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Identification of Volatile Constituents' Profile of *Zygophyllum aegyptium* A. Hosny by GC/MS Analysis

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Abstract: Zygophyllum aegyptium A. Hosny is an important medicinal plant natively grows in Mediterranean coastal regions of Egypt. Volatile secondary metabolites were extracted using the less polar solvents hexane and methylene chloride. The volatile constituents were identified using GC/MS technique to yield thirty-seven compounds belonging to acetogenins, sesquiterpenes, diterpenes and sterols classes. Saturated fatty esters (59.51%) and unsaturated fatty acids (22.21%) are the more dominant in hexane fraction while for methylene chloride extract, acetogenins (93.12%), diterpenes (0.82%) and sterols (2.21%) are the major classes. The most predominant constituents > 5% in the two extracts were methyl hexadecanoate, methyl linoleate, methyl docosanoate and methyl stearate which considered to be valuable constituents in industrial, pharmacological and biotechnological applications.

keywords: *Zygophyllum aegyptium* A. Hosny, GC/MS technique, volatile constituents' Profile

1.Introduction

Zygophyllum aegyptium A. Hosny is a medicinal plant belonging to family Zygophyllaceae. It is a perennial, woody undershrub with evergreen succulent leaves. This species is distributed in the Mediterranean region of Egypt, Tunisia, and Cyprus [1].*Z. aegyptium* is traditionally used in Rheumatism, gout, asthma and hypertension [2]. Biological studies on *Zygophyllum* species have indicated significant antioxidant, antidiabetic, antitumor, antimicrobial and anti-inflammatory activities [3-8]. Such activities were contributed to their phytochemical constituents. Various classes of compounds including triterpenes, flavonoids, saponins, sterols, phenolic, essential oils and esters have been isolated from different *Zygophyllum* species [9].

Several species of *Zygophyllum* were the subject of many chemical investigations by many workers. A number of natural constituents has been isolated and identified such as sterols, triterpenes, phenolics, flavonoids, saponins, lignans, organic acids and tannins [9, 10].

Investigation the previously published literature on the volatile constituents of *Zygophyllum aegyptium* revealed that no reports have highlighted this point. This motivated us to analyze the volatile constituents of *Z. aegyptium* less polar extracts using the GC/MS technique to predict the most biological activities, based on the most abundant constituents.

2. Materials and methods

2.1. General

GC/MS analysis was carried out using GC-TSQ mass spectrometer (Thermo Scientific, Austin, TX, USA) with a direct capillary column TG–5MS (30 m x 0.25 mm x 0.25 µm film thickness).

2.2. Chemicals

Hexane, methylene chloride, methanol and sodium sulphate anhydrous were obtained from Loba Company- India.

2.3. Plant material

Zygophyllum aegyptium was collected from Gamasa near Mansoura, Egypt at 31^{o} 25' 48.52" north to 31^{o} 32' 12.75" east in October 2019, the authenticity of the plant was

confirmed by Prof. Dr. Ibrahim Mashaly, Botany Department, Faculty of Science Mansoura University, Egypt.

2.4. Extraction process

The dried whole plant parts (2.5 kg) were soaked in methanol (5 x 10 L) at room temp. The methanolic extract was filtered, and evaporated to its 1/3 volume, then liquid-liquid extraction was performed using the less polar solvents hexane and methylene chloride, respectively to afford hexane (25 g) and methylene chloride (9 g) fractions.

3. Results and Discussion

GC/MS provides excellent separation capacity but is limited in the types of compounds that can be analyzed as samples must be sufficiently volatile for analysis. Liquid-liquid extraction was performed to the methanol extract of *Z. aegyptium* using hexane and methylene chloride to obtain the less polar and more volatile constituents, that fit the GC/MS analysis.

The volatile profiles for the two extracts were identified and tabulated (Table 1 and Figure 3). The composition of the volatile constituents in both hexane and methylene chloride extracts were qualitatively and quantitatively investigated using chromatographic retention data, mass spectra acquired by GC/MS. Each chromatographic GC peak (Fig 1, 2) was integrated and its EI MS spectra was matched with their analogue in the stored NIST and Wiley EI MS database libraries.

Thirty-seven compounds were identified representing 89.92% of the whole hexane fraction composition that belonging to various secondary metabolites'classes, comprising thirty-three acetogenins, one sesquiterpene, one diterpene and two sterols. Saturated fatty esters (59.51%) and unsaturated fatty acids (22.21%) are dominant in hexane fraction. Of the characterized constituents, methyl hexadecanoate (27.51%), methyl linoleate (11.33%) and methyl docosanoate (7.16%) were the most predominant constituents in hexane fraction.

Methylene chloride fraction afforded twenty volatile components and their quantities was estimated by GC/MS analysis to be 96.15% of the fraction composition. The identified constituents were belonging to acetogenins (93.12%), one diterpene (0.82%) and one sterol (2.21%). Four major constituents >5% methyl hexadecanoate (34.85%), methyl linoleate (18.63%) methyl docosanoate (7.23%) and methyl stearate (5.06%) were noticed.

A wide range of bioactivities were recorded previously for the major volatile constituents, so the potentiality of using these valuable fractions in industrial, pharmacological and biotechnological applications should be taken in consideration. Since, methyl hexadecanoate which detected in appreciable concentration exerted antioxidant, hypocholesterolemic, nematicide, pesticide, antiandrogenic, flavor, hemolytic and 5-Alpha reductase inhibitor [11, 12]. Also, it was used as antimicrobial and antifungal [11, 13] antitumor, immunostimulant, chemopreventive and lipoxygenase inhibitor [11, 14-17].

Methyl linoleate was possessed anti-oxidant [18], anti-inflammatory, hepatoprotective [19, 20], anticancer activities [21, 22] and mosquito vector control [23]. Methyl stearate was reported to have antimicrobial [24, 25], antifungal [26], antidiarrheal and antiproliferative activities [27].

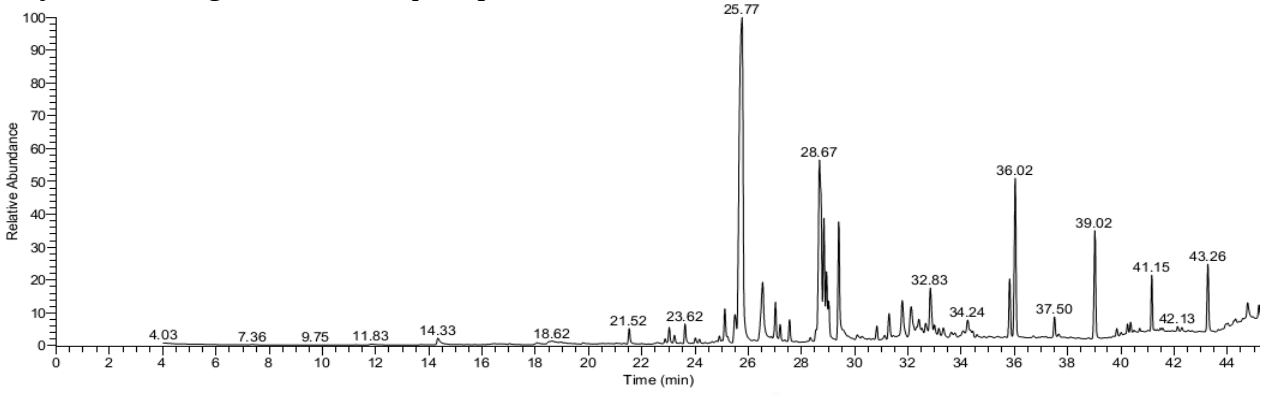

Fig. 1: GC chromatogram of hexane fraction of *Z. aegyptium* A. Hosny

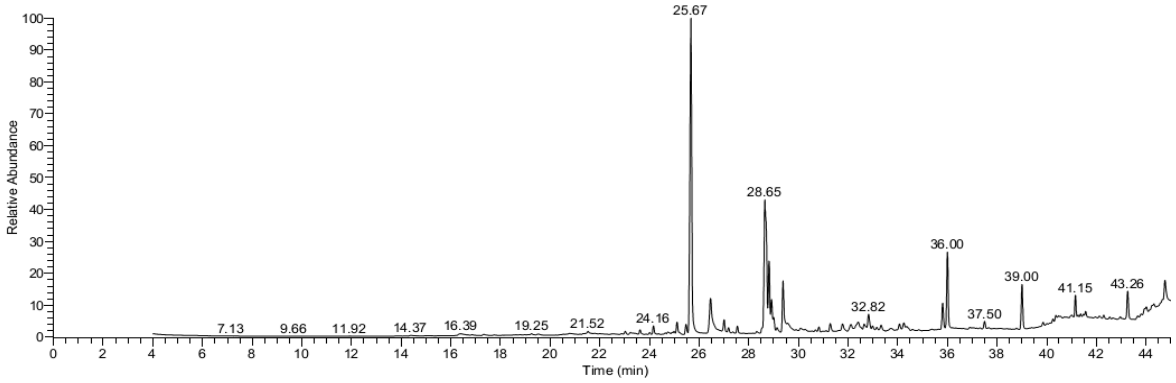

Fig. 2: GC chromatogram of methylene chloride fraction of *Z. aegyptium* A. Hosny

Table (1): Identification of the major volatile constituents of *Z. aegyptium* A. Hosny using GC/MS technique.

No.	Name	R.t.	Hexane Area %	CH_2Cl_2 Area %	Mol. Wt.	Mol. Formulae
1	9-Oxo-nonanoic acid methyl ester	14.33	0.33		186	$C_{10}H_{18}O_3$
2	Myristic acid methyl ester	21.52	0.65		242	$C_{15}H_{30}O_2$
3	Pentadecanoic acid methyl ester	22.87	1.01		256	$C_{16}H_{32}O_2$
4	12-Methyl- tetradecanoic acid methyl ester	23.02	0.67		256	$C_{16}H_{32}O_2$
5	Hexahydrofarnesyl acetone	24.00	0.22		268	$C_{18}H_{36}O$
6	3,7,11,15-Tetramethyl-2-hexadecen-1-ol	24.16	0.16	0.82	296	$C_{20}H_{40}O$
7	Methyl 14-methylpentadecanoate	24.91	0.21		270	$C_{17}H_{34}O_2$
8	Methyl Z-9-hexadecenoate	25.11	2.75	2.10	268	$C_{17}H_{32}O_2$
9	Methyl hexadecanoate	25.77	27.51	34.85	270	$C_{17}H_{34}O_2$
10	n-Hexadecanoic acid	26.54	2.87	3.77	256	$C_{16}H_{32}O_2$
11	Methyl 14-methylhexadecanoate	27.02	1.55	1.19	284	$C_{18}H_{36}O_2$
12	Z-10-Heptadecenoic acid, methyl ester	27.19	0.61		282	$C_{18}H_{34}O_2$
13	Methyl heptadecanoate	27.54	0.92	0..67	284	$C_{18}H_{36}O_2$
14	Methyl 5Z,9Z,12Z-octadecatrienoate	28.33	0.19		292	$C_{19}H_{32}O_2$
15	Methyl 5Z,9Z-octadecadienoate	28.53	0.17		294	$C_{19}H_{34}O_2$
16	Methyl linoleate	28.67	11.33	18.63	294	$C_{19}H_{34}O_2$
17	Methyl E-9-octadecenoate	28.84	3.51	4.56	296	$C_{19}H_{36}O_2$
18	Methyl E-11-octadecenoate	28.93	1.72	1.74	296	$C_{19}H_{36}O_2$
19	Methyl E-13-octadecenoate	29.01	1.05	0.75	296	$C_{19}H_{36}O_2$
20	Methyl stearate	29.39	4.92	5.06	298	$C_{19}H_{38}O_2$
21	Stearic acid	30.08	0.24		284	$C_{18}H_{36}O_2$
22	Octadecanedioic acid	31.28	1.19		314	$C_{18}H_{34}O_4$
23	Hexadecanedioic acid, dimethyl ester	31.28	0.88	0.88	314	$C_{18}H_{34}O_4$
24	11E,14E-Eicosadienoic acid, methyl ester	31.77	0.91	0.91	322	$C_{21}H_{38}O_2$
25	Methyl 8-(3-octyl-2-oxiranyl)octanoate	32.12	1.78		312	$C_{19}H_{36}O_3$
26	Methyl 15-hydroxy-9,12-octadecadienoate	32.65	0.47		310	$C_{19}H_{34}O_3$
27	Methyl icosanoate	32.83	2.36	1.63	326	$C_{21}H_{42}O_2$
28	Methyl 14-methyl-eicosanoate	34.43	0.21		340	$C_{22}H_{44}O_2$
29	Methyl docosanoate	36.03	7.16	7.23	354	$C_{23}H_{46}O_2$
30	Methyl tricosanoate	37.50	0.85	0.73	368	$C_{24}H_{48}O_2$
31	Methyl tetracosanoate	39.02	4.96	4.53	382	$C_{25}H_{50}O_2$
32	Bis(2-ethylhexyl) decanedioate	39.84	0.33		426	$C_{26}H_{50}O_4$
33	Methyl pentacosanoate	40.24	0.37		396	$C_{26}H_{52}O_2$
34	Methyl hexacosanoate	41.15	2.05	1.81	410	$C_{27}H_{54}O_2$
35	Methyl octacosanoate	43.26	2.90	2.75	438	$C_{29}H_{58}O_2$
36	3β-Ergost-5-en-3-ol	43.93	0.17		400	$C_{28}H_{48}O$
37	β-Sitosterol	44.76	0.74	2.21	414	$C_{29}H_{50}O$

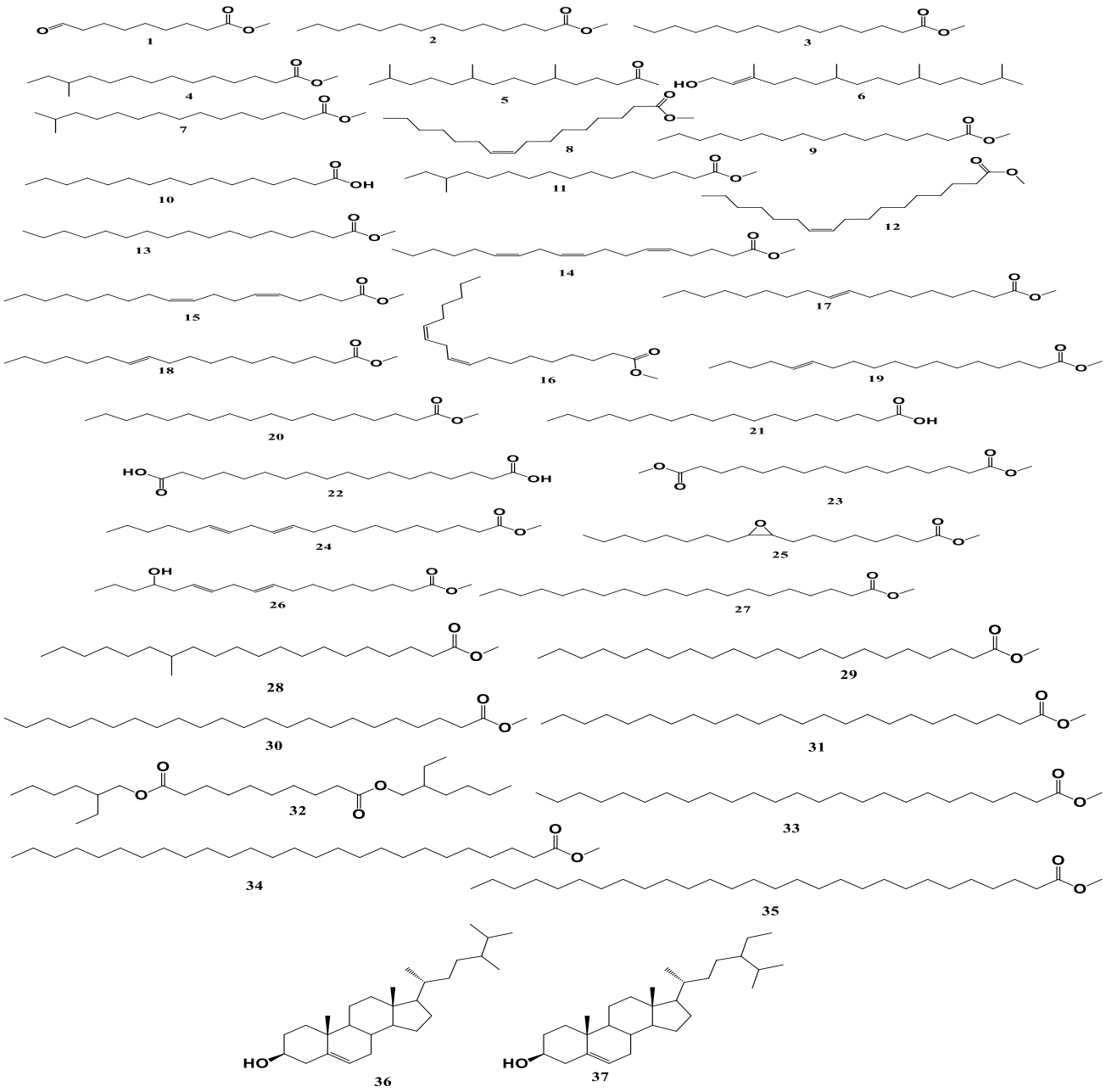

Fig. 3: Chemical formulas of the identified volatile constituents

4. Conclusion

Data obtained from GC/MS analysis of the less polar and more volatile constituents of *Zygophyllum aegyptium* A. Hosny supported that the medicinal plant is rich source of various biologically active volatile constituents which may be an added-value in the field of pharmaceutical and industrial uses.

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