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Hydro-distillation extraction of volatile components of cultivated *Bunium luristanicum Rech.f.* from west of Iran

Mohammad Hadi Meshkatalsadat^{*} Rashid Badri and Shahbanoo Zarei

^{*}Department of Chemistry, Lorestan University P.O.Box 465, Khoramabad, Iran

Abstract: Volatile constituents of *Bunium luristanicum Rech.f* was obtained by hydro-distillation and analyzed by gas chromatography-mass spectrometry (GC-MS). A total of fourty three compounds accounting for 95.16% of the black cumin(B.l) extracted by HD were identified as γ -Terpinene (5.6 %), Isopulegol acetate (24.64 %) , α -Pinene (5.16 %), Anethole(20.36%) and Camphor(10.43%), were the major compounds identified in the plant.

Keywords: Essential oil; Bunium luristanicum Rech.f y-terpinene Camphor Anethole

Introduction

Black cumin is an economically important umbellifer growing wild in the dry temperature regions of Jammu-Kashmir, Himachal Pradesh, Afghanistan, Baluchestan and Iran. The seeds, rich in essential oil, are consumed widely as condiment. In the indigenous system of medicines, seeds are regarded as stimulants and carminatives and found to be useful in diarrhoea and dyspepsia¹. Also, this plant is used for culinary purposes and for flavouring foods and beverages. It should be noted that the name of black cumin is sometimes given to entirely unrelated spice Nigella sativa L^{2,3}. The hydrodistillation of black cumin seeds has been reported previously. According to the reports, black cumin seeds contain essential oils rich in monoterpene aldehydes; the main components are cuminaldehyde, p-mentha-1,3-dien-7-al and *p*-mentha-1,4-dien-7-al; terpene hydrocarbons are γ -terpinene, *p*-cymene, β -pinene. The latter compounds are thought to reduce the quality of the spice^{1,4}. The objective of this study was to obtain composition of essential oil and analyzed by gas chromatography-mass spectrometry (GC-MS) ...

Materials and methods Plant materials

About100 g of fresh aerial part at maturity were collected from agriculture college garden of Lorestan university,Khoramabad Iran, on june 2007. The dried aerial parts were stored in a dark place at 4°C.

Hydro-distillation

The sample (100 g of dried material was charged with a particle size of about 500 μ m) was submitted to hydrodistillation for 1.5 h, using a Clevenger-type apparatus, according to the <u>European Pharmacopoeia (1975)</u>⁵. The volatile distillate was collected over anhydrous sodium sulphate and refrigerated until time of analysis. The yield of the oil was 3.1% v/w based on dry plant weight.

Gas chromatography-mass spectrometry

GC analyses were carried out on a Shimutzu 17A gas chromatograph and a BP-5 (non-polar and 95 % dimethyl polysiloxane) capillary column (30 m \times 0.25 mm; 0.25 µm film thickness). The oven temperature was held at 60 °C for 3 min then programmed at 5°C /min to 300 °C. Other operating conditions were as follows: carrier gas He, with a flow rate of 5 ml/min; injector temperature 230°C; detector temperature 300 °C; split analyses were performed on a ratio, 1:8. GC/MS Shimutsu 17A GC coupled with Shimutsu QGD5050 Mass system. The operating conditions were the same conditions as described above but the carrier gas was He Mass spectra were taken at 70 eV. Mass range was from m/z 50–450 amu. Quantitative data were obtained from the electronic integration of the peak areas. Retention indices were calculated using co-chromatographic standard hydrocarbons. The composition and the yields are listed in. Table 1.

NO	compounds	Tn	RI	(%)
1	alpha-thajene	9.51	930	0.1
2	-pinene α	9.75	939	2.8
3	Sabinene	10.64	975	0.3
4	- pinene β 2-	10.81	979	2.4
5	-Myrcene β	10.96	991	0.5
6	-Terpinene α	11.78	1017	0.1
7	Cymol	11.87	1016	1.7
8	dl-limonene	12.18	1029	9.7
9	gamma-ter pinene	12.91	1060	2.9
10	Fenchone	13.52	1087	1.8
11	- Terpinolene α	13.76	1089	1.1
12	O-Fenchylalcohol	14.5	1109	0.1
13	Camphor	15.04	1148	0.1
14	1,3-cyclohexadiene	16.38	1175	0.2
15	p-Allylanisole	16.51	1179	4.7
16	Fenchyl acetate	17.43	1220	0.4
17	Cuminaldehyde	17.62	1242	1.8
18	-fenchyl acetate α	17.84	1227	5.2
19	Anethole-Z	17.98	1253	1.2
20	Anethole-E	19.24	1225	60.9
21	p-Allylanisole	19.28	1279	0.1
22	Tetradecane	22.52	1400	0.1
23	trans β Caryophyllene-	23.23	1419	0.2
24	β Fa r nesene (E) -	23.75	1457	0.1
25	α Hamalene-	24.05	1455	0.1
26	Fenchyin-valepate	24.13	1465	0.1
27	Fenchyin-valepate	24.31	1472	0.1
28	Germacrene D	24.67	1485	0.2
29	(-) – spathalenol	25.6	1578	0.1
30	Nerolidol-E	26.25	1563	0.1
31	Hexadecane	27.42	1600	0.1
32	Benzyl benzoate	30.44	1760	0.3
33	Octadecane	31.92	1800	0.1
34	neophytaDine	32.74	1839	0.1
35	phytol	38.14	1943	0.8

 Table .1Chemical composition of Iranian Bunium luristanicum Rech.f; oils obtained by hydro-distillation (HD). The compounds were listed in order of elution time (Tn : Retention Time , RI: Kovats Constant)

Results and discussion

We compared our results with those of the Indian⁴ and Tajikistan¹ black cumin samples. In all oils, γ terpinene and cuminaldehyde were the major constituents, but our oil was higher in Isopulegol acetate(24.64%), Camphor(10.43%), Anethole (20.36%). On the other hand, in Indian and Tajikistan oil, *p*-mentha-1,4-dien-7-al was the major constituent, whereas, this component was not found in our oil. It is worthy to note that Indian and Tajikistan oils were obtained by hydro-distillation method. These differences might have been derived both from harvest time and local, climatic and seasonal factors or we may hypothesize

that the Iranian sample belongs to a different chemotype. However, further investigations are needed to elucidate this hypothesis.

The constituents of the oil were identified by calculation of their retention indices under temperature-programmed conditions for *n*-alkenes (C_6 - C_{24}) and the oil on BP-5 column under the same conditions. Identification of individual compounds was made by comparison of their mass spectra with those of the internal reference mass spectra library

(Wiley 5.0) or with authentic compounds and confirmed by comparison of their retention indices with authentic compounds and with those of reported in the literature.^{6,7} Quantitative data was obtained from FID area percentages without the use of correction factors. A total of nineteen compounds representing 95.16% of the total components in the oil, have been identified in the essential oil extracted from the aerial parts of the Bunium luristanicum Rech.f; y-Terpinene (5.6 %), Isopulegol acetate %) ,*α*-Pinene (5.16 (24.64 %). Anethole(20.36%) and Camphor(10.43%), were the major compounds identified in the plant. The oil of Bunium luristanicum Rech.f; consisted mainly of monoterpene hydrocarbons (%), oxygenated monoterpenes (%), sesquiterpene hydrocarbons (%) oxygenated sesquiterpenes (%), oxygenet diterpene (%) and one aliphatic aldehyde (%) Isopulegol acetate was the dominant compound of the essential oil.

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