

Composition of the essential oils of four endemic *Sideritis* species from Turkey

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ABSTRACT: *Sideritis phlomoides* Boiss. & Bal., *S. vulcanica* Hub.-Mor., *S. vuralii* Duman & Başer and *S. caesarea* Duman, Aytaç & Başer are endemic species in Turkey. Water-distilled essential oils from the flowering spike of these plants were analysed by GC–MS. β -Pinene (35%), 1,8-cineole (15%) and α -pinene (15%) were the main components in the oil of *S. vuralii*, while β -caryophyllene (8%) and caryophyllene oxide (7%) were the major constituents in the oil of *S. caesarea*. The oils from *S. phlomoides* and *S. vulcanica* were found to contain β -caryophyllene (31% and 10%) as major constituents. α -Bisabolol (16%) was also found as a major component in the oil of *Sideritis phlomoides*. Copyright © 1999 John Wiley & Sons, Ltd.

KEY WORDS: *Sideritis phlomoides* Boiss. & Bal.; *Sideritis vulcanica* Hub.-Mor.; *Sideritis vuralii* Duman & Başer; *Sideritis caesarea* Duman, Aytaç & Başer; Lamiaceae; β -caryophyllene; α -bisabolol; β -pinene; β -phellandrene; caryophyllene oxide

Introduction

The genus *Sideritis* (Lamiaceae) is represented by 46 species and 53 taxa in Turkey, 39 taxa being endemic (78.2%).^{1–7} Recently, one species (*S. scardica* ssp. *scardica*) as a new record³ and five species new to science have been described from Turkey. These are *S. gülendarii* H. Duman & F. A. Karavelioğulları; *S. akmanii* Z. Aytaç, M. Ekici & A. Dönmez;⁴ *S. vuralii*; *S. caesarea* Duman, Aytaç & Başer;⁵ and *S. öztürkii* Z. Aytaç & A. Aksoy.^{6–7}

Sideritis species are widely used as herbal tea and folk medicine in Turkey. These species are attributed to have anti-inflammatory, antispasmodic, carminative, analgesic, nervous system stimulant, sedative, anti-tussive, stomachic and anticonvulsant activities.^{8–17} Herbal teas prepared from these plants are taken as folk remedies for the treatment of cough due to cold and for curing gastrointestinal disorders.^{18,19} Our studies have indicated that the aqueous extracts of some *Sideritis* species of Turkey have nervous system stimulant or anti-stress activity in mice.²⁰

Here, we report on the essential oil compositions of *Sideritis phlomoides*, *S. vulcanica*, *S. vuralii* and

S. caesarea in Turkey, which have been described as endemic species. *S. phlomoides* is used as a herbal tea in Niğde province. The essential oil of *S. caesarea* was previously reported under the name *S. hispida*; β -caryophyllene (11%), carvacrol (9%) and caryophyllene oxide (5%) were characterized as its main constituents.²¹ Recently, the plant was identified as a new *Sideritis* species.⁵ Furthermore, two known entkaurenoids, siderol and epoxy-siderol, were isolated from dried inflorescences of *S. caesarea*.²²

Experimental

Collection sites, dates, oil yields and ESSE numbers of studied *Sideritis* species are given in Table 1. Voucher specimens are kept at the Herbarium of the Anadolu University Faculty of Pharmacy in Eskişehir, Turkey (ESSE). Flowering spikes were hydrodistilled for 3 h using a Clevenger-type apparatus.

GC–MS Analysis

The essential oils were analysed by GC–MS. The analysis was carried out using a Hewlett-Packard GC–MSD system. Innowax FSC column (60 m \times 0.25 mm, 0.25 μ m film thickness) was used with helium as carrier

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Table 1. Collection sites, dates, oil yields and ESSE numbers of *Sideritis* species

	Species	Collection site	Date	Oil yield (%)	ESSE
A	<i>S. phlomoides</i>	Niğde: Çamardı, Demirkazık, Narpuz Geçidi	8.1995	0.2	11477
B	<i>S. vulcanica</i>	Elazığ: Mastar Dağı, Kirkor Tepesi	8.1996	0.02	12155
C	<i>S. caesarea</i>	Kayseri: Sarız, Binboğa Mountain	7.1995	0.02	11952
D	<i>S. vuralii</i>	Antalya: Anamur-Ermenek road, Abanoz Yaylası	7.1995	0.1	11633

gas. GC oven temperature was kept at 60°C for 10 min and programmed to 220°C at a rate of 4°C/min and then held constant at 220°C for 10 min to 240°C at a rate of 1°C/min. The split ratio was adjusted to 50:1. The injector temperature was 250°C. MS were taken at 70 eV. Mass range was m/z 35–425. A library search was carried out using Wiley GC–MS Library and TBAM Library of Essential Oil Constituents. Relative percentage amounts of the separated compounds were calculated from total ion chromatograms by the computerized integrator.

Results and Discussion

Although 53 taxa belonging to 46 species of *Sideritis* known to occur in Turkey and dried inflorescences of most of these taxa are used as herbal tea, there is little work on their chemistry. Our group has collected all the recorded *Sideritis* taxa of Turkey and, as part of our systematic studies into essential oils of this genus, the results of four analyses are reported here. There are no previous data on the essential oils of these species. Compositions of the oils are given in Table 2.

Table 2. Composition of the essential oils of four *Sideritis* species

Compounds	KIP	A	B	C	D
Decane	1000	–	<0.1	–	–
α -Pinene	1032	3.9	1.1	0.4	14.5
α -Thujene	1035	–	<0.1	–	0.2
Camphene	1076	–	–	–	0.7
Undecane	1100	–	<0.1	–	–
β -Pinene	1118	4.8	2.3	0.9	35.3
Sabinene	1132	0.2	0.5	0.3	–
δ -3-Carene	1159	2.8	–	–	1.5
Myrcene	1174	0.4	0.3	0.1	1.6
α -Phellandrene	1176	–	<0.1	–	0.3
α -Terpinene	1188	–	<0.1	–	3.2
Heptanal	1195	–	0.2	0.4	–
Limonene	1203	1.5	3.1	1.7	0.2
1,8-Cineole	1213	0.2	1.0	0.1	14.6
β -Phellandrene	1218	0.9	0.4	0.1	0.5
(<i>Z</i>)- β -Ocimene	1246	–	–	0.3	–
γ -Terpinene	1255	0.1	0.3	0.1	0.2
(<i>E</i>)- β -Ocimene	1266	–	<0.1	<0.1	–
<i>p</i> -Cymene	1280	0.5	0.9	0.3	0.9
Terpinolene	1290	0.3	0.2	–	0.2
Octanal	1296	–	0.1	0.4	–
Tridecane	1300	–	0.1	–	–
6-Methyl-5-hepten-2-one	1348	–	0.1	–	–
Hexanol	1360	–	0.1	0.1	–
(<i>Z</i>)-3-Hexenol	1391	–	<0.1	–	–
3-Octanol	1393	–	0.4	0.3	–
Nonanal	1400	–	2.0	0.1	0.6
Tetradecane	1400	–	0.1	–	–
(<i>E</i>)-2-Octenal	1441	–	0.1	1.1	–
α , <i>p</i> -Dimethyl styrene	1444	–	0.1	–	–
1-Octen-3-ol	1452	–	3.7	2.0	0.4
Heptanol	1463	–	–	0.1	–
(<i>Z</i>)-4-Hexenyl-2-methyl butyrate	1482	–	–	0.1	–
α -Copaene	1497	–	0.4	–	–
α -Campholene aldehyde	1500	–	–	0.1	–
(<i>E,E</i>)-2,4-Heptadienal	1506	–	0.2	–	–
Decanal	1506	–	0.4	0.4	–
α -Bourbonene	1529	–	0.2	–	–
β -Bourbonene	1535	0.4	1.8	0.1	–
(<i>E</i>)-2-Nonenal	1547	–	0.2	2.9	–
Linalol	1553	0.5	3.3	2.1	1.3
Isopinocampone	1562	0.8	–	–	–

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Table 2. Continued

Compounds	KIP	A	B	C	D
Octanol	1562	–	0.4	0.9	1.5
8,9-Limonene-epoxide-I	1565	–	0.1	–	–
Pinocarvone	1586	–	0.3	0.3	–
Bornyl acetate	1591	–	–	0.2	–
(<i>E,Z</i>)-2,6-Nonadienal	1597	–	<0.1	–	–
β -Elemene	1600	–	0.4	–	–
Terpinen-4-ol	1607	0.2	0.6	0.4	<0.1
β -Caryophyllene	1612	30.7	10.2	8.3	0.5
β -Cyclocitral	1638	–	0.1	<0.1	–
Myrtenal	1648	–	0.4	0.5	–
(<i>E</i>)-2-Decanal	1655	–	0.4	1.5	–
Pulegone	1661	0.8	–	–	–
Nonanol	1664	–	0.4	0.2	–
(<i>E</i>)-Pinocarveol	1664	–	0.4	0.7	–
(<i>E</i>)- β -Farnesene	1671	0.1	1.9	1.8	0.4
(<i>E</i>)-Verbenol	1684	–	0.3	0.4	–
α -Humulene	1687	0.6	–	0.2	–
Methyl chavicol	1687	–	–	0.4	–
Cryptone	1690	–	–	–	0.2
Heptadecane	1700	–	–	0.2	–
γ -Muurolole	1704	–	0.5	–	–
α -Terpineol	1707	0.4	0.7	0.7	0.3
(<i>E,E</i>)-2,4-Nonadienal	1715	–	0.1	–	–
Borneol	1719	–	0.1	–	–
Dodecanal	1722	–	0.2	0.5	–
Germacrene D	1726	2.0	5.0	0.2	2.8
α -Zingiberene	1726	0.5	–	–	–
β -Bisabolene	1740	0.6	0.3	0.2	–
Bicyclogermacrene	1751	–	0.5	0.9	0.3
Carvone	1755	–	0.1	–	–
(<i>E,E</i>)- α -Farnesene	1758	0.5	0.1	–	–
(<i>E</i>)-2-Undecenal	1765	–	0.5	2.0	–
δ -Cadinene	1772	–	0.2	–	–
γ -Cadinene	1776	–	0.1	0.1	–
(<i>E,Z</i>)-2,4-Decadienal	1779	–	0.2	0.2	–
<i>ar</i> -Curcumene	1786	1.0	–	–	–
Myrtenol	1797	–	0.3	<0.1	–
Methyl salicylate	1800	0.1	0.4	0.7	–
Octadecane	1800	–	–	0.3	–
Cumin aldehyde	1804	–	0.3	–	–
Nerol	1808	–	0.1	<0.1	–
<i>p</i> -Mentha-1,3-dien-7-al	1811	–	0.1	–	–
(<i>E</i>)-2,4-Decadienal	1827	–	<0.1	0.5	–
Tridecanal	1830	–	0.1	0.2	–
β -Damascone	1830	–	0.1	<0.1	–
β -Damascenone	1838	–	2.1	0.4	–
Calamenene	1849	–	–	–	0.2
Geraniol	1856	–	0.6	0.4	–
(<i>E</i>)-Geranyl acetone	1868	–	1.2	1.2	–
Hexanoic acid	1871	–	–	0.2	–
(<i>E</i>)-2-Dodecanal	1875	–	0.4	–	–
Benzyl-2-methyl-butyrate	1882	–	0.1	0.2	–
Nonadecane	1900	–	<0.1	0.7	–
Benzyl isovalerate	1916	–	0.1	0.2	–
2-Methyl-butyl-benzoate	1929	–	0.1	–	–
Isoamyl benzoate	1933	–	0.1	<0.1	–
Tetradecanal	1937	–	0.1	0.2	–
β -Ionone	1957	–	–	0.3	–
2-Ethyl hexanoic acid	1965	–	0.5	–	–
1-Dodecanol	1973	–	0.4	–	–
Heptanoic acid	1981	–	–	1.2	–
(<i>E</i>)-2-Tridecanal	1988	–	–	0.1	–
Isocaryophyllene oxide	2000	0.8	–	0.8	–
Caryophyllene oxide	2008	5.6	4.7	7.4	0.3
Pentadecanal	2041	–	–	0.5	0.3
Norbourbonone	2045	–	0.3	–	–
(<i>E</i>)-Nerolidol	2053	1.7	–	0.3	–
Humulene epoxide II	2069	–	–	0.1	–
Tridecanol	2077	–	0.1	–	–

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Table 2. Continued

Compounds	KIP	A	B	C	D
Octanoic acid	2084	–	0.6	2.1	–
Hexyl benzoate	2096	–	0.1	0.3	–
Heneicosane	2100	–	–	1.0	–
Hexahydrofarnesyl acetone	2131	–	2.3	1.2	4.0
Spathulenol	2144	–	–	4.3	–
Valeranone	2144	–	–	–	1.6
(Z)-3-hexen-1-yl-benzoate	2148	0.9	0.3	0.8	0.4
1-Tetradecanol	2179	–	0.7	–	–
3,4-Dimethyl-5-pentylidene-2(5H)-furanone	2179	–	–	0.5	–
T-Cadinol	2187	–	0.1	–	–
Nonanoic acid	2192	–	1.0	–	–
Eugenol	2192	–	0.1	–	–
Docosane	2200	–	–	0.5	–
Thymol	2205	–	0.2	0.3	–
T-Muurolol	2209	–	0.2	–	–
δ -Cadinol	2219	–	0.1	–	–
α -Bisabolol	2232	16.2	–	–	–
(E)- α -Bergamotol	2241	–	0.1	–	–
Heptadecanal	2241	–	0.1	–	–
Carvacrol	2246	–	1.2	1.4	1.9
α -Cadinol	2251	–	0.3	–	–
(2Z, 6Z)-Farnesol	2287	–	0.1	–	–
Decanoic acid	2296	–	0.7	1.9	–
Tricosane	2300	–	–	1.0	–
Caryophylladienol I (= caryophylla-2(12),6(13)-dien-5 β -ol)	2312	–	1.6	–	–
Caryophylladienol II (= caryophylla-2(12),6(13)-dien-5 α -ol)	2316	4.8	0.1	1.0	–
Kaur-15-ene	2337	1.0	–	–	–
Caryophyllenol I (= caryophylla-2(12),6-dien-5 α -ol)	2353	–	0.2	–	–
Octadecanal	2357	–	0.1	0.3	–
Farnesyl acetone	2384	–	0.5	0.5	–
Caryophyllenol II (= caryophylla-2(12),6-dien-5 β -ol)	2392	–	0.6	0.9	–
Undecanoic acid	2400	–	–	1.1	–
Nonadecanal	2464	–	<0.1	–	–
Pentacosane	2500	–	<0.1	1.9	–
Dodecanoic acid	2503	–	0.9	0.1	–
Eicosanal	2571	–	0.1	–	–
Tridecanoic acid	2617	–	0.2	–	–
Phytol	2622	–	0.7	0.2	0.8
Benzyl benzoate	2655	–	0.6	3.1	–
Heptacosane	2700	–	1.1	1.5	–
Tetradecanoic acid	2713	–	–	1.5	–
Anthracene	2740	–	0.1	–	–
Octacosane	2800	–	0.1	–	0.7
Pentadecanoic acid	2822	–	0.6	–	–
Nonacosane	2900	–	–	0.8	0.4
Hexadecanoic acid	2931	–	9.7	4.1	4.6

KIP, Kováts index on a polar column (Innowax).

A, *Sideritis phlomoides*; B, *S. vulcanica*; C, *S. caesarea*; D, *S. vuralii*.

Identification was obtained by analysing them by GC–MS using a cross-linked PEG polar column. GC–MS analyses of the oils have revealed the occurrence of β -caryophyllene (30.8%, 10.2% and 8.3%, respectively) as the main constituent in *S. phlomoides*, *S. vulcanica* and *S. caesarea* oils. α -Bisabolol (16.2%) in the oil of *S. phlomoides*, hexadecanoic acid (9.7%) in the oil of *S. vulcanica* and caryophyllene oxide (7.4%) in *S. caesarea* were also among the major components. The main compounds characterized in the oil of *S. vuralii* were β -pinene (35.3%) and 1,8-cineole (14.6%).

The main components of the essential oils of some *Sideritis* species of Turkey are given in Table 3,^{23–31} which shows that oil yields of *Sideritis* species are poor. Monoterpene hydrocarbons such as α - and

β -pinene, myrcene and sabinene, or sesquiterpene hydrocarbons such as β -caryophyllene or germacrene D, are predominant components in most of the oils studied. The essential oils of *S. phlomoides*, *S. vulcanica* and *S. caesarea* fall into the sesquiterpene-rich group. *S. vuralii* is distinct from the other *Sideritis* species studied in that over 77% of the oil consists of monoterpenoids, with α - and β -pinenes making up almost 50% of the oil.

The occurrence of sesquiterpene alcohol α -bisabolol in *S. phlomoides* was interesting. It was previously detected in the oil of *Sideritis scordioides* L. var. *cavanillesii* Cav. as a major constituent (6.4%).³² (–)- α -Bisabolol is the main constituent in the oil of *Matricaria chamomilla*³³ and *Vanillosmopsis*

Table 3. Oil yields and the main components of the essential oils of some *Sideritis* species from Turkey

Species	Oil yield (%)	Main components (%)	Reference
<i>S. germanicopolitana</i> ssp. <i>germanicopolitana</i>	0.33	Myrcene 39, sabinene 21	23
<i>S. germanicopolitana</i> ssp. <i>viridis</i>	0.33	Myrcene 49, elemol 6	23
<i>S. trojana</i> *	0.05	(Z)-2-Decanal 7, β -bisabolene + bicyclogermacrene 7	24
<i>S. athoa</i>	0.25	Myrcene 39, β -pinene 12, α -pinene 6	25
<i>S. caesarea</i> **	> 0.01	β -Caryophyllene 11, carvacrol 9, caryophyllene oxide 5	21
<i>S. amasiaca</i>	0.04	β -Pinene 12, bicyclogermacrene 9, β -caryophyllene 7	26
<i>S. condensata</i>	> 0.01–0.65	β -Caryophyllene 9–19, germacrene D 5–14, hexadecanoic acid 6–15	27
<i>S. lycia</i>	0.7	β -Pinene 32, α -pinene 22	28
<i>S. arguta</i>	0.07	1,8-Cineole 23, β -pinene 8	28
<i>S. gulendamii</i>	0.07	β -Pinene 34, α -pinene 13	29
<i>S. scardica</i> ssp. <i>scardica</i>	0.03	β -Pinene 32, carvacrol 15	30
<i>S. erythrantha</i> var. <i>erythrantha</i>	0.39–0.49	α -Pinene 16–20, sabinene 6–10	31
<i>S. erythrantha</i> var. <i>cedretorum</i>	0.56–0.70	Myrcene 22–24, α -pinene 11–12	31

*This was reported as *Sideritis dichotoma* in the original paper.

**This was previously identified as *S. hispida* but later was proved to be a new species so named.⁵

erythropappa.³⁴ α -Bisabolol has been shown to have antiseptic, antibacterial, antifungal, ulcer-protecting and spasmolytic actions.³³

As indicated in the legend of Table 3, the oil composition of *S. caesarea* was previously reported as *S. hispida*.²¹ This was not a case of misidentification. The status of this taxon as a new *Sideritis* species has recently been reported.⁵ As indicated in Table 2, the main constituents were identified likewise as β -caryophyllene (8%) and caryophyllene oxide (7%) as opposed to 11% and 5%, respectively, reported earlier,²¹ with the exception of the reported higher percentage value of carvacrol (9%).

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