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A Germacrene –D, caracteristic essential oil from *A. microcarpus* Salzm and Viv. flowers growing in Algeria

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ABSTRACT

The present study deals with the essential oil composition of *Asphodelus microcarpus* (Liliaceae) growing in Algeria. The flowers essential oils were analyzed by GC/MS for the first time. Fourty nine compounds were identified in the crude oil characterized mainly by a high content of Germacrene D (**78.3**%), Germacrene B (3.9 %), á-Elemene (3.8 %)and Caryophyllene (3.3 %). To the best of our knowledge no studies have been reported on *A. microcarpus* essential oil so far.

Key words: *Asphodelus microcarpus*; (Liliaceae); Essential oil; Chemical composition; GC/MS.

Introduction

Essential oils are important natural products used for their flavor and fragrances in food, pharmaceutical and perfumery industries. they are also sources of aroma chemicals (Miguel, M.G., 2010).

They can be synthesized by all plant organs (flowers, buds, seeds, leaves, twigs, bark, herbs, wood, fruits and root) and therefore extracted from these parts. The proportions of the components present in essential oils vary greatly. Major components can constitute up to 85% of the essential oils, while the remaining components can be present in only trace amounts (Cavaleiro, C.M.F., 2001).

The subfamily Asphodeloideae (Asphodelaceae) comprises nine genera with approximately 261 species (Smith, G.F. and Van B.E. Wyk, 1991).

The genus *Asphodelus* (Liliaceae) is represented in the Algerian flora of by six species, namely : *A. acaulis* Desf., *A. cerasiferus* J. Gay, *A. refractus* Boiss., *A. fistulosus* L., *A. tenuifolius* Cav. and *A. microcarpus* Salzm et Viv. (Quezel, P., S. Santa, 1962)

A. microcarpus Salzm et Viv. (local name "berouaga") is a perennial tuberous geophyte of the family Asphodelaceae, widely distributed over the Mediterranean basin (Diaz Lifante, Z., 1996; Polunin, O., A. Huxley, 1965). Meat or white flowers, 15 mm long max. Tepals keeled, Keel green or purple. Capsule 6-14 mm long, oblong. ovoid or subglobose, distinctly wrinkled transversely to valves on dry (Quezel, P., S. Santa, 1962)

In Algerian folk medicine, *Asphodelus microcarpus* was used for the treatment of various diseases, such as ear pain, rheumatism, colds and eczema (Hammouda, F.M., *et al*., 1971).

Previous work on members of this species revealed that the main constituents are anthraquinones, flavonoids glycosides, resins, fixed oil and alkaloids (Hammouda, F.M., *et al*., 1971; Hammouda, F.M., *et al*., 1972; Rizk, A.M. and F.M. Hammouda, 1970; Rizk, A.M., *et al*., 1972; SISINI, A., *et al*., 1978; Zellagui A., *et al*., 2003).

To the best of the authors' knowledge, there are no reports about the chemical content of the essential oils of *Asphodelus microcarpus*.

Experimental:

Plant material:

The flowers of *Asphodelus microcarpus* were collected in Mars 2012 (flowering stage) in grarem-Mila , Algeria. The plant was identified by Dr. zellagui amar , department of life sciences and nature, University Larbi Ben M'hidi, Oum el Bouaghi Algeria. A voucher specimen was deposited at the life sciences and nature Department, University Larbi Ben M'hidi, Oum el Bouaghi, Algeria under the code number ZA11.

Extraction:

Essential oils were obtained by hydrodistillation of 100g of dried flowers using a Clevenger-type apparatus for 3 h. diethyl ether (10 ml) was used as the collector solvent as reported in literature. After evaporation of the

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solvent, the oil was dried over anhydrous sodium sulphate and stored in sealed vials protected from the light at −20 °C before analyses. The oil sample was subsequently analyzed by GC-MS .

Identification of components:

Gas chromatography/mass spectrometry (GC/MS):

The oil was analyzed by GC/MS using a Agilent 5973EI mass selective detector coupled with an Agilent GC6890A gas chromatograph, equipped with a cross-linked 5% PH ME siloxane HP-5MS capillary column (30m x 0.32mm, film thickness 0.25µm). Operating conditions: The carrier gas flow was 1.6 ml He/min, column pressure was 100 Kpa. The injector and detector temperatures were 220°C and 250°C respectively. The column temperature was held at 60°C for 1 min, then raised from 60°C to 200°C at 10°C/min and held there for 5 min and from 200°C to 240°C at 10°C /min and held there for 6 min. The program was run in the splitless mode with a mass range of 50–400 u, and the scan interval was 0.5 s. Detector voltage was set at 1.5 kV.

Identification of components:

Identification of oil components was achieved on the basis of their retention indices RI, (determined with reference to a homologous series of normal alkanes), and by comparison of

their mass spectral fragmentation patterns with those reported in the literature and stored on the MS library (NIST database). The concentration of the identified compounds was computed from the GC peak total area without any correction factor.

Results And Discussion

The hydrodistillation of the flowers of *A. microcarpus* yielded 0.05 % of a yellowish oil. The composition and percentage of the compounds are summarized in Table 1 and listed by their increasing order of retention times. Fifty one compounds were identified in the essential oils, representing **99.8%** of the total oil. The main constituents of the essential oil were Germacrene D (**68.3**%), Germacrene B (3.9 %), á-Elemene (3.8 %) and Caryophyllene (3.3 %) and some other compounds were only present in minor amounts. The essential oils were dominated by a large amount of sesquiterpenes (83.6 %) and oxygenated sesquiterpenes (3.9 %), while the monoterpenes hydrocarbons 0.5 %.

In total, essential oil composition of *A. microcarpus* was considered as a rich source of sesquiterpenes. This paper is the first study of the composition of the essential oil obtained from the leaves and fruits of *A. microcarpus* growing in Algeria.

Table 1: composition of the flowers essential oils of the *A. microcarpus*

no	tr	Constituent	%
1	6.763	á-Myrcene	0.4
2	8.737	á-trans-ocimene	0.1
3	17.172	2-Tridecenal, (E)-	0.1
4	18.910	Thymol	0.2
5	20.977	Elixene	0.1
6	21.466	Tetracyclo[5.2.1.0(2,6).0(3,5)]decane, 4,4-dimethyl-	tr
7	21.632	à -Cubebene	0.1
8	22.659	Copaene	0.8
9	23.345	á-Elemene	3.8
10	24.309	Caryophyllene	3.3
11	27.588	Germacrene D	78.3
12	25.303	à-Amorphene	0.2
13	25.616	à -Caryophyllene	0.7
14	27.806	Germacrene B	3.9
15	28.093	ë-Cadinene	1.6
16	28.255	ç-Muurolene	0.5
17	28.932	Naphthalene, 1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)-	tr
18	29.518	4,7-Octadecadiynoic acid, methyl ester	0.1
19	29.841	Aromadendrene oxide-(2	0.1
20	29.998	1R,3Z,9s-4,11,11-Trimethyl-8-methylenebicyclo[7.2.0]undec-3-ene	0.1
21	30.346	Germacrene D-4-ol	0.6
22	30.587	Thujopsene	0.1
23	30.727	2-(1,4,4-Trimethyl-cyclohex-2-enyl)-ethanol	0.3
24	30.911	1-Methyl-6-(3-methylbuta-1,3-dienyl)-7-oxabicyclo[4.1.0]heptane	0.2
25	31.149	1-Oxaspiro[2.5]octane, 5,5-dimethyl-4-(3-methyl-1,3-butadienyl)-	tr
26	31.280	Caryophyllene oxide	tr
27	31.462	Z-3-Hexadecen-7-yne	0.1
28	31.593	Andrographolide	0.8

29	31.796	Drimenol	0.1
30	32.346	Di-epi- à -cedrene	0.1
31	32.547	Cedrenol	0.1
32	32.814	(-)-ë-Cadinol= .(-)-Cedreanol	0.6
33	32.942	Ylangene	0.1
34	33.248	à -Cadinol	0.9
35	33.540	5-Ethyltricyclo[4.3.1.1(2,5)]undec-3-en-10-one	tr
36	33.935	ç-Eudesmol	0.1
37	35.028	1,3,3-Trimethylcyclohex-1-ene-4-carboxaldehyde, (+,-)-	0.1
38	35.694	cis,. à -Santalol,	0.1
39	35.867	Cyclopropanemethanol, 2,2-dimethyl-3-(2-methyl-1-propenyl)-	0.2
40	36.676	Cyclohexane-1-methanol, 3,3-dimethyl-2-(3-methyl-1,3-butadienyl)-	0.2
41	36.817	Tricyclo[4.4.0.0(2,7)]dec-3-ene-3-methanol, 1-methyl-8-(1-methylethyl)-	0.2
42	36.947	3-Isopropyl-6,7-dimethyltricyclo[4.4.0.0(2,8)]decane-9,10-diol	0.1
43	37.067	Cyclopenta[1,3]cyclopropa[1,2]cyclohepten-3(3aH)-one, 1,2,3b,6,7,8-hexahydro-6,6-dimethyl-	tr
44	38.187	4,7,10,13,16,19-Docosahexaenoic acid, methyl ester, (all-Z)-	0.1
45	41.065	1-Oxaspiro[2.5]octane, 5,5-dimethyl-4-(3-methyl-1,3-butadienyl)-	0.1
46	41.280	2-Methyl-4-(2,6,6-trimethylcyclohex-1-enyl)but-2-en-1-ol	tr
47	44.899	n-Hexadecanoic acid	0.1
48	45.548	Cycloisolongifolene, 8,9-dehydro-9-formyl-	tr
49	46.347	Falcarinol	0.1
total			99.8

Table 3: main classes of essential oils components of *A. microcarpus*

Sesquiterpenes hydrocarbons	83.6	
Monoterpene Hydrocarbons	0.5	
Oxygenated Monoterpenes	0.4	
Oxygenated Sesquiterpenes	3.9	
others	2.9	

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