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RESEARCH ARTICLE

Characterization of the endemic *Achillea teretifolia* Willd. essential oil

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Abstract
The genus *Achillea* L. (Asteraceae), which is commonly known as "yarrow", is mainly distributed in the Northern hemisphere. Turkey is one of the most important centers of diversity for the genus in the world. Many species of the genus *Achillea* have been known since the times of Dioscorides and used in the folk medicine to treat various illness and disorders in our country. In this study, the aerial parts of the endemic *Achillea teretifolia* Willd. collected from Afyon were investigated for essential oil composition. Hydrodistilled essential oil obtained from the aerial parts of the plant was analysed by gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS). Forty-eight compounds were identified representing 71.5% of the total oil, 1,8-cineole (16.1 %), camphor (12.7 %), p-cymene (10.6 %) and terpinen-4-ol (6.1 %) were characterized as the main constituents.

Keywords: *Achillea teretifolia*, Asteraceae, essential oil, GC-MS

Introduction

The genus *Achillea* L. (Asteraceae) constitutes of approximately 140 perennial herbaceous species worldwide. The genus is distributed in Europe and temperate areas of Asia and in North America and Middle east particularly in the Northern hemisphere (Nemeth, 2010). In Turkey, it is represented by 47 species, 24 of which are endemic (Huber-Morath, 1975; Duman, 2000; Arabacı & Budak, 2009).

In Anatolia, *Achillea* species are usually known as "civanperçemi" and various species of the genus are used as diuretic, emmenagogue, appetizer; carminative; in wound healing, abdominal pain, stomachache, symptomatic relief of colds, ulcer; and against diarrhea and flatulence in Turkish folk medicine (Honda et al., 1996; Baytop, 1999; Tuzlacı & Erol, 1999; Sezik et. al., 2001; Ezer & Arısan, 2006). Today, some therapeutic applications, such as antinociceptive, antiinflammatory wound healing, antispasmodic and hepatoprotective uses, are approved for several *Achillea* species by scientific experimental results (Agar et al., 2015). In addition to medical importance, *Achillea* species are also have economic value as they are consumed as vegetables, spices, beverages and additives in the food and cosmetic industry and in horticulture (Turkmenoglu et. al., 2015)

Numerous studies performed on various species have showed that the genus *Achillea* is rich in terpenoids and phenolics such as flavonoids and phenolic acids (Nemeth, 2005; Si et al., 2006; Saedina et al., 2011; Turkmenoglu et al., 2015; Agar et al., 2015), which are possible bioactive compounds. Monoterpenes were reported to be the major constituents of essential oil of the genus although high levels of sesquiterpenes were also quantified (Si et al., 2006; Saedina et al., 2011; Turkmenoglu et al., 2015; Başer, 2016).

Two main centers of diversity of *Achillea* occur in S.E. Europe and S.W. Asia. Diversified essential oil compositions from the Balkan Peninsula have been extensively studied and reported (Radulovic et al., 2007). However, report on essential oils of *Achillea* species growing in Turkey, which is one of the main centers of diversity, is still very limited. As a part of our ongoing studies of *Achillea* species, this paper represents the chemical compositions of the essential oil obtained by hydrodistillation from the aerial part of an endemic

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species *A. teretifolia* and analysed simultaneously by gas chromatography and gas chromatography-mass spectrometry.

Materials and Methods

Plant material

The aerial parts of *A. teretifolia* was collected during the flowering stage (July 2014) in Afyon, Şuhut, vicinity of Tekke Village, 1340 m. The collected samples were identified and the voucher specimens were deposited in the Herbarium of Hacettepe University, Faculty of Pharmacy (HUEF 14055).

Isolation of the essential oil

The air-dried aerial parts of the plant material were hydrodistilled for 3 h using a Clevenger-type apparatus to produce a small amount (<0.01%) of volatiles, which was trapped in *n*-hexane. Sample was stored at 4°C in the dark until analysed.

Gas chromatography analysis

The GC analysis was carried out using an Agilent 6890N GC system. FID detector temperature was 300 °C. To obtain the same elution order with GC/MS, simultaneous auto injection was done on a duplicate of the same column applying the same operational conditions. Relative percentage amounts of the separated compounds were calculated from FID chromatograms.

Gas chromatography-mass spectrometry analysis

The GC-MS analysis was carried out with an Agilent 5975 GC/MSD system. Innowax FSC column (60 m × 0.25 mm, 0.25 µm film thickness) was used with helium as carrier gas (0.8 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, and kept constant at 220 °C for 10 min and then programmed to 240 °C at a rate of 1 °C/min. Split ratio was adjusted at 40:1. The injector temperature was set at 250 °C. Mass spectra were recorded at 70 eV. Mass range was from m/z 35 to 450.

Identification of components

Identification of the oil components was carried out by comparison of their relative retention times with those of authentic samples or by comparison of their relative retention index (RRI) to series of n-alkanes. Computer matching against commercial (Wiley and MassFinder 3) (McLafferty & Stauffer, 1989; König et al., 2004) and in-house "Başer Library of Essential Oil Constituents" built up by genuine compounds and components of known oils, as well as MS literature data (Joulain & König, 1998) were also used for the identification.

Results and Discussion

Essential oil of *A. teretifolia* was obtained by hydrodistillation from air dried aerial parts and subsequently analyzed by GC and GC-MS systems. Forty-eight compounds representing 71.5% of the essential oil of *A. teretifolia* were characterized. Identified compounds with their relative percentages are listed in Table 1. The main constituents of *A. teretifolia* were 1,8-cineole (16.1 %), camphor (12.7 %), *p*-cymene (10.6 %) and terpinen-4-ol (6.1 %).

In the genus *Achillea*, composition of the essential oil is highly variable because of some biotic and abiotic factors, such as ontogenic and morphogenic differentiations, environmental factors as well as applied method of oil extraction (Kindlovits & Nemeth, 2012). Previous investigations demonstrated that, 1,8-

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cineole, found in almost every essential oil, was reported to be the most frequently identified component, ranging from trace levels to 47.7% in essential oils of Balkan *Achillea*, while camphor and borneol were the second and third repeatedly detected compounds, respectively. Moreover, caryophyllene oxide and β-caryophyllene were reported to be frequently identified sesquiterpenoids (Nemeth, 2005; Radulovic et al., 2007). The review on essential oil compositions of *Achillea* species growing in Turkey (Başer, 2016) indicated that 1,8-cineole and camphor were the most abundant constituents. In addition to these compounds, piperitone, ascaridol, *cis*-piperitol, *p*-cymene rich oils were also reported from Turkey.

Table 1. Main components (%) of the essential oil of *A. teretifolia*

No	RRI[a]	Compounds	%[b]	IM[c]
1.	1014	Tricyclene	0.1	MS
2.	1032	α-Pinene	1.3	RRI, MS
3.	1035	α-Thujene	0.2	MS
4.	1076	Camphene	2.3	RRI, MS
5.	1118	β-Pinene	0,5	RRI, MS
6.	1132	Sabinene	0,4	RRI, MS
7.	1188	α-Terpinene	1.1	RRI, MS
8.	1203	Limonene	0.2	RRI, MS
9.	1213	1,8-Cineole	**16.1**	RRI, MS
10.	1255	γ-Terpinene	0.3	RRI, MS
11.	1280	*p*-Cymene	**10.6**	RRI, MS
12.	1445	Filifolone	0.3	MS
13.	1451	β-Thujone	0.2	MS
14.	1522	Chrysanthenone	4.3	MS
15.	1532	Camphor	**12.7**	RRI, MS
16.	1553	Linalool	0.3	RRI, MS
17.	1556	*cis*-Sabinene hydrate	0.3	MS
18.	1571	*trans*-*p*-Menth-2-en-1-ol	1.0	MS
19.	1582	*cis*-Chrysanthenyl acetate	0.2	MS
20.	1586	Pinocarvone	0.3	RRI, MS
21.	1611	Terpinen-4-ol	**6.1**	RRI, MS
22.	1612	β-Caryophyllene	0.1	RRI, MS
23.	1638	*cis*-*p*-Menth-2-en-1-ol	0.4	MS
24.	1661	Alloaromadendrene	0.2	MS
25.	1670	*trans*-Pinocarveol	0.1	RRI, MS
26.	1682	δ-Terpineol	0.3	MS
27.	1689	*trans*-piperitol	0.3	MS
28.	1706	α-Terpineol	0.8	RRI, MS
29.	1719	Borneol	0.6	RRI, MS
30.	1748	Piperitone	0.2	RRI, MS
31.	1758	*cis*-Piperitol	0.3	MS
32.	1764	*cis*-Chrysanthenol	0.9	MS
33.	1776	γ-Cadinene	0.1	MS
34.	1786	*ar*-Curcumene	0.2	MS
35.	1804	Myrtenol	0.1	MS

36.	1969	*cis*-Jasmone	0.2	MS
37.	2008	Caryophyllene oxide	0.4	RRI, MS
38.	2071	Humulene epoxide-II	0.1	MS
39.	2074	Caryophylla-2(12),6(13)-dien-5-one	0.1	MS
40.	2144	Spathulenol	0.2	MS
41.	2186	Eugenol	0.1	RRI, MS
42.	2187	T-Cadinol	1.0	MS
43.	2257	β-Eudesmol	3.2	RRI, MS
44.	2260	15-Hexadecanolide	0.3	MS
45.	2300	Tricosane	0.4	RRI, MS
46.	2500	Pentacosane	1.1	RRI, MS
47.	2670	Tetradecanoic acid	0.3	RRI, MS
48.	2931	Hexadecanoic acid	0.7	RRI, MS
		Monoterpene Hydrocarbons	17.0	
		Oxygenated Monoterpenes	45.9	
		Sesquiterpene Hydrocarbons	0.6	
		Oxygenated Sesquiterpenes	5.0	
		Fatty acid	1.0	
		Others	2.0	
		Total	**71.5**	

[a] Relative retention indices calculated against *n*-alkanes; [b] % calculated from TIC data. [c] IM: Identification method based on the relative retention indices (RRI) of authentic compounds on the HP Innowax column; MS, identified on the basis of computer matching of the mass spectra with those of the Wiley and MassFinder libraries and comparison with literature data

Ünlü et al. (2002) previously reported 28 compounds in the essential oil of *A. teretifolia* collected from Sivas while main components of which were 1,8-cineole (19.9 %), camphor (11.1 %), borneol (11.9 %). In another study on *A. teretifolia* collected from Sivas, 1,8-cineole (15.9 %), borneol (8.1 %) and camphor (7.0 %) were the major identified compounds (Polatoğlu et al., 2013).

According to the report on *A. teretifolia* collected from Konya-Beyşehir, the main components of essential oil were 1,8-cineole (34 %), camphor (11 %), terpinen-4-ol (8 %), α-thujone (5 %) (Demirci et al., 2009).

1,8-cineole (34.3-54.5 %), camphor (14.2-19.8 %) and α-thujone (3.5-6.7 %) were reported to be the major identified compounds in essential oil of this plant collected from Isparta-Keçiborlu (Yaşar & Fakir 2016).

Kose et al. (2017) reported 1,8-cineole (18.8 %), camphor (17.6 %), *p*-cymene (13.7 %) and chrysanthenone (10.2 %) as the main constituents of essential oil of *A. teretifolia* collected from Afyon-Emirdağ.

Completely different oil compositions were also published. Aslan et al. (2009), identified 37 compounds in *A. teretifolia* essential oil and reported piperitone (21.37 %), linalool (18.99 %) as major compounds. However, location of the plant was not reported in the article. In another study, 3-cyclohexen-1-one (21.6 %), linalool (14,3 %), 1,8-cineole (12.7 %), chrysanthenone (8.6 %), *trans*-chrysanthenol (7.8 %), δ-cadinene (4.0 %) were reported as main constituents (Kocak et al., 2010). According to Başer (2016) as 3-cyclohexen-1-one is not found in nature, these data requires reconfirmation.

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