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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 $–CH_2$-, 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-planebending 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, 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 GC-MS analysis.

2.3. FT-IR Analysis

The analysis of the fatty acids in the *Cyperusesculentus* L. tuber oilsample 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 *Cyperusesculentus* L. tuber oilsample 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 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 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 (CH2)	Stretching (assym) M
2037.86 -----	-CNO	Stretching (Symm) S
1742.74 1743.71	C=O (ester)	Stretching M
1450.52 1449.55	C-H (CH3)	Bending (wagging) S
1364.68 1365.65	-CH2-C	Bending (twisting) M
718.51 718.51	cis- olefinic CH2	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 esculentusL.tubers.*

S/N Name of fatty acid MF MM RI SI% to 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 brownvariety of Cyperus esculentus L. tubers.*

S/N Name of fatty acid MF MM RI SI% to 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.

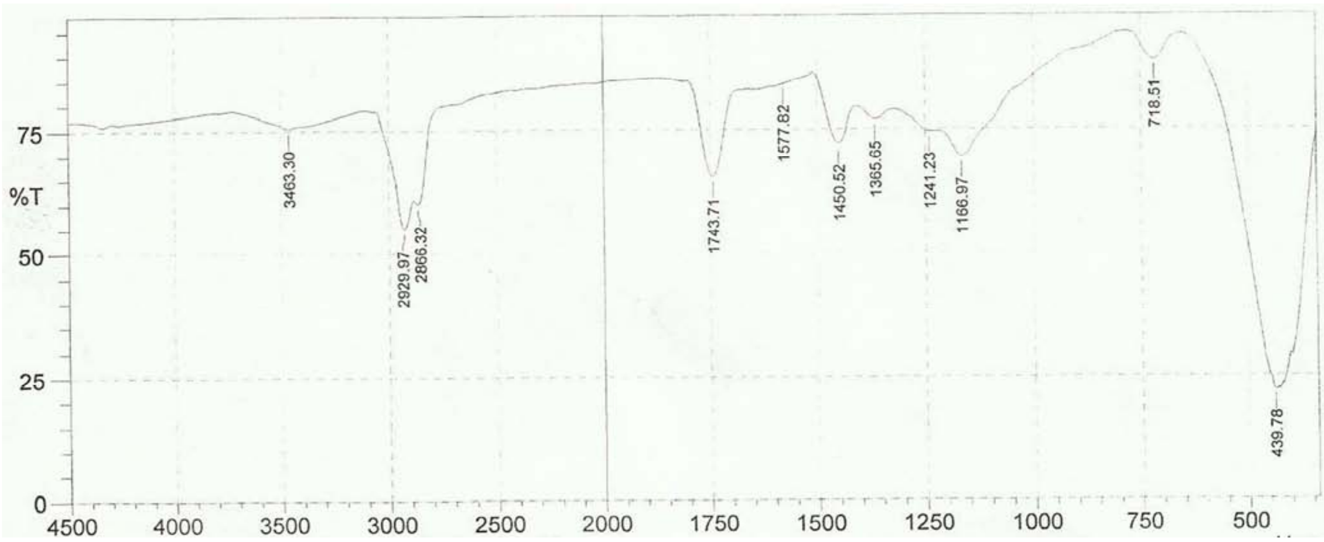


Figure 5. *%T against wave no. plot of typical Infra-Red Spectrum of Brown variety of Cyperusesculentus L. tuber oil.*

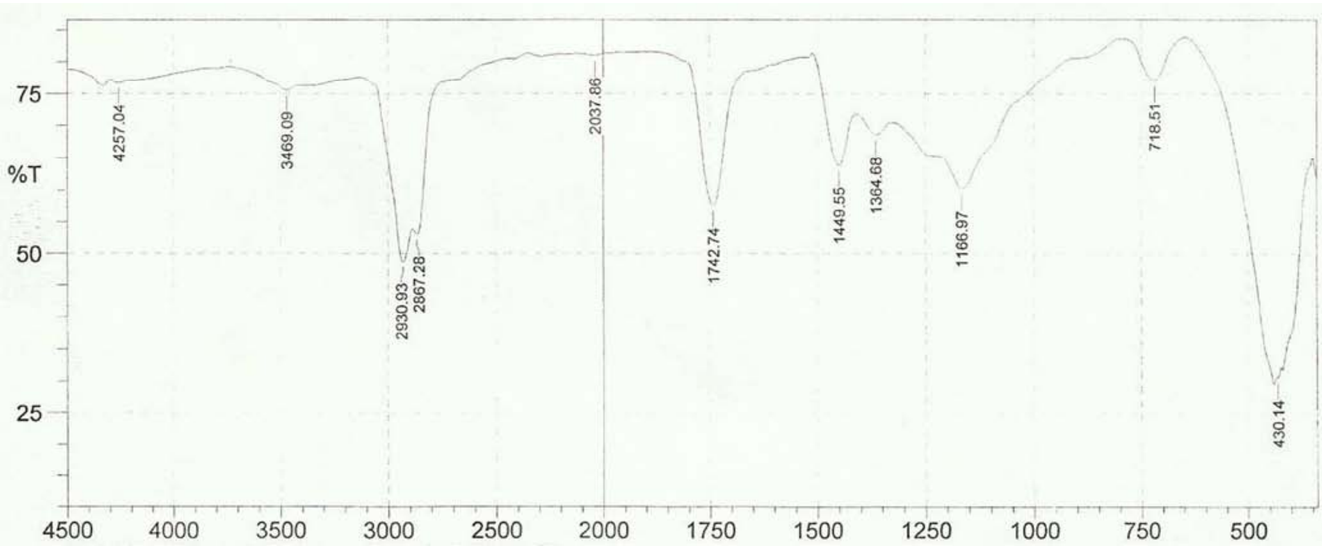


Figure 6. *%T against wave no. plot of typical Infra-Red Spectrum of Yellow variety of Cyperus esculentus L. tuber oil.*

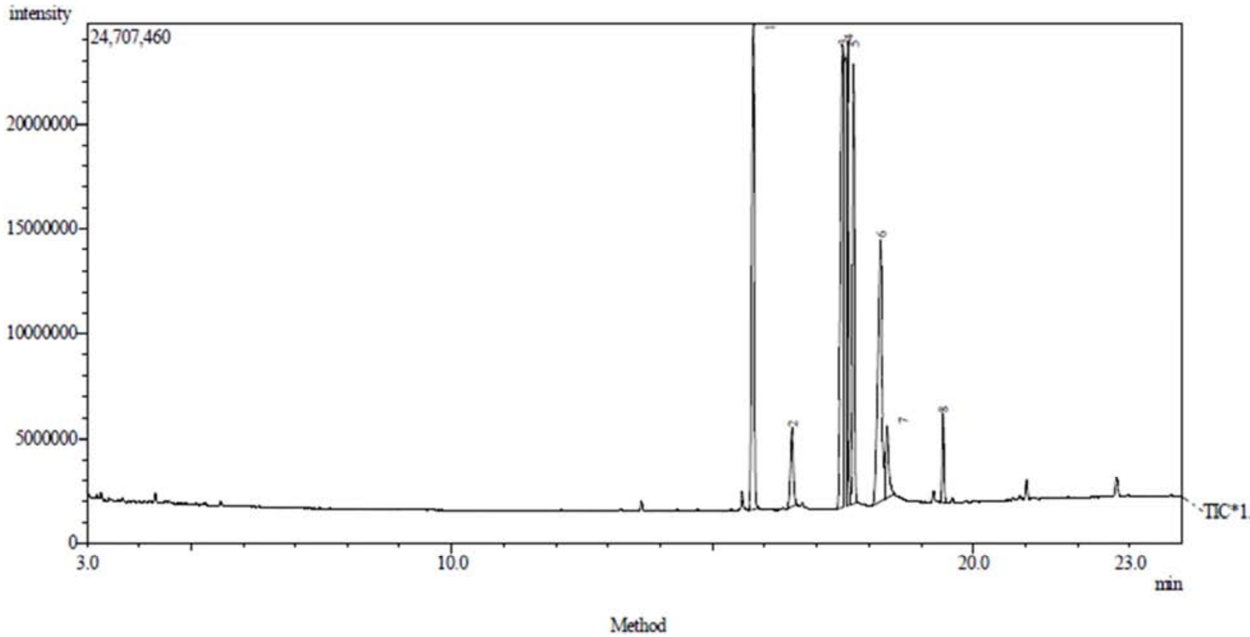
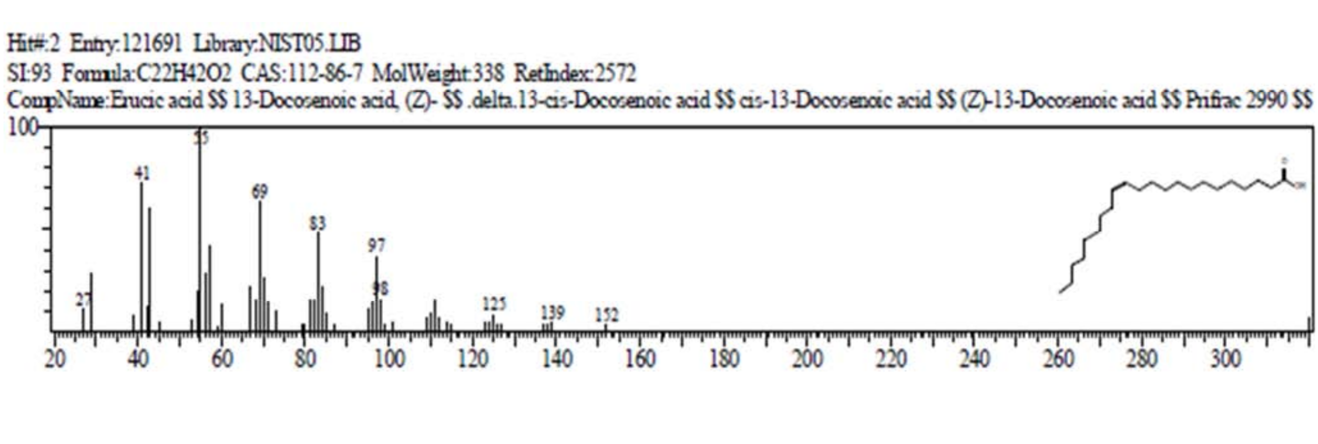
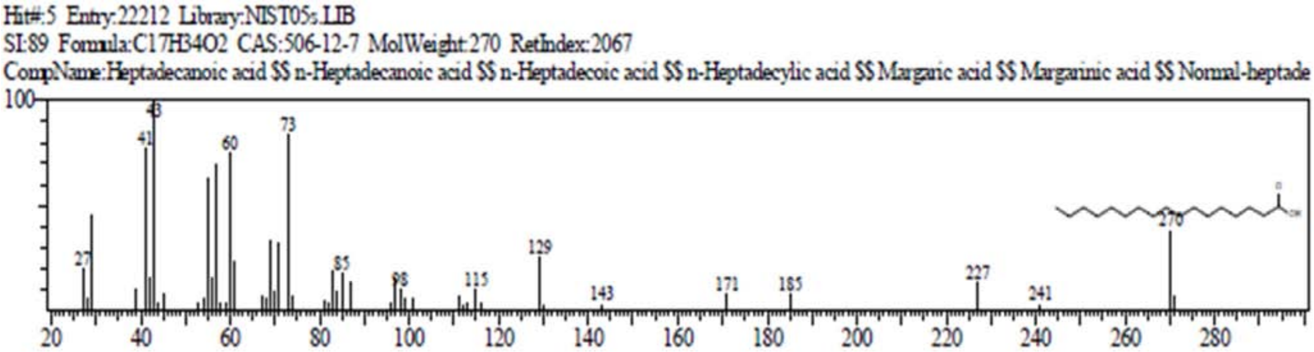
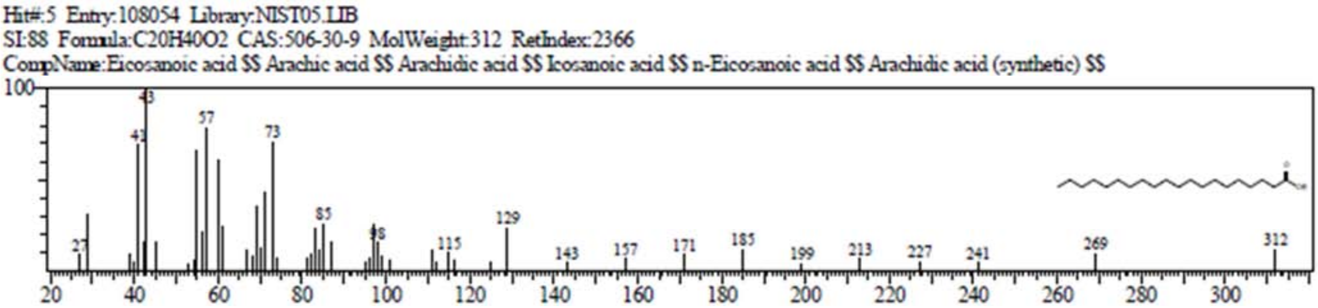
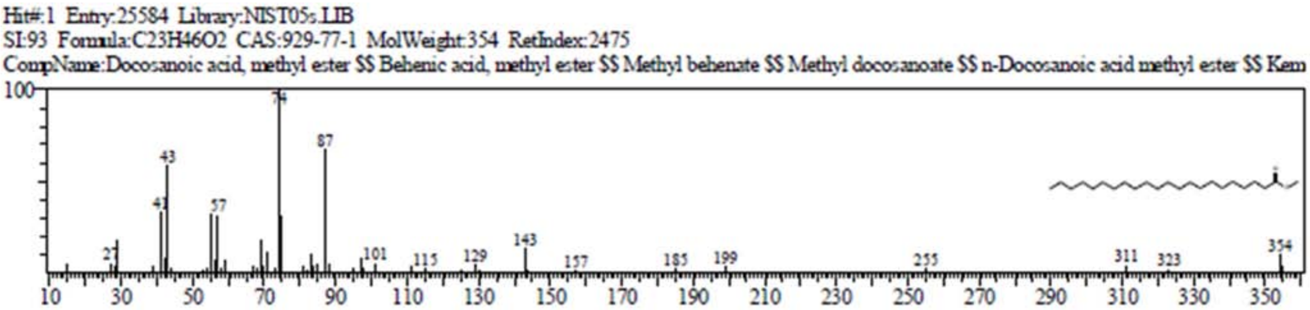


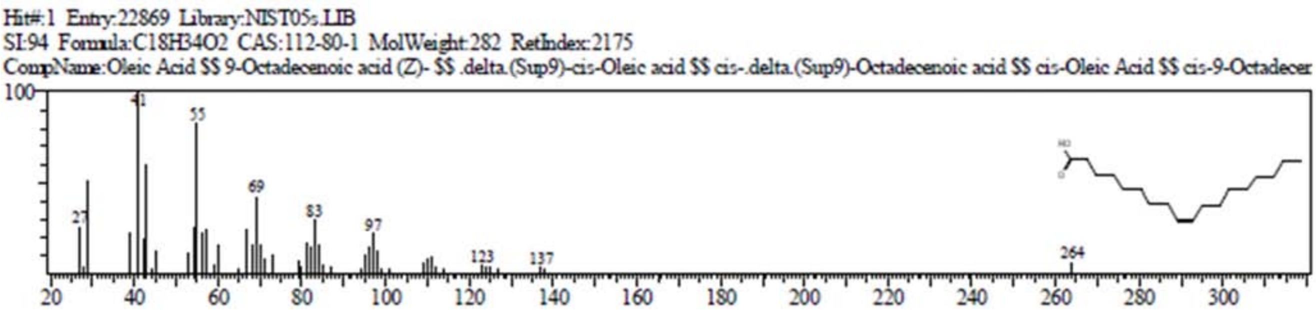
Figure 7. *Typical GC-MS total ionic chromatogram (TIC) of hexane extract of brown variety of Cyperusesculentus L. tuber oil.*

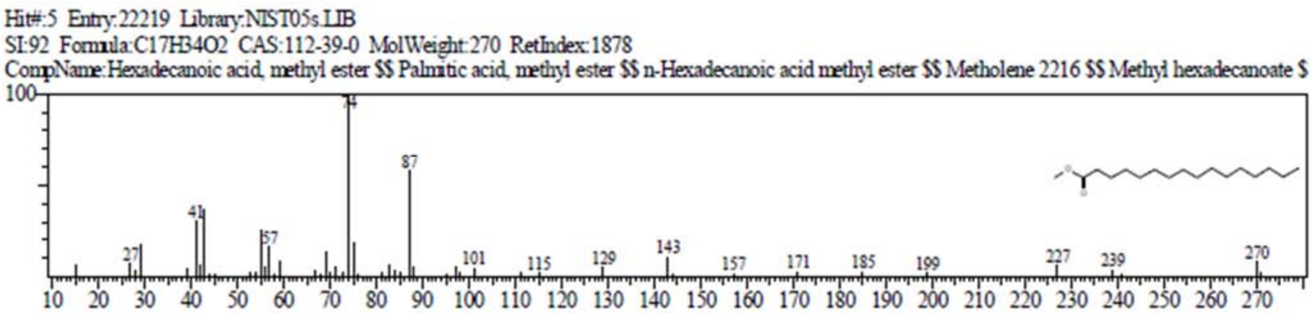



Hit#:5 Entry:22212 Library:NIST05s.LIB
SI:89 Formula:C17H34O2 CAS:506-12-7 MolWeight:270 RetIndex:2067
CompName:Heptadecanoic acid $$ n-Heptadecanoic acid $$ n-Heptadecoic acid $$ n-Heptadecylic acid $$ Margaric acid $$ Margarinic acid $$ Normal-heptade
100
27 41 43 60 73 85 98 115 129 143 171 185 227 241 270
20 40 60 80 100 120 140 160 180 200 220 240 260 280


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 $$ Icosanoic acid $$ n-Eicosanoic acid $$ Arachidic acid (synthetic) $$
100
27 41 43 57 73 85 98 115 129 143 157 171 185 199 213 227 241 269 312
20 40 60 80 100 120 140 160 180 200 220 240 260 280 300


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
100
27 41 43 57 74 87 101 115 129 143 157 185 199 255 311 323 354
10 30 50 70 90 110 130 150 170 190 210 230 250 270 290 310 330 350


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-Octadecer
100
27 41 55 69 83 97 123 137 264
20 40 60 80 100 120 140 160 180 200 220 240 260 280 300


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 $
100
27 41 57 74 87 101 115 129 143 157 171 185 199 227 239 270
10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270

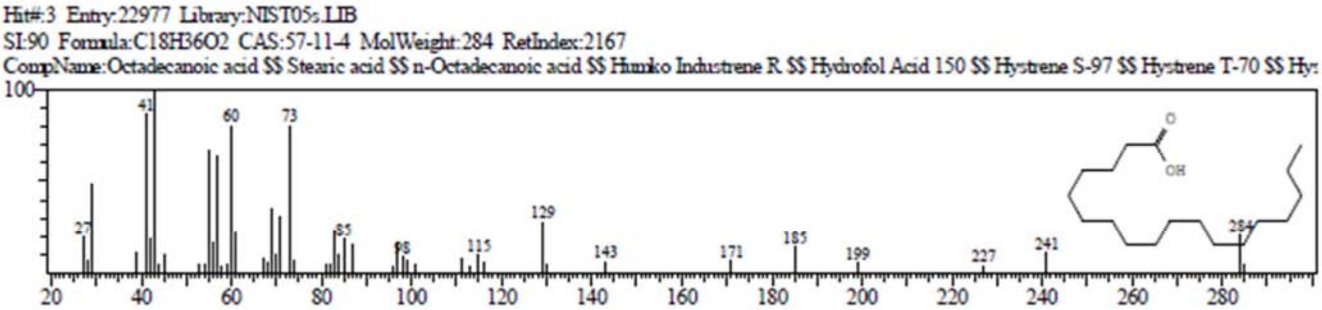


Figure 8. GC-MS Fragments of hexane extract of brown variety of Cyperus esculentus L. tuber oil.

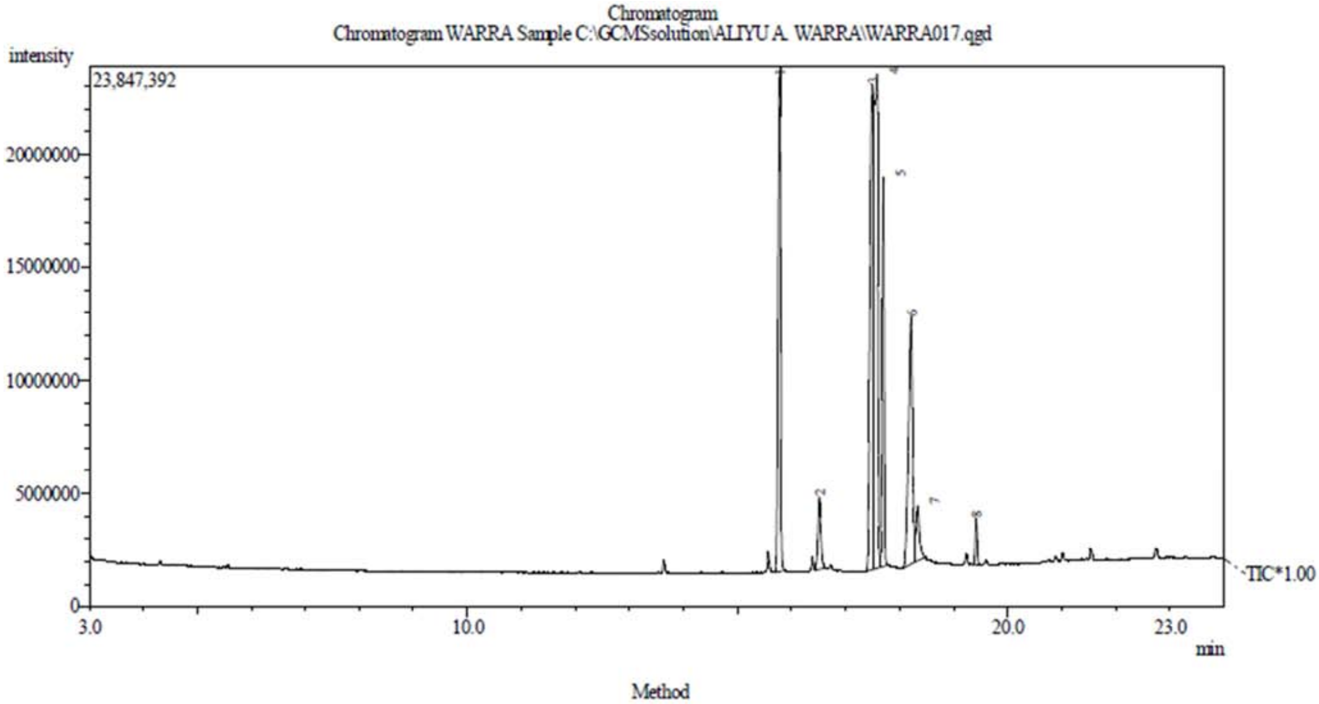
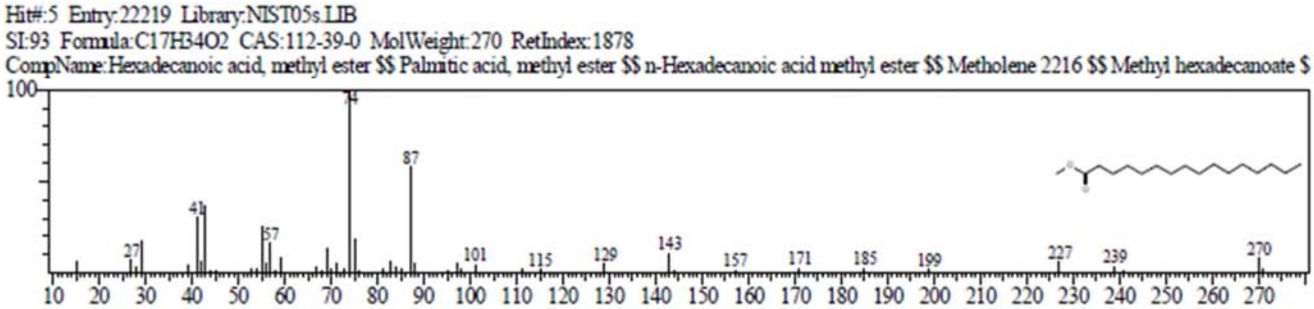
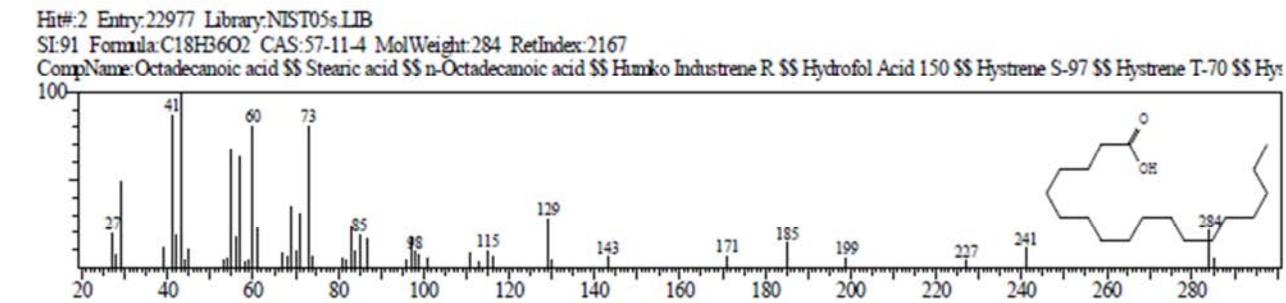


Figure 9. Typical GC-MS total ionic chromatogram (TIC) of hexane extract of Yellow variety of Cyperus esculentus L. tuber oil.





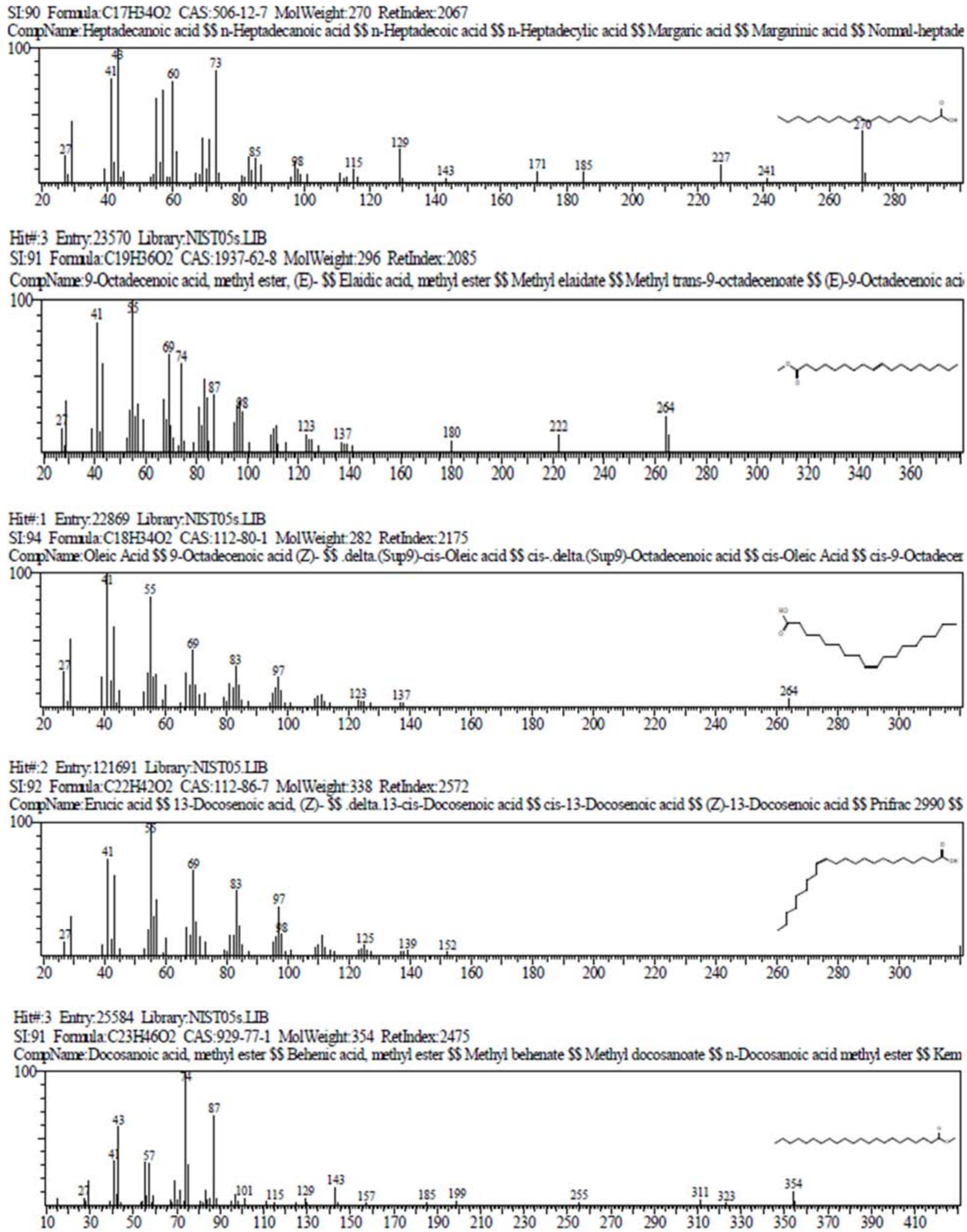


Figure 10. *GC-MS Fragments of hexane extract of yellow variety of Cyperus esculentus L. tuber oil.*

4. Discussion

The following major fatty acids were qualitatively identified form the GC-MS analysis; Palmitic acida natural saturated acid, present in plants, animals, and microorganisms [11]. It is among the fatty acids that isusedin high concentration in cosmetics [12]. Stearic acida 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 $–CH_2$-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 C=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,(C-CO-O- and O-CH_2-C) and the in-plane bending vibration of CH cis-olefinic groups are seen at 1449.5 5 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 C-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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