

Essential oil composition of Turkish mountain tea (*Sideritis* spp.)

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Abstract

The essential oil components of aerial parts from *Sideritis bilgerana*, *Sideritis tmolea* and *Sideritis congesta* were investigated by GC and GC–MS. The oil yields of dried plants obtained by hydro-distillation were 0.26, 0.33 and 0.83 (v/w), respectively. Fifty compounds representing 94.6% of the *S. bilgerana* oil were identified. The main ones were β -pinene (51.2%) and α -pinene (30.2%). Thirty-six components, representing 79.7% of the *S. congesta* oil were determined with high contents of muurol-5-en-4-a-ol (11.7%) and muurol-5-en-4-b-ol (33.0%). Forty-four components were identified accounting for 89.6% of the *S. tmolea* oil. Major constituents were α -cadinol (21.9%), β -caryophyllene (10.6%), calamenene (7.05%), muurol-5-en-4-b-ol (7.05%) and α -pinene (5.1%). All oils consist of monoterpenic hydrocarbons, oxygenated monoterpenes and sesquiterpenes. Muurol-5-en-4a-ol and muurol-5-en-4b-ol, at high percentages, were distinct components of *S. congesta*. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: *Sideritis bilgerana*; *Sideritis tmolea*; *Sideritis congesta*; Labiatae; Essential oils; β -Pinene; α -Pinene; α -Cadinol; Muurol-5-en-4-b-ol

1. Introduction

The genus *Sideritis*, a member of the family Labiatae, is widely distributed in subtropical and moderate regions and is a shrub 20–75 cm high (Davis, 1982). *Sideritis* species are a group of plants known as “mountain tea” in Anatolia. Some species are used as tea, flavouring agents and for medicinal purposes in several regions. (Local names are “dağ çayı” or “yayla çayı” in Turkish; Kırimer, Özek, Tanrıverdi, Koca, Kaya, & Başer, 1991). Infusion of aerial parts of a number of *Sideritis* species are used as tonics, carminatives, as anti-inflammatory agents, antispasmodics, diuretics and digestives, and in the treatment of colds (Ezer, Sezik, Erol, & Özdemir, 1991; Koedam, 1986; Villar, Navaro, Zafra-Pollo, & Rios, 1984; Yeşilada & Ezer, 1989).

The composition of the essential oils of several species of *Sideritis* grown in Turkey have been studied (Başer, 1992, 1994; Başer, Özek, Tümen, & Karaer, 1996; Başer, Kırimer, & Tümen, 1997; Ezer, Vila, Canigual, &

Adzet, 1995, 1996; Nakipoglu, 1995; Özek, Başer, & Tümen, 1993; Tanker, İlisulu, Koyuncu, & Coşkun, 1986; Tümen, Başer, Kırimer, & Ermin, 1995). The most recent contribution in this area is by Kırimer et al. (1991); Kırimer, Başer, Özek, and Tümen (1994); Kırimer, Başer, Tümen, and Sezik (1992); Kırimer, Kürkçüoğlu, Özek, and Başer (1996); Kırimer, Özek, Tanrıverdi, Koca, Kaya, and Başer (1992); Kırimer, Tabanca, Özek, Başer, and Tümen (1999). Koedam (1986) has reported the presence of β -copaene, α -pinene, δ -cadinene, β -pinene, β -caryophyllene and limonene. The dried inflorescences of a number of species of *Sideritis* are used to prepare a popular beverage in the Balkan countries and, especially in Greece, the so-called mountain tea (Gergis, Argyriadou, & Poulo, 1989; Koedam, 1986), and the composition of the essential oils of several species of *Sideritis* which grow wild in Greece has been reported by Gergis et al. (1989). Galati, Germano, Rossitto, Tzakou, Skaltsa, and Roussis (1996) studied the chemical composition of the oil of *Sideritis rasei* ssp. *rasei*, and determined camphor and 1,8-cineole as the major components. Also, Başer et al. (1997) established β -pinene, carvacrol and α -pinene in oil of *Sideritis scardica* Griseb. subsp. *scardica*.

Kırimer, Başer et al. (1992) and Kırimer, Özek et al. (1992) established the presence of β -pinene and α -pinene

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as major constituents in the essential oil of *Sideritis dichotoma*. Kirimer et al. (1994) detected limonene, linalool, β -caryophyllene, γ -elemene, (*Z*)- β -farnesene, caryophyllene oxide, spathulenol and carvacrol in the volatile oil of *Sideritis hispida*. Many studies on essential oils of *Sideritis* species have also been carried out (Adzet, Conigueral, Ibanez, & Vila, 1989; Burzaco, Velasco-Negueruela, & Perez-Alonso, 1992; Flamini, Cioni, Morelli, Maccioni, & Tomei, 1994; Galati et al., 1996; Gergis et al., 1989; Gergis, Spiliotis, Argyriadou, & Poulos, 1991; Koedam, 1986; Laer, Glombitza, & Neugebauer, 1996; Manez, Jemenez, & Villan, 1991; Mateo, Sanz, & Calderon, 1984; Papageorgiu, Kokkini, & Argyriadou, 1982; Zafra-Polo & Blaquez, 1989). The aim of this investigation was to determine the composition of volatile oils of *Sideritis bilgerana*, *Sideritis congesta* and *Sideritis tmolea* collected in Turkey.

2. Materials and methods

2.1. Plant material

S. bilgerana P.H. Davis, *S. congesta* P.H. Davis et Hub.Mor. and *S. tmolea* P.H. Davis were collected from plants growing wild in Turkey. The plants were identified by H. Dural, director of the herbarium section. Herbarium specimens were deposited at the Department of Biology, Faculty of Science and Education, Selçuk University, Turkey.

2.2. Recovery of the essential oils

Air-dried aerial parts of the plants were subjected to hydrodistillation for 3 h using a Clevenger-type apparatus. The percentage yields of the oils were calculated on a dry weight basis.

2.3. GC–MS analysis

For identification of components, analytical gas chromatography (GC) was performed using a DELSI 121 C apparatus fitted with a flame ionization detector and a CP WAX 51 fused silica column (25 m \times 0.3 mm i.d., 0.25 μ m film thickness). Temperature was programmed from 50 °C (5 min) to 220 °C at 3 °C/min. A CP WAX 51 fused silica WCOT column (60 m \times 0.3 mm) for GC–MS was used with helium as carrier gas. Identifications were made by comparison of the mass spectrum of each compound with that of known compounds. Quantification was elaborated as the percentage contribution of each compound to the total amount present. For GC–MC a CPWAX 52 fused silica CB column (50 m \times 0.25 mm i.d.) was used with helium as carrier gas and coupled to a HP mass spectrometer: ionization energy 70 eV. Temperature programming

was from 50–240 °C at 3 °C/min. The samples were injected at injector temperature 240 °C.

3. Results and discussion

The percentage of the volatile oils of the dried aerial parts from *S. bilgerana*, *S. tmolea* and *S. congesta* were light yellow with yields of 0.26, 0.33, and 0.83%, v/w, on dry weight basis, respectively. The yields in essential oil of *Sideritis* species previously examined were: *Sideritis cilicica*, *Sideritis perfoliata*, *Sideritis germanicopolitana* subsp. *germanicopolitana* and *S. germanicopolitana* subsp. *viridis* (0.33%; Kirimer et al., 1991). *S. dichotoma* (0.045%; Kirimer, Başer et al., 1992; Kirimer, Özek et al., 1992), *S. germanicopolitana* (0.33%; Kirimer, Başer et al., 1992; Kirimer, Özek et al., 1992), *Sideritis clandestina* subsp. *clandestina* and subsp. *raeseri* (0.09% and 0.12%; Koedam, 1986) and *S. raeseri* subsp. *raeseri* (0.14%; Galati et al., 1996).

GC–MS analyses of *S. bilgerana*, *S. tmolea* and *S. congesta* oil resulted in the identification of 50, 44 and 36 components, respectively (Table 1). Fifty compounds were characterized, representing 94.6% of the *S. bilgerama* oil. The oil from *S. bilgerama* contained mainly monoterpenoids (81.4%) with β -pinene (51.2%) and α -pinene (30.2%) as major components.

The others with smaller amounts were limonene (1.47%), β -phellandrene (0.83%), sabinene (0.79%), myrcene (0.36%) and camphene (0.36%). Decreasing percentages of sabinene (dominating the *Sideritis* distillates) was probably due to effects of hydrodistillation at ambient pressure. Thirty-six components, representing 79.7% of the *S. congesta* oil, were identified. Muurol-5-en-4-b-ol and muurol-5-en-4-a-ol, linalool, bornyl acetate, α -cadinol, α -pinene and 1 epicubenol were the major constituents of *S. congesta* oil; others were present in small amounts.

Forty-four components were identified, accounting for 89.6% the *S. tmolea* oil. The major constituents detected were α -cadinol (21.9%), β -caryophyllene (10.6%), calamenene (7.05%), muurol-5-en-4-b-ol (7.05%), α -pinene (5.1%) and sabinene (4.0%). Other components, such as β -caryophyllene oxide (3.31%), terpinene-4-ol (2.49%), *trans*-sabinene hydrate (2.49%), *cis*-sabinene hydrate (2.08%), α -terpineol (2.03%), muurol-5-en-4-a-ol (1.97%) and δ -cadinene (1.92%), were detected in lower amounts.

The principal components detected in the oil of *S. tmolea* were α -pinene (5.1%), sabinene (4.0%) *cis*-sabinene hydrate (2.08%), *trans*-sabinene hydrate (2.49%) and *p*-cymene (1.07%). They were accompanied by small amounts of usual monoterpene hydrocarbons.

The amounts of the major monoterpene hydrocarbons found in the *S. congesta* oil were α -pinene (2.16%), sabinene (1.52%), β -phellandrene (0.53%), *p*-

Table 1
Components of *Sideritis bilgerana*, *Sideritis tmolea* and *Sideritis congesta* essential oils

RI		Percentage (%)		
		<i>S. bilgerama</i>	<i>S. tmolea</i>	<i>S. congesta</i>
5.96	α -pinene	30.2	5.1	2.16
6.07	α -thujene	0.03	0.8	0.21
6.38	thuja-2,4(10)-diene	— ^a	0.03	t ^b
7.20	camphene	0.36	—	—
8.44	β -pinene	51.2	0.89	0.36
9.00	sabinene	0.79	4.00	1.52
10.36	δ -3-carene	0.04	—	—
11.18	myrcene	0.36	0.54	0.17
12.76	limonene	1.47	0.21	0.11
13.11	β -phellandrene	0.83	0.35	0.53
16.29	<i>p</i> -cymene	0.20	1.07	0.44
22.32	α -pinene oxide	0.01	—	—
24.22	octanol	0.09	—	—
26.78	<i>cis</i> -linalool oxide	0.01	—	—
27.76	oct-1-en-3-ol	0.30	—	—
27.92	α -cubenene	—	0.28	—
28.30	<i>cis</i> -sabinene hydrate	—	2.08	—
28.36	<i>cis</i> -thujanol-4	0.11	—	—
30.01	α -copaene	0.05	1.07	0.24
31.42	β -bourbonene	—	1.01	0.41
31.50	<i>trans</i> -linalool oxide	—	2.49	—
32.12	α -guijunene	—	0.16	—
33.37	<i>trans</i> -sabinene hydrate	—	2.49	—
33.43	<i>trans</i> thujanol-4	0.05	—	—
33.77	linalool	0.64	0.93	2.64
34.13	pinocarvone	0.70	—	—
34.53	nopinone	0.05	—	—
35.24	isobornyl acetate	0.89	—	—
35.73	bornyl acetate	0.15	—	2.62
36.59	terpinene-4-ol	0.08	2.49	—
37.78	myrtenal	0.70	—	—
38.12	β -caryophyllene	—	10.63	0.77
38.89	aromadendrene	—	0.93	—
39.03	linalyl isobutanoate	0.19	—	—
39.70	<i>trans</i> -pinocarveol	1.26	—	0.30
40.15	cryptone	0.60	—	—
40.28	α -humulene	—	0.39	—
40.57	estragol	—	0.76	—
41.01	(<i>Z</i>)- β -farnesene	—	0.63	—
41.03	<i>cis</i> -verbenol	—	—	0.11
42.16	verbenone	0.21	—	—
42.29	borneol	—	—	0.21
42.29	α -terpineol	—	2.03	0.35
42.38	α -terpineol + borneol	0.47	—	—
42.86	germacrene-D	—	1.31	—
42.87	myrtenol	0.01	—	0.53
42.89	γ -muurolene	—	0.09	—
43.44	piperitone	—	—	0.79
43.83	α -muurolene	—	0.20	—
43.93	carvone	0.01	0.14	—
44.20	β -bisabolene	—	0.67	—
44.37	<i>cis</i> -isobornyl-2-methylbutanoate	0.52	—	—
45.67	α -cadinene	—	1.92	0.28
45.71	<i>trans</i> -isobornyl-2-methylbutanoate	0.08	—	—
46.62	cuminal	—	0.31	—
47.33	<i>cis</i> -pinocarveol	0.04	—	—
47.69	myrtenol	0.19	—	—
49.79	calamenene	0.11	7.05	0.67
50.08	<i>cis</i> -carveol	0.05	—	—
50.81	<i>p</i> -cymene-8-ol	0.03	0.19	—

(continued on next page)

Table 1 (continued)

RI		Percentage (%)		
		<i>S. bilgerana</i>	<i>S. tmolea</i>	<i>S. congesta</i>
51.36	geranyl acetone	0.02	–	–
52.95	muurol-5-en-4-b-ol	0.17	7.05	33.02
53.97	bourbonenol	–	0.25	–
53.98	2-methylbutylbenzoate	0.05	–	–
54.66	ledol	–	0.23	0.35
55.36	copaenone	0.05	–	–
55.71	muurol-5-en-4-a-ol	0.02	1.97	11.65
56.79	<i>iso</i> -caryophyllene oxide	–	0.59	0.35
57.26	β -caryophyllene oxide	–	3.31	1.90
57.35	β -caryophyllene	0.15	–	–
58.59	salvial-4(14)-en-1-one	–	0.10	0.33
59.86	globulol	0.03	1.66	1.51
61.40	cubenol-1-10-diepi	–	–	1.42
61.73	cubenol	–	0.61	–
61.79	1 epicubenol	–	–	3.92
61.91	epiglobulol	0.12	–	–
63.65	cuminol	0.03	–	–
64.81	spathulenol	0.25	0.25	0.83
67.23	θ -cadinol	–	–	0.82
67.76	epi- α -muurol	–	0.90	1.42
67.96	α -cadinol	0.62	21.94	6.63

^a Nondetermined.

^b Trace.

cymene (0.44%), β -pinene (0.36%), myrcene (0.17%) and limonene (0.11%).

However, sesquiterpene hydrocarbon contents (β -caryophyllene, globulol, germacrene-D, spathulenol, α -humulene) of *S. tmolea* oil were found at higher levels than in *S. bilgerana* (globulol, spathulenol) and *S. congesta* (globulol, spathulenol, β -caryophyllene) oils. Also, all of the oils contained less oxygenated monoterpenes, such as linalool, terpinene-4-ol, *trans*-pinocarveol and α -terpinol.

As a result, α -pinene and β -pinene were the major constituents of the volatile oil of *S. bilgerana*, while α -cadinol, β -caryophyllene, calamenene, α -pinene, muurol-5-en-4-b-ol and sabinene were the principal components of *S. tmolea* oil. However, it is of interest to note the presence of muurol-5-en-4a-ol and muurol-5-en-4b-ol in very high percentages, which was distinctive of *S. congesta*.

References

- Adzet, T., Conigueral, S., Ibanez, C., & Vila, R. (1989). Essential oil of *Sideritis reverchonii*. *Herba Hungarica*, 28, 59–62.
- Başer, K. H. C. (1992). Essential oils of Anatolian Labiatae: a profile. *Acta Horticulturae*, 333, 217–238.
- Başer, K. H. C. (1994). Essential oils of Labiatae from Turkey: recent results. *Lamiales Newsletter*, 3, 6–11.
- Başer, K. H. C., Kırımer, N., & Tümen, G. (1997). The essential oil of *Sideritis scardica* Griseb. subsp. *scardica*. *Journal of Essential Oil Research*, 9, 205–207.
- Başer, K. H. C., Özek, T., Tümen, G., & Karaer, F. (1996). Essential oil composition of three Labiatae endemic to Turkey (*Micromeria fruticosa* (L.) Druce subsp. *giresunica* P.H. Davis, *Sideritis lycia* Boiss et Heldr. and *S. arguta* Boiss et Heldr.). *Journal of Essential Oil Research*, 8, 699–701.
- Burzaco, A., Velasco-Negueruela, A., & Perez-Alonso, M. S. (1992). Essential oil analysis of *Sideritis pouli* Pau. *Flavour and Fragrance Journal*, 7, 47–48.
- Davis, P. H. (1982). *Flora of Turkey and East Egean Island, Vol.7*. Edinburgh: Edinburgh University Press.
- Ezer, N., Sezik, E., Erol, K., & Özdemir, M. (1991). The antispasmodic activity of some *Siderites* species. In K. H. C. Başer (Ed.), *Proc. 9th Symp. on Plant Drugs, 16–19 May* (pp. 88–93). Turkey: Eskişehir (in Turkish).
- Ezer, N., Vila, R., Canigueral, S., & Adzet, T. (1995). Essential oil of *Sideritis lycia* Boiss et Heldr. *Journal of Essential Oil Research*, 7, 183–185.
- Ezer, N., Vila, R., Canigueral, S., & Adzet, T. (1996). Essential oil composition of four Turkish species of *Sideritis*. *Phytochemistry*, 41, 203–205.
- Flamini, G., Cioni, P. L., Morelli, I., Maccioni, S., & Tomei, P. E. (1994). Characterization of the volatile fraction of a *Sideritis rornana* population from Montemarcello (Easten Liguria). *Journal of Essential Oil Research*, 6, 239–242.
- Galati, E. M., Germano, M. P., Rossitto, A., Tzakou, O., Skaltsa, H., & Roussis, V. (1996). Essential oil of *Sideritis raeseri* Boiss. et Heldr. ssp. *raeseri*. *Journal of Essential Oil Research*, 8, 303–304.
- Gergis, V., Argyriadou, N., & Poulos, C. (1989). Composition of the essential oils of *Sideritis cladestina* ssp. *cyllenea* and *Sideritis sipylea*. *J. Sci. Food Agric.*, 47, 501–507.
- Gergis, V., Spiliotis, V., Argyriadou, N., & Poulos, C. (1991). Relation between the antimicrobial activity and the chemical composition of the essential oil of *Sideritis sipylea* Boiss. (Labiatae). *Flavour and Fragrance Journal*, 6, 93–95.

- Kırimer, N., Özek, T., Tanrıverdi, H., Koca, F., Kaya, A., & Başer, K. H. C. (1991). Composition of essential oils of *Sideritis germanicopolitana*. In K. H. C. Başer (Ed.), *Proc. 9th Symp. on Plant Drugs, 16–19 May* (pp. 371–381). Turkey: Eskişehir (in Turkish).
- Kırimer, N., Özek, T., Tanrıverdi, H., Koca, F., Kaya, A., & Başer, K. H. C. (1992). Composition of the essential oils of two subspecies of *Sideritis germanicopolitana* Bornm. *Journal of Essential Oil Research*, 4, 533–534.
- Kırimer, N., Başer, K. H. C., Tümen, G., & Sezik, E. (1992). Characterization of the essential oils of *Sideritis dichotoma*. *Journal of Essential Oil Research*, 4, 641–642.
- Kırimer, N., Başer, K. H. C., Özek, T., & Tümen, G. (1994). Essential oil of *Sideritis hispida* species from Turkey. *Journal of Essential Oil Research*, 6, 435–436.
- Kırimer, N., Kürkçüoğlu, M., Özek, T., & Başer, K. H. C. (1996). Composition of the essential oil of *Sideritis condensata* Boiss. et Heldr. *Flavour and Fragrances Journal*, 11, 315–320.
- Kırimer, N., Tabanca, N., Özek, T., Başer, K. H. C., & Tümen, G., (1999). Composition of essential oils from two endemic *Sideritis* species of Turkey. In: *Chemistry of natural compounds. 3rd Symp. on the Chemistry of Natural Compounds (SCNC)* (pp. 76–80), 19–22 October, Uzbekistan: Bukhara.
- Koedam, A. (1986). Volatile oil composition of Greek mountain tea (*Sideritis* spp.). *J. Sci. Food Agric.*, 36, 681–684.
- Laer, U., Glombitza, K. W., & Neugebauer, M. (1996). The essential oil of *Sideritis syriaca*. *Planta. Med.*, 62, 81–82.
- Manez, S., Jemenez, A., & Villan, A. (1991). Volatiles of *Sideritis mugronensis* flower and leaf. *Journal of Essential Oil Research*, 3, 395–397.
- Mateo, C., Sanz, J., & Calderon, J. (1984). The essential oils of some Eastern Spain *Sideritis*. *Phytochemistry*, 23, 319–322.
- Nakipoğlu, M. (1995). Chemotaxonomical studies on the *Sideritis* species of Turkey. I. *Sideritis sipylea* Boiss., and *Sideritis perfoliata* L. *Anadolu*, 5, 118–129 (in Turkish).
- Ozek, T., Başer, K. H. C., & Tümen, G. (1993). The essential oils of *Sideritis athoa* Papanikolaou et Kokkini. *Journal of Essential Oil Research*, 5, 669–670.
- Papageorgiou, V. P., Kokkini, S., & Argyriadou, N. (1982). Chomotaxonomy of Greek species of *Sideritis* I. Components of the volatile fraction of *Sideritis raeseri* ssp. *raeseri*. In N. Margaritis, A. Koedam, & D. Vakou (Eds.), *Aromatic plants: basic and applied aspects* (pp. 211–220). The Hague: Martinus Nijhoff.
- Tanker, N., İlisulu, F., Koyuncu, M., & Coşkun, M. (1986). Chytochemical screening of plants from the Ermenek–Mut–Gülнар (Turkey) area, III. Labiatae. *International Journal of Crude Drug Research*, 24, 177–182.
- Tümen, G., Baser, K. H. C., Kirimer, N., & Ennin, N. (1995). The essential oil of *Sideritis amasiaca* Bornm. *Journal of Essential Oil Research*, 7, 699–700.
- Villar, A., Navaro, A., Zafra-Pollo, M. C., & Rios, J. L. (1984). Constituents of the essential oil of *Sideritis mugronensis*. *Planta Medicinales et Phytotherapie*, 18, 150–153.
- Yeşilada, E., & Ezer, N. (1989). The antiinflammatory activity of some *Sideritis* species growing in Turkey. *International Journal of Crude Drug Research*, 27, 38–40.
- Zafra-Polo, M. C., & Blazquez, M. A. (1989). Chemical constituents of the essential oil from *Sideritis scordloides* L. subsp. *cavanillesii* Cav. Herb. *Plant. Med. Phytother.*, 23, 288–292.