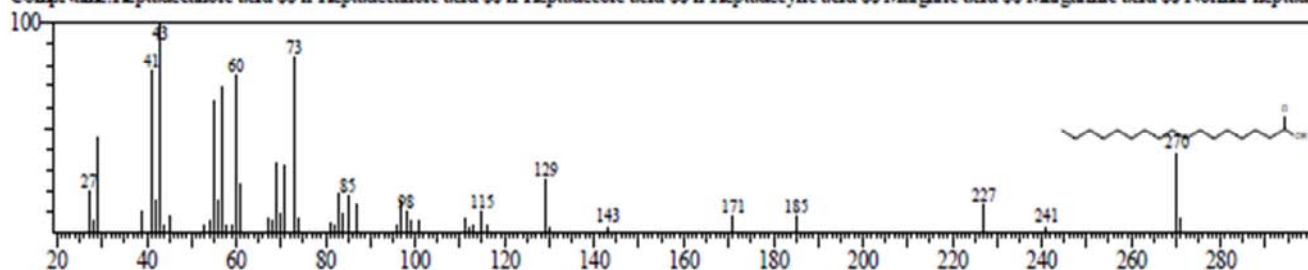
CompName:Erucic acid SS 13-Docosenoic acid, (Z)- SS .delta.13-cis-Docosenoic acid SS cis-13-Docosenoic acid SS (Z)-13-Docosenoic acid SS Prifrac 2990 SS



Hit# 5 Entry: 22212 Library: NIST05s.LIB

SI: 89 Formula: C₁₇H₃₄O₂ CAS: 506-12-7 MolWeight: 270 RetIndex: 2067

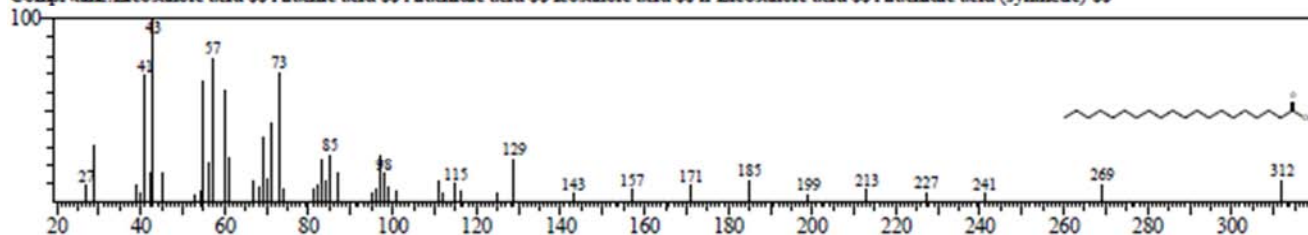
CompName: Heptadecanoic acid \$\$ n-Heptadecanoic acid \$\$ n-Heptadecanoic acid \$\$ n-Heptadecanoic acid \$\$ Margaric acid \$\$ Margaric acid \$\$ Normal-heptade



Hit# 5 Entry: 108054 Library: NIST05.LIB

SI: 88 Formula: C₂₀H₄₀O₂ CAS: 506-30-9 MolWeight: 312 RetIndex: 2366

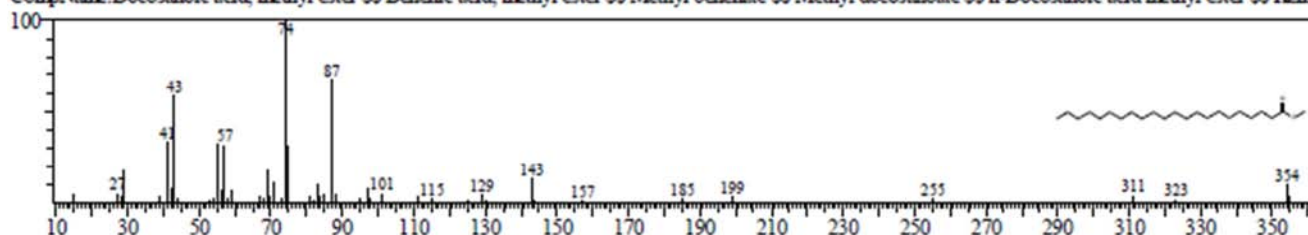
CompName: Eicosanoic acid \$\$ Arachic acid \$\$ Arachidic acid \$\$ Icosenoic acid \$\$ n-Eicosanoic acid \$\$ Arachidic acid (synthetic) \$\$



Hit# 1 Entry: 25584 Library: NIST05s.LIB

SI: 93 Formula: C₂₃H₄₆O₂ CAS: 929-77-1 MolWeight: 354 RetIndex: 2475

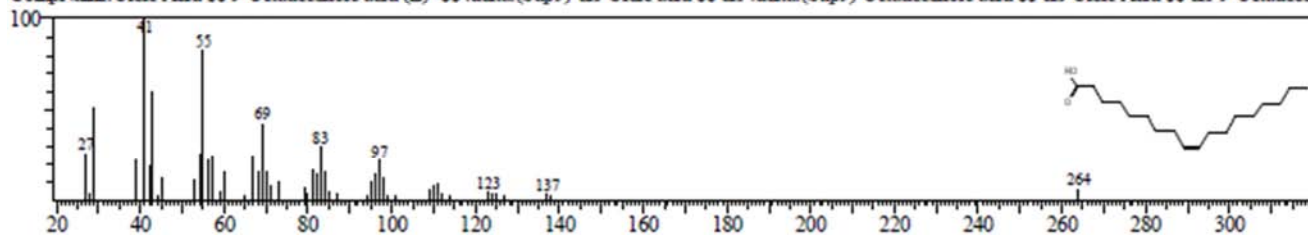
CompName: Docosanoic acid, methyl ester \$\$ Behenic acid, methyl ester \$\$ Methyl behenate \$\$ Methyl docosanoate \$\$ n-Docosanoic acid methyl ester \$\$ Kem



Hit# 1 Entry: 22869 Library: NIST05s.LIB

SI: 94 Formula: C₁₈H₃₄O₂ CAS: 112-80-1 MolWeight: 282 RetIndex: 2175

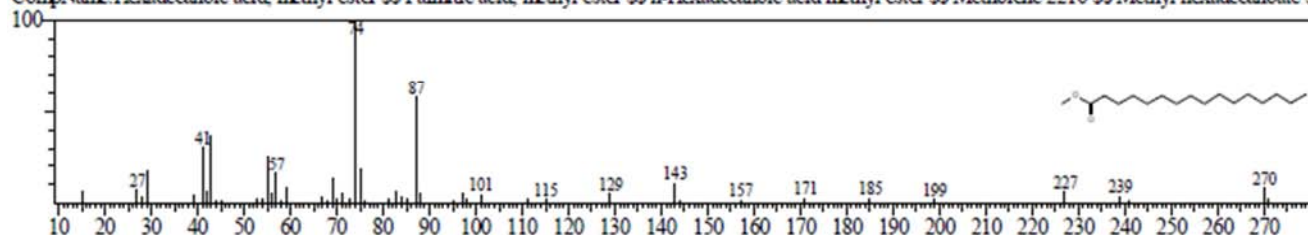
CompName: Oleic Acid \$\$ 9-Octadecenoic acid (Z)- \$\$.delta.(Sup9)-cis-Oleic acid \$\$ cis-.delta.(Sup9)-Octadecenoic acid \$\$ cis-Oleic Acid \$\$ cis-9-Octadec



Hit# 5 Entry: 22219 Library: NIST05s.LIB

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 \$



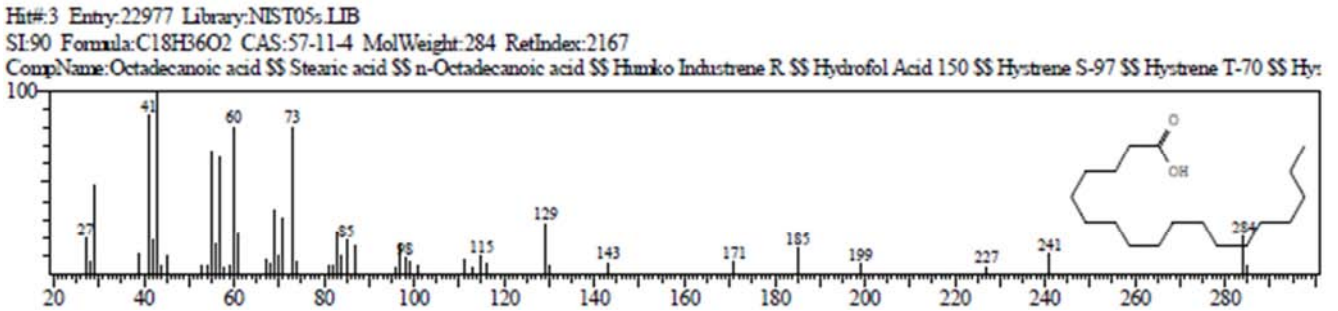


Figure 8. GC-MS Fragments of hexane extract of brown variety of *Cyperus esculentus L. tuber oil*.

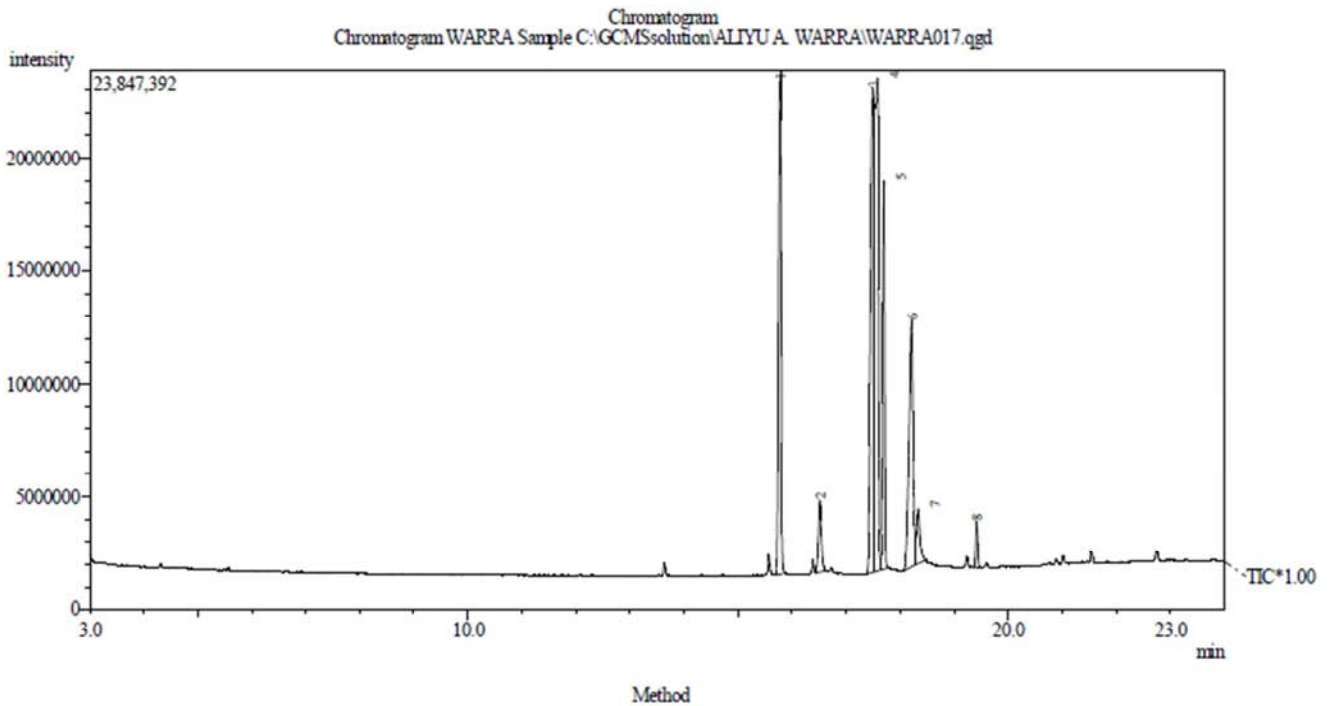
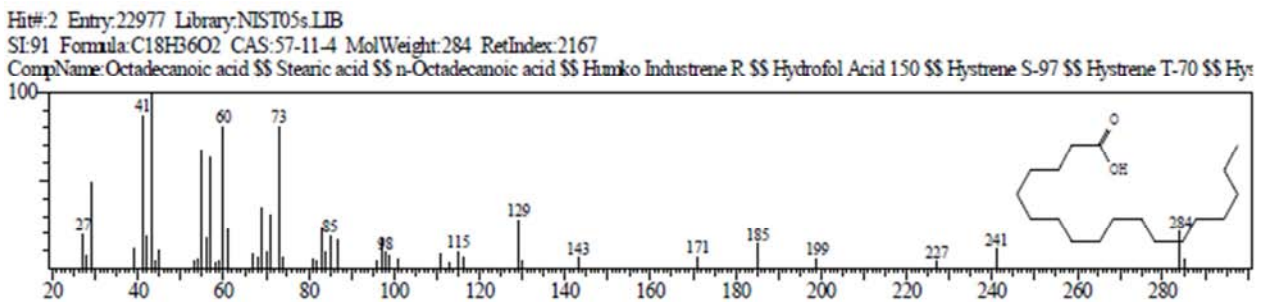
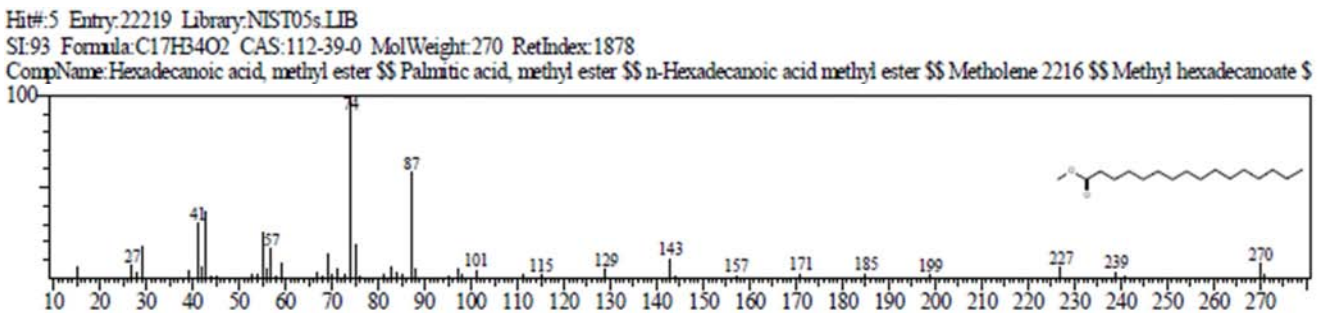


Figure 9. Typical GC-MS total ionic chromatogram (TIC) of hexane extract of Yellow variety of *Cyperus esculentus L. tuber oil*.



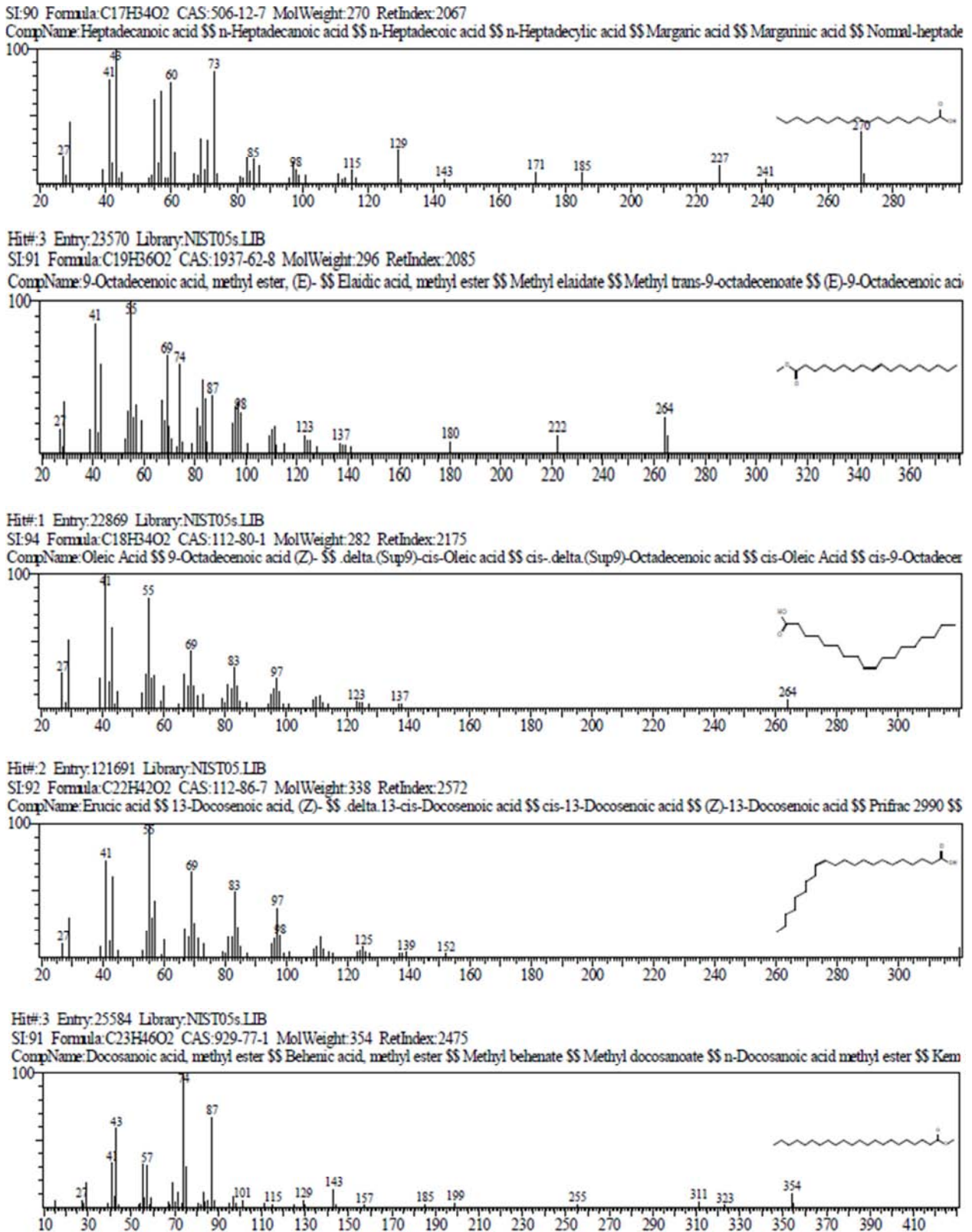


Figure 10. GC-MS Fragments of hexane extract of yellow variety of *Cyperus esculentus L.* tuber oil.

4. Discussion

The following major fatty acids were qualitatively identified from the GC-MS analysis; Palmitic acid natural

saturated acid, present in plants, animals, and microorganisms [11]. It is among the fatty acids that is used in high concentration in cosmetics [12]. Stearic acid saturated fatty acid with an 18-carbon chain and the IUPAC name octadecanoic acid was found, Stearic acid is mainly used in

the production of detergents, soaps, and cosmetics such as shampoos and shaving cream products. Soaps are not made directly from stearic acid, but indirectly by saponification of triglycerides consisting of stearic acid esters. Esters of stearic acid with ethylene glycol, glycol stearate, and glycol distearate are used to produce a pearly effect in shampoos, soaps, and other cosmetic products. They are added to the product in molten form and allowed to crystallize under controlled conditions. Detergents are obtained from amides and quaternary alkylammonium derivatives of stearic acid. Surfactants, cosmetics and personal hygiene products are in fact prospects of stearic acid [13]. Heptadecanoic acid or margaric acid is a saturated fatty acid was also detected. Oleic Acid's presence makes it a great moisturizer, and a number of cosmetic companies add it to lotions and soaps in order to boost their ability to nourish the skin [14] Behenic acid detected is often used to give hair conditioners and moisturizers their smoothing properties. Also used as anti-foam in the manufacturing of detergents [15]. Arachidic acid also called eicosanoic acid is a saturated fatty acid was detected. It is found in appreciable quantities only in some vegetable fats and oils where it occurs as glycerol ester [16]. Erucic acid was also found, products produced using erucic acid include cosmetics [17].

The FT-IR bands of 3469.09 cm^{-1} , 3463.3 cm^{-1} observed in yellow and brown tiger nut oil respectively are the region of unsaturated fatty acid. The asymmetrical and symmetrical modes of vibration of $-\text{CH}_2$ -exhibit very strong and sharp bands at 2867.28 cm^{-1} , 2037.86 cm^{-1} and 2866.32 cm^{-1} for yellow and brown tiger nut oil. The band at 1742.74 cm^{-1} and 1743.71 cm^{-1} is very strong and sharp band due to ester carbonyl functional group of the triglycerides i.e $\text{C}=\text{O}$ stretching vibrations no absorption around 1710 cm^{-1} this shows that the fatty acids is exclusively in ester forms. [18]. The bending vibrations of the CH_2 and CH_3 aliphatic groups, ($\text{C}-\text{CO}-\text{O}$ - and $\text{O}-\text{CH}_2-\text{C}$) and the in-plane bending vibration of CH cis-olefinic groups are seen at 1449.55 cm^{-1} , 1364 cm^{-1} and 1450.52 cm^{-1} , 1365.65 cm^{-1} for yellow and brown tiger nut oil respectively. The vibration frequency at, 1166.97 cm^{-1} for brown tiger nut oil and yellow tiger nut oil is finger print of the stretching vibration of the $\text{C}-\text{O}$ ester group. The frequencies of the in-and out-of plane rocking of the cis-olefinic CH_2 group are at 718.51 cm^{-1} for the brown and yellow tiger nut. The observed differences in frequency positions of the ester carbonyl, and the inactivity or absence of some in-plane bending are attributed to the variation in fatty acids composition, chain length, degree and position of double bonds in the triacylglycerols [19]. However, polymeric applications of quality characterized plants oils in general found useful in polymer preparations. A plant oil with a metathesis catalyst produces cyclohexadiene (CHD), which can then be converted to other monomers through isomerization or polymerized to mimic petroleum-based polymers. The preparation of cyclohexadiene (CHD) can be performed with little to no plant oil purification, minimal catalyst loading, no organic solvents, and simple product recovery by distillation. These monomers can be used to

supplement or substitute petroleum-based monomers traditionally used in the preparation of polymeric materials [20].

5. Conclusion

The results of FT-IR and fatty acid composition of oil from two varieties of tiger nut (*Cyperus esculentus* L.) tubers through GC-MS analysis indicated that there is no different as such in the two varieties and they are suitable for industrial production of cosmetic and polymeric products.

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