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SHORT COMMUNICATION

Analytical Characterization of Volatile Active Principles from the Leaves of the Alboroseum Backer Plant

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Abstract

The study presents for the first time the data concerning the qualitative and quantitative determination of the volatile active principles from the leaves of the Alboroseum Backer plant (Crassulaceae). After the extraction of volatile active principles in water, analytical separation and quantitative determination using a GC/MS technique was performed. The compounds detected, are belonging to the following classes: aldehydes, ketones, aromatic hydrocarbons and alcohols.

Keywords Alboroseum Backer; Crassulaceae; GC/MS; 2,4-dimethylpentanal.

Introduction

Alboroseum Backer is known as a decorative herb. Lately, a polyuronide type active principle having antibacterial and immunomodulators properties was separated from Alboroseum Backer fresh leaves [1-3]. Since other active principles could exist in composition of the leaves, gas chromatography coupled with mass spectrometry was utilized to identify and quantify them. The GC/MS technique is usually used for the identification and quantitative analyses of volatile active principles in herbal.

Experimental

Materials and Method

In order to obtain a watery extract of volatile compounds from dried leaves, a dynamic thermal stripper method for collection and thermal desorbtion was used. To accomplish this, 2 g sample of dried leaves were washed with a carrier gas (He), then the carrier gas passed through an absorbant cartridge (CARBOTRAP), which presents four different absorbants for compounds with different molecular mass. Desorption was accomplished in ultrafiltrated distilled water, obtaining from this procedure an aqueous extract that contains the volatile compounds.

The sample designated for GC/MS analysis was prepared into a SUPELCO 890-891 Thermal Desorption Unit (TDU) as follows: 1 μL from vegetal extract previously obtained was absorbed into a C_{400} SUPELCO cartridge under 350^0C, for 4 minutes, in a flow of 2.65 L min^{-1} of helium current. This cartridge was coupled with a C_{301} type desorption cartridge, of 1.5 mm diameter at 350^0C and a flow of 5 mL min^{-1} of carrier gas. During thermal desorption, the cartridge is appropriately located inside the desorption device so that the carrier gas passes vice-versa than in the case of absorption.

The separation and identification of volatile active principles were made by means a gas chromatograph of VARIAN - 3400 type coupled with a quadripolar mass spectrometer of NERMAG R-10-10B4-1500 amu type. To separate the volatile compounds a PTE-5 SUPELCO chromatographic capillary column having 30 m x 0.32 mm x 0.25 μm was used. The stationary phase was a mixture of 5% diphenyl-

siloxan and 95% dimethylsiloxan. The temperature programme was: 70^0C (8 min), 250^0C - 4^0C min^{-1}, 295^0C - 15^0C min^{-1} (5 min). The carrier gas was He (3mL min^{-1}).

Identification of individual organic compounds was performed comparing the obtained mass spectrums with NERMAG spectrum' s libraries [4,5].

Results and Discussions

Figure 1 shows the chromatogram of volatile compounds. The results obtained in the chromatogram (Figure 1) were compared with data belonging to spectrum' s libraries of mass spectrometer. The concentration of volatile compounds was reported to the amount of dried leaves of 2 g that underwent extraction.

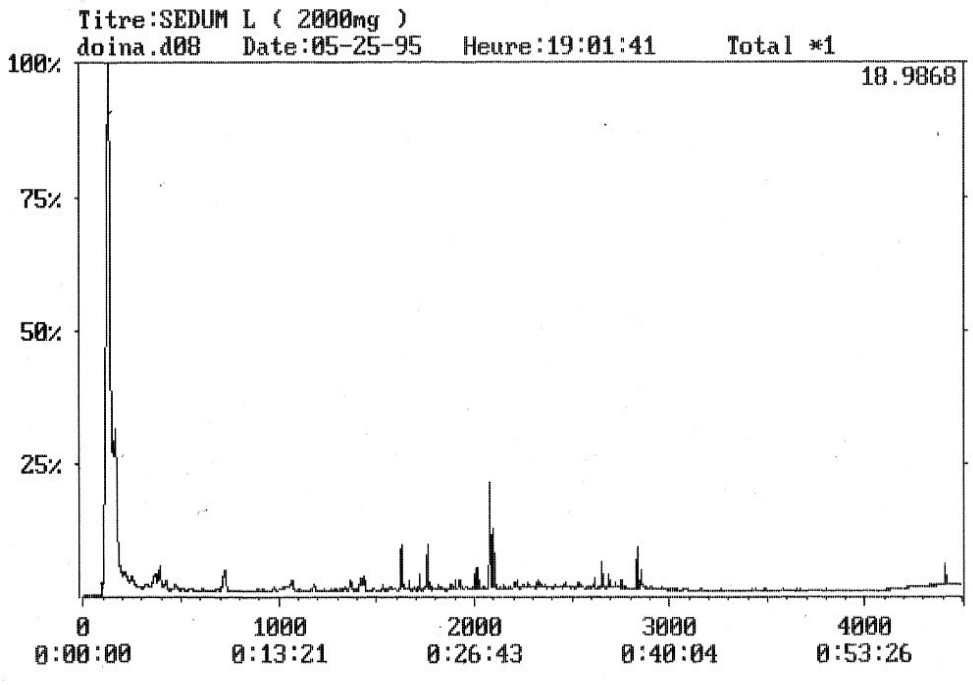

Figure 1 The chromatogram of volatile compounds.

Table 1 comprises all the identified volatile substances that were characterized from the point of view of molecular mass and their concentration in the leaves. The highest percentage of 33.5 was found for 2,4-dimethylpentanal, the interval ranging from 1 to 33.5% comprises three compounds belonging to different classes: aldehydes, hydrocarbons, and alcohols.

Table 1 The quantitative evaluation of volatile compounds from Alboroseum Backer leaves.

No.	Compounds	Molecular Mass	Conc. mg g^{-1} dried leaves	% (w/w)
1.	2,4-Dimethylpentanal	114	669.70	33.5
2.	2-Tridecanol	200	27.50	1.38
3.	2-Propenylidenecyclobutene	92	24.40	1.20
4.	2-Methyl,1-(1,1-dimethylethyl)-2-methyl-1,3-propanediyl	286	17.97	0.90
5.	1a,2,3,5,6,7,7a,7b-octahydro-1,1,7,7a-tetramethyl-1*H*-cyclopropa[*a*]naphthalene	204	17.23	0.86
6.	Butyl 2-methylpropyl 1,2-benzenedicarboxylate	278	15.54	0.78
7.	3,7-Octadien-2-one	124	11.80	0.60
8.	5,9-Dimethyl-1-decanol	186	8.86	0.44
9.	2-Tridecanone	198	8.16	0.41
10.	Palmitic acid	256	7.67	0.38
11.	1-Butylcyclobutanol	128	7.10	0.36
12.	[5,1-*c*]-*as*-Triazin-4-pyrazole	240	6.39	0.30
13.	1-(Ethenyloxy)hexadecane	268	5.80	0.30
14.	1-[3-Methyl-2-(1-pyrrolidinyl)]cyclobutylethanone	181	4.40	0.22
15.	2,3-Dimethylpentanal	114	4.35	0.22
16.	4-Methyl-2,6-ditert.-butylphenol	220	3.92	0.20
17.	4,11,11,trimethyl-8-methylenebicyclo[7.2.0]un-dec-4-ene	204	4.00	0.20
18.	6-10-Dimethyl-5,9-undecadien-2-one	194	3.60	0.18
19.	Cyclopropa[*d*]naphthalen-2(4a*H*)-one	204	3.36	0.17
20.	6,10-Dimethyl-2-undecanone	198	3.26	0.16
21.	2-Heptadecanol	256	2.84	0.14
22.	Bicyclo[4.4.11]undeca-1,3,5,7,9-pentaen-11-one	156	2.60	0.13
23.	2-Tetradecanol	214	2.30	0.12
24.	6,10,14-Trimethyl-5,9,13-pentadecatrien-2-one	262	2.30	0.12
25.	1-Methylnaphthalene	142	1.85	0.09
26.	4,7-Dimethyl-1,6-octadien-4-ol	154	1.70	0.09
27.	2-Chloro-6-methylphenol	142	1.5	0.08
28.	2-Undecanol	172	1.60	0.08
29.	Methyl 2-ethyl-2-propylhexanoate	200	1.67	0.08
30.	(*E,E*)-2,4-Decadienal	152	1.46	0.07
31.	3,7,11-Trimethyl-1,6,10-dodecatrien-3-ol	222	0.90	0.05

All volatile ketones were detected in the range between 0.1 – 0.5%. Four of seven compounds having the concentrations below 0.1% are alcohols.

Conclusions

The study presents for the first time data concerning the qualitative and quantitative determination of the volatile active principles from the leaves of Alboroseum Backer plant. The majority of the volatile compounds are alcohols followed by ketones. 2,4-Dimethylpentanal was detected in the highest proportion. Therefore, it is considered the main component responsible for the pharmaceutical activity of the herb. Accordingly, a separation method for this component should be established in order to study its full biological properties.

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