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Chemical Composition of the Essential Oil of *Haplophyllum tuberculatum* (Forssk.) L.A. Juss. From Algeria.

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ABSTRACT

The chemical composition of the essential oil of *Haplophyllum tuberculatum* (Forssk.) L. A. Juss. was analysed by GC and GC–MS, the identified components constituting 82.5% of the oil. The main constituents were □-phellandrene (2.1%), □-phellandrene (3.0%), terpinene-4-ol (3.2.%) , p-cymene-8-ol (2.9%), piperitone (17.8), 2,4-bis(1,1-dimethylethyl)-phenol (28.3%), (1E,4E)-germacrene B (2.1%), hexadec-1-ene (3.2%) and octadec-1-ene (2.1%).

Keywords: *Haplophyllum tuberculatum*, essential oil, piperitone, 2,4-bis(1,1-dimethylethyl)-phenol (28.3%).

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INTRODUCTION

The genus *Haplophyllum* belonging to the Rutaceae family comprises about 70 species distributed from the Mediterranean area to eastern Siberia [1]. It is represented by three species in the Algerian Flora [2]. *Haplophyllum tuberculatum* (Forssk.) L. A. Juss. is used in traditional medicine as a remedy for headaches and arthritis, the juice is applied as a wart removal, skin discoloration, infections and parasitic diseases [3]. It is also used to treat nervous system, infertility and fever [4]. Previous investigations on the essential oil of this species showed variable chemical composition. Sample from Iran was found to contain limonene (27,3%) and α-pinene (21,9 %) as major constituents [5]. Oil of this species from Oman revealed that α -phellandrene (23.3%), limonene (12.6%), (Z)- β-ocimene (12.3%), β-caryophyllene (11.6%), myrcene (11.3%) and β-phellandrene (10.9%) were the most abundant components [3]. In other sample the major components were trans-p-menth-2-en-1-ol (19.2%), cis-p-menth-2-en-1-ol (13.2%), myrcene (10.1%), *δ*-3-carene (8.8%), β-phellandrene (6.9%), limonene (6.6%) and cis-piperitol (6.4%) [6].

In continuation of our research program on Saharian species [7-16], we report in this work, the chemical composition of the essential oil of *Haplophyllum tuberculum* from Algeria. To the best of our knowledge the essential oil of this sample was not previously investigated.

MATERIAL AND METHODS

Experimental

Plant materiel

The studied sample was collected in April 2012 from flowering plants from the region of Beni Abbes in the Southwest of Algeria and identified by M. Benabdelhakem from the National Agency of Preservation of Natural Resources of Bechar. Voucher specimens are kept in the Herbarium of the Research Unity of Valorization of Natural Resources and Bioactive Molecules, University of Constantine, (HP-N°156-2012).

Isolation of the essential oil

The aerial parts (315g) of *Haplophyllum tuberculatum* were steam distilled in a Kaiser Lang apparatus.

GC and GC-MS analysis

The essential oil was analyzed on an Agilent gas chromatograph (GC-FID) Model 6890, equipped with a HP-5 ms fused silica capillary column having (5%-phenyl) methylpolysyloxane stationary phase (25 m x 0.25 mm, film thickness 0.25 m), programmed from 50°C (5 mn) to 250 °C at 3°/mn and held for 10 mn . Injector and flame ionization detector temperatures were 280 and 300 °C, respectively. The essential oils were diluted in acetone 3.5% (v/v) and injected in split mode (1/60), helium was used as a carrier gas (1.0 mL/mn). Solutions of standard alkanes (C8-C20) were analyzed under the same conditions to calculate retention indices (RI) with Van del Dool and Kratz equation.

Mass spectrometry was performed on an Agilent gas chromatograph-mass spectrometer (GCMS) Model 7890/5975, equipped with HP-5 capillary column (25 m x 0.25 mm, film thickness 0.25 m) programmed with the same conditions as for GC-FID. The mass spectrometer (MS) was in electron impact mode at 70 eV and electron multiplier was at 2200 V. Ion source and MS quadrupole temperatures were 230°C and 180°C, respectively. Mass spectral data were acquired in the scan mode in the *m/z* range 33-450. The essential oil constituents were identified by matching their mass spectra and retention indices (RI) with those of reference compounds from libraries such as Adams [17] and Mc Lafferty & Stauffer [18]. The proportions of the identified compounds were calculated by internal normalization.

RESULTS AND DISCUSSION

The yield of steam distillation was 0.91% (w/w) in relation to the dry weight of the plant. A total of twenty four constituents were determined which account for about 82.5% of the essential oil of *Haplophyllum*

tuberculatum. The components identified in the essential oil are listed in table 1 in order of their experimental retention times and retention indices.

The main constituents were α-phellandrene (2.1%), β-phellandrene (3.0%), terpinene-4-ol (3.2.%) , p-cymene-8-ol (2.9%), piperitone (17.8), 2,4-bis(1,1-dimethylethyl)-phenol (28.3%), (1E,4E)-germacrene B (2.1%), hexadec-1-ene (3.2%) and octadec-1-ene (2.1%). Oxygenated compounds represented 63,5 % of the total oil composition.

Table 1: Volatile oil composition of *Haplophyllum tuberculatum*

RI	RT	Compounds	%
1006	12,655	α-phellandrene	2.1
1017	13,015	α-terpinene	0.6
1031	13,491	β-phellandrene	3.0
1033	13,553	1,8-cineole	0.8
1069	14,726	cis-linalol oxide	0.5
1071	14,794	n-octanol	1.3
1099	15,725	linalol	0.5
1125	16,531	cis p-ment-2-en-1-ol	1.3
1144	17,082	menth-2-en-1-ol	1.7
1164	17,686	mentha-1,5-diene-8-ol	0.5
1182	18,241	terpinene-4-ol	3.2
1188	18,416	p-cymene-8-ol	2.9
1210	19,058	cis-piperitol	1.7
1212	19,122	trans-piperitol	0.7
1257	20,403	piperitone	17.8
1391	24,001	tetradecene-1	1.8
1494	26,567	valencene	0.6
1505	26,833	2,4-bis(1,1-dimethylethyl)-phenol	28.3
1559	28,093	(1E,4E)-germacrene B	2.1
1591	28,841	hexadec-1-ene	3.2
1675	30,72	gamma dodecalactone	0.9
1790	33,185	octadec-1-ene	2.1
1838	34,145	pentadec-2-one	0.5
1855	34,49	Dibutyl-phtalate	1.9
1949	36,345	phtalate	0.8
1990	37,132	eicos-1-ene	1.7

CONCLUSION

This study reported the chemical composition of the essential oil of *Haplophyllum tuberculatum* (Forssk.) L. A. Juss. Our results are different from the various results reported on this species and showed that the major compounds were piperitone (17.8%) and 2,4-bis(1,1-dimethylethyl)-phenol (28.3%).

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