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RESEARCH ARTICLE

Essential oil composition of two endemic *Centaurea* species from Turkey

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Abstract

Essential oil composition of *Centaurea austroanatolica* Hub.-Mor. and *Centaurea kizildaghensis* Uzunh., E. Doğan & H. Duman were analyzed by means of gas chromatography-mass spectrometry (GC-MS). Sixty and fourty six compounds were identified in the essential oils of *C. austroanatolica* and *C. kizildaghensis*, respectively. The major components were determined hexadecanoic acid (21.3%), caryophyllene oxide (4.8%), dodecanoic acid (4.2%) and heptacosane (3.5%) in *C. austroanatolica* oil, hexadecanoic acid (24.4%), phytol (9.3%), caryophyllene oxide (3.8%) and salvial-4(14)-en-1-one (3.1%) in *C. kizildaghensis* oil.

Keywords: *Centaurea austroanatolica*, *Centaurea kizildaghensis*, essential oil, GC-MS

Introduction

Centaurea L. species are member of Asteraceae family and distributed mainly in Southwest and Central and Eastern regions in Turkey. *Centaurea* has 226 species in 34 sections, of which 66% are endemic of the country. Turkey is the main center of diversity for *Centaurea* (Kültür et al., 2016). The aerial parts of several species of *Centaurea* are used in the traditional medicine for the treatment of diabetes, diarrhea, hypertension, malaria, microbial infections, rheumatism and ulcers (Baytop, 1999; Sarker et al., 1997; Uğur et al., 2009). But there is no report any medicinal uses of *C. austroanatolica* and *C. kizildaghensis* in traditional medicine in Turkey. Even though *Centaurea* L. is one of the largest genera of Asteraceae family, reports on the analysis of the essential oils of this genus are limited. There is only one report chemical composition and antimicrobial activity of endemic *Centaurea austroanatolica*. The main components of the chloroform extract of *Centaurea austroanatolica* were caryophyllene oxide (21.32%), spathulenol (10.86%), *n*-tricosanol (9.58%) and geranyl isovalerate (8.71%). The chloroform extract showed significant antibacterial activity toward all bacteria test (Uğur et al., 2009). Additionally, anthocyanin content was detected in *C. kizildaghensis* (Gokbel et al., 2015). Flavonoids from Turkish *C. austroanatolica* and *C. kizildaghensis* were investigated. Apigenin was identified from both species, genkwanin and quercetin from *C. austroanatolica* and genkwanin-4'-methyl ether from *C. kizildaghensis* (Uddin et al., 2017).

According to our literature survey there is only one report on the essential oil composition of any *C. austroanatolica*. This prompted us to investigate the essential oil composition of these species. To the best of our knowledge this is the first report on the essential oil composition of *C. austroanatolica* and *C. kizildaghensis*.

Materials and Methods

Plant materials

Plant materials were collected during the flowering period; *C. austroanatolica* on 19.06.2015 from Antalya, Kumluca District (250 m) and *C. kizildaghensis* on 11.07.2015 from Konya, Derebucak District (1960 m) in Turkey. Voucher specimens have been deposited in the Herbarium of Akdeniz University (Voucher no. AKDU 4139 and AKDU 4140 for *C. kizildaghensis* and *C. austroanatolica* respectively), Turkey.

Isolation of the essential oils

Aerial parts of the air dried plants subjected to hydrodistillation for 3 h, using a Clevenger-type apparatus to produce essential oils. Condenser of the Clevenger was attached to a microchiller that set to 4°C. *C. kizildaghensis* and *C. austroanatolica* afforded oils from the aerial parts with 0.01 and 0.02% (v/w) yields, respectively. The oils were recovered with 1 mL *n*-hexane and preserved in amber vials under -20°C until the day they were analyzed.

Gas chromatography/mass spectrometry analysis

The GC-MS analysis was performed with an Agilent 5975C Inert XL EI/CI MSD system operating in EI mode. Essential oil of *C. kizildaghensis* and *C. austroanatolica* were diluted 1/100 and 1/65 (v/v) with *n*-hexane, respectively. Injector and MS transfer line temperatures were set at 250˚C. Innowax FSC column (60 m (×) 0.25 mm, 0.25 µm film thickness) and helium as carrier gas (1 mL/min) were used in both GC/MS analyses. Splitless injection was employed. Oven temperature was programmed to 60˚C for 10 min. and raised to 220˚C at rate of 4˚C/min. Temperature kept constant at 220˚C for 10 min. and then raised to 240˚C at a rate of 1˚C/min. Mass spectra were recorded at 70 eV with the mass range *m*/*z* 35 to 425. Relative percentage amounts of the separated compounds were calculated from integration of the peaks in MS chromatograms. Identification of essential oil components were carried out by comparison of their relative retention indices (RRI) obtained by series of n-alkanes (C5 to C30) to the literature and with mass spectra comparison (Baser et al., 2006a, 2006b, 2008; Demirci et al., 2013; Maggio et al., 2012; Moronkola et al., 2009a, 2009b; Noorizadeh and Farmany, 2010; Özcan et al., 2001; Polatoğlu et al., 2010, 2011, 2013, 2014, 2015, 2016, 2017; Saidana et al., 2008; Schepetkin et al., 2016; Tabanca et al., 2001, 2006; Viegas and Bassoli, 2007). Mass spectra comparison was done by computer matching with commercial Wiley 8th Ed./NIST 05 Mass Spectra library, Adams Essential Oil Mass Spectral Library and Pallisade 600K Complete Mass Spectra Library. The analysis was carried out in triplicate and the results were given as the mean ± standard deviation.

Results and Discussion

Essential oil composition of *Centaurea austroanatolica* Hub.-Mor. and *Centaurea kizildaghensis* UzunH., E. Doğan & H. Duman were analyzed by means of gas chromatography-mass spectrometry (GC-MS). Sixty compounds were identified in the essential oil of *C. austroanatolica* that represent 76.1 ± 1.8% (*n*=3) of the oil. The main components of the essential oil were hexadecanoic acid (21.3 ± 0.9%), caryophyllene oxide (4.8 ± 0.1%), dodecanoic acid (4.2 ± 0.1%) and heptacosane (3.5 ± 0.1%). Forty six compounds were identified in the essential oil of *C. kizildaghensis* that represent 76.1 ± 0.5% (*n*=3) of the oil. The main components of the essential oil were hexadecanoic acid (24.4 ± 0.2%), phytol (9.4 ± 0.3%), caryophyllene oxide (3.8 ± 0.1%) and salvial-4(14)-en-1-one (3.1 ± 0.1%). The essential oil composition of *C. austroanatolica* and *C. kizildaghensis* are given in Table 1.

Hexadecanoic acid is the main compound of the both species. Hexadecanoic acid was previously found as the major compound of essential oils of *C. wagenitzii*, *C. tossiensis*, *C. luschaniana*, *C. iberica*, *C. hyalolepis*, *C. polyclada*, *C. aladagensis*, *C. hierapolitana*, *C. cadmea*, *C. calolepis*, *C. reuterana* var. *reuterana*, *C. depressa*, *C. urvillei* ssp. *urvillei*, *C. solstitalis* ssp. *solstitalis*, *C. balsamita*, *C. behen*, *C. aggregata* subsp. *aggregata* and *C. pichleri* ssp. *pichleri* from Turkey (Erdoğan et al., 2017; Erel et al., 2013; Karamenderes 2008; Köse et al., 2007, 2008; Tastan et al., 2017; Tuzun et al., 2017). Hexadecanoic acid was shown to increase the possibility of coronary heart diseases. People should be worried to use of *C. austroanatolica* and *C. kizildaghensis* essential oil might cause serious cardiac problems (Connor, 1999). The essential oil obtained from *C. austroanatolica* have considerable differences than the chloroform extract of *C. austroanatolica*. These differences in the previous literature and present data could be related to different collection times, climatic and soil conditions, ecological factors, methods and instruments employed in analysis or different genotypes. We believe the results obtained from this research will stimulate further research on the chemistry of *Centaurea* species.

Table 1. The essential oil composition of *C. kizildaghensis* and *C. austroanatolica*

				C. kizildaghensis					***C. austroanatolica***					
No	**RRI[1]**	**RRI Lit.[2]**	**Compound**	**I[3]**	**II**	**III**	**Mean[4]**	**SD[5]**	**I**	**II**	**III**	**Mean**	**SD**	**Id. Met.[6]**
1	1236	1239	2-pentyl furan	0.8	0.8	0.7	0.8	0.1	0.3	0.3	0.3	0.3	0.0	RI, MS
2	1398	1399	Nonanal	0.7	0.7	0.6	0.7	0.1	0.2	0.2	0.2	0.2	0.0	RI, MS
3	1401	1400	Tetradecane	0.1	0.1	0.1	0.1	0.0	-	-	-	-	-	RI, MS, Ac
4	1489	1497	α-Copaene	-	-	-	-	-	0.5	0.5	0.5	0.5	0.0	RI, MS
5	1499	1505	Dihydroedulan II	0.5	0.5	0.4	0.5	0.1	0.2	0.2	0.2	0.2	0.0	RI, MS
6	1504	1505	Decanal	-	-	-	-	-	0.4	0.4	0.4	0.4	0.0	RI, MS
7	1530	1535	Dihydroedulan I	-	-	-	-	-	0.1	0.1	0.2	0.1	0.1	RI, MS
8	1549	1553	(*Z*)-Theaspirane	-	-	-	-	-	0.2	0.2	0.3	0.2	0.1	RI, MS
9	1549	1553	β-linalool	0.4	0.4	0.4	0.4	0.0	-	-	-	-	-	RI, MS
10	1599	1600	β-Elemene	-	-	-	-	-	0.1	0.1	0.1	0.1	0.0	RI, MS
11	1602	1600	Hexadecane	-	-	-	-	-	0.2	0.2	0.2	0.2	0.0	RI, MS, Ac
12	1608	1608	β-caryophyllene	0.6	0.6	0.7	0.6	0.1	0.9	1.0	1.0	1.0	0.1	RI, MS
13	1633	1638	β-cyclocitral	0.4	0.4	0.4	0.4	0.0	-	-	-	-	-	RI, MS
14	1659	1661	Safranal	0.4	0.4	0.4	0.4	0.0	-	-	-	-	-	RI, MS
15	1661	1664	1-nonanol	0.4	0.4	0.3	0.4	0.1	0.2	0.2	0.2	0.2	0.0	RI, MS
16	1683	1687	α-humulene	0.1	0.1	0.1	0.1	0.0	-	-	-	-	-	RI, MS
17	1723	1723	Germacrene D	0.4	0.4	0.3	0.4	0.1	0.4	0.4	0.4	0.4	0.0	RI, MS
18	1735	1742	β-Selinene	-	-	-	-	-	0.3	0.3	0.3	0.3	0.0	RI, MS
19	1762	1763	Naphthalane	0.9	0.9	0.9	0.9	0.0	-	-	-	-	-	RI, MS
20	1764	1766	Decanol	-	-	-	-	-	0.1	0.1	0.1	0.1	0.0	RI, MS
21	1770	1773	δ-Cadinene	-	-	-	-	-	0.2	0.2	0.2	0.2	0.0	RI, MS
22	1776	1779	(*E*,*Z*)-2,4-Decadienal	-	-	-	-	-	0.2	0.2	0.2	0.2	0.0	RI, MS
23	1823	1827	(*E*,*E*)-2.4-Decadienal	-	-	-	-	-	0.6	0.6	0.5	0.6	0.1	RI, MS
24	1825	1830	Tridecanal	-	-	-	-	-	0.3	0.3	0.3	0.3	0.0	RI, MS
25	1835	1838	β-damascenone	1.5	1.5	1.4	1.5	0.1	0.6	0.7	0.7	0.7	0.1	RI, MS
26	1849	1849	Dihydro-β-ionone	0.7	0.7	0.7	0.7	0.0	-	-	-	-	-	RI, MS

27	1863	1864	(*E*)-Geranyl acetone	1.0	1.1	0.9	1.0	0.1	0.5	0.5	0.5	0.5	0.0	RI, MS
28	1901	1900	Nonadecane	0.3	0.3	0.3	0.3	0.0	0.1	0.1	0.1	0.1	0.0	RI, MS, Ac
29	1929	1932	Neophytadiene isomer	0.3	0.3	0.4	0.3	0.1	-	-	-	-	-	RI, MS
30	1936	1941	α-Calocarene	-	-	-	-	-	0.3	0.3	0.3	0.3	0.0	RI, MS
31	1953	1958	(*E*)-β-ionone	1.8	1.8	1.8	1.8	0.0	0.8	0.8	0.9	0.8	0.1	RI, MS
32	2001	2000	Eicosane	1.3	1.4	1.3	1.3	0.1	-	-	-	-	-	RI, MS, Ac
33	2005	2007	Caryophyllene oxide	3.8	3.9	3.8	3.8	0.1	4.6	4.9	4.8	4.8	0.2	RI, MS
34	2029	2031	Salvial-4(14)-en-1-one	3.0	3.1	3.0	3.0	0.1	0.5	0.5	0.5	0.5	0.0	RI, MS
35	2037	2037	Pentadecanal	0.5	0.4	0.5	0.5	0.1	-	-	-	-	-	RI, MS
36	2039	2036	Hexadecanal	-	-	-	-	-	0.9	0.9	0.9	0.9	0.0	RI, MS
37	2044	2050	(*E*)-Nerolidol	0.3	0.3	0.3	0.3	0.0	0.3	0.3	0.3	0.3	0.0	RI, MS
38	2064	2063	Humulene epoxide II	-	-	-	-	-	0.8	0.9	0.9	0.9	0.1	RI, MS
39	2098	2092	β-Oplopenone	-	-	-	-	-	0.3	0.3	0.3	0.3	0.0	RI, MS
40	2104	2100	Heneicosane	2.3	2.3	2.3	2.3	0.0	1.0	1.0	1.0	1.0	0.0	RI, MS, Ac
41	2133	2131	Hexahydro farnesyl acetone	1.9	1.9	1.9	1.9	0.0	1.8	1.8	1.8	1.8	0.0	RI, MS
42	2140	2142	Spathulenol	1.4	1.5	1.4	1.4	0.1	2.4	2.5	2.4	2.4	0.1	RI, MS
43	2171	2192	Nonanoic acid	0.1	0.1	0.1	0.1	0.0	2.0	2.1	2.2	2.1	0.1	RI, MS
44	2173	2150	Nor-copaanone	1.5	2.0	2.0	1.8	0.3	-	-	-	-	-	RI, MS
45	2187	2187	t-Cadinol	-	-	-	-	-	1.8	1.7	1.6	1.7	0.1	RI, MS
46	2195	2198	1-Docosene	-	-	-	-	-	0.9	0.9	0.9	0.9	0.0	RI, MS
47	2203	2200	Docosane	0.7	0.8	0.8	0.8	0.1	1.3	1.3	1.3	1.3	0.0	RI, MS, Ac
48	2226	2239	Carvacrol	-	-	-	-	-	0.9	0.8	0.8	0.8	0.1	RI, MS
49	2241	2228	Isospathulenol	0.1	0.1	0.1	0.1	0.0	0.4	0.4	0.4	0.4	0.0	RI, MS
50	2251	2253	β-Eudesmol	-	-	-	-	-	1.1	1.2	1.2	1.2	0.1	RI, MS
51	2277	2282	Decanoic acid	0.7	0.7	0.7	0.7	0.0	1.0	1.0	1.0	1.0	0.0	RI, MS
52	2282	2289	Oxo-α-ylangene	0.5	0.5	0.5	0.5	0.0	0.5	0.5	0.5	0.5	0.0	RI, MS
53	2295	2296	Isophytol	-	-	-	-	-	0.3	0.3	0.3	0.3	0.0	RI, MS
54	2303	2300	Tricosane	2.0	2.0	2.0	2.0	0.0	1.9	1.9	1.9	1.9	0.0	RI, MS, Ac
55	2311	2316	Caryophylla-2(12),6(13)dien-5-α-ol	-	-	-	-	-	0.4	0.4	0.4	0.4	0.0	RI, MS
56	2315	2315	2,4-bis(tert-butyl)phenol	0.6	0.6	0.6	0.6	0.0	-	-	-	-	-	RI, MS
57	2352	2324	Caryophylladienol-II	-	-	-	-	-	1.5	1.6	1.6	1.6	0.1	RI, MS
58	2353	2389	Caryophyllenol-I	-	-	-	-	-	0.7	0.7	0.7	0.7	0.0	RI, MS
59	2383	2381	Farnesyl acetone	0.9	0.9	0.8	0.9	0.1	0.2	0.2	0.3	0.2	0.1	RI, MS
60	2393	2399	Aromadendrene oxide	0.5	0.5	0.4	0.5	0.1	-	-	-	-	-	RI, MS
61	2394	2392	Caryophyllenol-II	-	-	-	-	-	1.4	1.4	1.4	1.4	0.0	RI, MS
62	2399	2423	γ-Cadinene-15-al	-	-	-	-	-	0.3	0.3	0.3	0.3	0.0	RI, MS
63	2402	2400	Tetracosane	0.3	0.3	0.3	0.3	0.0	0.7	0.7	0.7	0.7	0.0	RI, MS, Ac
64	2488	2492	Dodecanoic acid	1.3	1.3	1.3	1.3	0.0	4.2	4.2	4.3	4.2	0.1	RI, MS
65	2504	2500	Pentacosane	0.7	0.7	0.	0.7	0.0	1.6	1.6	1.9	1.7	0.2	RI, MS, Ac
66	2542	2551	Geranyl linalool	-	-	-	-		0.2	0.4	0.4	0.3	0.1	RI, MS
67	2608	2600	Hexacosane	-	-	-	-	-	0.6	0.6	0.5	0.6	0.1	RI, MS, Ac

68	2618	2614	Phytol	9.6	9.6	9.1	9.4	0.3	1.8	1.9	1.8	1.8	0.1	RI, MS
69	2700	2713	Tetradecanoic acid	1.1	1.2	1.2	1.2	0.1	2.1	2.2	2.1	2.1	0.1	RI, MS
70	2705	2700	Heptacosane	1.7	1.7	1.7	1.7	0.0	3.6	3.5	3.4	3.5	0.1	RI, MS, Ac
71	2776	2783	1-Docosanol	1.5	1.5	1.5	1.5	0.0	-	-	-	-	-	RI, MS
72	2806	2809	Pentadecanoic acid	1.0	1.0	1.0	1.0	0.0	1.3	1.4	1.3	1.3	0.1	RI, MS
73	2916	2931	Hexadecanoic acid	24.3	24.3	24.7	24.4	0.2	20.2	22.0	21.6	21.3	0.9	RI, MS
74	2985	2990	Docosanal	1.5	1.5	1.5	1.5	0.0	-	-	-	-	-	RI, MS
			Total	75.8	76.7	75.8	76.1	0.5	74.1	77.3	77.0	76.1	1.8	

In addition to the above data, diisobutyl phthalate is a common plasticizer contaminant and it was detected as a considerable component as 1.0 percentage for *C. austroanatolica*. [1]RRI: Relative retention time indices calculated against *n*-alkanes (C5-C30). [2]RRI Lit.: Relative retention time given in the literature for the compound in similar columns and analysis conditions. [3]The results of the analysis in each replicate. [4,5]The analysis were carried out in triplicate results are given as mean % area ± standard deviation (SD), calculated from MS data. [6]Identification method: RI: identification based on the relative retention times (RRI) of genuine compounds on the HP Innowax column and the literature data; MS: identification based on MS comparison with the database or the literature data, Ac: Identification is done according to RRI and MS values of the authentic compounds.

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