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## ESSENTIAL OILS OF ANNUAL *SIDERITIS* SPECIES GROWING IN TURKEY\*

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### ABSTRACT

Water distilled essential oils of five annual *Sideritis* species collected from different regions of Turkey were analysed by GC/MS. Results are tabulated and compared with main components of the essential oils of perennial *Sideritis* species from Turkey.

### INTRODUCTION

The genus *Sideritis* (Lamiaceae) is now represented in Turkey by 46 species and altogether 53 taxa (5 taxa belong to annual *Sideritis* species), 39 taxa being endemic in Turkey. Annual *Sideritis* species of sect. *Hesiodia* Bentham. are represented in Anatolia by four species and altogether five taxa. These are *Sideritis lanata* L., *S. romana* L. subsp. *romana*, *S. curvidens* Stapf, *S. montana* L. subsp. *montana*, *S. montana* L. subsp. *remota* (d' Urv.) P.W. Ball ex Heywood (Huber-Morath, 1982; Davis, 1988; Baßer & Kırımer, 1998).

Dried flowering spikes of *S. montana* subsp. *montana* are used as herbal tea. The plant is locally known as "Kuyruk çay" and "Dağ çayı" in Kırklareli province. Only one report dealing with the compositions of these oils has previously been reported in the literature (Flamini et al., 1994). The occurrence of flavonoids and alkaloids in *S. montana* and flavonoids in *S. romana*

were reported (Pulatova, 1969; Aynehchi et al., 1985; Barberan et al., 1988; Menkovis et al., 1993; Sattar et al., 1993). We report here the investigation of essential oils of annual *Sideritis* species from Turkey.

### MATERIALS AND METHODS

#### Plant Materials

Plant materials were collected during the flowering stage. Regions, collection dates of the materials are given Table 1. Voucher specimens are kept at the Herbarium of the Anadolu University Faculty of Pharmacy in Eskişehir, Turkey (ESSE).

#### Distillation

Plant materials were hydrodistilled for 3 h using a Clevenger-type apparatus. The percentage yields of the oils calculated on a moisture free basis were as follows: *S. lanata*, 0.03%; *S. romana* subsp. *romana*, 0.05%; *S. curvidens*, 0.02%; *S. montana* subsp. *montana*, 0.05%; *S. montana* subsp. *remota*, 0.03%.

#### GC/MS

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 × 0.25 mm Ø with 0.25 mm film thickness) was used with helium as carrier 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 kept constant at 220°C for 10 min to 240°C at rate of 1°C/min. Split ratio was adjusted at 50:1. The injector and detector temperatures were at 250°C. MS were taken at 70 eV. Mass range was from *m/z* 35 to 425. 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.

**Keywords:** *Sideritis lanata*, *S. romana* ssp. *romana*, *S. curvidens*, *S. montana* ssp. *montana*, *S. montana* ssp. *remota*, Lamiaceae, essential oil, distillation, GC/MS.

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Table 1. Regions, collection dates and ESSE number of the annual *Sideritis* species.

Species	Region	Date	ESSE
<i>S. lanata</i>	Aydin: Pirene	03.06.1995	11366
<i>S. romana</i> subsp. <i>romana</i>	Istanbul: Beykoz, Tophane village	31.05.1996	12052
<i>S. curvidens</i>	Balikesir: Erdek	16.06.1996	12113
<i>S. montana</i> subsp. <i>montana</i>	Kirkkareli: Demirköy, İslambeyli	31.07.1996	12171
<i>S. montana</i> subsp. <i>remota</i>	Eskişehir: Eskişehir-Kütahya road, Musaözü	13.07.1994	10724

Table 2. Classification of *Sideritis* species according to main components in essential oil.

Monoterpene hydrocarbons	<i>amasiaca, argyrea, armeniaca, athoa, bilgerana, brevidens, congesta, dichotoma, erythrantha</i> var. <i>erythrantha</i> , <i>erythrantha</i> var. <i>cedretorum</i> , <i>galatica, germanicopolitana</i> ssp. <i>germanicopolitana</i> , <i>germanicopolitana</i> ssp. <i>viridis</i> , <i>gulendamii, hispida, huber-morathii, libanotica</i> ssp. <i>libanotica, libanotica</i> ssp. <i>kurdica, lycia, niveotomentosa, phrygia, rubriflora, scardica</i> ssp. <i>scardica, serratifolia, sipylea, stricta, syriaca</i> ssp. <i>nusairiensis, trojana, vuralii</i>
Oxygenated monoterpenes	<i>arguta, libanotica</i> ssp. <i>microchlamys, romana</i> ssp. <i>romana</i>
Sesquiterpene hydrocarbons	<i>akmanii, albiflora, brevibracteata, caesarea, cilicina, condensata, curvidens, hololeuca, leptoclada, libanotica</i> ssp. <i>linearis, libanotica</i> ssp. <i>violascens, montana</i> ssp. <i>montana, montana</i> ssp. <i>remota, ozturkii, pisidica, tmolea, vulcanica</i>
Oxygenated sesquiterpenes	<i>phlomoides, taurica</i>
Diterpenes	<i>perfoliata</i>
Others	<i>lanata</i>

## RESULTS AND DISCUSSION

The results of GC/MS analyses of the essential oils are given in Table 2. Major components characterized in the oils were as follow: *S. curvidens*, bicyclogermacrene (20.60%), β-caryophyllene (8.93%); *S. montana* subsp. *montana* and *S. montana* subsp. *remota* bicyclogermacrene (10.80; 13.86%) and germacrene D (24.59; 10.33%); *S. romana* subsp. *romana* thymol (24.98%). Carvacrol (20%) was previously reported as the major component of the oil of *S. romana* from Italy (Flamini et al., 1994). The oil of *S. lanata* was quite distinct from the other annual *Sideritis* species with hexadecanoic acid (10.67%) and spathulenol (9.45%) as major constituents.

Continuing our research on the composition of the essential oils of Turkish *Sideritis* species (Başer, 1992; Kirimer et al., 1992a,b, 1993, 1994, 1995, 1996, 1997, 1998a,b; Özük et al., 1993; Tümen et al., 1993, 1995; Başer et al., 1996a,b, 1997; Ermin et al., 1997) we have classified them into six main groups, namely “monoterpene hydrocarbon-rich”, “oxygenated monoterpene-rich”, “sesquiterpene hydrocarbon-rich”, “oxygenated sesquiterpene-rich”, “diterpene-rich” and “others”. Fifty-seven percent of the *Sideritis* species existing in Turkey belong to the “monoterpene hydrocarbon-rich” group. *S. curvidens* and two subspecies of *S. montana* oils were included in sesquiterpene hydrocarbon-rich

group, while *S. romana* ssp. *romana* was in oxygenated monoterpene-rich group. *S. lanata* formed a distinct group named “other” with the occurrence of a fatty acid as main constituent in the oil (Table 2).

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Table 3. Percentage composition of the essential oil of annual *Sideritis* species.

Compounds	KIP	A	B	C	D	E
$\alpha$ -pinene	1032	—	0.52	0.32	1.27	3.58
$\alpha$ -thujene	1035	—	0.23	<0.01	—	—
camphene	1076	—	0.57	—	—	—
$\beta$ -pinene	1118	—	1.03	1.50	2.22	7.33
sabinene	1132	—	0.08	0.16	0.12	0.47
myrcene	1174	—	0.50	—	0.22	0.61
$\alpha$ -phellandrene	1176	—	0.11	—	—	—
$\alpha$ -terpinene	1188	—	0.49	—	—	—
heptanal	1195	—	0.14	—	—	—
limonene	1203	0.07	1.29	0.61	3.73	2.87
1,8-cineole	1213	0.08	1.97	—	0.28	0.16
$\beta$ -phellandrene	1218	0.09	0.35	0.36	0.32	0.32
(E)-2-hexenal	1232	—	0.21	—	—	—
$\gamma$ -terpinene	1255	—	4.42	—	—	0.20
p-cymene	1280	—	3.67	—	0.54	0.30
terpinolene	1290	—	0.06	—	—	0.04
octanal	1296	—	0.03	0.11	—	—
hexanol	1360	—	0.09	—	—	—
(Z)-3-hexen-1-ol	1391	—	0.08	—	—	—
3-octanol	1393	<0.01	0.42	—	—	—
nonanal	1400	0.65	0.38	2.01	0.94	0.95
1-octen-3-ol	1452	0.22	12.62	0.67	<0.01	0.45
heptanol	1463	—	0.07	—	—	—
$\alpha$ -cubebene	1466	0.10	—	—	—	—
$\beta$ -cubebene	1466	—	—	—	—	0.13
trans-sabinene hydrate	1474	0.97	—	—	—	—
$\delta$ -elemene	1479	—	—	0.74	—	—
octyl acetate	1482	—	—	—	—	0.16
cyclosativene	1492	—	—	—	—	0.05
$\alpha$ -copaene	1497	1.79	—	—	1.04	2.83
$\alpha$ -campoholene aldehyde	1500	—	—	—	—	0.19
(E,E)-2,4-heptadienal	1506	—	0.09	—	—	—
decanal	1506	0.35	0.46	1.04	—	0.03
$\alpha$ -bourbonene	1529	<0.01	—	—	—	0.23
camphor	1532	—	0.35	—	—	—
$\beta$ -bourbonene	1535	0.96	0.09	0.94	1.10	3.03
$\alpha$ -gurjunene	1544	—	—	—	—	0.17
$\beta$ -cubebene	1547	—	—	—	<0.01	—
(E)-2-nonenal	1547	0.10	0.05	0.12	—	—
linalool	1553	0.35	3.12	0.30	—	0.11
cis-sabinene hydrate	1556	—	0.32	—	—	—
octanol	1562	0.24	1.15	0.23	—	0.42
linalyl acetate	1565	—	—	—	—	0.08
trans-p-menth-2-en-1-ol	1571	—	0.04	—	—	—
pinocarvone	1586	—	—	0.17	—	0.28
bornyl acetate	1591	—	0.29	—	—	0.22
$\beta$ -elemene	1600	—	—	0.20	0.43	0.45
2-undecanone	1604	—	—	—	—	0.17
thymol methyl ether	1607	—	0.46	—	—	—
terpinen-4-ol	1607	0.20	1.27	0.27	—	0.30
$\beta$ -gurjunene	1610	—	—	—	0.14	0.17
$\beta$ -caryophyllene	1612	2.39	2.63	8.93	4.04	—
cis-dihydrocarvone	1624	—	0.12	—	—	—
aromadendrene	1628	0.18	—	—	—	—
$\beta$ -cyclocitral	1638	—	0.04	—	—	0.05
trans-dihydrocarvone	1645	—	0.09	—	—	—
myrtenal	1648	—	—	0.21	—	0.36
(E)-2-decenal	1655	0.35	—	0.45	—	0.15
pulegone	1661	—	0.09	—	—	—
alloaromadendrene	1661	—	—	0.21	—	—
trans-pinocarveol	1664	—	—	0.40	—	0.48
nonanol	1665	—	—	0.19	—	0.34
(E)- $\beta$ -farnesene	1671	—	0.49	—	7.22	2.93
trans-verbenol	1684	—	—	0.27	—	0.33
$\alpha$ -humulene	1684	0.26	0.24	—	0.36	0.12

Table 3 continues

Table 3. (cont.).

Compounds	KIP	A	B	C	D	E
cryptone	1687	<0.01	—	—	—	—
$\alpha$ -acoradiene	1690	—	—	—	—	0.56
$\gamma$ -curcumene	1704	—	—	—	1.13	1.24
myrtenyl acetate	1704	—	—	—	—	0.07
ledene	1707	—	—	1.09	—	0.29
$\alpha$ -terpineol	1707	0.85	0.59	0.21	—	—
borneol	1719	—	9.20	0.54	—	—
dodecanal	1722	—	—	2.10	—	0.17
2-undecanol	1722	0.44	—	—	—	—
germacrene D	1726	1.74	1.62	4.34	24.59	10.33
$\alpha$ -zingiberene	1726	—	—	—	1.76	1.38
$\beta$ -bisabolene	1737	—	6.28	—	—	—
$\alpha$ -cadinene	1740	0.42	—	—	—	0.35
bicyclogermacrene	1751	3.42	0.11	20.60	10.80	13.86
carvone	1755	—	0.05	—	—	—
naphthalene	1765	—	—	—	0.25	—
(E)-2-undecenal	1765	0.53	—	0.78	—	—
1-decanol	1766	—	0.16	—	—	0.34
$\delta$ -cadinene	1772	2.56	0.18	0.37	1.53	2.97
$\gamma$ -cadinene	1776	0.20	0.05	—	0.14	0.38
ar-curcumene	1786	—	—	—	0.46	0.14
myrtenol	1797	—	—	<0.01	—	0.32
methyl salicylate	1800	—	0.16	—	0.13	0.35
cuminaldehyde	1804	—	—	0.33	<0.01	—
3,7-guaiadiene	1811	—	—	—	—	0.05
p-mentha-1,3-dien-7-al	1811	—	—	0.11	—	—
(E,E)-2,4-decadienal	1827	0.44	—	0.23	—	0.22
tridecanal	1830	—	—	—	—	0.04
$\beta$ -damascenone	1838	0.08	—	—	—	0.04
calamenene	1849	<0.01	—	—	—	—
geraniol	1856	0.08	—	—	—	—
p-cymen-8-ol	1864	—	0.11	—	—	—
thymyl acetate	1868	—	0.09	—	—	—
(E)-geranyl acetone	1868	0.96	0.07	0.30	—	0.34
undecanol	1871	—	—	0.08	—	—
epicubebol	1900	0.20	—	—	—	—
tetradecanal	1937	—	—	—	—	0.07
$\alpha$ -calacorene I	1941	0.15	—	—	—	0.05
1,5-epoxy - salvial-4(14)-ene	1945	—	—	—	—	0.08
cubebol	1957	0.20	—	—	—	0.12
$\beta$ -ionone	1957	0.63	0.13	—	—	0.16
dodecanol	1973	0.59	—	0.13	—	0.60
$\alpha$ -calacorene II	1984	—	—	—	—	0.05
isocaryophyllene oxide	2000	—	0.04	—	—	0.09
caryophyllene oxide	2008	1.62	0.50	2.60	—	0.65
methyl eugenol	2029	—	—	—	—	0.04
pentadecanal	2041	0.47	—	0.38	—	0.29
11-norbourbonan-1-one	2045	—	—	—	—	0.25
1,6-germacradiene-5-ol	2069	0.51	—	—	—	—
humulene epoxide II	2069	—	—	0.19	—	—
cubenol	2080	0.15	—	—	—	—
octanoic acid	2084	—	—	0.27	—	—
elemol	2096	0.93	—	—	—	—
globulol	2096	—	—	0.46	—	0.29
viridiflorol	2100	0.66	—	0.61	5.46	0.44
hexahydrofarnesyl acetone	2131	3.45	0.40	0.30	1.66	1.36
hexadecanal	2135	—	—	1.42	—	—
spathulenol	2144	9.45	0.50	12.37	1.10	4.84
3,4-dimethyl-5-pentylidene -2(5H)-furanone	2179	0.93	—	—	—	0.51
T-cadinol	2187	—	—	—	0.47	1.02
nonanoic acid	2192	<0.01	0.27	2.95	—	0.12
thymol	2205	—	24.90	1.06	<0.01	—
T-muurolol	2209	0.54	—	—	—	0.37
$\delta$ -cadinol	2219	0.12	—	—	—	—
$\alpha$ -bisabolol	2232	—	—	—	—	0.19

Table 3 continues

Table 3. (cont.).

Compounds	KIP	A	B	C	D	E
<i>trans</i> - $\alpha$ -bergamotol	2241	1.59	—	1.39	—	0.61
carvacrol	2246	3.07	4.68	2.11	0.86	1.72
$\alpha$ -cadinol	2255	1.57	—	0.36	0.19	0.55
decanoic acid	2296	0.48	0.22	1.34	—	0.16
tricosane	2300	—	—	—	0.27	—
caryophylladienol II (=caryophylla-2(12),6(13)-dien-5 $\alpha$ -ol)	2316	—	—	0.17	—	—
farnesyl acetone	2384	0.80	—	0.16	—	—
hexadecanol	2384	0.06	—	—	—	—
undecanoic acid	2400	0.47	—	1.28	—	—
8 $\alpha$ -acetoxy elemol	2486	2.13	—	—	—	—
dodecanoic acid	2503	3.32	0.11	3.91	—	—
9-hexacosene	2594	—	—	—	3.87	—
tridecanoic acid	2617	—	—	0.28	—	—
phytol	2622	1.09	1.21	0.61	0.75	0.08
heptacosane	2700	7.50	—	—	—	—
tetradecanoic acid	2713	—	—	2.10	5.94	1.01
pentadecanoic acid	2822	0.20	—	—	0.76	—
nonacosane	2900	1.08	—	—	—	—
hexadecanoic acid	2931	10.67	2.56	6.46	5.80	5.75

KIP: Kovats index on a polar Innowax column

A: *Sideritis lanata*B: *Sideritis romana* ssp. *romana*C: *Sideritis curvifrons*D: *Sideritis montana* ssp. *montana*E: *Sideritis montana* ssp. *remota*

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