

Chemical Compositions of *Achillea sivasica*: Different Plant Part Volatiles, Enantiomers and Fatty Acids

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Abstract: In the present work, Microsteam distillation - Solid phase microextraction (MSD-SPME) and hydrodistillation (HD) techniques were applied to obtain volatiles from *Achillea sivasica*, an endemic species from Turkey. GC-FID and GC/MS analysis revealed that 1,8-cineole (22.1%) and α -pinene (9.3%) were the main constituents of the hydrodistilled flower volatiles. (*Z*)- β -Farnesene (23.9%), decanoic acid (10.1%), β -eudesmol (8.0%), tricosane (7.3%) and hexadecanoic acid (7.2%) were the main volatiles obtained from flowers by MSD-SPME. The leaf volatiles obtained by HD contained camphor (9.0%), β -pinene (6.9%), 1,8-cineole (6.7%), α -pinene (6.7%) and α -bisabolol (6.6%) as the main constituents while the leaf volatiles obtained by MSD-SPME technique were rich in (*E*)-geranyl acetone (10.5%), (*E*)- β -ionone (10.3%), camphor (10.2%), 1,8-cineole (9.6%), longiverbenone (7.9%), β -eudesmol (7.5%), isopropyl myristate (6.7%) and epi- α -bisabolol (6.4%). The root volatiles were rich in longiverbenone (14.1%), (*E*)-geranyl acetone (9.3%), nonanol (12.1%) and decanol (12.5%). The enantiomeric distribution of the major volatile constituents was analyzed by using different β -cyclodextrin chiral columns. (*IR*)-(+)- α -Pinene, (*IS*)-(-)- β -pinene, (*4R*)-(+)-limonene, (*IR,3S,5R*)-(-)-*trans*-pinocarveol, (*IS,2R,4S*)-(-)-borneol, (*2S*)-(-)- α -bisabolol were detected as dominant enantiomers. The lipids extracted from the flower and leaf with Folch method and methylated with BF₃ reagent contained common acids: linolenic, linoleic, hexadecanoic acids. Oleic and stearic acids were detected particularly in high amount in the flower lipids.

Keywords: *Achillea sivasica*; volatiles; enantiomer; fatty acids; MSD-SPME; GC/MS. © 2018 ACG Publications. All rights reserved.

1. Introduction

The genus *Achillea* L. (Asteraceae) is a perennial herb and comprise about 140 species around the worldwide [1, 2]. The name of *Achillea* originates from the Trojan warrior *Achilles*, who used yarrow (*Achillea millefolium*) on soldiers for wound healing [3].

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Achillea species are used as traditional treatment for analgesic purposes at abdominal pain, menstrual pain, headache, rheumatics pain. They are used for ulcer, appetizing, carminative, diuretic and against fleas as insecticidal [4-7] *A. millefolium* reviewed by German Commission E advert to its astringent, choleric, antibacterial, antispasmodic effects [8]. In literature survey, the essential oil of *Achillea* species showed antioxidant [9-11], antimicrobial and antifungal [12-16], antimelanogenesis [17], antiinflammatory and for healing [18, 19], cytotoxic and insecticidal activities [20-22].

The survey of previous studies on essential oil contents revealed that main (major) volatile components of *Achillea* species are oxygenated monoterpenes, mostly 1,8-cineole and camphor. Essential oil contents from literature survey are summarized in Table 1.

Table 1. Essential oil compositions of *Achillea* species (literature survey).

Species	Main compound, %	Location	Ref.
<i>A. abrotanoides</i> (Vis.) Vis	1,8-cineole +camphor 32.7		[23]
<i>A. aleppica</i> DC.	camphor 34, 1,8-cineole 20, <i>p</i> -cymene 14.2	Turkey (Elazig /Hazar)	[24]
	camphor 33, 1,8-cineole 26.1, α -pinene 4	Turkey (Elazig/Keban)	[24]
<i>A. asplenifolia</i> Vent.	β -caryophyllene 17.6, germacrene-D 15.6 chamazulene 13.3	Yugoslavia	[25]
<i>A. biebersteinii</i> Afan.	β -eudesmol 19.1, piperitone 9.2, camphor 4.2 borneol 3.3	Turkey (Ankara Beynam forest)	[26]
	piperitone 31.1, camphor 12.5, 1,8-cineole 10.9	Turkey (Erzurum)	[27]
	1,8-cineole, 30.6 piperitone, 28.9, camphor, 11.7	Turkey (Agri Mountain)	[28]
	1,8-cineole 31.1, camphor 14.4	Turkey (Dumluca–Sivas)	[28]
	1,8 cineole 38.1, camphor 23.6, borneol 5.8	Turkey (Erzurum)	[29]
<i>A. biserrata</i> M. Bieb.	camphor 36.8, 1,8-cineole 19.4, camphene 16.4, artemisia alcohol 14.3	Turkey (Gümüşhane, Trabzon)	[13]
<i>A. boissieri</i> Hausskn. Ex Boiss	1,8-cineole 27.4, camphor 25.3, caryophyllene oxide 4.60	Turkey (Elbistan)	[30]
<i>A. clavennae</i> L.	camphor 29.5, myrcene 5.5, 1,8-cineole 5	Montenegro (Velebit Mountain, Dalmaçya)	[31]
	camphor 46.9, 1,8-cineole 43.9, caryophyllene oxide 2.25	Serbia (Stara Planina eastern)	[32]
<i>A. collina</i> Becker	chamazulene 30-67 in flower, β -caryophyllene 2- 17, β -cubebene 1-9		[33]
<i>A. coarctata</i> Poir.	1,8-cineole 20.1, camphor 15.6, viridiflorol 11.8	Turkey (South Anatolia)	[34]
<i>A. cretica</i> L.	caryophylladienol-II 13.4, β -maaliene 6.1, neointermedeol 6.0	Turkey (Muğla/Datça)	[35]
<i>A. cucullata</i> Bornm.	camphor 32.6, 1,8-cineole 29.2, isoborneol 3,5	Turkey (Malatya)	[24]
<i>A. distans</i> W.&K.	1,8-cineole 16.8, <i>trans</i> -thujone 9.8, sabinene 8.2		[36]
<i>A. eriophora</i> DC.	1,8-cineole 34, α -pinene 7.6, β -pinene 6.2	Iran (Badjgah)	[37]
<i>A. falcata</i> L.	1,8-cineole 14-24, camphor 4-24, α -pinene 2-12	Turkey (Antalya-Elmalı, Antalya-Anamur)	[38]
<i>A. filipendulina</i> Lam.	1,8 cineole 22.9, 2,7 dimethyl octa-3,6dien-2-ol, 21.9, borneol 8.1	Turkey (Diyarbakır)	[11]
<i>A. formosa</i> (Boiss.) Sch. Bip. subsp. <i>amanica</i> (Rech. f) Ehrend & Y.P. Guo	borneol 12.8, hexadecanoic acid 11.6, caryophyllene oxide 5.5	Turkey (Osmaniye)	[39]
<i>A. gonioccephala</i> Boiss. et Bal.	camphor 32.6, 1,8-cineole 23.2	Turkey (Kahraman Maraş: Göksun-Yeşilkent)	[40]
<i>A. gypsicola</i> Hub-Mor.	camphor 40.2, 1,8-cineole 22, piperitone 11.3	Turkey (Çorum)	[41]
<i>A. hamzaoglu</i> Arabaci & Budak	1,8-cineole 24.1, linalool 12.2, camphor 6.7	Turkey (Kırşehir)	[42]
<i>A. holosericea</i> Sibth & Sm.	1,8-cineole 47.4, camphor 23.5, borneol 17.11	Macedonia (Ohrid)	[32]
<i>A. ketenoglu</i> H.	1,8-cineole 13.8, borneol 14.1, camphor 13.4	Turkey (Ankara)	[43]

Species	Main compound, %	Location	Ref.
Duman	terpinen-4-ol 14.5, <i>trans</i> -sabinene hydrate 10.9, <i>cis</i> -sabinene hydrate 5.9	Turkey (Eskişehir)	
<i>A. kotschy</i> Boiss.subs. <i>kotschy</i>	1,8-cineole 22.5, caryophyllene oxide 10.1, <i>p</i> -cymene 8.4	Yozgat	[42]
<i>A. ligustica</i> All.	leaf: 4-terpineol 19.3, carvone 8.9, γ -terpinene 7.2 flower: 4-terpineol 12.0, carvone 10.0, β -phellandrene 5.4	Sicily (Liguria) Sicily (Liguria)	[44] [44]
<i>A. lingulata</i> W&K.	santolina alcohol 21.1, borneol 15.0 T-cadinol 22.4, caryophyllene oxide 16.6, α -bisabolone oxide 12.79	Italy (Sardina) Macedonia (Ohrid)	[45] [32]
<i>A. lycaonica</i> Boiss & Heldr.	camphor 43.19 <i>trans</i> -sabinene hydrate 9.3, terpinen-4-ol 9 caryophyllene oxide 7.2	Turkey (Karaman)	[46] [43]
<i>A. millefolium</i> L.	1,8-cineole 6.6, δ -cadinol 6.2, caryophyllene epoxide 5.6	Turkey (Erzurum)	[29]
	β -pinene 10.6, 1,8-cineole 3.0, borneol 0.2	Himalaya	[47]
	β -pinene 17.7, 1,8-cineole 15.1, borneol 12.1	Himalaya	[47]
	1,8-cineole, 5.2 germacrene D 14.1	Iran	[48]
	artemisia ketone 14.9, camphor 11.6, linalyl acetate 11.5		[17]
	β -pinene 32.6, β -caryophyllene 16.5, sabinene 11.5	River Nisava Eastern Serbia	[32]
<i>Achillea millefolium</i> subsp. <i>millefolium</i> Afan.	1,8 cineole 24.6, camphor 16.7, α -terpineol 10.2 3-cyclohexen-1-one 21.6, linalool 14.3, 1,8- cineole 12.7	Turkey (Sivas/Kizildağ) Turkey (Elazığ)	[14] [49]
<i>A. millefolium</i> subs. <i>elborsensis</i>	chamazulene 48.9, isoborneol 10.2, camphor 9.5		[50]
<i>A. monocephala</i> Boiss. Bal.	camphor 30.2, borneol 15.6	Turkey (Çukurova/Adana)	[51]
<i>A. multifida</i> (DC.) Boiss.	camphor 34.6, borneol 23.6, 1,8-cineole 12.4 α -thujone 60.9, β -thujone 9.1, sabinene 4.1, camphor 3.7	Çukurova/Adana Turkey (Uludağ/Bursa)	[51] [12]
<i>A. nobilis</i> L. subsp. <i>neilreichii</i> (Kerner)	fragranyl acetate 32, fragranol 24, 3-eudesmol 8	Turkey (Beyşehir)	[10]
<i>A. odorata</i> L. var. <i>microphylla</i> (Willd.) Wilk	camphor 26.3, 1,8-cineole 17.8, α -pinene 12.5	Algeria	[52]
<i>A. odorata</i> L. subsp. <i>pectinata</i> (Lamk) var <i>microphylla</i> (Wild.) Wilk.	camphor 22.9, 1,8-cineole 15.7, α -pinene 11.3	Algeria	[52]
<i>A. oligocephala</i> DC	1,8-cineole 18.6, α -terpineol 6.8, linalool 6.0	Turkey (South Anatolia)	[34]
<i>A. phrygia</i> Boiss. & Bal.	<i>cis</i> -piperitol 11.2, <i>trans</i> - <i>p</i> -menth-2-en-1-ol 11.0, <i>cis</i> - <i>p</i> -menth-2-en-1-ol 7.2, 1,8-cineole 9.1 <i>cis</i> -piperitole 31.2, <i>trans</i> - <i>p</i> -menth-2-en-1-ol 14.7, <i>cis</i> - <i>p</i> -menth-2-en-1-ol 9.9, 1,8-cineole 9.9 camphor 14.5, linalool 9.9	Turkey (Seyitgazi/Eskişehir) Turkey (Alpu/Eskişehir)	[53] [53]
	camphor 35.6, 2-furaldehyde 16.6, 1,8-cineole 10.1	Turkey (Mihalliççik/ Eskişehir)	[53]
<i>A. salicifolia</i> subsp. <i>salicifolia</i>	camphor 55.3, 1,8-cineole 22.8, camphene 3.2	Turkey (Nevşehir, Avanos)	[54]
<i>A. sieheana</i> Stapf.	camphor 43.3, artemisia ketone 25.9, 1,8-cineole 6.3, camphene 4.8	Turkey (Göle/Ardahan)	[13]
<i>A. sieheana</i> Stapf.	camphor 39.9, 1,8-cineole 15.5, camphene 8.3	Turkey (eveli, Sindelhöyük, Kayseri)	[55]
<i>A. schischkinii</i> Sosn.	1,8- cineole 31.0, camphor 20.0	Turkey (Kayseri/Develi)	[56]
		Turkey (Kızıldağ Imranli/ Sivas)	[57]
<i>A. sintenisii</i> Hub.- Mor.	camphor 14.8, 1,8 cineole 12.9, β -pinene 12.8, borneol 10.8	Turkey (Sivas)	[58]

Species	Main compound, %	Location	Ref.
<i>A. taygetea</i> Boiss&Heldr	1,8-cineole 26.6, camphor 24.7	Taygetos	[59]
<i>A. teretifolia</i> Wild.	piperitone 21.4, linalool 19, 1,8-cineole 6.8	Turkey (Beyşehir)	[60]
	1,8-cineole 34, camphor 11, terpinen-4-ol 8.0, α -thujone 5		[10]
	1,8-cineole 15.9, borneol 8.1, camphor 7.0	Turkey (Dumluca– Sivas)	[28]
	3-cyclohexen-1-on 21.6, linalool 14.3, 1,8-cineole 12.7	Turkey (Elaziğ)	[49]
<i>A. vermicularis</i> Trin.	1,8-cineole 29.2, camphor 25.8, borneol 5.2	Turkey (Van–Muradiye)	[28]
<i>A. wilhelmstii</i> C. Koch.	camphor 39.6		[46]
	carvacrol 22.5, dihydrocarvone 13.2, linalool 12, 1,8-cineole 11.42	Iran (Golmakan, Horasan Razavi)	[61]
	camphor 46.6, 1,8-cineole 14.4	Iran	[62]

Several methods are applied to obtain volatiles from plant material. Nowadays, simple, rapid, cheap and “green” techniques which require small amount of plant materials for analysis are in demand. There is increasing requirement for fast, safe and repeatable extraction techniques which give possibility to obtain volatiles within a short period of time. MSD-SPME technique is such a technique combining solid phase microextraction with hydrodistillation [63]. The technique gives us the possibility to obtain the volatiles in a short time from a small amount of sample [64, 65]. In this context, the tandem of MSD-SPME method with GC-FID and GC/MS techniques was applied for the analysis of the volatiles obtained from the flower, leaf and root of *A. sivasica*. MSD-SPME technique combined with GC-FID and GC/MS methods has earlier been successfully applied for the extraction of volatiles from diverse species such as *Salvia rosifolia* Sm. [66], *Heracleum* spp. [67], *Prangos turcica* [68], *Xanthogalum purpurascens* Lallemand [69] and *Angelica sylvestris* var. *syvestris* [70].

It is known that the quality of flavoring components is dependent on enantiomeric profile. Chiral capillary GC (CC-GC) has proven to be a convenient method for characterizing enantiomeric profile of essential oils. CC-GC technique allows to differentiate natural flavors from those of synthetic origin. Chiral compounds from natural origins usually exist as one predominant optical isomer [71]. Also, the inspection of enantiomeric ratios can characterize regional differences between oils. Enantiomers are important in the determination of essential oils’ sensorial properties such as smell and flavor. On the other hand, they may have different biological activity and toxicity properties. For example, (*S*)-(+)-carvone inhibited *Fusarium* spp. fungi and the elongation of the potato sprouts [72], while (*R*)-(-)-carvone showed important toxicity properties [73]. Variations in enantiomeric ratios of specific components which may be related to sensory characteristics, indicate substitution or adulteration, or improper storage and handling of the material. Therefore, investigation of enantiomeric distribution is important. In scope of the present work, the volatiles of *A. sivasica* were separated on chiral column to obtain information about the distribution of major enantiomeric compounds.

Hence, we aimed to carry out comprehensive investigation of the flower, leaf and root volatiles of *A. sivasica*. In scope of the present work, we attempted to evaluate the enantiomeric distribution of the major volatile constituents of *A. sivasica* by separating them on β -cyclodextrin chiral columns. In addition, we investigated the lipid composition, namely chemical composition of fatty acids which were extracted with Folch method for subsequent analysis of their composition after methylation with Boron trifluoride reagent (BF₃). So, the present work is the first comprehensive investigation of the volatiles from flower, leaf and root of *Achillea sivasica* using a tandem of MSD-SPME, GC-FID and GC/MS techniques. The enantiomeric distribution of major volatiles, and chemical composition of the fatty acids of *A. sivasica* were carried out for the first time.

2. Materials and Methods

2.1. Chemicals

Boron trifluoride reagent (BF₃), hydrochloric acid, *n*-hexane (Sigma-Aldrich, Germany), calcium chloride, anhydrous sodium sulfate (Fluka, Germany), diethyl ether (JT Baker, Holland), chloroform (Sigma-Aldrich, France), methanol (Sigma-Aldrich, Poland) were of analytical grade. Standard compounds of definite enantiomeric purity were used to assess the enantiomeric ratios. (1*R*)-(+)- α -Pinene, (1*S*)-(-)- α -pinene, (1*R*)-(+)- β -pinene, (1*S*)-(-)- β -pinene, (4*R*)-(+)-limonene, (4*S*)-(-)-limonene, (4*R*)-(+)-terpinen-4-ol, (4*S*)-(-)-terpinen-4-ol, (1*S*,3*R*,5*S*)-(+)-*trans*-pinocarveol, (1*R*,3*S*,5*R*)-(-)-*trans*-pinocarveol, (1*R*,2*S*,4*R*)-(+)-borneol, (1*S*,2*R*,4*S*)-(-)-borneol, (2*R*)-(+)- α -bisabolol, (2*S*)-(-)- α -bisabolol were purchased from Sigma-Aldrich (USA). A C₈–C₄₀ *n*-alkane standard solution was purchased from Fluka (Buchs, Switzerland).

2.2. General

Agilent 5975 GC-MSD system (Agilent, USA; SEM Ltd., Istanbul, Turkey) was equipped with the HP-Innowax FSC column (60 m \times 0.25 mm id with 0.25 μ m film thickness, Agilent, USA). The GC-FID analysis was carried out with capillary GC using an Agilent 6890N GC system (SEM Ltd., Istanbul, Turkey). Separation of enantiomer was performed with an Agilent 5975 inert MSD with Triple-Axis Detector and Agilent 7890A GC systems (Agilent, USA; SEM Ltd., Istanbul, Turkey). The manual SPME holder (57330-U, Supelco, Bellefonte, PA) and the device for MSD-SPME system were used for MSD-SPME experiments.

2.3. Plant Material

Plant material of *A. sivasica* was collected in Sivas province of Turkey: Kangal to Gürün, 8. km, calcareous area, 39°07'56.3"N, 37°13'48.0"E, on June 10, 2015. The herb was dried under the shade. Botanical identification was performed by Dr. M. Tekin (Trakya University). Voucher specimen is deposited at the Herbarium of Cumhuriyet University, Faculty of Science (CUFH) under the voucher number M. Tekin 1570.

2.4. Hydrodistillation of Essential Oil

A. sivasica flowers and leaves were separately subjected to hydrodistillation in a Clevenger type apparatus to yield essential oils (3 hours) [74].

2.5. Microsteam Distillation - Solid Phase Microextraction (MSD - SPME)

Microsteam distillation - solid phase microextraction of the volatiles was carried out using an assembly reported previously [66]. MSD-SPME involved concurrent solid-phase microextraction combined with continuous hydrodistillation of the volatiles. This method significantly makes shorter the time required for the isolation of volatiles. It should be noted that this technique allows the isolation of volatiles from a very small amount of plant material. Tandem of MSD-SPME with GC-FID and GC/MS techniques is simple, sensitive, rapid, solvent-less and non-toxic technique for analysis of the volatile compounds at microscale level. Briefly, the ground plant material (0.3 g) was placed in 25 mL round bottom flask along with 10 mL of water. The flask was fitted with a Claisen distillation head with plug and a condenser set up for refluxing rather than distillation. The threaded plug was used for SPME fiber assembly. A manual SPME holder (57330-U, Supelco, Bellefonte, PA) and the PDMS-DVB 65 μ m fiber "blue type" were used for the extraction of volatiles. Previously, the fiber was conditioned at 250 °C for 10 min before the experiment. After the SPME needle pierced the plug, the fiber was expressed through the needle and

exposed to the headspace above the plant sample. Different extraction periods were applied to optimize experiment conditions. The extraction time for leaf and flower volatiles was 3, 5, 8, 10 min, while the roots were extracted for 5, 8, 10 and 15 min. After trapping of the volatiles, the loaded SPME fiber was withdrawn into the needle, and then the needle was removed from the plug and subsequently used for GC/FID and GC/MS analyses. Thermal desorption of analytes from the fiber coating was performed by injection of the fiber in the injection port (at 250°C) for 5 min. Experiments were performed in triplicate.

2.6. Fatty Acid Extraction and Derivatization

The ground plant material was subjected to maceration with chloroform: methanol (2:1) at room temperature for 24 h. The extract was filtered and the residue material was macerated twice (for 30 min) more with new portions of the solvent. All filtrates were combined and half of the solvent was evaporated under vacuum in a rotary evaporator. Then, half amount of chloroform was added into the extract. The obtained extract was washed (three times) with CaCl₂ solution (0.4%) in separatory funnel. At the end of the procedure, the chloroform extract was filtered through anhydrous sodium sulfate to remove moisture, then chloroform was removed under vacuum. The dried extract was subjected to saponification. To do this, the crude extract was boiled in KOH-H₂O-MeOH (1:1:8) solution for 2 hours in a refluxing system. After the saponification process, 1-2 mL of *n*-hexane was added to remove non-saponified compounds [75]. The fatty acids were extracted with diethyl ether after acidification of the extract with HCl (15% solution). The methylation of the free fatty acids was performed using BF₃ reagent [76]. The fatty acids methyl esters were subjected to analysis with GC/MS and GC/FID techniques.

2.7. Gas Chromatographic Analysis

The GC/MS and GC-FID analyses were carried out with the HP-Innowax FSC column (60 m × 0.25 mm id with 0.25 μm film thickness, Agilent, USA) in conditions reported earlier [77].

2.8. Enantioselective GC Analysis

Enantioselective GC analyses were performed with GC/MS and GC-FID systems, Agilent 5975 inert MSD with Triple-Axis Detector and Agilent 7890A GC systems (Agilent, USA; SEM Ltd., Istanbul, Turkey) equipped with three different chiral columns to determine enantiomeric distribution of the constituents. The scanned mass spectra were recorded at 70 eV. The acquisition mass range was from *m/z* 35 to 450 amu. The temperature conditions of chromatographic separation and column types are given in Table 2.

2.9. Identification and Quantification of Compounds

Identification of the individual compounds was based on the following: (i) comparison of the GC/MS Relative Retention Indices (RRI) of the compounds on polar column determined relative to the retention times of a series of *n*-alkanes (C₈-C₄₀), with those of authentic compounds or literature data; (ii) computer matching with commercial mass spectral libraries: MassFinder software 4.0, Adams Library, Wiley GC/MS Library (Wiley, New York, NY, USA) and Nist Library, and comparison of the recorded spectra with literature data [78, 79]. Confirmation was also achieved using the in-house “Başer Library of Essential Oil Constituents” database, obtained from chromatographic runs of pure compounds performed with the same equipment and conditions [80]. Percent composition was obtained for each constituent on the basis of GC-FID analyses of the oils

Table 2. Chromatographic separation conditions applied for the separation of enantiomers (experimental data).

	Method 1	Method 2	Method 3
Enantiomers	α -Pinene	Terpinen-4-ol, borneol	β -Pinene, limonene, α -bisabolol, <i>trans</i> -pinocarveol
Column	Lipodex-G (25 m \times 0.25 mm id, 0.125 μ m film thickness) (6-methyl-2,3-pentyl- γ -cyclodextrin), (60% in polysiloxane PS086)	Rt TM - β -DEXse (25 m \times 0.32 mm id, 0.25 μ m film thickness) (6- <i>tert</i> -butyldimethylsilyl-2,3-diethyl- β -cyclodextrin)	Rt TM - β -DEXse (25 m \times 0.32 mm id, 0.25 μ m film thickness) (6- <i>tert</i> -butyldimethylsilyl-2,3-diethyl- β -cyclodextrin)
Carrier gas	Helium (5 mL/min)	Helium (5 mL/min)	Helium (7 mL/min)
Flow rate average	77.985 cm/sec.	63.943 cm/sec.	63.943 cm/sec.
Temperature Program	50 min@35°C; 40°C/min at 200°C; 10.875 min @ 200°C; Total: 65 min	0 min@40°C; 2°C/min at 200°C; Total: 80 min	10 min@35°C; 2°C/min at 130°C; 57.5 min@130°C; 40°C/min with 230°C; 2.5 min@230°C; Total: 120 min.
Injection Mode	Split (10:1)	Split (40:1)	Split (40:1)
Injection Temperature	250°C	250°C	250°C

3. Results and Discussion

3.1. Composition of Volatiles

In the course of our ongoing studies on Turkish aromatic plants, we report here on the composition of endemic *A. sivasica* essential oil, determined by means of GC-FID and GC/MS techniques. The flower and leaf oils were obtained with conventional hydrodistillation method. Yields of the essential oils of *A. sivasica* were calculated on moisture free basis: flower (0.048%) and leaf (0.036%). Additionally, the volatiles of the flower, leaf and root were obtained with modern rapid MSD-SPME technique and subsequently analyzed with GC-FID and GC/MS. Table 3 lists the detected compounds with their RRI values, relative percentages and methods of identification. 1,8-Cineole (22.1%) and α -pinene (9.3%) were found to be the main constituents of the flower volatiles obtained with HD. In the flower volatiles obtained with MSD-SPME, (*Z*)- β -farnesene (23.9%), decanoic acid (10.1%), β -eudesmol (8.0%), tricosane (7.3%) hexadecanoic acid (7.2%), 1,8-cineole (5.3%), epi- α -bisabolol (4.9%) and germacrene D (4.7%) were found as the main constituents. Hence, MSD-SPME technique was more sensitive in trapping flower volatiles rich in sesquiterpenes.

Camphor (9.0%), β -pinene (6.9%), 1,8-cineole (6.7%) α -pinene (6.7%), α -bisabolol (6.6%) and longiverbenone (4.9%) were found to be the main constituents of the leaf volatiles obtained with HD. MSD-SPME technique trapped leaf volatiles rich in (*E*)-geranyl acetone (10.5%), (*E*)- β -ionone (10.3%), camphor (10.2%), 1,8-cineole (9.6%), longiverbenone (7.9%), β -eudesmol (7.5%), isopropyl myristate (6.7%), epi- α -bisabolol (6.4%), and borneol (5.9%). Similar to the flowers, MSD-SPME technique extracted leaf volatiles particularly rich in sesquiterpenes. The root volatiles were rich in the sesquiterpenes, longiverbenone (14.1%) and (*E*)-geranyl acetone (9.3%) as well as in aliphatic alcohols, nonanol (12.1%) and decanol (12.5%).

Table 3. *A. sivasica* essential oil composition.

No	RI ^{a)}	RRI ^{b)}	Compound	% ^{c)}					IM*
				Flower		Leaf		oot ^{d)}	
				HD	MSD-SPME	HD	MSD-SPME		
1.	1032	1032[81]	α -Pinene	9.3	2.0	6.7	4.3	-	e), f), g)
2.	1076	1076[81]	Camphene	0.5	-	1.2	-	-	e), f), g)
3.	1118	1118[81]	β -Pinene	3.0	-	6.9	2.1	-	e), f), g)
4.	1132	1132[81]	Sabinene	1.7	-	1.0	-	-	e), f), g)
5.	1188	1188[81]	α -Terpinene	0.5	-	0.2	-	-	e), f), g)
6.	1203	1203[81]	Limonene	-	-	1.4	-	-	e), f), g)
7.	1213	1213[82]	1,8-Cineole	22.1	5.3	6.7	9.6	-	e), f), g)
8.	1255	1256[82]	γ -Terpinene	1.1	-	0.7	-	-	e), f), g)
9.	1280	1278[82]	<i>p</i> -Cymene	0.5	-	0.2	-	-	e), f), g)
10.	1290	1290[81]	Terpinolene	0.2	-	0.2	-	-	e), f), g)
11.	1474	1474[81]	<i>trans</i> -Sabinene hydrate	2.1	-	0.6	-	-	e), f), g)
12.	1480	1469[81]	α -Longipinene	-	-	0.2	-	-	e), f), g)
13.	1492	1492[83]	Cyclosativene	t	-	-	-	-	e), f), g)
14.	1497	1497[81]	α -Copaene	2.8	-	0.7	-	-	e), f), g)
15.	1499	1496[84]	α -Campholene aldehyde	-	-	0.1	-	-	e), f), g)
16.	1506	1510[82]	Decanal	-	-	0.1	-	-	e), f), g)
17.	1512	-	Longicyclene	-	-	t	-	-	e), f), g)
18.	1532	1532[82]	Camphor	4.1	1.3	9.0	10.2	-	e), f), g)
19.	1549	1547[85]	β -Cubebene	t	-	-	-	-	e), f), g)
20.	1556	1555[85]	<i>cis</i> -Sabinene hydrate	0.6	-	-	-	-	e), f), g)
21.	1562	-	Isopinocampone	t	-	-	-	-	e), f), g)
22.	1563	1557[86]	Octanol	-	-	-	-	4.1	e), f), g)
23.	1570	-	<i>trans-p</i> -Ment-2-en-3-ol	t	-	-	-	-	e), f), g)
24.	1586	1590[87]	Pinocarvone	2.5	-	1.3	-	-	e), f), g)
25.	1611	1611[82]	Terpinen-4-ol	3.2	1.1	1.4	-	-	e), f), g)
26.	1628	1628[85]	Aromadendrene	-	-	t	-	-	e), f), g)
27.	1638	1628[83]	β -Cyclocitral	-	-	t	-	-	e), f), g)
28.	1648	1648[85]	Myrtenal	0.6	-	0.5	-	-	e), f), g)
29.	1664	-	Nonanol	-	-	-	-	12.1	e), f), g)
30.	1668	1666[88]	(<i>Z</i>)- β -Farnesene	2.5	23.9	-	1.2	-	e), f), g)
31.	1670	1671[87]	<i>trans</i> -Pinocarveol	2.0	-	1.6	-	-	e), f), g)
32.	1682	1687[87]	δ -Terpineol	-	-	t	-	-	e), f), g)
33.	1683	1685[82]	<i>trans</i> -Verbenol	0.1	-	0.3	-	-	e), f), g)
34.	1686	-	Lavandulol	0.9	-	0.3	-	-	e), f), g)
35.	1704	-	α -Terpinyl acetate	-	t	-	t	-	e), f), g)
36.	1706	1706[85]	α -Terpineol	0.3	-	t	-	-	e), f), g)
37.	1719	1719[85]	Borneol	3.7	-	2.0	5.9	-	e), f), g)
38.	1726	1726[85]	Germacrene D	3.5	4.7	2.0	-	-	e), f), g)
39.	1765	1755[89]	Geranyl acetate	0.2	-	0.6	-	-	e), f), g)
40.	1766	-	Decanol	-	-	-	-	12.5	e), f), g)
41.	1733	1729[82]	Neryl acetate	-	-	-	t	t	e), f), g)
42.	1740	1740[85]	α -Muurolene	0.5	-	t	-	-	e), f), g)
43.	1742	-	Geraniol	-	-	t	-	-	e), f), g)
44.	1755	1756[85]	Bicyclogermacrene	0.3	-	0.4	-	-	e), f), g)
45.	1773	1773[85]	δ -Cadinene	0.6	1.2	0.5	-	-	e), f), g)
46.	1776	1776[85]	γ -Cadinene	0.3	-	t	-	-	e), f), g)
47.	1786	1786[85]	<i>ar</i> -Curcumene	-	-	t	-	-	e), f), g)

No	RI ^{a)}	RRI ^{b)}	Compound	% ^{c)}					IM*
				Flower		Leaf		oot ^{d)}	
				HD	MSD-SPME	HD	MSD-SPME		
48.	1779	1779[85]	(<i>E,Z</i>)-2,4-Decadienal	t	-	-	-	-	e), f), g)
49.	1804	1804[85]	Myrtenol	1.0	-	0.5	-	-	e), f), g)
50.	1830		Tridecanal	t	-	-	-	-	e), f), g)
51.	1838		2-Phenylethyl acetate	t	-	0.4	-	-	f), g)
52.	1857	1857[85]	Geraniol	e	-	0.5	-	-	e), f), g)
53.	1868	1868[85]	(<i>E</i>)-Geranyl acetone	0.2	-	1.1	10.5	9.3	e), f), g)
54.	1871		<i>p</i> -Mentha-1,8-dien-10-yl acetate	-	-	t	-	-	e), f), g)
55.	1882		1-Isobutyl-4-isopropyl-3-isopropyl-2,2-dimethyl succinate	-	-	0.2	2.1	-	g)
56.	1889		Myrtenol	-	-	0.5	-	-	e), f), g)
57.	1896		Phenylethyl isobutyrate	0.8	2.2	-	-	-	e), f), g)
58.	1900		Nonadecane	t	-	-	-	-	e), f), g)
59.	1941	1941[81]	α -Calacorene	-	-	t	-	-	e), f), g)
60.	1957		Cubebol	1.1	-	0.3	-	-	e), f), g)
61.	1958	1957[81]	(<i>E</i>)- β -Ionone	-	-	0.4	10.3	-	e), f), g)
62.	1973		Dodecanol	-	-	-	-	1.5	e), f), g)
63.	1988		2-Phenylethyl-2-methylbutyrate	0.3	-	-	-	-	f)
64.	1992		2-Phenylethyl-3-methylbutyrate	-	-	-	t	t	g)
65.	2008	2008[90]	Caryophyllene oxide	0.2	-	0.4	-	-	e), f), g)
66.	2037	2037[81]	Salvial-4(14)-en-1-one	t	-	t	-	-	f), g)
67.	2041		Pentadecanal	0.3	-	0.3	-	-	e), f), g)
68.	2050	2050[82]	(<i>E</i>)-Nerolidol	-	-	0.2	-	-	e), f), g)
69.	2052		Isopropyl myristate	-	-	-	6.7	-	e), f), g)
70.	2088		1- <i>epi</i> -Cubanol	-	2.4	-	-	-	e), f), g)
71.	2090		Isogymnomitrol	-	-	0.3	-	-	e), f), g)
72.	2094		1,10-di- <i>epi</i> -Cubanol	0.3	-	0.7	-	-	e), f), g)
73.	2096	2096[81]	Elemol	-	-	0.3	-	-	e), f), g)
74.	2100		Heneicosane	0.4	-	-	-	-	e), f), g)
75.	2131	2131[81]	Hexahydrofarnesyl acetone	0.4	-	0.4	-	-	e), f), g)
76.	2144	2136[87]	Spathulenol	1.1	3.8	0.5	-	-	e), f), g)
77.	2178		(<i>Z</i>)-3-Hexen-1-ol benzoate	-	-	-	-	-	f), g)
78.	2179		3,4-Dimethyl-5-pentylidene-2(5H)-furanone	-	-	0.4	-	-	f), g)
79.	2187	2185[82]	T-Cadinol	1.5	3.9	1.0	4.3	-	e), f), g)
80.	2192		Muurolo-4,10(14)dien-1-ol	0.3	4.2	-	-	-	e), f), g)
81.	2194		3,4-Dimethyl-5-pentylidene-2(5H)-furanone	-	-	-	4.1	-	f), g)
82.	2198	2187[44]	Thymol	0.2	-	0.2	-	-	e), f), g)
83.	2209	2209[82]	T-Muurolo	-	-	-	t	t	e), f), g)
84.	2214	2214[81]	<i>ar</i> -Turmerol	-	-	-	<i>t</i>	<i>t</i>	e), f), g)
85.	2222	2208[91]	Copaborneol	0.4	-	0.4	-	-	e), f), g)
86.	2232	2236[85]	α -Bisabolol	4,3	-	6.6	-	-	e), f), g)
87.	2255		β -Sinensal	0.6	-	0.9	-	-	e), f), g)
88.	2256	2245[82]	<i>epi</i> - α -Bisabolol	-	4.9	-	6.4	-	e), f), g)
89.	2257	2258[81]	β -Eudesmol	3.9	8.0	3.6	7.5	-	e), f), g)
90.	2262	2278[92]	Torilenol	-	2.1	-	-	-	e), f), g)
91.	2265		Longiverbenone	1.0	-	4.9	7.9	14.14	e), f), g)
92.	2298	2285[83]	Decanoic acid	-	10.1	-	-	-	e), f), g)

No	RI ^{a)}	RRI ^{b)}	Compound	% ^{c)}					IM*
				Flower		Leaf		oot ^{d)}	
				HD	MSD-SPME	HD	MSD-SPME		
93.	2300	2300[81]	Tricosane	2.6	7.3	-	-	-	e), f), g)
94.	2353		(6S-7R)-Bisabolone	0.5		0.3			e), f), g)
95.	2392		Eudesma-4(15),7-dien-1- β -ol	-		0.6			e), f), g)
96.	2400	2400[81]	Tetracosane	0.2		-			e), f), g)
97.	2411		Hexyl cinnamic aldehyde	-		0.3			g)
98.	2415	2389[83]	Farnesyl acetone	-		0.6			e), f), g)
99.	2430		Chamazulene	t		-			e), f), g)
100.	2500	2500[81]	Pentacosane	0.8		0.4			e), f), g)
101.	2622	2606[88]	Phytol	-		2.8			f), g)
102.	2670		Tetradecanoic acid	-		0.7			e), f), g)
103.	2683	2655[81]	Benzyl benzoate	-		0.2			e), f), g)
104.	2700	2700[85]	Heptacosane	0.6		0.6			e), f), g)
105.	2766		Phenylethyl benzoate	0.2		-			e), f), g)
106.	2900	2900[85]	Nonacosane	0.3		4.5			e), f), g)
107.	2931	2931[81]	Hexadecanoic acid	1.0	7.2	4.5	6.8	15.1	e), f), g)
Total				94.2	95.6	87.3	99.9	68.7	

RRI: Relative retention indices; *IM: Identification Method ^{a)} RRI calculated against *n*-alkanes (C₉-C₄₀) on polar HP-Innowax column; ^{b)} RRI on polar column reported in literature ^{c)} Percent composition was obtained for each constituent on the basis of GC-FID analyses of the oils; ^{d)} The root volatiles were obtained only with MSD-SPME technique; ^{e)} Identification based on retention index of genuine compounds on the HP-Innowax column; ^{f)} Identification on the basis of computer matching of the mass spectra from Başer, Adams, MassFinder, Wiley, and NIST libraries; ^{g)} Tentative identified on the basis of computer matching of the mass spectra from Wiley Library.

Distribution of the major compound groups in the flower, leaf and root volatiles obtained with hydrodistillation (HD) and MSD-SPME techniques from *A. sivasica* is presented on Figure 1.

In hydrodistilled flower and leaf oils, oxygenated monoterpenes predominated constituting 43.6% and 26.1% of the oil, respectively. Application of MSD-SPME technique to the flowers and the leaves resulted in the extraction of oxygenated sesquiterpenes ranging from 15.8% to 29.3% and 22.8% to 36.6%, respectively. In the leaf oils, total amount of the sesquiterpene hydrocarbons was scarce. It was interesting to note that MSD-SPME extracted volatiles with lower percentage of the oxygenated monoterpenes (7.7%) from flowers. The root volatiles obtained with MSD-SPME technique were found as rich in aliphatic alcohols (30.1%) and oxygenated sesquiterpenes (23.4%). The monoterpenes have not been detected in the roots at all.

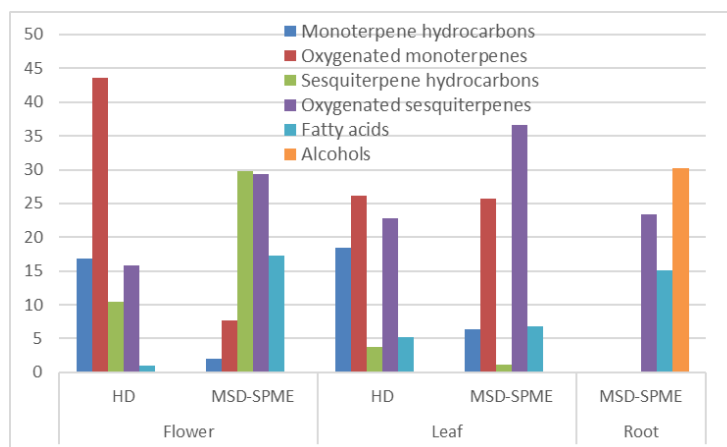


Figure 1. Distribution of the major compound groups in the flower, leaf and root volatiles obtained with hydrodistillation (HD) and MSD-SPME techniques from *A. sivasica*.

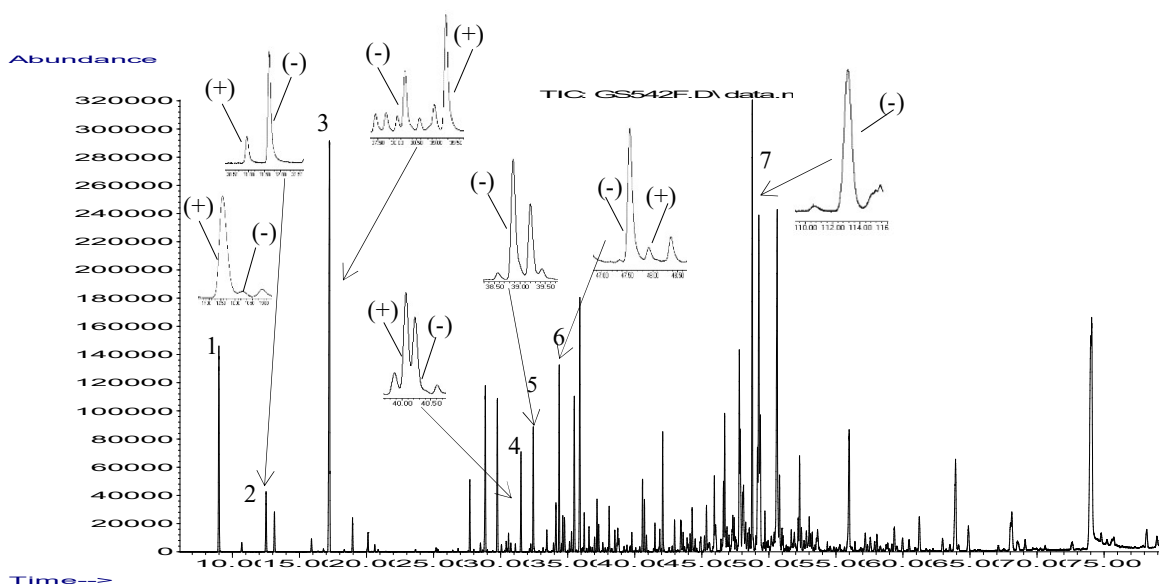


Figure 2. Representative chromatogram of *A. sivasica* flower essential oil and separation of major enantiomers: 1) α -pinene; 2) β -pinene; 3) limonene; 4) terpinen-4-ol; 5) *trans*-pinocarveol; 6) borneol; 7) α -bisabolol.

3.2. Enantiomeric Distribution

Stereochemical configurations of the major volatile constituents were measured by chiral GC-FID and GC/MS using three different chiral stationary phases. Chiral gas chromatography is the most popular method for the enantioselective determination of volatile phytochemicals. In scope of the present work, the essential oils of *A. sivasica* were analyzed by GC-FID and GC/MS using a split injection onto a capillary column with a stationary phase containing derivatized cyclodextrin macromolecules, achieving optimum separations of enantiomers. Before analyzing the oils, the chromatographic separation was optimized. The parameters were selected based on the information provided in the “Restek Guide to the Analysis of Chiral Compounds by GC”[93]. Representative chromatogram of separated enantiomers in the flower oil of *A. sivasica* is presented in Figure 2.

The enantiomeric ratios of certain enantiomeric compounds in the essential oil were determined, (-)- α -bisabolol and (-)-borneol were identified by 100% purity, α -pinene, β -pinene, limonene, *trans*-pinocarveol, terpinen-4-ol compound are presented by enantiomer pairs. Percentage distribution of the enantiomeric compounds in *A. sivasica* oils is presented in Table 5.

In the literature, there is scarce information about enantiomeric distribution of *Achillea* volatiles. The enantiomeric distribution of 1*S*-(-)-camphor was measured as 93.3% and 99.6% in *A. falcata* from two localities [38]. The enantiomeric distribution of 8 chiral constituents in *A. ligustica* were determined by GC-MS using two enantioselective stationary phases (DIME- α -CD and Lipodex-E) [94].

Table 4. Enantiomeric distribution of major volatiles from *A. sivasica*

No	Enantiomer	Flower oil	Leaf oil
1	(<i>IR</i>)-(+)- α -Pinene	95	98
2	(<i>IS</i>)-(-)- α -Pinene	5	2
3	(<i>IR</i>)-(+)- β -Pinene	21	14
4	(<i>IS</i>)-(-)- β -Pinene	79	86
5	(<i>4R</i>)-(+)-Limonene	66	80
6	(<i>4S</i>)-(-)-Limonene	34	20
7	(<i>4R</i>)-(+)-Terpinen-4-ol	57	66
8	(<i>4S</i>)-(-)-Terpinen-4-ol	43	34
9	(<i>1S,3R,5S</i>)-(+)- <i>trans</i> -Pinocarveol	7	21
10	(<i>1R,3S,5R</i>)-(-)- <i>trans</i> -Pinocarveol	93	79
11	(<i>1R,2S,4R</i>)-(+)-Borneol	-	-
12	(<i>1S,2R,4S</i>)-(-)-Borneol	100	100
13	(<i>2R</i>)-(+)- α - Bisabolol	-	-
14	(<i>2S</i>)-(-)- α -Bisabolol	100	100

3.3. Fatty Acid Contents

As part of our research on the phytochemical profile of essential oil bearing plants, *A. sivasica* was investigated also for its fatty acid composition. The present work is the first fatty acid analysis report on *A. sivasica*. Liquid-liquid extraction with subsequent methylation and GC-FID and GC/MS analyses revealed that *A. sivasica* fatty acids are comprised of 16 compounds representing 99.7% of the flower and 13 compounds representing 97.7% of the leaf lipids.

Table 5. Fatty acid composition of *A. sivasica*

No	Compound*	Flower	Leaf	
		%		
1.	C8:0	Methyl octanoate	0.1	-
2.	C10:0	Methyl decanoate (=Methyl caproate)	1.5	-
3.	C12:0	Methyl dodecanoate (=Methyl laurate)	0.2	-
4.	C14:0	Methyl tetradecanoate (=Methyl myristate)	0.9	0.5
5.	C15:0	Methyl pentadecanoate	0.2	0.1
6.	C16:0	Methyl hexadecanoate (=Methyl palmitate)	22.2	17.3
7.	C16:1 D-7 <i>cis</i>	(Z)-7-Methyl hexadecenoate	0.1	0.2
8.	C16:1 D-9 <i>cis</i>	(Z)-9-Methylhexadecenoate (= Methyl palmitoleate)	0.2	0.2
9.	C17:0	Methyl heptadecanoate	-	0.3
10.	C18:0	Methyl octadecanoate (=Methyl stearate)	6.3	0.7
11.	C18:1 D-9 <i>cis</i>	(Z)-9-Methyl octadecenoate (=Methyl oleate)	20.9	3.9
12.	C18:1 D-7 <i>cis</i>	(Z)-7-Methyl octadecenoate	2.1	0.7
13.	C18:2 D-9,12 <i>cis</i>	(Z,Z)-9,12-Methyl octadecadienoate (=Methyl linoleate)	26.9	16.9
14.	C18:1 D-9 <i>cis</i>	Ethyl linoleate	2.6	-
15.	C18:3 D-9,12,15 <i>cis</i>	Methyl linolenate	13.2	56.2
16.	C20:0	Methyl eicosanoate (=Methyl arachidate)	1.2	0.3
17.	C22:0	Methyl docosanoate (=Methyl behenate)	1.1	0.4
		Total	99.7	97.7

Seventeen fatty acids in total were identified and quantified in the flower and the leaf lipids. Fatty acids were classified as total saturated (SFA), unsaturated (UFA), monounsaturated (MUFA) and polyunsaturated (PUFA) fatty acids. Among SFAs, palmitic (22.2% and 17.3%) acid was found to be the main constituent in the flower and the leaf lipids, respectively. It was followed by stearic acid (6.3% and 0.7%). Eight SFA, C8:0, C10:0, C12:0, C14:0, C15:0, C17:0, C20:0 and C22:0 were detected in smaller amounts (0.1-1.5%). Oleic acid (20.9% and 3.9%) was detected as the major representative of MUFA in the flower and the leaf oils, respectively. PUFA fraction was presented by linoleic (26.9% and 16.9%) and linolenic (13.2% and 56.2%) acids. Over half of the leaf lipids consisted of linolenic acid. The ratio of PUFA/SFA ranged between 1.18 and 3.7, while the ratio of UFA/SFA was 1.95 and 3.98 in the flower and the leaf oils, respectively. Fatty acid chemical contents are summarized in Table 6.

In literature, there is scarce information about fatty acids of *Achillea* species. Oleic (34.7%), palmitic (23.1%) and linoleic (20.6%) acids were determined as the main fatty acids in *A. cappadocica* Hausskn, et Bornm, (whole plant) [95]. Achene fatty acids composition data for *A. biebersteinii* Hub.-Mor., *A. bisserata* M.Bieb., *A. multifida* (DC) Griseb., *A. wilhelmsii* K. Koch and *A. millefolium* L. were reported recently [96]. *A. millefolium* was characterized by low amounts of C16:0 (4%), C18:2 (5,6%) and C18:3 (10,68%) [97]. In contrast, *A. sivasica* was characterized by extremely high percentage of C18:3 fatty acid. A paper reported the occurrence of 24.3%-25.9%, 21.6%-16.8% and 41.3%-7.4% C18:2 and C18:3 in *A.*

linguata, *A. nobilis* and *A. crithmifolia*, respectively [98]. *A. tenuifolia* L. was characterized with high contents of linoleic C18:2 (69.4%), oleic C18:1 (14.5%) and linolenic C18:3 (1.7%) acids in the seed oil [99].

4. Conclusion

In scope of the present work, essential and fatty oils from different parts of *A. sivasica* obtained variously by conventional hydrodistillation, rapid MSD-SPME and solvent extraction (Folch method) techniques were analyzed by GC/MS and GC-FID. Enantiomeric distribution of main components was investigated with capillary enantioselective gas chromatography. This is the first contribution on chemical profile of *A. sivasica* volatiles, enantiomers, essential and fatty oils.

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References

- [1] N. Çelik and A. Akpulat (2008). *Achillea sivasica* (Asteraceae: sect. Babounya (DC.) O. Hoffm.), a new species from Inner Anatolia, Turkey, *Kew Bulletin* **63**, 485-489.
- [2] E. Nemeth (2010). *Achillea* species used medicinally in Hungary, *Isr. J. Plant Sci.* **58**, 279-289.
- [3] B. Benedek and B. Kopp (2007). *Achillea millefolium* L. sl revisited: recent findings confirm the traditional use, *Wiener Medizinische Wochenschrift*. **157**(13-14), 312-314.
- [4] G. Honda, E. Yesilada, M. Tabata, E. Sezik, T. Fujita, Y. Takeda and T. Tanaka (1996). Traditional medicine in Turkey VI. Folk medicine in West Anatolia: Afyon, Kütahya, Denizli, Muğla, Aydın provinces, *J. Ethnopharmacol.* **51**, 75-87.
- [5] T. Fujita, E. Sezik, M. Tabata, E. Yeşilada, G. Honda, Y. Takeda, T. Tanaka and Y. Takaishi (1995). Traditional medicine in Turkey VII. Folk medicine in middle and west Black Sea regions, *Econ. Bot.* **49**(4), 406-422.
- [6] E. Sezik, E. Yesilada, M. Tabata, G. Honda, Y. Takaishi, T. Fujita, T. Tanaka and T. Yoshio (1997). Traditional medicine in Turkey. VIII. Folk medicine in East Anatolia; Erzurum, Erzincan, Agri, Kars, Iğdir province, *Econ. Bot.* **51**, 195-211.
- [7] T. Baytop (1999). Türkiye'de Bitkiler ile Tedavi. Geçmişte ve Bugün, ed. B. T. Vol. 1-177. İstanbul Üniversitesi: 480.
- [8] M. Blumenthal, W.R. Busse, A. Goldberg, J. Gruenwald, T. Hall, C.W. Riggins and R.S. Rister (2000). Yarrow In: Herbal medicine expanded commission monographs, *Integr. Med. Commun. Boston.* 419-423.
- [9] A. Akçın, A. T.A., F. Seyis, A.Y. Coban and B. Durupınar (2014). Antimicrobial and antioxidant activity of the essential oil of the Turkish endemic species *Achillea phyrgia* Boiss.&Bal., *J. Essent. Oil Bearing Plants.* **17**(2), 219-227.
- [10] B. Demirci, I. Gurbuz, E. Yesilada and K.H.C. Baser (2009). Characterization and biological activity of *Achillea teretifolia* Willd. and *A. nobilis* L. subsp. *neilreichii* (Kerner) Formanek essential oils, *Turk. J. Biol.* **33**(2), 129-136.
- [11] N. Hasimi, S. Kizil and V. Tolan (2015). Essential oil components, microelement contents and antioxidant effects of *Nepeta italica* L. and *Achillea filipendulina* LAM., *J. Essent. Oil Bear. Plants.* **18**(3), 678-686.
- [12] K.H.C. Baser, B. Demirci, F. Demirci, S. Kocak, C. Akinci, H. Malyer and G. Guleryuz (2002). Composition and antimicrobial activity of the essential oil of *Achillea multifida*, *Planta Med.* **68**(10), 941-943.
- [13] A.D. Azaz, T. Arabaci and M.K. Sangun (2009). Essential oil composition and antimicrobial activities of *Achillea biserrata* M. Bieb. and *Achillea salicifolia* Besser subsp. *salicifolia* collected in Turkey, *As. J. Chem.* **21**(4), 3193-3198.

- [14] F. Candan, M. Unlu, B. Tepe, D. Daferera, M. Polissiou, A. Sokmen and H.A. Akpulat (2003). Antioxidant and antimicrobial activity of the essential oil and methanol extracts of *Achillea millefolium* subsp *millefolium* Afan. (Asteraceae), *J. Ethnopharmacol.* **87(2-3)**, 215-220.
- [15] C. Avancini, J.M. Wiest, Dall'Angol and G.L. Von Poser (2008). Antimicrobial activity of plants used in the prevention and control of bovine mastitis in Southern Brazil, *Latin Am. J. Pharm.* **27(6)**, 894-899.
- [16] S. Barel and J. Yashphe (1989). Effect of the essential oil from *Achillea fragrantissima* on *Escherichia coli* cells, *Curr. Microbiol.* **19**, 337-341.
- [17] A. Judzentiene (2016). Atypical chemical profiles of wild yarrow (*Achillea millefolium* L.) essential oils, *Rec. Nat. Prod.* **10(2)**, 262-268
- [18] G. Iscan, N. Kirimer, M. Kurkuoglu, T. Arabaci, E. Kupeli and K.H.C. Baser (2006). Biological activity and composition of the essential oils of *Achillea schischkinii* Sosn. and *Achillea aleppica* DC. subsp *aleppica*, *J. Agr. Food Chem.* **54(1)**, 170-173.
- [19] A. Dadkhah, F. Fatemi, S. Ababzadeh, K. Roshanaei, M. Alipour and B.S. Tabrizi (2014). Potential preventive role of Iranian *Achillea wilhelmsii* C. Koch essential oils in acetaminophen-induced hepatotoxicity, *Bot. Stud.* **55**:37. doi:10.1186/1999-3110-55-37
- [20] S.Kordali, A. Cakir, T.A. Akcin, E. Mete, A. Akcin, T. Aydin and H. Kilic (2009). Antifungal and herbicidal properties of essential oils and n-hexane extracts of *Achillea gypsicola* Hub-Mor. and *Achillea biebersteinii* Afan. (Asteraceae), *Ind. Crops & Prod.* **29(2/3)**, 562-570.
- [21] G. Benelli, S. Bedini, G. Flamini, F. Cosci, P.L. Cioni, S. Amira, F. Benchikh, H. Laouer, G. Di Giuseppe and B. Conti (2015). Mediterranean essential oils as effective weapons against the West Nile vector *Culex pipiens* and *Echinostoma intermediate* host *Physella acuta*: what happens around? An acute toxicity survey on non-target mayflies, *Parasit. Res.* **114**, 1011-1021.
- [22] B. Conti, A. Canale, A. Bertoli, F. Gozzini and L. Pistelli (2010). Mediterranean aromatic plants against the mosquito *Aedes albopictus* (Diptera: Culicidae), *Parasit. Res.* **107**, 1455-1461.
- [23] C. Bicchi, C. Frattini, L. Cantamessa and L. Poggio (1988). On the composition of *Achillea abrotanoides* (Vis.) Vis. essential oil, *Flav. Fragr. J.* **3(3)**, 101-104.
- [24] O. Toncer, S. Basbag, E. Diraz and M. Basbag (2010). Chemical composition of the essential oils of some *Achillea* species growing wild in Turkey, *Int. J. Agric. Biol.* **12(4)**, 527-530.
- [25] N. Simic, R. Palic, V. Vajs, S. Milosavljevic and D. Djokovic (2002). Composition and antibacterial activity of *Achillea asplenifolia* essential oil, *J. Essent. Oil Res.* **14**, 76-78.
- [26] E.K. Akkol, U. Koca, I. Pesin and D. Yilmazer (2011). Evaluation of the wound healing potential of *Achillea biebersteinii* Afan. (Asteraceae) by in vivo excision and incision models, *Evid-Based Compl Alt.* 474026.
- [27] Ö. Barış, M. Gulluce, F. Sahin, H. Ozer, H. Kilic, H. Ozkan, M. Sokmen and T. Ozbek (2006). Biological activities of the essential oil and methanol extract of *Achillea biebersteinii* Afan. (Asteraceae), *Turk. J. Biol.* **30(2)**, 65-73.
- [28] K. Polatoglu, O.C. Karakoc and N. Goren (2013). Phytotoxic, DPPH scavenging, insecticidal activities and essential oil composition of *Achillea vermicularis*, *A. teretifolia* and proposed chemotypes of *A. biebersteinii* (Asteraceae), *Ind. Crop. Prod.* **51**, 35-45.
- [29] R. Kotan, A. Cakir, F. Dadasoglu, T. Aydin, R. Cakmakci, H. Ozer, S. Kordali, E. Mete and N. Dikbas (2010). Antibacterial activities of essential oils and extracts of Turkish *Achillea*, *Satureja* and *Thymus* species against plant pathogenic bacteria, *J. Sci. Food Agr.* **90(1)**, 145-160.
- [30] F.Z. Kucukbay, E. Kuyumcu and T. Arabaci (2010). The essential oil oil *Achillea boissieri*, *Chem. Nat. Comp.* **46(5)**, 824-825.
- [31] N. Bezic, M. Skocibusic, V. Dunkic and A. Radonic (2003). Composition and antimicrobial activity of *Achillea clavennae* L. essential oil, *Phytother. Res.* **17(9)**, 1037-1040.
- [32] Z. Boskovic, N. Radulovic and S. G. (2005). Essential oil composition of four *Achillea* species from the Balkans and its chemotaxonomic significance, *Chem. Nat. Comp.* **41(6)**, 674-678.
- [33] E. Nemeth, B. J. and G. Tarjan (2008). Quantitative and qualitative studies of essential oils of Hungarian *Achillea* populations, *J. Herbs, Spices & Med. Pl.* **13(1)**, 57-69.
- [34] Z. Toker, H.C. Ozen, R.A. Clery and N.E. Owen (2003). Essential oils of two *Achillea* species from Turkey, *J. Essent. Oil Res.* **15(2)**, 100-101.
- [35] F.Z. Kucukbay, E. Kuyumcu, T. Bilenler and B. Yildiz (2012). Chemical composition and antimicrobial activity of essential oil of *Achillea cretica* L. (Asteraceae) from Turkey, *Nat. Prod. Res.* **26(18)**, 1668-1675.
- [36] A. Konakchiev, M. Todorova, B. Mikhova, A. Vitkova and H. Najdenski (2011). Composition and antimicrobial activity of *Achillea distans* essential oil., *Nat. Prod. Comm.* **6(6)**, 905-906.
- [37] P. Weyerstahl, H. Marschall, I. Seelmann and A. Rustaiyan (1997). Constituents of the essential oil of *Achillea eriophora* DC., *Flav. Fragr. J.* **12**, 71-78.
- [38] M. Kurkuoglu, B. Demirci, N. Tabanca, T. Ozek and K.H.C. Baser (2003). The essential oil of *Achillea falcata* L., *Flav. Fragr. J.* **18(3)**, 192-194.

- [39] F.Z. Kucukbay, E. Kuyumcu, S. Gunal and T. Arabaci (2011). Composition and antimicrobial activity of essential oil of *Achillea formosa* subsp. *amanica*, *Chem. Nat. Comp.* **47(2)**, 300-302.
- [40] K.H.C. Baser, B. Demirci, H. Duman, Z. Aytac and N. Adiguzel (2001). Composition of the essential oil of *Achillea gontiocephala* Boiss. et Bal. from Turkey, *J. Essent. Oil Res.* **13(4)**, 219-220.
- [41] S. Kordali, A. Cakir, T.A. Akcin, E. Mete, A. Akcin, T. Aydin and H. Kilic (2009). Antifungal and herbicidal properties of essential oils and n-hexane extracts of *Achillea gypsicola* Hub-Mor. and *Achillea biebersteinii* Afan. (Asteraceae), *Ind. Crop Prod.* **29(2-3)**, 562-570.
- [42] F.P. Turkmenoglu, O.T. Agar, G. Akaydin, M. Hayran and B. Demirci (2015). Characterization of volatile compounds of eleven *Achillea* species from Turkey and biological activities of essential oil and methanol extract of *A. hamzaoglu* Arabaci & Budak, *Molecules.* **20(6)**, 11432-11458.
- [43] K.H.C. Baser, B. Demirci and H. Duman (2001). Composition of the essential oils of two endemic species from Turkey: *Achillea lycaonica* and *A. ketenoglu*, *Chem. Nat. Comp.* **37(3)**, 245-252.
- [44] A. Bader, L. Panizzi, P.L. Cioni and G. Flamini (2007). *Achillea ligustica*: composition and antimicrobial activity of essential oils from the leaves, flowers and some pure constituents. *Cent. Eur. J. Biol.* **2(2)**, 206-212.
- [45] C.I.G. Tuberoso, A. Kowalczyk, V. Coroneo, M.T. Russo, S. Dessi and P. Cabras (2005). Chemical composition and antioxidant, antimicrobial, and antifungal activities of the essential oil of *Achillea ligustica* All., *J. Agr. Food Chem.* **53(26)**, 10148-10153.
- [46] A.D. Azaz, T. Arabaci, M.K. Sangun and B. Yildiz (2008). Composition and the in vitro antimicrobial activities of the essential oils of *Achillea wilhelmsii* C. Koch. and *Achillea lycaonica* Boiss & Heldr., *As. J. Chem.* **20(2)**, 1238-1244.
- [47] V.K. Agnihotri, S.K. Lattoo, R.K. Thappa, P. Kaul, G.N. Qazi, A.K. Dhar, A. Saraf, B.K. Kapahi, R.K. Saxena and S.G. Agarwal (2005). Chemical variability in the essential oil components of *Achillea millefolium* Agg. from different Himalayan habitats (India), *Planta Med.* **71(3)**, 280-283.
- [48] A. Barghamadi, M. Mehrdad, F. Sefidkon, Y. Yamini and M. Khajeh (2009). Comparison of the volatiles of *Achillea millefolium* L. obtained by supercritical carbon dioxide extraction and hydrodistillation methods, *J. Essent. Oil Res.* **21(3)**, 259-263.
- [49] A. Kocak, E. Bagci and A. Bakoglu (2010). Chemical composition of essential oils of *Achillea teretifolia* Willd. and *A. millefolium* L. subsp. *millefolium* growing in Turkey, *As. J. Chem.* **22(5)**, 3653-3658.
- [50] S. Alinezhad, A. Kamalzadeh, M. Shams-Ghahfarokhi, M.B. Rezaee, K. Jaimand, M. Kawachi, Z. Zamani, R. Tolouei and M. Razzaghi-Abyaneh (2011). Search for novel antifungals from 49 indigenous medicinal plants: *Foeniculum vulgare* and *Platycladus orientalis* as strong inhibitors of aflatoxin production by *Aspergillus parasiticus*, *Ann. Microbiol.* **61**, 673-681.
- [51] F. Gogus, M.Z. Ozel and A.C. Lewis (2006). Extraction of essential oils of leaves and flowers of *Achillea monocephala* by superheated water, *Flav. Fragr. J.* **21(1)**, 122-128.
- [52] C. Bekkechi, F.A. Bekkara, J. Casanova and F. Tomi (2011). Composition and antimicrobial activity of the essential oil of *Achillea odorata* L. subsp. *pectinata* (Lamk) var. *microphylla* (Willd.) Willk. from Northwestern Algeria, *J. Essent. Oil Res.* **23(3)**, 42-46.
- [53] K.H.C. Baser, B. Demirci, R. Kaiser and H. Duman (2000). Composition of the essential oil of *Achillea phrygia* Boiss. et Ball, *J. Essent. Oil Res.* **12(3)**, 327-329.
- [54] A. Akcin, T.A. Akcin, F. Seyis, A.Y. Coban and B. Durupinar (2014). Antimicrobial and antioxidant activity of the essential oil of the Turkish endemic species *Achillea phrygia* Boiss. & Bal., *J. Essent. Oil Res.* **17(2)**, 219-227.
- [55] S. Albayrak (2013). The volatile compounds and bioactivity of *Achillea sieheana* Stapf. (Asteraceae), *Iran J. Pharm. Res.* **12(1)**, 37-45.
- [56] N. Tabanca, T. Ozek, K.H.C. Baser and M. Vural (2004). Composition of the essential oil of *Achillea sieheana* stapf and the enantiomeric distribution of camphor, *J. Essent. Oil Res.* **16(3)**, 180-181.
- [57] E. Donmez, B. Tepe, D. Daferera and M. Polissiou (2005). Composition of the essential oil of *Achillea schischkinii* Sosn. (Asteraceae) from Turkey, *J. Essent. Oil Res.* **17(5)**, 575-576.
- [58] A. Sokmen, G. Vardar-Unlu, M. Polissiou, D. Daferera, M. Sokmen and E. Donmez (2003). Antimicrobial activity of essential oil and methanol extracts of *Achillea sintenisii* Hub. Mor. (Asteraceae), *Phytother. Res.* **17(9)**, 1005-1010.
- [59] P. Magiatis, A.L. Skaltsounis, I. Chinou and S.A. Haroutounian (2002). Chemical composition and in-vitro antimicrobial activity of the essential oils of three Greek *Achillea* species, *Zeitschrift Fur Naturforschung C-a J. Bioscie.* **57(3-4)**, 287-290.
- [60] S. Aslan, H. Evren, B. Konuklugil, I. Turkoglu and M. Kartal (2009). Essential oil composition of *Achillea teretifolia* from Turkey, *Chem. Nat. Comp.* **45(2)**, 274-275.
- [61] S.M.H. Alfatemi, J.S. Rad, M.S. Rad, S. Mohsenzadeh and J.A.T. da Silva (2015). Chemical composition, antioxidant activity and in vitro antibacterial activity of *Achillea wilhelmsii* C. Koch essential oil on methicillin-susceptible and methicillin-resistant *Staphylococcus aureus* spp., *3 Biotech.* **5(1)**, 39-44.

- [62] A. Çakir, H. Özer, T. Aydın, S. Kordali, A.T. Çavusoglu, T. Akçin, T. and A. Akçin (2016). Phytotoxic and insecticidal properties of essential oils and extracts of four *Achillea* species. *Rec. Nat. Prod.*, **10(2)**, 154-167.
- [63] M.R. Tellez, I.A. Khan, B.T. Schaneberg, S.L. Crockett, Rimando A.M. and M. Kobaisu (2004). Steam distillation–solid-phase microextraction for the detection of *Ephedra sinica* in herbal preparations, *J. Chromatogr. A.* **1025**, 51–56
- [64] G. Özek, T. Özek and K.H.C. Baser (2006). Comparison of the essential oils of *Prangos turcica* A. Duran, M. Sağiroğlu et H. Duman fruits obtained by different isolation techniques, *J. Essent. Oil Res.* **18**, 511-514.
- [65] T. Özek, G. Özek and K.H.C. Baser (2005). Comparison of the essential oils of three endemic Turkish *Heracleum* species obtained by different isolation techniques, *J. Essent. Oil Res.* **17**, 605-610.
- [66] G. Özek, F. Demirci, T. Özek, N. Tabanca, D.E. Wedge, S.I. Khan, K.H.C. Başer, A. Duran and E. Hamzaoglu (2010). Gas chromatographic–mass spectrometric analysis of volatiles obtained by four different techniques from *Salvia rosifolia* Sm., and evaluation for biological activity, *J. Chromatogr. A.* **1217(5)**, 741-748.
- [67] T. Özek, G. Özek, K. Baser and A. Duran (2005). Comparison of the essential oils of three endemic Turkish *Heracleum* species obtained by different isolation techniques, *J. Essent. Oil Res.* **17(6)**, 605-610.
- [68] G. Özek, T. Özek, K. Başer, A. Duran, M. Sağiroglu and H. Duman (2006). Comparison of the essential oils of *Prangos turcica* A. Duran, M. Sağiroglu et H. Duman fruits obtained by different isolation techniques, *J. Essent. Oil Res.* **18(5)**, 511-514.
- [69] G. Özek, T. Özek, K. Baser, A. Duran and M. Sağiroglu (2006). Comparison of essential oil of *Xanthogalum purpurascens* Lallem. obtained via different isolation techniques, *J. Essent. Oil Res.* **18(2)**, 181-184.
- [70] T. Özek, G. Özek, K.H.C. Başer, A. Duran and M. Sağiroglu (2008). Composition of the essential oils of *Angelica sylvestris* L. var. *sylvestris* isolated from the fruits by different isolation techniques, *J. Essent. Oil Res.* **20(5)**, 408-411.
- [71] W.A. König, R. Krebber, P. Evers and G. Bruhn (1990). Stereochemical analysis of constituents of essential oils and flavor compounds by enantioselective capillary gas chromatography, *J. Sep. Sci.* **13(5)**, 328-332.
- [72] K. Oosterhaven, K. Hartmans and J. Scheffer (1995). Inhibition of potato sprout growth by carvone enantiomers and their bioconversion in sprouts, *Potato Res.* **38(2)**, 219-230.
- [73] A.K. Tripathi, V. Prajapati and S. Kumar (2003). Bioactivities of l-carvone, d-carvone, and dihydrocarvone toward three stored product beetles, *J. Econ. Entomol.* **96(5)**, 1594-1601.
- [74] EDQM (2005). Determination of Essential Oils in Vegetable Drugs. European Pharmacopea 5.0. Vol. 1. European Directorate for the Quality of Medicines. Strasbourg: 217-218.
- [75] J. Folch, M. Lees and S. G.H.S. (1957). A simple method for the isolation and purification of total lipides from animal tissues, *J. Biol. Chem.* **226**, 497-509.
- [76] W.R. Morrison and L.M. Smith (1964). Preparation of fatty acid methyl esters and dimethylacetals from lipids with boron fluoride–methanol, *J. Lipid Res.* **5(4)**, 600-608.
- [77] I.A. Schepetkin, S.V. Kushnarenko, G. Özek, L.N. Kirpotina, P. Sinharoy, G.A. Utegenova, K.T. Abidkulova, T. Özek, K.H. Baser and A.R. Kovrizhina (2016). Modulation of human neutrophil responses by the essential oils from *Ferula akitschkensis* and their constituents, *J. Agr. Food Chem.* **64(38)**, 7156-7170.
- [78] D. Joulain, W.A. König and D.H. Hochmuth (2001). Library of MassFinder-4, in *Terpenoids and related constituents of essential oils*, D.H. Hochmuth, Editor. Hochmuth Scientific Consulting: Hamburg, Germany.
- [79] R.P. Adams (1995). Identification of Essential Oil Components By Gaz Chromatography/Mass Spectroscopy. Allured Publishing Corporation. Carol Stream.
- [80] K.H.C. Baser and F. Demirci (2007). Chemistry of Essential Oils, in *Flavours and Fragrances. Chemistry, Bioprocessing and Sustainability*, R.F. Berger, Editor. Springer-Verlag: Berlin, Heidelberg. p. 43-83.
- [81] H. Noorizadeh, A. Farmany and M. Noorizadeh (2011). Application of GA-PLS and GA-KPLS calculations for the prediction of the retention indices of essential oils, *Química Nova.* **34(8)**, 1398-1404.
- [82] A. Maggio, S. Rosselli, M. Bruno, V. Spadaro, F.M. Raimondo and F. Senatore (2012). Chemical composition of essential oil from Italian populations of *Artemisia alba* Turra (Asteraceae), *Molecules.* **17(9)**, 10232-10241.
- [83] D. Rigano, M. Marrelli, C. Formisano, F. Menichini, F. Senatore, M. Bruno and F. Conforti (2017). Phytochemical profile of three *Ballota* species essential oils and evaluation of the effects on human cancer cells, *Nat. Prod. Res.* **31(4)**, 436-444.
- [84] J.J. Brophy, R.J. Goldsack, A. Punruckvong, A.R. Bean, P.I. Forster, B.J. Lepschi, J.C. Doran and A.C. Rozefelds (2000). Leaf essential oils of the genus *Leptospermum* (Myrtaceae) in eastern Australia. Part 7. *Leptospermum petersonii*, *L. liveridgei* and allies, *Flav.Fragr. J.* **15(5)**, 342-351.
- [85] H. Noorizadeh and A. Farmany (2010). Exploration of linear and nonlinear modeling techniques to predict of retention index of essential oils, *J. Chinese Chem. Soc.* **57(6)**, 1268-1277.
- [86] A. Mangia and M. Careri (2001). Gas chromatography-mass spectrometry analysis of flavors and fragrances, in *Current Practice of Gas Chromatography-Mass Spectrometry*. CRC Press. p. 409-440.
- [87] C. Bignell, P. Dunlop and J. Brophy (1998). Volatile leaf oils of some south-western and southern Australian species of the genus *Eucalyptus* (series 1). Part XIX, *Flav. Fragr. J.* **13(2)**, 131-139.

- [88] J. Palá-Paúl, J. Brophy, R. Goldsack and B. Fontaniella (2004). Analysis of the volatile components of *Lavandula canariensis* (L.) Mill., a Canary Islands endemic species, growing in Australia, *Biochem. Syst. Ecol.* **32(1)**, 55-62.
- [89] A. Orav (2001). Identification of terpenes by gas chromatography-mass spectrometry, in *Current Practice of Gas Chromatography-Mass Spectrometry*. CRC Press. p. 483-494.
- [90] E. Mancini, L. De Martino, A. Marandino, M.R. Scognamiglio and V. De Feo (2011). Chemical composition and possible in vitro phytotoxic activity of *Helichrysum italicum* (Roth) Don ssp. *italicum*, *Molecules* **16(9)**, 7725-7735.
- [91] T. Aşkun, K.H.C. Başer, G. Tümen and M. Kürkçüoğlu (2010). Characterization of essential oils of some *Salvia* species and their antimycobacterial activities, *Turk. J. Biol.* **34(1)**, 89-95.
- [92] F. Ayaz, N. Küçükboyacı and B. Demirci (2017). Chemical composition and antimicrobial activity of the essential oil of *Conyza canadensis* (L.) Cronquist from Turkey, *J. Essent. Oil Res.*, **29(4)**, 336-343.
- [93] R. Tabacchi, G. Saturnin, C. Porret, M. Biedermann, S. Sponsler and L. Bitzer (1997). Guide to the analysis of chiral compounds by GC, *Restek Corp.* Lit. Cat.#59889
- [94] J.J. Filippi, D.A. Lanfranchi, S. Prado, N. Baldovini and U.J. Meierhenrich (2006). Composition, enantiomeric distribution, and antibacterial activity of the essential oil of *Achillea ligustica* All. from Corsica, *J. Agr. Food Chem.* **54(17)**, 6308-6313.
- [95] A. Ertaş, M. Boğa, N. Haşimi, Y. Yeşil, A.C. Gören, G. Topçu and U. Kolak (2014). Antioxidant, anticholinesterase, and antimicrobial activities and fatty acid constituents of *Achillea cappadocica* Hausskn. et Bornm, *Turk. J. Chem.* **38(4)**, 592-599.
- [96] F.A. Ayaz, H. Inceer, S. Hayirlioglu-Ayaz and N. Aksu-Kalmuk (2016). Achene fatty acid composition in the Tribe Anthemideae (Asteraceae), *Rom. Biotechnol. Lett.* **21(3)**, 11576-11584.
- [97] D. Warner, S.K. Jensen, J.W. Cone and A. Elgersma (2010). Fatty acid composition of forage species. in *the 23rd General Meeting of the European grassland Federation*. Kiel, Germany Organising Committee of the 23th General Meeting of the European Grassland Federation
- [98] R. Palić, G. Stojanović, N. Randelović, V. Randelović and J. Veličković (2000). The fatty acids from plants of the genus *Achillea*, *Facta universitatis-series: Physics, Chem. Technol.* **2(2)**, 101-104.
- [99] S.A.H. Goli, M. Rahimmalek and B.E.S. Tabatabaei (2008). Physicochemical characteristics and fatty acid profile of yarrow (*Achillea tenuifolia*) seed oil, *Int. J. Agr. Biol.* **10**, 355-357.

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