



Analysis of *Helichrysum oligocephalum* DC. essential oil

N. Ghassemi-Dehkordi, M. Sadeghi, M.R. Kaviani, B. Zolfaghari*

Department of Pharmacognosy and Isfahan Pharmaceutical Sciences Research Center, School of Pharmacy, Isfahan University of Medical Sciences, Hezar Jarib Avenue, Isfahan, Iran.

Abstract

Background and objectives: *Helichrysum oligocephalum* DC. (Compositae) is an endemic plant in Iran that has been recommended by Iranian traditional and folk medicine practitioners for gastrointestinal complaints. The plant is rich in essential oil and in the present investigation, the volatile composition of the species has been determined. **Methods:** The light yellow essential oil from the aerial parts of the plant was prepared according to the method which was recommended in British Pharmacopoeia by using hydrodistillation. The chemical composition of the oil was investigated by gas chromatography mass spectroscopy (GC/MS). **Results:** Thirty-seven compounds were identified; among them β -caryophyllene, α -humulene and epimanoyl oxide were dominant. Sesquiterpenoids were the most dominant compounds in the essential oil while monoterpenoids, fatty acids and diterpenoids were found to be less. These outcomes are a little bit different from what has been reported before. **Conclusion:** The differences between the results of the present study with previous works could be due to the diversity of variety, polymorphism, stage of plant growth and environmental factors.

Keywords: GC/MS, *Helichrysum oligocephalum*, Iran, volatile oil

Introduction

The genus *Helichrysum* is one of the members of Asteraceae (Compositae) family [1]. The name has been originated from Greek words: helios (Sun) and chrysos (gold); that represents the bright yellow flowers of the genus [2]. *Helichrysum* includes about 600 species widespread through in Eurasia, Africa and Australia [3]. It is represented, in flora of Iran, by 19 species, eight of which are endemic [4]. *Helichrysum* species are herbaceous, perennials or shrubs, their leaves are dense, oblong to lanceolate, simple entire. The bracts are numerous white or colored [5]. Some members of this genus have been used for

nervousness and hysteria [6], as anti-inflammatory, antiallergic [7], diuretic agents [7, 8], for healing wound, infections and respiratory complaints [9], and as hepatoprotective and anti-psoriasis agents [10] traditionally. Nowadays their antimicrobial [11-13], antioxidant [14,15] and anti-inflammatory [15] activities have been approved. The genus contains monoterpenes and sesquiterpenoids [9,10,13,16-18], diterpenoids [19,20], triterpenoids [21], flavonoids [6-8] and phenolic compounds [22]. Additionally some species such as *H. italicum* have been used in the fragrance industries [23]. *Helichrysum*

oligocephalum DC. is an endemic genus that is found in the north, west and center of Iran [24]. It has been used as adulteration of *Artemisia absinthium* which is called "Afsantin" in Persian language. It has been usually recommended for gastrointestinal complaints. In the present investigation the volatile composition of the species has been studied.

Experimental

Plant material

The plant material was collected from Baneh (Kordestan province, Iran) in June 2013. The plant was distinguished by Dr. L. Ghaemmaghami, Herbarium of Department of Biology, Faculty of Sciences, Isfahan, Iran, (voucher specimen no. 1925). The dried plant material was powdered at room temperature and stored at 4 °C.

Extraction

H. oligocephalum aerial parts powder (100 g) was hydro-distilled (with 1.2 L water) in a Clevenger-type apparatus for 4 h according to British Pharmacopoeia method [25]. The volatile oil was collected with pentane and was dried by anhydrous sodium sulfate then stored in a sealed vial at 4 °C until analysis. The yield of the oil was calculated based on the dried weight of the plant material.

GC/MS analysis

The GC/MS analysis was used for identification of the components. The procedure was performed on a Hewlett-Packard 5792A mass selective detector coupled with a Hewlett-Packard 6890 GC, equipped with a HP-5MS capillary column (30 m × 0.25 mm; film thickness 0.25 µm). The oven temperature was programmed from 60-280 °C increasing at 4 °C/min. Helium was used as the carrier gas at a flow rate of 2 mL/min. The injector temperatures was 280 °C and the split ratio was 1:50. The MS operating parameters were: ionization voltage, 70 eV; ion source temperature, 250 °C; ionization current 750 µA, mass range 35-425.

Identification of the constituents was performed on the basis of calculating Kovats index using a homologous series of *n*-alkanes (C₈-C₂₅) and by comparing the mass spectra with those in Database Wiley 275L library and the literature data (26,27).

Results and Discussion

The light yellow essential oil was obtained from *H. oligocephalum* aerial parts (0.8% v/w) bearing the characteristic aromatic odor of the plant. Thirty-seven compounds, representing 73.1% were distinguished (table 1) according to their mass spectra and retention indices. β-caryophyllene, α-humulene and epimanoyl oxide consisted more than 5% of the essential oil each. Sesquiterpenoids (44.0%) were the dominant compounds. Monoterpenoids, fatty acids and diterpenoids were found to be 7.8%, 15.4% and 5.4%, respectively. Other reports have demonstrated high concentration of ortho-vaniline [28] and thymol [29]. There is not much convergence between our results and the previously reported ones which could be due to the diversity of variety, polymorphism, stage of plant growth and environmental factors [23,30]. On the other hand, thymol was the most abundant monoterpene in our sample which has been the main constituent of *H. oligocephalum* oil reported by Sajjadi *et al.* although it is not a common compound in this genus. The main compound of some *Helichrysum* species have identified as 1,8-cineol, α-pinene, E-caryophyllene and β-selinene [6,9,31]. The main metabolites in the oil of *H. oligocephalum* have been distinguished as sesquiterpene hydrocarbons such as α-humulone (5.9%), β-caryophyllene (5.5%) and caryophyllen oxide (4.2%). Dominance of this class of volatile compounds is similar to the results of some other species of the genus such as *H. cordifolium* and *H. faradifani* [6,31]. Some constituents of the essential oil have demonstrated biologic effects in previous studies. α-Humulone has shown anti-inflammatory [32] and cytotoxic activities [33]. Caryophyllene has acted as selective agonist of

Table 1. Chemical constituents of *H. oligocephalum* essential oil

NO	Compound	Calculated KI	Reported KI	percentage
1	linalool	1099	1098	0.8
2	<i>Endo</i> -borneol	1166	1165	0.8
3	terpinene-4-ol	1177	1177	0.4
4	octanoic acid	1184	1179	0.9
5	α -terpineol	1189	1189	0.7
6	geraniol	1254	1253	0.3
7	bornyl acetate	1283	1289	1.8
8	thymol	1290	1290	3.0
9	α -longipinene	1346	1353	1.3
10	α -copaene	1372	1377	1.4
11	decanoic acid	1383	1385	3.5
12	tetradecane	1395	1400	0.1
13	β -caryophyllene	1415	1419	5.5
14	aromadendrene	1434	1439	0.8
15	α -himachalene	1444	1451	0.7
16	α -humolene	1450	1455	5.9
17	alloaromadendrene	1457	1460	2.6
18	δ -muroloene	1473	1480	1.0
19	β -selinene	1482	1490	3.9
20	α -selinene	1490	1498	2.1
21	α -muroloene	1495	1495	1.1
22	δ -amorphene	1509	1512	1.6
23	δ -cadinene	1519	1523	3.0
24	α -cadinene	1533	1539	0.4
25	α -calacorene	1538	1546	0.9
26	Lauric acid	1573	1571	1.4
27	Caryophyllene oxide	1578	1581	4.2
28	viridiflorole	1596	1593	1.5
29	1,2-epoxide humulene	1603	1608	3.1
30	α -cadinol	1637	1640	3.0
31	benzyl benzoate	1757	1762	0.2
32	myristic acid	1768	1768	2.8
33	methyl palmitate	1926	1927	0.3
34	palmitic acid	1973	1984	5.3
35	epimanoyl oxide	1987	2010	5.2
36	Z-phytol	2113	2114	0.2
37	linoleic acid	2136	2152	1.4

cannabinoid receptor type 2 [34], antinoceptive [35], neuroprotective, anxiolytic and antidepressant [36]. Caryophyllene oxide has anti-inflammatory and analgesic activities [37]. Partly high amount of epimanoyl oxide has been found in the oil of *H. oligocephalum* (5.2%), that has also shown anti-inflammatory activity [38]. These pharmacological activities could explain the anti-inflammatory activity of the total extract of the plant that has been previously reported by Minaian *et al.* [39].

Declaration of interest

The authors declare that there is no conflict of interest. The authors alone are responsible for the content of the paper.

References

- [1] Judd WS, Campbell CS, Kellogg EA, Stevens PF, Donoghue MJ. *Plant systematics: a phylogenetic approach*. 2nd ed. Sunderland: Sinauer Association Inc, 2002.

- [2] Pankhurst R, Tlyam R. *Plant and their names: A Concise Dictionary*. Edinburgh: Oxford University Press, 1995.
- [3] Suntar I, Keles H, Yesilada E, Sarker SD. Exploration of the wound healing potential of *Helichrysum graveolens* (Bieb) Sweet: Isolation of apigenin as an active component. *J Ethnopharmacol*. 2013; 49: 103-110.
- [4] Mozaffarian V. *Dictionary of Iranian plant names*. Tehran: Farhang Moaser Publishers, 1996.
- [5] Ghahreman A. *Iranian choromophites (Botanical systematic)*. Tehran: Iran University Press, 1994.
- [6] Lourens AC, Viljoen AM, van Heerden FR. South African *Helichrysum* species: A review of the traditional uses, biological activity and phytochemistry. *J Ethnopharmacol*. 2008; 119(3): 630-652.
- [7] Al-Rehaily AJ, Albishi OA, El-Olemy MM, Mossa JS. Flavonoids and terpenoids from *Helichrysum forskahlii*. *Phytochemistry*. 2008; 69(9): 1910-1914.
- [8] Süzgeç S, Meriçli AH, Houghton PJ, Çubukçu B. Flavonoids of *Helichrysum compactum* and their antioxidant and antibacterial activity. *Fitoterapia*. 2005; 76(2): 269-272.
- [9] Lourens AC, Reddy D, Başer KH, Viljoen AM, Van Vuuren SF. *In vitro* biological activity and essential oil composition of four indigenous South African *Helichrysum* species. *J Ethnopharmacol*. 2004; 95 (2-3): 253-258.
- [10] Satta M, Tuberoso CIG, Angioni A, Pirisi FM, Cabras P. Analysis of the essential oil of *Helichrysum italicum* G. Don. ssp. *microphyllum* (Willd) Nym. *J Essent Oil Res*. 1999; 11(4): 711-715.
- [11] Antunes Viegas D, Palmeira-de-Oliveira A, Salgueiro L, Martinez-de-Oliveira J, Palmeira-de-Oliveira R. *Helichrysum italicum*: From traditional use to scientific data. *J Ethnopharmacol*. 2014; 151(1): 54-65.
- [12] Oji KA, Shafaghat A. Constituents and antimicrobial activity of the essential oil from leaf and stem of *Helichrysum armenium*. *Nat Prod Commun*. 2012; 7(5): 671-674.
- [13] Bougatsos C, Ngassapa O, Runyaro DK, Chinou IB. Chemical composition and *in vitro* antimicrobial activity of the essential oil of two *Helichrysum* species from Tanzania. *Z Naturforsch C*. 2004; 59(5-6): 368-372.
- [14] Bigovic D, Savikin K, Jankovic T, Menkovic N, Zdunic G, Stanokovic T, Djuric Z. Antiradical and cytotoxic activity of different *Helichrysum plicatum* flower extracts. *Nat Prod Commun*. 2011; 6(6): 819-822.
- [15] Legoalea PB, Mashimbyeb MJ, Van Rec T. Antiinflammatory and antioxidant flavonoides from *Helichrysum kraussii* and *H. odoratissimum* flower. *Nat Prod Commun*. 2013; 8(10): 1403-1404.
- [16] Bougatsos C, Meyer JJM, Magitatis P, Vagias C, Chinou JB. Composition and antimicrobial activity of essential oil of *Helichrysum Kraussii* and *Helichrysum rugulosum* Less from South Africa. *Flavour Frag J*. 2003; 18: 45-51.
- [17] Baser KHC, Demirci B, Kirimer N. Compositions of essential oils of 4 *Helichrysum* species from Madagascar. *J Essent Oil Res*. 2002; 14: 53-55.
- [18] Formisano C, Mignola E, Rigano D, Senatore F, Arnold NA, Bruno M, Rosselli S. Constituents of leaves and flowers essential oils of *Helichrysum pallasi* (Spreng.) Ledeb

- Growing wild in Lebanon. *J Med Food*. 2009; 12(1): 203-207.
- [19] Zanetsie Kakam AM, Franke K, Ndom JM, Dongo E, Mpondo TN, Wessjohann LA. Secondary metabolites from *Helichrysum foetidum* and their chemotaxonomic significance. *Biochem Syst Ecol*. 2011; 39: 166-167.
- [20] Drewes SE, Mudau KE, van Vuuren SF, Viljoen AM. Antimicrobial monomeric and dimeric diterpenes from the leaves of *Helichrysum tenax* var *tenax*. *Phytochemistry*. 2006; 67(7): 716-722.
- [21] Mezzetti T, Orzalesi G, Rossi C, Bellavita V. A new triterpenoid lactone, alpha-amyrin and uvaol from *Helichrysum italicum*. *Planta Med*. 1970; 18(4): 326-331.
- [22] Jakupovic J, Zedro C, Grenz M, Tschritzis F, Lehmann L, Hasheminejad SM, Bohlman F. Twenty-one acyclophloroglucinal and further constituents from South African *Helichrysum* species. *Phytochemistry*. 1989; 28: 119-131.
- [23] Bianchini A, Santoni F, Paolini J, Bernardini AF, Mouillot D, Costa J. Partitioning the relative contributions of inorganic plant composition and soil characteristics to the quality of *Helichrysum italicum* subsp. *italicum* (Roth) G. Don fil. essential oil. *Chem Biodivers*. 2009; 6(7): 1014-1033.
- [24] Ghahreman A. *Flora of Iran*. Tehran: The Publication of Research Institute of Forests and Rangelands, 1992.
- [25] *British Pharmacopoeia*. London: HMSO Publication, 1988.
- [26] Adams RP. *Identification of essential oil components by gas chromatography/mass spectroscopy*. Illinois: Allured Publishing Corporation, 1995.
- [27] Swigar AA, Silverstein RM. *Monoterpenes: infrared, mass, ¹H-NMR, ¹³C-NMR spectra and Kovats indices*. Wisconsin: Aldrich Chemical Company, 1981.
- [28] Esmeili A. Biological activity and chemical composition of the stems and roots of *Helichrysum oligocephalum* DC. growing in Iran. *Pak J Pharm Sci*. 2013; 26(3): 599-604.
- [29] Sajjadi SE, Jafari A, Naderian M. Chemical composition of the essential oil of *Helichrysum oligocephalum*. *Chem Nat Compd*. 2009; 45(2): 269-271.
- [30] Roussis V, Tsoukatou M, Petrakis PV, Chinou I, Skoula M, Harborne JB. Volatile constituents of four *Helichrysum* species growing in Greece. *Biochem Syst Ecol*. 2000; 28(2): 163-175.
- [31] Cavalli JF, Ranarivelo L, Ratsimbason M, Bernardini AF, Casanova J. Constituents of the essential oil of six *Helichrysum* species from Madagascar. *Flavour Frag J*. 2001; 16(4): 253-256.
- [32] Rogerio AP, Andrade EL, Leite DFP, Figueiredo CP, Calixto JB. Preventive and therapeutic anti-inflammatory properties of the sesquiterpene α -humulene in experimental airways allergic inflammation. *Brit J Pharmacol*. 2009; 158: 1074-1087.
- [33] El Hadri A, Gomez Del Rio M, Sanz J, Coloma A, Idaomar M, Ozanas B. Cytotoxic activity of α -humulene and transcaryophyllene from *Salvia officinalis* in animal and human tumor cells. *An R Acad Nac Farm*. 2010; 76: 343-356.
- [34] Al Mansouri S, Ojha S, Al Maamari E, Al Ameri M, Nurulain SM, Bahi A. The cannabinoid receptor-2 agonist, β -caryophyllene, reduced voluntary alcohol

- intake and attenuated ethanol-induced place preference and sensitivity in mice. *Pharmacol Biochem Be.* 2014; 124: 260-268.
- [35] Paula-Freire LI, Andersen ML, Gama VS, Molska GR, Carlini EL. The oral administration of trans-caryophyllene attenuates acute and chronic pain in mice. *Phytomedicine.* 2014; 21(3): 356-362.
- [36] Bahi A, Al Mansouri S, Al Memari E, Al Ameri M, Nurulain SM, Ojha S. β -caryophyllene, a CB2 receptor agonist produces multiple behavioral changes relevant to anxiety and depression in mice. *Physiol Behav.* 2014; 135:119-124.
- [37] Chavan MJ, Wakte PS, Shinde DB. Analgesic and anti-inflammatory activity of caryophyllene oxide from *Annona squamosa* L. bark. *Phytomedicine.* 2010; 17(2):149-151.
- [38] Alcaraz MJ, Jimenez MJ, Valverde S, Sanz J, Rabanal RM, Villar A. Anti-inflammatory compounds from *Sideritis javalambrensis* n-hexane extract. *J Nat Prod.* 1989; 52(5): 1088-1091.
- [39] Minaiyan M, Ghassemi-Dehkordi N, Mahzouni P, Ahmadi N. Anti-inflammatory effect of *Helichrysum oligocephalum* DC. extract on acetic acid-induced acute colitis in rat. *Adv Biomed Res.* 2014; 3: 87.