

Comparative chemical composition of the essential oils of Iranian *Achillea oxyodonta* from different ecological regions

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ARTICLE INFO

Article history:

Received on: 29/04/2015

Revised on: 08/05/2015

Accepted on: 22/05/2015

Available online: 27/05/2015

Key words:

Essential oil; Chemical composition; *Achillea oxyodonta*; endemic; GC-MS.

ABSTRACT

Hydro-distilled volatile oils from the aerial parts of *Achillea oxyodonta* (collected from two different locations), which is endemic to Iran, was analysed by GC and GC-MS. In the oil of *A. oxyodonta* from Shemshak sample, 54 compounds representing 95.68% of the total oil were characterized with camphor 13.18%; spathulenol 11.19%; 1,8-cineole 10.51%; salvia-4(14)-en-1-one 4.82%; eudesm-4-en-6-one 3.17%; caryophyllene oxide 3.07%; filifolone 3.03% as the major components. In the oil obtained from *A. oxyodonta* sample collected in Soleghan, 49 compounds representing 97.98% of the oil were characterized. spathulenol 13.13%; camphor 12.83%; 1,8-cineole 11.15%; cis- β -Farnesene 8.21%; α -Cadinol 4.83%; salvia-4(14)-en-1-one 4.19%; bornyl acetate 4.16%; isospathulenol 3.64%; germacrene D 3.45%; endo-1,5-Epoxy-salvia-4(14)-ene 3.09% was found as the main components. The results showed that there are qualitative similarities between the oils although the amounts of some corresponding compounds are different indicating that environmental factors strongly influence its chemical composition.

INTRODUCTION

The genus *Achillea* L. (Asteraceae), with about 115 species, is widely distributed in Europe, Asia and northern Africa and is naturalised in other parts of the world. The plants are perennial herbs and sub-shrubs with alternate, ordinary dentate to pinnatisect leaves and flower heads in dense corymbs (Oberprieler *et al.*, 2007). Nineteen species of this genus have been recognized in Iran; seven of these species are endemic (Rechinger, 1963; Mozaffarian, 2009).

These plants have been used as a medicinal herb for a long time (Fiume, 2001; Teixeira da Silva, 2004). Nowadays, different medicinal functions of *Achillea* such as spasmolytic, choleric, treatment of wounds and anti-inflammatory activities, make it as an important medicinal plant (Benedek *et al.*, 2007). Many species of the *Achillea* are traditionally used in Iran as diuretic and menstrual regularity agents for wound healing, diarrhea, flatulence and abdominal pain (Mazandarani *et al.*, 2013). Some of the *Achillea* species were also reported to be

used as herbal tea and additives in food and cosmetic products (Kirimer and Mat, 1999; Rauchensteiner *et al.*, 2004). Previous phytochemical investigations on *Achillea* genus reported essential oils that contain 1,8-cineole, camphor (Polatoglu *et al.*, 2013; Azaz *et al.*, 2009; Bader *et al.*, 2003; Baser *et al.*, 2001; Boskovic *et al.*, 2005; Candan *et al.*, 2003; Donmez *et al.*, 2005; Feizbakhsh *et al.*, 2003; Ghani *et al.*, 2008; Kordali *et al.*, 2009; Kundakovic *et al.*, 2007; Ozen *et al.*, 2003; Pavlovic *et al.*, 2008; Rustaiyan *et al.*, 1999; Suleimenov *et al.*, 2001), borneol (Agnihotri *et al.*, 2005; Kovacevic *et al.*, 2005; Kundakovic *et al.*, 2007; Rahimmalek *et al.*, 2009; Simic *et al.*, 2000;), α -thujone (Baser *et al.*, 2002; Ghani *et al.*, 2008; Azizi *et al.*, 2010; Tuberoso *et al.*, 2005), β -thujone (Tzakou and Loukis, 2009), β -pinene (Boskovic *et al.*, 2005; Kundakovic *et al.*, 2007; Simic *et al.*, 2005; Suleimenov *et al.*, 2001), santolina alcohol (Tuberoso *et al.*, 2005) and in some cases sesquiterpenes germacrene D (Maffei *et al.*, 1989; Rahimmalek *et al.*, 2009), β -caryophyllene (Agnihotri *et al.*, 2005; Simic *et al.*, 2002), carvacrol (Javidnia *et al.*, 2004) and spathulenol (Afsharypuor *et al.*, 1996; Rahimmalek *et al.*, 2009) as major compounds. In the literature there are many reports indicating the chemotype variation of the essential oils from *Achillea* species (Agnihotri *et al.*, 2005; Nemeth *et al.*, 1999; Nemeth, 2005).

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The plant *Achillea oxyodonta* investigated in this report is an endemic species of Iran which finds habitat in centre and west of Iran (Podlech, 1986). The study concerning the composition of *A. oxyodonta* oil is very limited. Until now, we have found only one published report on phytochemical composition of this species collected from Latian region in East of Tehran (Esmaeili *et al.*, 2006).

In the present work, we investigated the essential oil content and chemical composition of Iranian *A. oxyodonta* collected from two different localities in North and Northwest of Tehran province.

MATERIALS AND METHODS

Plant materials

The aerial parts of *A. oxyodonta* (sample 1,2) were collected during the flowering period in 14 June 2013 from Shemshak, the North of Tehran, at 2240 m altitude (sample 1) and 13 June 2013 from Soleghan, the Northwest of Tehran, at 1450 m altitude (sample 2) respectively. Voucher specimens have been deposited at the Herbarium Ministerii Iranici Agriculture (Voucher no. 63062-IRAN (sample 1) and 63061-IRAN (sample 2)).

Isolation of the essential oils

Aerial parts (150 g each) of the air dried plant samples 1, 2 from two various locations in Tehran province were separately subjected to hydro distillation for 4 hours using a Clevenger type apparatus according to the method recommended in the British Pharmacopoeia. The essential oil content determined was based on dry matter. Oils were obtained in 0.89% (sample 1) and 0.78% (sample 2) (w/w) yields. The oils were dissolved in n-hexane (Merck), dried over anhydrous sodium sulphate and stored at +4 °C until tested and analysed.

Essential oil analysis

Gas chromatography-flame ionization detector (GC-FID) analyses of the oil were conducted using a Thermoquest-Finnigan instrument (Thermo Fisher Scientific, USA) equipped with a DB-5 fused silica column (60m × 0.25mm i.d., film thickness 0.25µm). Nitrogen was used as the carrier gas at the constant flow of 1.1ml/min. The split ratio was 1/50. The oven temperature was raised from 60°C to 250°C at a rate of 5°C/min. The injector and detector (FID) temperatures were kept at 250°C and 280°C, respectively. GC-MS analysis was carried out on a Thermoquest-Finnigan Trace GC-MS instrument equipped (Thermo Fisher Scientific, USA) with the same column and temperature programming as mentioned for GC. Transfer line temperature was 250°C. Helium was used as the carrier gas at a flow rate of 1.1ml/min with a split ratio equal to 1/50. Mass spectra were taken at 70 eV.

Identification of the oil components

The constituents of the essential oils were identified by calculation of their retention indices under temperature-

programmed conditions for n-alkanes (C6-C24) and the oil on a DB-5 column under the same conditions. Identification of individual compounds was made by comparison of their mass spectra with those of the internal reference mass spectra library (Wiley7n.1) or with authentic compounds and confirmed by comparison of their retention indices with authentic compounds or with those of reported in the literature (Adams, 1995). Semi-quantitative data was obtained from FID area percentages without the use of correction factors.

RESULTS AND DISCUSSION

The hydrodistillation of the aerial parts of *A. oxyodonta* samples collected from two different locations in Tehran province gave greenish yellow oils with a yield of 0.89% (sample 1) and 0.78% (sample 2), on dry weight basis (w/w). The general chemical profiles of the tested oils, the percentage content of the individual compounds except the components with trace amounts (<0.20%) and retention indices are summarized in Table 1. Fifty-four compounds were identified in *A. oxyodonta* oil from Shemshak sample representing 95.68% of the oil; 49 compounds were identified in the oil from Soleghan sample representing 97.98% of the oil (Table 1). Main components of the first sample from Shemshak were camphor 13.18%; spathulenol 11.19%; 1,8-cineole 10.51%; salvia-4(14)-en-1-one 4.82%; eudesm-4-en-6-one 3.17%; caryophyllene oxide 3.07%; filifolone 3.03% and second sample Soleghan were spathulenol 13.13%; camphor 12.83%; 1,8-cineole 11.15%; cis-β-Farnesene 8.21%; α-Cadinol 4.83%; salvia-4(14)-en-1-one 4.19%; bornyl acetate 4.16%; isospathulenol 3.64%; germacrene D 3.45%; endo-1,5-Epoxysalvia-4(14)-ene 3.09%. Other components were present in amounts less than 3% (Table 1). Both *A. oxyodonta* oils were rich in camphor, spathulenol and 1,8-cineole however, other components with rather high amounts showed variation. For example, Sample from Soleghan had bornyl acetate, cis-β-Farnesene and α-Cadinol in rather high amounts unlike Shemshak sample. From Table, it is evident that there are many qualitative similarities between the oils although the amounts of some corresponding compounds are different; it may be related to the different geographical origins of the samples. In a previous investigation on *A. oxyodonta*, twenty-eight components were identified in the oil of plant, making up 96.4% of total composition (Esmaeili *et al.*, 2006). The main constituents were 1,8-cineole (38.5%), artemisia ketone (23.0%), α-pinene (4.4%), sabinene (4.1%), chrysantenone (3.7%) and piperitone (3.0%). In regard to the previously reported chemical composition of *A. oxyodonta* essential oil, it is interesting to point out that there are important qualitative and quantitative differences between the present work and that study suggesting that the environmental factors strongly influence its chemical composition. For instance, spathulenol was found to be the major constituent of *A. oxyodonta* essential oil in our research (Table 1); it was not detected in previous report. On the contrary, artemisia ketone, which was not present in our samples, was assayed to be the main component in the previous report.

Table 1: Essential oil compositions of *Achillea oxyodonta* from two different locations in Iran.

S. No.	Compound	RI	<i>A. oxyodonta</i>	
			Sample 1 RA%	Sample 2 RA%
1	α -Pinene	936	1.76	0.68
2	Camphene	952	1.11	1.52
3	Sabinene	974	tr	0.39
4	β -Pinene	980	0.63	0.90
5	2-Pentylfuran	987	0.81	0.71
6	dehydro-1,8-Cineol	990	0.19	tr
7	α -Terpinene	1016	0.86	0.93
8	p-Cymene	1024	0.55	0.79
9	Limonene	1029	0.31	0.45
10	1,8-Cineol	1032	10.51	11.15
11	γ -Terpinene	1057	0.68	0.35
12	α -Terpinolene	1088	tr	tr
13	Linalool	1094	tr	tr
14	Trans-Sabinene hydrate	1097	0.42	tr
15	2-Methyl butyl isovalerate	1100	0.32	0.25
16	Filifolone	1103	3.03	1.81
17	cis-p-Menth-2-en-1-ol	1122	tr	tr
18	Chrysanthenone	1125	tr	tr
19	trans-Pinocarveol	1143	0.41	tr
20	Camphor	1147	13.18	12.83
21	Menthone	1154	0.51	0.39
22	Pinocarvone	1164	0.96	-
23	Terpinen-4-ol	1178	2.18	1.25
24	α -Terpineol	1191	0.84	tr
25	Myrtenal	1198	1.30	1.08
26	Safranal	1200	0.50	-
27	Betacyclocitral	1221	0.61	0.50
28	Pulegone	1240	0.67	0.28
29	Carvone	1244	1.18	-
30	Piperitone	1255	2.24	0.78
31	cis-Chrysanthenyl acetate	1259	-	0.68
32	Bornyl acetate	1285	tr	4.16
33	Bicycloelemene	1339	0.72	0.29
34	α -Copaene	1382	1.04	0.61
35	β -Bourbonene	1393	0.56	0.60
36	trans-Caryophyllene	1429	1.85	1.37
37	cis- β -Farnesene	1450	2.96	8.21
38	α -Humulene	1464	0.26	0.23
39	Germacrene D	1489	1.41	3.45
40	Bicyclogermacrene	1505	1.80	1.26
41	δ -Cadinene	1526	0.93	2.82
42	trans-Nerolidol	1558	-	0.75
43	endo-1,5-Epoxy-salvial-4(14)-ene	1578	2.98	3.09
44	Spathulenol	1587	11.19	13.13
45	Caryophyllene oxide	1595	3.07	2.55
46	Salvial-4(14)-en-1-one	1605	4.82	4.19
47	Eudesm-4-en-6-one	1618	3.17	-
48	Isospathulenol	1621	1.97	3.64
49	epi- α -Cadinol	1646	0.75	0.91
50	α -Muurolool	1647	0.60	1.03
51	α -Cadinol	1659	0.60	4.83
52	α -Bisabolol	1689	1.45	2.40
53	Bisabolol oxide A	1753	1.01	-
54	14-Oxy- α -muurolole	1779	2.47	-
55	14-Hydroxy- α -muurolole	1787	1.53	-
56	6, 10,14-trimethyl-Pentadecanone	1839	2.78	-
57	6, 10,14-trimethyl-Pentadecanone	1840	-	0.74
	Total identified		95.68	97.98

RI, Retention index measured relative to n-alkanes (C6-C24) on the non-polar DB-5 column; RA, relative area; tr, traces (<0.2%), - not detected. Sample 1 - collection at Shemshak; Sample 2 - collection at Soleghan.

CONCLUSIONS

In general, these findings confirmed that essential oil composition of plant can be different in quality and quantities in different geographical and environmental conditions and period of

growth of plant. Thus to obtained uniform chemical contents, we recommend that the plants should be grown in cultural conditions as the next step.

ACKNOWLEDGMENT

The authors wish to thank from research department of Islamic Azad University, Roudehen Branch because of financial support for this study.

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How to cite this article:

Leila Shafiee Dastjerdi, Ali Mazoji. Comparative chemical composition of the essential oils of Iranian *Achillea oxyodonta* from different ecological regions. J App Pharm Sci, 2015; 5 (05): 106-109.