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Effect of reaping time on volatile constituents of *Achillea teretifolia* Willd.

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Abstract: In this study, *Achillea teretifolia* Willd. collected in June, July and August 2012 were compared on the basis of the chemical compositions of oils obtained by hydrodistillation. In the case of June sample, 40 compounds were identified representing the 97.7 % of the total oil. Thirty eight constituents consisting 97.3 % of the total composition were determined in the oil of July sample. On the other hand, 28 components were obtained representing 98.7 % of the oil of August sample. The major constituents of three oils were described as 1,8-Cineole (35.8, 34.3 and 54.5 %), Camphor (14.2, 15.1 and 19.8 %), α-Thujone (6.7, 6.6 and 3.5 %) and Terpinen-4-ol (5.2, 4.1 and 3.7 %), respectively. The main compound groups of *A. teretifolia* Willd. oil samples from three reaping times were terpene oxides, ketones and alcohols with their contents being 37.9, 23 and 23.3 % in the June sample, 36.6, 25.2 and 22 % in the July sample and 55.7, 24.4 and 14.1 % in the August sample, respectively.
Keywords: *Achillea teretifolia* Willd., Reaping time, Essential oil, GC-MS

Toplama zamanının *Achillea teretifolia* Willd.'in uçucu bileşenlerine etkisi

Özet: Bu çalışmada haziran, temmuz ve ağustos 2012 aylarında toplanan *Achillea teretifolia* Willd. bitkisine ait su buharı destilasyonu ile elde edilen yağlardaki kimyasal bileşenler karşılaştırılmıştır. Haziran ayına ait örnekte 40 bileşen tespit edilmiş olup toplam yağın % 97.7'sini oluşturmuştur. Temmuz ayında toplanan örnekte 38 bileşen belirlenmiş ve bileşenler toplam yağın % 97.3'ünü kapsamıştır. Diğer taraftan, 28 bileşen saptanan ağustos ayına ait olan örnekte ise bileşenler toplam yağın % 98.7'sini meydana getirmiştir. Her üç örneğe ait yağlarda ana bileşenler 1,8-Cineole (% 35.8, 34.3 ve 54.5), Camphor (% 14.2, 15.1 ve 19.8), α-Thujone (% 6.7, 6.6 ve 3.5) ve Terpinen-4-ol (% 5.2, 4.1 ve 3.7) olarak belirlenmiştir. Örneklerde saptanan bileşenler yüksek oranda 3 ana grupta toplanmış olup, bunlar terpen oksitler, ketonlar ve alkollerdir. Terpen oksitler, ketonlar ve alkoller sırasıyla haziran ayı örneğinde % 37.9, 23 ve 23.3, temmuz ayı örneğinde % 36.6, 25.2 ve 22 ve ağustos ayı örneğinde % 55.7, 24.4 ve 14.1 şeklinde yer almıştır.
Anahtar kelimeler: *Achillea teretifolia* Willd., Toplama zamanı, Uçucu yağ, GC-MS

1. Introduction

The genus *Achillea* (Asteraceae) is represented by 44 species and altogether 50 taxa, in which 21 are endemic to Turkey (Arabaci, 2006). Various species of the plant are traditionally used in Turkey for wound healing, against diarrhea and flatulence, as a diuretic, as emmenagog agents and also for abdominal pain (Baytop, 1999; Sezik and Yesilada, 1999; Sezik et al., 2001). *A. teretifolia* Willd. is a perennial plant and endemic to Turkey. *A. teretifolia* Willd. has 20-35 cm height, terete stem, linear filiform leaves and white corolla. This plant chooses rock slopes, coniferous forest and subalpine meadows as growth place (Davis et al., 1975).

In the present study, we investigated the effect of reaping time on chemical composition of the essential oils from aerial parts of *A. teretifolia* Willd. Plant samples were collected three times and compared the volatile constituents after gas chromatographic analysis using flame ionization and mass selective detections.

2. Material and method

2.1. Plant material

The flowering aerial parts of *A. teretifolia* Willd. were collected from Aydogmus-Keciborlu/Isparta in mediterranean region of Turkey about 1100 m in the middle of June, July and August 2012. The aerial parts were dried in darkness at the room temperature.

2.2. Isolation of the essential oil

The dried aerial parts of plants collected were submitted to water distillation for 3 h using a Clevenger-type apparatus. The obtained essential oils were used in GC and GC-MS analysis.

2.3. GC/MS and GC analysis

GC-MS analysis was performed on a Hewlett-Packard 6890 gas chromatograph equipped with a Hewlett Packard 5973 mass selective detector in the electron impact mode

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(70 eV) and HP-5MS capillary column (30 m x 0.25 mm i.d., 0.25 μm film thickness). Injector and interface were set at 250 and 300 °C, respectively. The oven temperature was programmed from 70 to 290 °C at a rate of 5 °C/min and then held for 10 min. Helium was the carrier gas, at a flow rate of 1 mL/min. The injection volume was 1 μL and GC/MS analysis was repeated three times for each sample.

GC analysis of the essential oil was performed under the same conditions with GC-MS (injection, column, oven temperature, flow rate of the carrier gas) using a flame ionization detector.

2.4. Identification of components

The retention indices of the essential oil components were determined in relation to homologous series of C_7–C_{31} *n*-alkanes on the HP-5MS column under the same chromatographic conditions. Further identification of components in the oils was based on computer matching with the Wiley library, as well as by comparison of their mass spectra with data in the literature (Adams, 2007). The relative percentages of the constituents were obtained from the chromatogram by computer.

3. Results and discussion

A. teretifolia Willd. yielded 0.42, 0.4 and 0.24 % (v/w, on dry basis) essential oil for sample from June, July and August 2012, respectively. GC and GC/MS analyses of the essential oils extracted from *A. teretifolia* Willd. collected in June, July and August 2012 enabled the identification of 106 constituents (40 in sample from June, 38 in sample from July and 28 in sample from August), representing 97.7, 97.3 and 98.4 % of the total GC peak areas, respectively. The major contributors to all three oils were 1,8-Cineole (35.8, 34.3 and 54.5 %), Camphor (14.2, 15.1 and 19.8 %), α-Thujone (6.7, 6.6 and 3.5 %) and Terpinen-4-ol (5.2, 4.1 and 3.7 %) (Table 1).

In the literature, there were several reports on the components of essential oil of *A. teretifolia* (Demirci et al., 2009; Unlu et al., 2002; Aslan et al., 2009). The essential oil (0.48 %, v/w) of *A. teretifolia* was analyzed, and 1,8-cineole (34 %), camphor (11 %), terpinen-4-ol (8 %), and α-thujone (5 %) were identified as the major components of the sample from Beysehir-Sarkikaraagac road/Turkey (Demirci et al., 2009). 1,8-cineole (19.9 %), borneol (11.9 %), camphor (11.1 %) and thujone (5.1 %) were reported as the main constituents in the volatile oil (0.43 %, v/w) of *A. teretifolia* collected in Kizildag Pass-Sivas/Turkey (Unlu et al., 2002). In another study, piperitone (21.37 %), linalool (18.99 %), 1,8-cineole (6.79 %), α-terpineol (5.88 %), and borneol (4.29 %) were found as most abundant compounds in the essential oil (0.4 %, v/w) of *A. teretifolia* from Turkey (Aslan et al., 2009).

1,8-Cineole was the major constituent essential oil of three *Achillea teretifolia* Willd. samples. 1,8-Cineole is mainly used in flavorings, fragrances, and cosmetics. 1,8-Cineole based eucalyptus oil is utilized as a flavoring at low levels (0.002%) in various products (baked goods, confectionery, meat products and beverages) (Harborne and Baxter, 2001). 1,8-Cineol is an effective substance in many mouthwash and cough suppressants and controls airway mucus hypersecretion and asthma via anti-inflammatory cytokine inhibition (Juergens et al., 2003; Juergens et al., 2004). 1,8-Cineole is also an effective treatment for nonpurulent rhinosinusitis (Kehrl et al., 2004). A study (Santos and Rao, 2000) showed that treated subjects experienced fewer headaches on bending, frontal headache, sensitivity of pressure points of trigeminal nerve, impairment of general condition, nasal obstruction and rhinological secretion. Side effects from treatment were in minimal level and 1,8-Cineole decreases inflammation and pain when applied topically. 1,8-Cineole is utilized as an insecticide and insect repellent (Klocke et al., 1987; Sfara et al., 2009). On the other hand, 1,8-Cineole is attractive to males of various species of orchid bees, who apparently gather the chemical to synthesize pheromones. It is generally used as bait to attract and collect these bees (Schiestl and Roubik, 2004).

Table 1. The volatile compounds of *Achillea teretifolia* Willd. collected at different times

No	Constituent	RI	A (%)	B (%)	C (%)
1	α-Pinene	946	0.2	0.3	0.1
2	Sabinene	973	0.3	1.1	0.1
3	β-Pinene	980	0.2	0.2	-
4	α-Phellandrene	1009	0.1	0.2	-
5	α-Terpinene	1020	0.4	0.2	-
6	Limonene	1026	0.2	0.3	0.1
7	p-Cymene	1029	3.2	1.3	0.4
8	1,8-Cineole	1031	35.8	34.3	54.5
9	γ-Terpinene	1059	2.1	3.4	0.2
10	cis-Sabinene hydrate	1069	0.3	3.3	-
11	Terpinolene	1088	0.2	0.5	-
12	Linalool	1101	-	2.5	-
13	trans- Sabinene hydrate	1103	0.5	-	-
14	α-Thujone	1111	6.7	6.6	3.5
15	β- Thujone	1121	1.2	2.2	0.9
16	cis-p-menth-2-en-1-ol	1126	3.4	4.1	3.8
17	trans-Pinocarveol	1145	0.1	0.2	-
18	Camphor	1150	14.2	15.1	19.8
19	Pinocarvone	1167	0.2	0.8	0.2
20	α-Phellandren-8-ol	1171	1.1	0.7	0.3
21	Epoxylinalool	1178	0.1	-	-
22	Terpinen-4-ol	1182	5.2	4.1	3.7
23	p-Cymen-8-ol	1189	0.2	0.1	-
24	α-Terpineol	1191	1.2	0.9	0.6
25	cis-Piperitol	1196	2.1	2.3	1.6
26	Myrtenal	1200	0.2	0.2	-
27	trans-Piperitol	1213	3.7	2.5	0.8
28	trans-carveol	1223	0.8	0.5	0.2
29	β-Cyclocitral	1226	0.1	0.2	0.8
30	2-undecanone	1295	0.7	0.5	-
31	p-Menth-1,4-dien-7-ol	1330	0.5	0.4	0.1
32	Eugenol	1361	0.6	0.7	0.3
33	Decanoic acid	1370	0.2	0.4	0.1
34	β-Caryophyllene	1418	0.4	0.4	0.2
35	allo-Aromadendrene	1458	0.3	0.2	0.1
36	Germacrene D	1480	0.6	-	-
37	Bicyclogermacrene	1494	3.2	0.6	1.6
38	Spathulenol	1577	3.4	1.2	2.1
39	Caryophyllene oxide	1584	2.1	2.3	1.2
40	α-Cadinol	1648	0.9	1.8	0.6
41	Hexadecanoic acid	1969	0.8	0.7	0.5
		Total:	97.7	97.3	98.4

RI: Retention indices on HP-5MS column.
A: Essential oil of *A. teretifolia* Willd. collected on 15th June of 2012.
B: Essential oil of *A. teretifolia* Willd. collected on 15th July of 2012.
C: Essential oil of *A. teretifolia* Willd. collected on 15th August of 2012.
%: Percentages calculated from FID data.

Camphor was second main component in the essential oils of the study. Camphor oil is commonly an ingredient in pain-numbing rubs or ointments, to be used externally. It can use for pain from arthritis, muscle aches and bruising, but should not be utilized on open wounds as it can scar internal tissue and slow healing. It also can be applied as a stimulant rub, encouraging circulation in stiff or cold limbs. Internally, camphor is a dependable remedy for sinus or lung congestion, and acts as an expectorant when steam with camphor is breathed in. It also can help to decrease fever as an inhalant (Anonymous, 2012a; Anonymous, 2012b). Camphor oil is an important ingredient in antibacterial and astringent cosmetic washes, to reduce acne or oily skin. It also is found in a treated white crystalline form, in which it can be utilized as a soap-making ingredient, as an insect repellent around the house and as a preservative. Cabinets produced from the wood of camphor are popular for natural history collections, as they keep away moths, worms and other insects. In Japan and China, it was utilized as a varnish, as a paint remover for oil paints, as fragrant oil for burning and as a diluting ingredient in inks. Camphor was used as a popular ingredient in traditional recipes in Europe, China and India (Anonymous, 2012b).

α-Thujone was the third main compound in the oil of June and July samples and the fourth major component in the oil of August samples. Thujone is a neurotoxic substance, acting as a convulsant and hallucinogen. Regulations in many countries limit the sale of oral–use produces including certain of these herbs and their essential oils (McGuffin et al., 1997). However, many of the plant oils containing thujone are used as flavoring ingredient in the alcoholic drink industry. *Thuja occidentalis* oil is also utilized as flavoring substance in most categories of foods, including alcoholic and nonalcoholic beverages, frozen dairy dessert, candy, baked goods, gelatins and puddings, meat and meat products, condiments and relishes (Leung and Foster, 1996).

Terpinen-4-ol was the fourth main substance in the oil of June and July samples and the third major constituent in the oil of August samples.Terpinen-4-ol, which is the main component in tea tree oil, is thought to be active germicidal compound (Anonymous, 2012c). On the other hand, odor of Terpinen-4-ol is intended for use in fragrances. It is utilized in many soap perfumes, perfume specialties, and in artificial oils such as geranium, pepper, and rose (Anonymous, 2012d).

The main groups of determined constituents of three oils obtained from *A. teretifolia* Willd. are given in Table 2. The major components, belonging to terpene oxides, were found as 39.9, 36.6 and 55.7 % in the oil samples from June, July and August. Ketones consisted of 23, 25.2 and 24.4 % in the oil of *A. teretifolia* Willd. collected in June, July and August. Alcohols were determined as 23.3, 22 and 14.1 % in June, July and August oil samples of *A. teretifolia* Willd. Terpenes with their contents being 11.4, 8.7 and 2.8 % were identified in the oil samples from June, July and August. Other components including terpene hydrates, acids and aldehydes constituted 2.1, 4.8 and 1.4 % in the oil of three *A. teretifolia* Willd. samples.

Table 2. The main groups of identified constituents of essential oils of *Achillea teretifolia* Willd.

Main Groups	A (%)	B(%)	C (%)
Terpene oxides	37.9	36.6	55.7
Ketones	23	25.2	24.4
Alcohols	23.3	22	14.1
Monoterpenes	6.9	7.5	0.9
Sesquiterpenes	4.5	1.2	1.9
Terpene hydrates	0.8	3.3	-
Acids	1	1.1	0.6
Aldehydes	0.3	0.4	0.8
Total	97.7	97.3	98.4

A: Essential oil of *A. teretifolia* Willd. collected on 15th June of 2012.
B: Essential oil of *A. teretifolia* Willd. collected on 15th July of 2012.
C: Essential oil of *A. teretifolia* Willd. collected on 15th August of 2012.

4. Conclusion

The results of the study indicated that different amounts of major components and main groups of determined components were obtained from oils of *A. teretifolia* Willd. collected in June, July and August 2012. *A. teretifolia* Willd. provides more 1,8-Cineole, camphor, terpene oxide and sesquiterpene content in August oil, α-Thujone, terpinen-4-ol and alcohol content in June oil and monoterpene and ketone content in July oil, when three reaping times were compared.

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