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# COMPOSITION OF THE ESSENTIAL OILS OF TWO ENDEMIC SPECIES FROM TURKEY: *Achillea lycaonica* AND *A. ketenoglui**

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*The hydrodistilled essential oils of* Achillea lycaonica *Boiss. & Heldr. and* A. ketenoglui *H. Duman were analyzed by GC/MS. The main components in the oils were* trans*-sabinene hydrate (9.3%) in* A. lycaonica *and borneol (14.1%) and terpinen-4-ol (14.5%) in* A. ketenoglui.

**Key words:** Compositae, *Achillea lycaonica*, *Achillea ketenoglui*, essential oil, *trans*-sabinene hydrate, borneol, terpinen-4-ol.

The genus *Achillea* (Compositae) is represented by 42 species and altogether 47 taxa in Turkey, 23 taxa being endemic [1,2]. Several *Achillea* species are used for their pharmaceutical, cosmetic, and fragrance properties. *A. wilhelmsii* C. Koch has been used as medicine mainly for the treatment of gastrointestinal disorders [3, 4]. *A. millefolium* L., which is known as yarrow or milfoil, has been used internally as herbal tea and externally in lotions and ointments [5].

The essential oils of *Achillea* species have been the subject of several investigations. For example, *A. millefolium* L. has been the most widely studied because of its economic value and therapeutic properties [6]. Previous results are summarized in Table 1.

The compositions of the oils are given in Table 3. In the oil of *A. lycaonica*, 105 compounds representing 91.5% of the total components were identified with *trans*-sabinene hydrate (9.3%), terpinen-4-ol (9.0%) and caryophyllene oxide (7.2%) as major components.

Two samples of *A. ketenoglui* were investigated. In the oil of *A. ketenoglui* from Ankara, 105 compounds representing 91.3% of the total oil were characterized with borneol (14.1%), 1,8-cineole (13.8%), and camphor (13.4%) as the major components. In the oil obtained from *A. ketenoglui* samples collected in Eskisehir, 109 compounds representing 91.8% of the oil were characterized. Terpinen-4-ol (14.5%) and *trans*-sabinene hydrate (10.9%) was found as the main components.

It was interesting to note that the oil compositions of two samples of *A. ketenoglui* were different. In fact, one sample showed similarity in composition to that of *A. lycaonica*. Such an occurrence is not uncommon in *Achillea* oils. Previously, different chemotypes of *A. biebersteinii* Afan., *A. millefolium* L., *A. wilhelmsii* C. Koch, etc. have been reported (Table 1).

## EXPERIMENTAL

**GC/MS Analysis.** The oils were analyzed by GC/MS using a Hewlett Packard GCD system. An HP-Innowax FSC column (60m × 0.25 mm, with 0.25 mm film thickness) was used with helium as a carrier gas (1 mL/min). GC oven temperature was kept at 60°C for 10 min and programmed to 220°C at a rate of 4°C/min, then kept constant at 220°C for 10 min and then

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TABLE 1. Previous Studies on *Achillea* Species

| *Achillea* species | Plant part | Main component (%) | Ref. |
|---|---|---|---|
| *A. abrotanoides* | Aerial parts | $\alpha$-Thujone (35) | 7 |
| *A. abrotanoides* | Leaves | 1,8-Cineole (18.5) | 8 |
| *A. ageratum* | Full bloom | Artemisia acetate (45.6) | 9 |
| *A. ageratum* | Aerial parts | Artemisia ketone (55.7) | 10 |
| *A. asiatica* | Aerial parts | $\beta$-Pinene (18.6) | 11 |
| *A. asiatica* | Flowers | $\beta$-Pinene (31.3-36.8) | 12 |
| *A. asiatica* | Leaves | $\beta$-Pinene (15-27.5) | 12 |
| *A. aspleniifolia* | Full bloom | Chamazulene (27.9) | 9 |
| *A. biebersteinii* | Leaves + Flowers | Ascaridole (37) | 13 |
| *A. biebersteinii* | Aerial parts | 1,8-Cineole (46.1) | 14 |
| *A. biebersteinii* | Aerial parts | 1,8 -Cineole (29.9) | 15 |
| *A. biebersteinii* | Aerial parts | Piperitone (49.9) | 15 |
| *A. chrysocoma* | Aerial parts | 1,8-Cineole (17) | 16 |
| *A. clavenae* | Aerial parts | Camphor (29.6) | 17 |
| *A. collina* | Full bloom | Germacrene-D (41.4) | 9 |
| *A. collina* | Aerial parts | $\beta$-Pinene (33.8) | 18* |
| *A. collina* | Aerial parts | 1,8-Cineole (27.6) | 17 |
| *A. corabensis* | Aerial parts + Seeds | Sabinene (25) | 19 |
| *A. crithmifolia* | Flowers | $\alpha$-Terpineol (25) | 20 |
| *A. crithmifolia* | Flowers | Camphor (8-45) | 21 |
| *A. crithmifolia* | Leaves | Camphor (3-26) | 21 |
| *A. cuneatiloba* | Aerial parts | Chamazulene (22.4) | 22 |
| *A. cuneatiloba* | Flowers | Chamazulene (26.0) | 22 |
| *A. distans* | Full bloom | Bisabolene oxide (30.3) | 9 |
| *A. erba-rotta* | Full bloom | Camphor (31.2) | 3 |
| *A. eriphora* | Leaves + Flowers | 1,8-Cineole (34.2) | 23 |
| *A. fragrantissima* | Flowers | Artemisia alcohol (32)<br>Santolina alcohol (32) | 24* |
| *A. fragrantissima* | Aerial parts | $\alpha$-Thujone (25.5-36.5) | 25 |
| *A. grandifolia* | Aerial parts | Camphor (25.6) | 6 |
| *A. ligustica* | Full bloom | Artemisia ketone (43.9) | 9 |
| *A. ligustica* | Leaves | Linalool (28.2) | 26 |
| *A. ligustica* | Flowers | Linalool (70.8) | 26 |
| *A. lingulata* | Aerial parts | Borneol (20.3) | 17 |
| *A. millefolium* | Aerial parts | $\beta$-Thujone (8.3-21.7) | 27 |
| *A. millefolium* | Aerial parts | Ascaridole (47.2) | 28 |
| *A. millefolium* | Aerial parts | Caryophyllene oxide (20) | 29 |
| *A. millefolium* | Aerial parts | Germacrene-D (36.3) | 11 |
| *A. millefolium* | Not stated | Chamazulene (13.0) | 30* |
| *A. millefolium* | Flowers | Camphor (20.6) | 31* |
| *A. millefolium* | Not stated | Guaiazulene (9.5) | 32* |
| *A. millefolium* ssp. *millefolium* | Flowers | 1,8-Cineole (28.7) | 33 |
| *A. millefolium* ssp. *millefolium* | Leaves (flowering period) | 1,8-Cineole (24.5) | 33 |

TABLE 1. (Continued)

| *Achillea* species | Plant part | Main component (%) | Ref. |
|---|---|---|---|
| *A. millefolium* ssp. *millefolium* | Leaves (vegetative phase) | Germacrene-D (65.1) | 33 |
| *A. millefolium* ssp. *millefolium* | Aerial parts (flowering period) | $\alpha$-Bisabolol (22.9) | 34 |
| *A. moschata* | Full bloom | Terpinen-4-ol (22.3) | 3 |
| *A. nana* | Full bloom | *trans*-Isoeugenol (21.6) | 3 |
| *A. nobilis* | Full bloom | Germacrene-D (46) | 3 |
| *A. phrygia* | Aerial parts | *cis*-Piperitol (11.2-31.2) | 35 |
| *A. ptarmica* | Full bloom | Germacrene-D (32.3) | 3 |
| *A. serbica* | Aerial parts | $\beta$-Sabinyl acetate (39.9) | 36 |
| *A. setacea* | Full bloom | Caryophyllene oxide (18.1) | 9 |
| *A. talagonica* | Leaves + Flowers | 1,8-Cineole (27) | 13 |
| *A. tenuifolia* | Flowers | Camphor (36.9) | 37 |
| *A. vermicularis* | Leaves + Flowers | Camphor (32) | 13 |
| *A. wilhelmsii* | Aerial parts | Fragranyl acetate (25.5) | 4 |
| *A. wilhelmsii* | Aerial parts | Camphor (36.1) | 4 |
| *A. wilhelmsii* | Aerial parts | Caryophyllene oxide (12.5) | 38 |

*Napralert (Natural Products Alert) database search.

TABLE 2. Collection Site, Dates, Plant Part, ESSE Number, and Oil Yields of the Achillea Species Studied

| Data | *Achillea lycaonica* | *Achillea ketenoglui* | |
|---|---|---|---|
| | Collection site | | |
| | Karaman: Hadim-Ermenek road | Ankara: Kirbasi-Beypazari road | Eskisehir: Polatli-Sivrihisar road |
| Collection date | 19.07.1998 | 16.06.2000 | 21.06.2000 |
| Plant part | Aerial parts | Aerial parts | Aerial parts |
| ESSE number | 12711 | 13248 | 13247 |
| Oil yields (v/d.w.) | 0.1 | 0.1 | 0.1 |

programmed to 240°C at a rate of 1°C/min. Alkanes were used as reference points in the calculation of relative retention indices (RRI). The split ratio was adjusted at 50:1. The injector temperature was at 250°C. MS were taken at 70 eV. Mass range was from 35 to 425 *m/z*. Library search was carried out using the Wiley GC/MS Library and the TBAM Library of Essential Oil Constituents. Relative percentage amounts were calculated from TIC by computer. The compounds identified in the oil are listed in Table 3.

**Plant Material.** Information on plant materials is given in Table 2. Voucher specimens are kept at the Herbarium of the Faculty of Pharmacy of Anadolu University in Eskisehir, Turkey (ESSE).

**Oil Isolation.** Air dried aerial parts of the plant material were subjected to hydrodistillation for 3 h using a Clevenger-type apparatus.

TABLE 3. The Composition of the Essential Oil of *Achillea* Species

| RRI | Compound | A (%) | B (%) | C (%) |
|---|---|---|---|---|
| 1032 | $\alpha$-Pinene | 0.8 | 1.0 | 0.4 |
| 1076 | Camphene | Tr. | 0.9 | 0.1 |
| 1118 | $\beta$-Pinene | 0.2 | 0.6 | 0.3 |
| 1132 | Sabinene | 0.1 | 0.4 | 0.1 |
| 1136 | 2-Methyl butyl acetate | - | 0.1 | - |
| 1176 | $\alpha$-Phelandrene | Tr. | - | 0.1 |
| 1185 | Isobutyl 2-methyl butyrate | - | 0.1 | 0.1 |
| 1188 | $\alpha$-Terpinene | 0.1 | 0.2 | 0.7 |
| 1195 | Dehydro-1,8-cineole | Tr. | 0.3 | 0.1 |
| 1198 | Isobutyl 3-methyl butyrate (=Isobutyl isovalerate) | - | 0.1 | 0.2 |
| 1203 | Limonene | 0.1 | Tr. | 0.1 |
| 1212 | Isoamyl alcohol | - | 0.1 | - |
| 1213 | 1,8-Cineole | 2.6 | 13.8 | 2.2 |
| 1218 | $\beta$-Phellandrene | 0.1 | - | - |
| 1244 | Amyl furan (=2-Pentyl furan) | Tr. | 0.1 | 0.1 |
| 1255 | $\gamma$-Terpinene | 0.6 | 0.5 | 2.5 |
| 1266 | (*E*)-$\beta$-Ocimene | - | 0.1 | 0.1 |
| 1280 | *p*-Cymene | 1.3 | 0.5 | 3.2 |
| 1286 | 2-Methyl butyl 2-methyl butyrate | - | 0.5 | 0.3 |
| 1290 | Terpinolene | 0.2 | 0.1 | 0.6 |
| 1296 | Octanal | Tr. | 0.1 | 0.1 |
| 1299 | 2-Methylbutyl isovalerate | Tr. | 0.5 | 0.6 |
| 1360 | Hexanol | Tr. | Tr. | 0.1 |
| 1400 | Nonanal | 0.8 | 0.3 | 0.4 |
| 1435 | $\gamma$-Campholene aldehyde | 0.2 | Tr. | - |
| 1441 | (E)-2-Octenal | Tr. | Tr. | 0.1 |
| 1450 | *trans*-Linalool oxide (Furanoid) | 0.1 | 0.1 | Tr. |
| 1452 | 1-Octen-3-ol | 0.1 | - | 0.1 |
| 1474 | *trans*-Sabinene hydrate | **9.3** | 1.1 | 10.9 |
| 1478 | *cis*-Linalool oxide (Furanoid) | 0.1 | - | - |
| 1479 | (*E*,*Z*)-2,4-Heptadienal | - | | 0.1 |
| 1480 | Nerol oxide | - | 0.1 | Tr. |
| 1487 | Isoneroloxide | Tr. | - | - |
| 1495 | Bicycloelemene | - | Tr. | 0.1 |
| 1497 | $\alpha$-Copaene | - | 0.2 | - |
| 1499 | $\alpha$-Campholene aldehyde | 1.0 | 0.1 | 0.3 |
| 1506 | Decanal | 0.2 | 0.3 | 0.2 |
| 1507 | (*E*,*E*)-2,4-Heptadienal | - | - | 0.1 |
| 1522 | Chrysanthenone | 0.1 | - | - |
| 1532 | Camphor | 2.0 | 13.4 | 5.5 |
| 1546 | (*Z*)-4-Decenal | - | - | 0.1 |
| 1547 | Dihydroachillene | - | 0.1 | - |
| 1548 | (*E*)-2-Nonenal | 0.1 | - | 0.1 |
| 1553 | Linalool | 4.4 | 3.7 | 0.4 |
| 1556 | *cis*-Sabinene hydrate | 4.2 | 1.0 | 5.9 |
| 1562 | Isopinocamphone | 0.4 | 0.2 | 0.2 |
| 1571 | *trans*-*p*-Menth-2-en-1-ol | 2.0 | 0.2 | 1.7 |
| 1586 | Pinocarvone | 2.3 | 1.4 | 1.5 |
| 1597 | Bornyl acetate | 0.3 | 0.1 | 3.0 |
| 1600 | Hexadecane | - | Tr. | Tr. |
| 1602 | 6-Methyl-3,5-heptadien-2-one | 0.1 | - | - |
| 1611 | Terpinen-4-ol | 9.0 | 3.0 | **14.5** |
| 1616 | Hotrienol | - | 0.3 | - |
| 1621 | Sylveterpenyl acetate | - | 0.1 | - |
| 1630 | 4-Terpinenyl acetate | - | - | 0.2 |
| 1638 | cis-*p*-Menth-2-en-1-ol | 1.6 | 0.2 | 1.0 |
| 1642 | Thuj-3-en10-al | Tr. | 0.1 | 0.1 |

TABLE 3. (Continued)

| RRI | Compound | A (%) | B (%) | C (%) |
|---|---|---|---|---|
| 1648 | Myrtenal | 0.3 | 0.3 | 0.2 |
| 1655 | (*E*)-2-Decenal | 0.1 | 0.1 | 0.1 |
| 1663 | *cis*-Verbenol | 0.2 | - | - |
| 1664 | *trans*-Pinocarveol | 2.5 | 1.0 | 1.1 |
| 1668 | (*Z*)-$\beta$-Farnesene | - | Tr. | 0.1 |
| 1681 | (*Z*)-3-Hexenyl tiglate | Tr. | 0.1 | 0.1 |
| 1682 | $\delta$-Terpineol | 0.2 | 0.3 | Tr. |
| 1683 | *trans*-Verbenol | 2.1 | 0.3 | - |
| 1689 | *trans*-Piperitol (=*trans*-p-Menth-1-en-3-ol) | - | - | 0.9 |
| 1694 | Neral | 0.1 | - | - |
| 1700 | *p*-Mentha-1,8-dien-4-ol (=Limonen-4-ol) | - | - | 0.2 |
| 1700 | Heptadecane | - | - | 0.1 |
| 1704 | $\gamma$-Curcumene | - | 0.2 | - |
| 1706 | $\alpha$-Terpineol | 1.9 | 2.2 | 0.8 |
| 1719 | Borneol | 0.5 | **14.1** | 5.6 |
| 1725 | Verbenone | 0.2 | - | - |
| 1726 | Germacrene D | - | 6.0 | 2.9 |
| 1729 | *cis*-1,2-Epoxy-terpin-4-ol | 0.1 | - | - |
| 1737 | $\gamma$-Campholenal* | 0.9 | - | - |
| 1740 | $\alpha$-Muurolene | - | 0.1 | - |
| 1742 | $\beta$-Selinene | - | - | 0.2 |
| 1748 | Piperitone | 1.8 | - | 0.2 |
| 1751 | Carvone | 0.1 | - | - |
| 1755 | Bicyclogermacrene | - | 1.9 | 0.8 |
| 1758 | *cis*-Piperitol | 2.7 | - | 0.7 |
| 1763 | Naphthalene | - | 4.1 | 0.1 |
| 1764 | *cis*-Chrysanthenol | 0.2 | 0.3 | 0.1 |
| 1764 | (*E*)-2-Undecenal | - | - | 0.1 |
| 1773 | $\delta$-Cadinene | - | 0.4 | 0.3 |
| 1776 | $\gamma$-Cadinene | - | 0.1 | - |
| 1779 | (*E,Z*)-2,4-Decadienal | 0.1 | 0.1 | 0.1 |
| 1786 | *ar*-Curcumene | - | 0.1 | - |
| 1793 | $\gamma$-Geraniol* | 1.9 | - | - |
| 1797 | *p*-Methyl acetophenone | - | Tr. | 0.1 |
| 1802 | Cumin aldehyde | - | - | 0.1 |
| 1804 | Myrtenol | 0.8 | 0.5 | 0.2 |
| 1814 | *p*-Mentha-1,5-dien-7-ol | - | 0.1 | Tr. |
| 1827 | (*E,E*)-2,4-Decadienal | 0.2 | 0.3 | 0.5 |
| 1838 | 2-Phenylethyl acetate | - | 0.1 | 0.1 |
| 1838 | (*E*)-$\beta$-Damascenone | - | 0.1 | 0.1 |
| 1845 | *trans*-Carveol | 0.9 | 0.2 | 0.2 |
| 1857 | Geraniol | 0.6 | 0.1 | 0.1 |
| 1864 | *p*-Cymen-8-ol | 0.3 | 0.3 | 0.3 |
| 1868 | (*E*)-Geranyl acetone | 0.1 | 0.1 | 0.2 |
| 1882 | *cis*-Carveol | 0.1 | 0.3 | - |
| 1901 | Geranyl butyrate | 0.1 | - | - |
| 1889 | Ascaridol | Tr. | - | 0.2 |
| 1900 | *epi*-Cubebol | - | 0.2 | - |
| 1924 | Geranyl isovalerate | 0.1 | - | - |
| 1908 | Isobutyl phenyl acetate | - | 0.3 | 1.5 |
| 1929 | 2-Methylbutyl benzoate | - | 0.1 | - |
| 1945 | 1,5-Epoxy-salvial(4)14-ene | - | 0.1 | 0.1 |
| 1948 | *trans*-Jasmone | - | - | 0.1 |
| 1957 | Cubebol | - | 0.1 | - |
| 1958 | (*E*)-$\beta$-Ionone | 0.2 | 0.1 | - |
| 1969 | *cis*-Jasmone | 0.1 | 0.2 | 0.4 |
| 1988 | 2-Phenylethyl-2-methylbutyrate | - | - | 0.1 |

TABLE 3. (Continued)

| RRI | Compound | A (%) | B (%) | C (%) |
|---|---|---|---|---|
| 2001 | Isocaryophyllene oxide | 0.2 | - | - |
| 2008 | Caryophyllene oxide | 7.2 | 0.2 | 0.1 |
| 2016 | Isoamyl phenylacetate | - | 0.5 | 1.2 |
| 2030 | Methyl eugenol | 0.3 | 0.6 | 0.7 |
| 2037 | Salvial-4(14)-en-1-one | - | 0.2 | 0.1 |
| 2041 | Pentadecanal | 0.2 | 0.1 | 0.1 |
| 2050 | (*E*)-Nerolidol | 0.2 | 0.2 | 0.1 |
| 2056 | 13-Tetradecanolide | 1.3 | - | - |
| 2069 | Germacrene D-4$\beta$-ol(=1(10),5-Germacradien-4$\beta$-ol) | - | 0.3 | 0.1 |
| 2071 | Humulene epoxide-II | 0.3 | - | - |
| 2074 | Caryophylla-2(12),6(13)-dien-5-one | 0.9 | - | - |
| 2100 | Heneicosane | Tr. | 0.2 | 0.2 |
| 2112 | Caryophylla-2(12),6-dien-5-one | 0.2 | - | - |
| 2131 | Hexahydrofarnesyl acetone | 0.5 | 0.5 | 0.4 |
| 2135 | Benzyl tiglate | - | - | 0.3 |
| 2144 | Spathulenol | - | 1.2 | 1.2 |
| 2148 | (*Z*)-3-Hexen-1-yl benzoate | 0.1 | - | - |
| 2157 | (*E*)-Ethyl cinnamate | 0.1 | - | - |
| 2178 | $\gamma$-Decalactone | 0.2 | - | - |
| 2179 | 3,4-Dimethyl-5-penthylidene-2(5H)-furanone | 0.2 | 0.1 | 0.1 |
| 2181 | Isothymol (=2-Isopropyl-4-methyl phenol) | - | - | 0.1 |
| 2186 | Eugenol | 0.8 | 0.4 | 0.6 |
| 2192 | Nonanoic acid | - | 0.2 | - |
| 2198 | Thymol | 0.9 | - | 3.6 |
| 2209 | T-Muurolol | - | 0.6 | - |
| 2214 | Phenyl ethyl tiglate | - | - | 0.1 |
| 2239 | Carvacrol | 3.4 | - | 0.7 |
| 2247 | *trans*-$\alpha$-Bergamotol | - | - | 0.3 |
| 2257 | $\beta$-Eudesmol | 1.1 | 1.7 | 2.0 |
| 2260 | 15-Hexadecanolide | 2.6 | - | - |
| 2287 | 1-Pentadecanol | 0.1 | - | - |
| 2300 | Tricosane | 0.2 | 0.4 | 0.2 |
| 2316 | Caryophylla-2(12),6(13)-dien-5$\beta$-ol (=Caryophylladienol I) | 0.9 | - | - |
| 2324 | Caryophylla-2(12),6(13)-dien-5$\alpha$-ol (=Caryophylladienol II) | 2.8 | - | - |
| 2388 | (*E*)-Nuciferal | - | 0.2 | 0.1 |
| 2392 | Caryophylla-2(12),6-dien-5$\beta$-ol (Caryophyllenol II) | 1.0 | - | 0.7 |
| 2456 | Methyl oleate | 0.3 | - | - |
| 2500 | Pentacosane | 0.4 | 0.5 | 0.2 |
| 2533 | $\gamma$-Costol | 0.1 | - | - |
| 2571 | Eicosanal | 0.1 | - | - |
| 2586 | (*E*)-Nuciferol | - | 0.2 | 0.1 |
| 2622 | Phytol | 0.3 | 0.2 | - |
| 2655 | Benzyl benzoate | 0.1 | - | - |
| 2700 | Heptacosane | 0.3 | - | 0.2 |
| 2811 | Benzyl salicylate | 0.1 | - | - |
| 2900 | Nonacosane | 0.3 | 0.2 | - |
| 2931 | Hexadecanoic acid | - | 1.7 | 0.8 |
| **Total** | | **91.5** | **91.3** | **91.8** |

A : *Achillea lycaonica*.

B : *Achillea ketenoglui* (Ankara).

C : *Achillea ketenoglui* (Eskisehir).

RRI: Relative retention indices calculated against n-alkanes.

% calculated from TIC data.

Tr.: Trace (< 0.1 %).
*Tentative.
γ-Campholenal: $C_{10}H_{18}O$, EIMS, 70 eV, *m/z* (rel. int. %): 154($M^+$, 12), 139(67), 121(41), 109(26), 105(26), 95(100), 93(49), 79(32), 77(24), 67(21), 55(16), 41(28).
γ-Isogeraniol: $C_{10}H_{18}O$, EIMS, 70 eV, *m/z* (rel. int. %): 154($M^+$, 0.3), 136(2), 121(5), 111(27), 93(17), 77(5), 69(100), 53(9), 41(67).

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