Content of essential oil obtained from flower heads of *Echinacea purpurea* L. and identification of selected components

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Summary

Hydrodistilled essential oil from flower heads of *Echinacea purpurea* (L.) Moench cultivated in Slovakia was analyzed by GC/MS method. Seventy-two components were identified and determined. As a result of studying the content and composition of the essential oil obtained from flower heads of *E. purpurea* it was established that the content of the essential oil was 1.85% (v/w) of the weight of dry material. The main components in the studied samples were palmitic acid, α-pinene, germacrene-D, β-pinene, and α-phelandrene. The main components of the analyzed oil were palmitic acid (8.3%), nerolidol (6.6%), α-pinene (5.1%), germacrene-D (4.8%), α-phelandrene (4.3%), and β-pinene (4.1%).

Key words: Echinacea, flower-heads, essential oil, gas chromatography, mass spectrometry, germacrene

Echinacea purpurea L. was used as a testing material. *Echinacea purpurea* L. is a perennial plant of the *Asteraceae* family, native to the Canadian prairies. For some time the plants have been cultivated in Europe (mainly Germany). Three species of *Echinacea* are generally used in medicine: *E. angustifolia* DC, *E. pallida* (Nutt.) Nutt. and *E. purpurea* (L.) Moench, which is also known as "purple coneflower". Plant parts used in herbal medicine include flowers, tops, roots and aerial parts. Several components of *Echinacea*, such as alkamides, caffeic acid derivatives (especially cichoric acid), glycoproteins, polysacharides, polyacetylenes have been reported to have immunostimulatory, anti-inflammatory, antiviral and antioxidant properties, but the mechanism behind these effects is not understood well [1-6]. It was concluded that the pharmacological activity of the *Echinacea* preparations depended on the combined activities of several plant constituents [6]. The headspace volatile components of roots, stems, leaves and flowers of *E. angustifolia*, *E. pallida* and *E. purpurea* were analysed and identified [7]. Extracts were anal-

ysed by GC/MS methods and over 70 components were identified in the samples. GC/MS methods were used to verify the authenticity of extracts of roots from different species of *Echinacea* [8]. Various extraction methods were applied, i.e. Soxhlet extraction, supercritical fluid extraction and maceration with three different solvents. The volatile components of *Echinacea*, however, have not been studied thoroughly, and no results have been published. Recently an analysis of the essential oils from the aerial parts of *E. purpurea* from healthy and infected (with cucumber mosaic cucumovirus) plants has been published [9]. The hydrodistilled essential oils were analysed by GC/MS.

MATERIAL AND METHODS

Plant

Echinacea purpurea L. was used as a testing material. The plant material was collected in the phase of full flowering flower heads. The medicinal plants were cultivated in the vicinity of Nitra in Slovakia. A detailed agro-ecological characteristic of individual localities is available at the offices of the authors.

Determination of oil content in drug

Twenty grams of dry flower heads were subjected to hydrodistillation for 3.5 hours in accordance to the European Pharmacopoea [12]. Isolated oil was diluted in n-hexane and dried over anhydrous sodium sulfate.

Oil analysis by GLC method

Oil samples were analysed using a Hewlett Packard HP 5971A mass selective detector directly coupled to an HP 5890 Series II FID gas chromatograph. A capillary column DB-WAX/25m x 0.20 mm, 0.2 mm film thickness (Hewlett Packard, USA) was used. The temperature program was as follows: 40°C – 250°C at 3°C/min. The injection port temperature was 220°C. Helium was used as a carrier gas, split ratio 1:50. Mass spectra were recorded at ionisation energy (EI) 70 eV.

Oil components were identified by comparison of their mass spectra with those from the databases NBS 75 K, INRA MASS (LRSA, Dijon, France), Wiley 138 and NIST.

RESULTS AND DISCUSSION

The oil isolated by hydrodistillation from the flowers of *Echinacea purpurea* (L.) Moench was found to be a pale yellow and the yield was 1.85% (v/w) of the weight of dry plant material.

In Table 1 a list of the chromatographic peaks identified by GC/MS methods with their percentage contents is presented, according to their elution order. The oil was separated to ninety components, representing 97.1% of the total yield. Seventy-two components were identified and their relative percentage determined, comprising 82% of the total yield. The components were identified by comparison with library mass spectra. The percentage composotion was calculated from FID area values without the use of correction factor.

The results in Table 1 show the occurrence of palmitic acid (8.3%), nerolidol (6.6%), α-pinene (5.1%), germacrene-D (4.8%), α-phelandrene (4.3%) and β-pinene (4.1%). Germacrene-D has frequently been reported as a typical component of *E. purpurea* flower oil [2, 4, 6, 9]. Comparing the oil composition (Table 1) with the composition of the oil analysed by Hudaib et al. [9], we can see many differences, as far as qualitative and quantitative compositions of these two samples are concerned. The flower oil analysed by some authors [9-11] contained high amount of germacrene-D (57.8%), while in the flower oil from *Echinacea purpurea* (L.) that was analysed by us we detected significantly lower levels of the compound (4.8%).

In conclusion, from the the data presented and those from literature [7] it is evident that the content of germacreme-D in oil from flower heads of *Echinacea purpurea* from diverse sources is different. We suppose that this variability may be caused by the origin of the plant, as well as part of the locality of its cultivation, environmental conditions and development stage of the plant. Furthermore, the possible contribution of some other factors, like viral infection (CMV) to the oil yield and composition is to be considered [9].

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Table 1

Composition of essential oil from flower heads of *Echinacea purpurea* (L.) Moench.

peak	Rt (min)	component	area %
1	4.17	α-pinene	5.2
2	4.90	camphene	0.3
3	6.13	sabinene	0.4
4	5.80	β-pinene	4.2
5	6.19	verbene	0.5
6	7.27	α-phelandrene	4.3
7	7.33	myrcene	0.3
8	8.15	limonene	1.4
9	8.36	β-phelandrene	0.1
10	8.62	mentha-1,4,8-triene	0.1
11	9.37	6-metyhyl-heptanon	0.1
12	10.25	p-cymene	2.4
13	17.09	α-campholene	0.2
14	19.39	pinocarvone	0.3

15	20.36	β-caryophyllene	1.0
16	21.21	myrtenal	0.4
17	22.29	pinocarvol	0.5
18	22.42	cis-verbenol	0.3
19	22.47	α-humulene	0.3
20	22.60	α-phelandrenol	0.2
21	23.53	verbenone	0.2
22	23.70	germacrene-D	4.8
23	24.36	D-carvone	0.2
24	24.48	p-mentha-1,5-dien-8-ol	0.4
25	25.15	Δ-cadinene	0.8
26	26.31	myrtenol	0.2
27	27.52	trans-carveol	0.2
28	29.47	5-epoxy-salvial-4-en	1.0
29	30.27	naphtalene	0.3
30	31.10	caryophyllene oxide	2.5
31	31.74	dihydromayurone	1.9
32	31.97	lasiocaprenolol	0.2
33	31.11	albicanol	1.0
34	32.41	pentadecan-2-one	0.2
35	32.50	humulene oxide	0.7
36	32.61	1-butyl-2,3,6-trimethyl-benzene	0.2
37	33.11	nerolidol	6.6
38	33.87	mezitylaceton	0.8
39	34.54	cyclodeca-1,5-diene	3.9
40	34.83	(+)-spatulenol	1.6
41	35.10	nor-copanon	0.9
42	35.50	α-cedrane	0.6
43	35.76	silvenone	0.3
44	35.89	cyclonona-1,4-diene	1.1
45	36.10	(-)isoledene	1.2
46	36.34	α-cadinol	0.3
47	37.04	thymol	0.4
48	37.34	widrol	3.7
49	37.34	viridiflorol	0.3
50	38.43	valerenol	0.7
51	38.51	decanoid acid	1.0
52	38.75	6-amino-1-imidazole-carboxilic acid	1.1
53	39.20	ethylpenthanylbenzene	0.4
54	39.32	9-methyl-nonadecane	0.2
55	39.45	3-methyl-3-ethenyl-cyclohexanone	0.6
56	39.87	1-acetyl-2-fenyl-3-methyl-pyrolidine	0.4
57	40.62	1,1-dimethyl-2,4-bis-methenyl-ethenyl-cyclohexane	1.0
58	40.98	paramentha-1,3,8-triene	2.7
59	41.51	linalic acetate	0.7
60	42.35	cyclooctene	0.2
61	43.17	lauric acid	0.5
62	43.43	camphane	0.3
63	43.66	n-licosane	0.4
64	43.94	copaenol	0.3
65	45.22	1-acetyl-trans-3-(hydroxymethyl)2-pyridil-pyrolidine	0.5
66	45.46	5-azulencarboxaldehyde	1.0
67	47.32	alkylphtalate	1.5
68	47.54	myristic acid	1.8
69	48.56	1-(3-buthenyl)cyclobutabenzene	1.4
70	50.08	pentadecanoic acid	0.4
71	53.37	palmitic acid	8.3
72	67.62	linolic acid	1.9

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OLEJEK ETERYCZNY Z KOSZYCZKÓW KWIATOWYCH ROŚLINY *ECHINACEA PURPUREA* (L.) MOENCH

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Streszczenie

Badano pochodzące ze Słowacji rośliny jeżówki purpurowej *Echinacea purpurea* (L.) Moench. Olejek otrzymano metodą destylacji z parą wodną, a następnie analizowano za pomocą GC i MS. Zidentyfikowano i oznaczono ilościowo 72 jego składniki. Najważniejszymi były kwas palmitowy (8,3%), nerolidol (6,6%), α-pinen (5,1%), germakren-D (4,8%), α-felandren (4,3%) i β-pinen (4,1%).

Słowa kluczowe: Echinacea purpurea, koszyczki kwiatowe, Asteraceae, skład chemiczny olejku eterycznego, chromatografia gazowa, spektroskopia masowa