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## **Chemical Composition Of The Volatile Fractions Of Mesembryanthemum Forskahlei Flowers And Herb**

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### **Abstract**

Halophytes are succulent native plants to the northern region of the Kingdom of Saudi Arabia. They have therapeutic properties and can be potentially useful as a new source of bioactive compounds. Our study aims to investigate the chemical composition of the volatile fraction of Herb and Flowers of *Mesembryanthemum forskahlei* by the application of Gas Chromatography method. Our study showed that palmitic acid (420mg/kg) and squaline (352,32mg/kg) are the main components in the herb. Furthermore, we identified that palmitic acid is the main component of the flower. Finally, *Mesembryanthemum forskahlei* herb has higher content of volatile compounds than the flower.

Keywords: forskahlei fractions, chromatography, Phytochemical, Palmitic acid, Squaline.

### **Introduction**

These plants are used as ornaments due to their bright blue, red, pink and light violet flowers. Their lustrous leaves have glandular hairs, which iridescent in the sunlight. In addition, plants of *Mesembryanthemum* genus are used in medicine and as a high interest to herbal medicine has recently been observed because the synthetic drugs' possess a considerable side effects on the human. Herbal drugs are better tolerated but their therapeutic effect is achieved a

little later than synthetic drugs. In addition, Herbal medicines acquire great significance in prevention and complex treatment of various diseases. The arsenal of herbal drugs is permanently widened due to the profound study of well-known plants with proven efficacy, as well as the research of previously unexplored species. In this study ,we intended to deeply explore halophytes belonging to the above mentioned plants.

*Mesembryanthemum crystallinum* is specifically used in cell cultures for the studying of water transport involving aquaporins. [4, 7].

Plants of *Mesembryanthemum* genus are used as antibacterial remedies in folk medicine prescriptions and as a gargle for inflammatory diseases of mucous membranes [5].

*M. crystallinum* ethanol extract was shown to possess the ability to inhibit adipocytes growth and differentiation which allows its use in the prevention and treatment of obesity [2]. The study performed on rats showed anti-cancer activity of *M. crystallinum* frozen-dried extract [3].

Phenolic compounds of *M. edule*, in particular flavonoids and tannins, have been proven to exert expressed antioxidant activity [1, 6].

Despite the wide study of *Mesembryanthemum crystallinum*, another representative of this genus, *Mesembryanthemum forskahlei* Hochst. ex Boiss, which is an endemic species in Jordan and Israel, has not been studied enough.

Thus, our research is directed to study the chemical composition of the volatile fraction of *M. forskahlei*.

## Materials

Methylene chloride ( $\text{CH}_2\text{Cl}_2$ ) solvent of high purity 99.9% was purchased from Fisher Scientific / (Ontario, Canada). Tridecane ( $\text{C}_{13}\text{H}_{28}$ ) as internal standard with purity of 99.5% was purchased from Tokyo Chemical Industry Co, LTD/(Kumagaya , Japan).

Herb and Flowers of *Mesembryanthemum forskahlei* were collected from the northern region of the Kingdom of Saudi Arabia and dried at room temperature for 15 days then grinded and passed through a (100  $\mu\text{m}$  stainless steel sieve mesh). Grinder Small Scale Automatic Feeding Mill Machine (Henan New

Elephants Machinery Co, LTD/China)

## Methods

The dried *M. forskahlei* herb and flowers were used as a raw material collected in the spring season from the north of Saudi Arabia at Al Jouf region in 2013. The Agilent Technology 6890 gas chromatograph with mass-spectrometric detector 5973(USA) was used for the experiment with the following analytical parameters: capillary chromatography column DB-5 (length 30m, internal diameter 0.25 mm), carrier gas (helium) flow rate 1.2 mL/min, the sample injection temperature was 250°C; thermostat temperature is programmed from 50°C to 320°C with the speed 4 degrees/min [8, 9].

About (0.05 g) of the dried grinded plant sample was placed into a 2ml vial that contains 0.6ml of methylene chloride which was used as a solvent and 50 µg of tridecane as internal standard. The samples were then kept at the temperature at 50°C for 2-3 hours or for twenty-four hours at room temperature. The extracts transferred to 2 ml vial and concentrated by stream of ultrapure nitrogen with the speed of 100 ml/min. until the samples residual volume reached about 10 µl.

Sample injection in the chromatography column was conducted at splitless rate, which allowed to inject the sample without loss on division and 10-20 times increase in the method sensitivity.

The sample components identification was carried out by comparing mass-spectra of the products with data in the NIST05 and WILEY 2007 mass-spectra libraries with the help of AMDIS and NIST identification softwares.

Quantitative content of the volatile fraction components (X, mg/kg) was calculated by the internal standard method using formula:

$$X = \frac{S_1 \cdot D}{S_2 \cdot M},$$

Where:

S1 – the studied sample peak area;

S2 – the standard sample peak area;

50 – mass of the injected internal standard,  $\mu\text{g}$ ;

M – sample mass, g.

### Results and Discussion:

59 components, 55 of which are identified

Table 1 shows the integrated results of quantitative determination of *M. forskahlei* herb and flowers volatile fraction components.

Chromatograms of *M. forskahlei* plant material volatile fraction components are given in Fig. 1 and 2.

In this report ,we found substances like squaline, limonene, Trans-Caryophylline, geranyl acetate in the volatile fraction of herb. The maximum concentration of Palmitic acid was found in the volatile fraction of flowers.

The comparative analysis of *M. forskahlei* plant material volatile fractions has shown that the chemical composition of herb has higher content than that of the flowers.

$\gamma$ -Cadinene, dodadecanal, aromadendrenepoxide and terpenyl acetate dominated are among the terpene compounds (essential oil components) of *M. forskahlei* flowers. Farnesylacetone C was found to be the main component of *M. forskahlei* volatile fractions.

Table 1. Results of the determination of the volatile fraction components in *Mesembryanthemum*

*forskahlei* raw material

№	Retention time	Compound	Content, mg/kg	
			Flowers	Herb
1	2	3	4	5
.1	7,47	Benzeneacetaldehyde	-	2,23
.2	7,74	Limonene	-	0,87
.3	8,99	Trans-linalool oxide	7,27	-
.4	9,16	Cis-linalool oxide	3,56	-
.5	9,71	Heptanal	3,53	-

.6	10,37	Nonanal	5,98	-
.7	10,55	2-Methyl octane	2,65	-
.8	10,77	2-Methyl nonane	1,43	-
.9	13,04/13,01	Decanal	1,86	3,85
.10	16,24	Dodecanal	15,49	-
.11	16,8/16,78	IS) Internal Standard) (Tridecane	mg/kg 5000	
.12	17,70/17,67	Terpenyl acetate	11,67	4,08
.13	18,01	*	4,58	-
.14	19,59	ol- $\gamma$ -en- $\gamma$ -Oct	-	0,84
.15	19,93	Pentadecane	2,10	2,49
.16	20,05	Trans-caryophyllene	-	3,33
.17	20,78	Geranyl acetone	-	10,39
.18	21,31	$\alpha$ -Farnesene	-	1,95
.19	21,55	Trans- $\beta$ -ionone	-	2,01
.20	21,65	Cis- $\beta$ -ionone	-	5,19
.21	22,58	Cedrane epoxide	-	4,50
.22	22,80/22,06	$\beta$ -Bisabolene	2,04	1,59
.23	22,97	$\gamma$ -Cadinene	21,13	-
.24	23,25	$\delta$ -Cadinene	3,51	-
.25	23,42	6-Methyl octadecane	4,96	-
.26	24,18	Megastigmatrienone	-	1,92
.27	24,27	Aromadendrene epoxide	13,17	-
.28	24,55	Spathulenol	3,72	-
.29	24,82	Caryophyllene oxide	4,38	-
.30	25,13	4-Methyl-pentadec-2-one	2,31	-
31	25,131	2,2,4-Trimethyl-3-carboxyisopropylpentanoic acid isobutyl ester	-	6,48
32	28,05	Octadecane	4,67	-

33	28,25/28,35	Phenyl methylene) oc-)2-tanal	13,70	11,14
34	29,06	Myristic acid	15,53	-
35	29,07	**	-	72,48
36	29,13	Nonadecane	13,84	-
37	29,83	***	6,03	-
38	30,04	1-Hexylheptyl)-benzene)	14,25	10,68
39	30,28	6,10,14-Pentadec-2-one	5,99	26,04
40	30,68	Pentadecanoic acid	12,17	61,87
41	31,26/31,23	Farnesyl acetone C	9,80	35,95
42	31,50	Methyl palmitate	16,70	13,38
43	31,81	Palmitoleic acid	42,44	141,24
44	32,19/32,22	Palmitic acid	<b>150,02</b>	<b>420,60</b>
45	32,45	Ethyl palmitate	-	49,41
46	32,80	14-β-H-pregnane	-	15,90
47	33,73/33,66	Methyl linolenoate	3,55	7,49
48	34,16/34,18	Linolic acid	2,91	39,82
49	34,23/34,27	Linolenic acid	3,33	26,67
50	34,26/34,30	Oleic acid	13,57	95,33
51	34,58/34,44	Stearic acid	6,36	3,68
52	34,59	****	-	40,95
53	35,99	Farnesol	-	3,29
54	36,23	Tricosane	6,06	14,27
55	36,97	Tetracosane	2,81	5,77
56	37,22/37,28	Pentacosane	3,57	11,40
57	38,28/38,17	Hexacosane	2,96	4,86
58	40,14	Heptacosane	12,70	68,94
59	41,12/41,14	Squalene	25,52	<b>353,32</b>
60	41,88	Nonacosane	35,48	73,19

Note: \*\*\*\* - Unidentified.

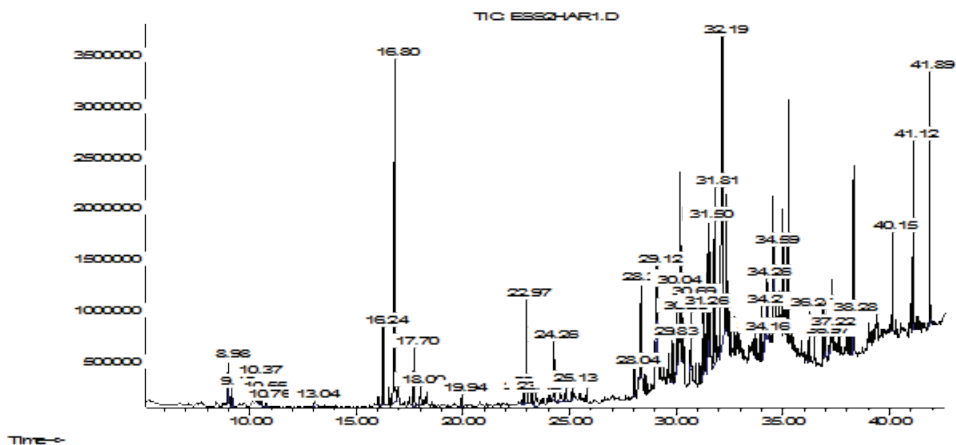


Fig. 1. Chromatogram of *M. forskahlei* flowers volatile fraction components

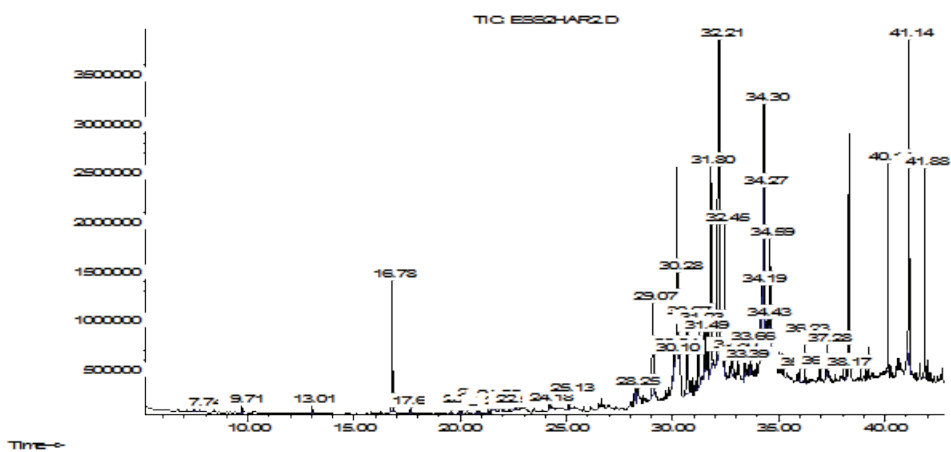


Fig. 2. Chromatogram of *M. forskahlei* herb volatile fraction components

Our research has shown the high content of fatty acids in *M. forskahlei* herb, in particular palmitoleic, palmitic and oleic acids.

High content of squalene – a precursor of steroidal compounds – was found in *M. forskahlei* herb.

A study done earlier mentioned the importance of squalene as one of the contributing factor for the epidemiological observations of reduced risk for



several cancers [10]. Squalene has been added in low dose with pravastatin in hyperlipidemic patients [11]. In addition, it might act as a “sink” for highly lipophilic xenobiotics [12].

### Conclusions

Our research has allowed detecting at least 59 compounds, 55 of which were identified. Furthermore *M. forskahlei* herb had higher content of volatile compounds than the flowers. Also our study of the chemical composition of *M. forskahlei* shows the existence of various amino acids, which allows us to consider the possibility of the use of this species to be used as a component of phytoremedies.

We also found that palmitic acid is one of the main components in the flower. In addition we discovered that squalene and palmitic acid are the important components that exist in the herb.

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