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Research Article

Volatile components of *Ferulago aucheri* Boiss. (Apiaceae)

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ABSTRACT

Ferulago aucheri Boiss, which belongs to the Apiaceae family, is distributed from Türkiye to the Caucasus. Its chemical composition may vary depending on the region where it grows, climate conditions and topography. Essential oil components were determined using two different techniques (hydrodistilled and headspace-solid phase microextraction HS-SPME) with the species collected from Nallıhan district of Ankara, Türkiye. In the present study, essential oil and headspace volatiles of aerial parts of *Ferulago aucheri* Boiss. were analyzed by GC-GC/MS. Thirty-five components were characterized, representing 99.6% of the oil by hydrodistilled and thirty components were characterized, representing 98.7% by HS-SPME. In both techniques, the main substances were identified as α-pinene, limonene, and δ-3-carene. In previous studies, it has been observed that the main components are in different amounts, and some studies even have different main components.

Keywords: Apiaceae, essential oil, *Ferulago aucheri*, GC- GC/MS, HS-SPME

1. INTRODUCTION

The Apiaceae family, which is a cosmopolitan, is widespread in the temperate zone of the Northern Hemisphere with 466 genera and approximately 3.820 species in the world [1]. The *Ferulago* W. Koch (Apiaceae) includes about 49 species worldwide [2]. In Türkiye, the genus *Ferulago* consists of about 34 species, 19 of which are endemic [3].

Ferulago species are used in folk medicine as antidiabetic, aphrodisiac, cancer, dermal wounds, eye pains, enhancing body strength, menstrual regulator and sedative [4-8]. There have been extensive phytochemical studies conducted with the roots and aerial parts of *Ferulago* taxa, which have shown that they are extremely rich in coumarins. Many flavonoids, terpenoids and other metabolites have also been identified. In the literature, volatile components of the genus *Ferulago* have been investigated with different techniques (headspace solid phase microextraction, hydrodistillation, microdistillation) in many studies [9-18]. In addition, essential oils of some *Ferulago* species were showed antimicrobial activity [9,10,12,14,16,17]. Stated that *F. sylvatica* (Besser) Rchb. may have different chemical components in samples collected from diverse geographical districts [19].

Ferulago aucheri Boiss. is known as 'Yayla kişnişi', it is a perennial and endemic species [3, 20]. The aerial parts of *F. aucheri* contains aromatic components, coumarins and flavonoids [21].

This study aims to investigate and compare *F. aucheri* the essential oil and the headspace volatile components.

2. MATERIALS AND METHODS

2.1. Plant materials

F. aucheri was collected in July 2020, from Nallıhan in Türkiye. The specimen had been stored in Anadolu University, Faculty of Pharmacy Herbarium (ESSE: 15826).

2.2. Isolation and analysis of essential oil

The air dried and crushed aerial parts of *F. aucheri* (25 g) were hydrodistilled for three hours using Clevenger apparatus. The essential oil obtained was stored at 4°C in the dark until analyzed. The oil was analyzed by capillary GC and GC/MS using an Agilent GC-MSD system [22].

2.3. Headspace-solid phase microextraction (HS-SPME) method, GC/MS analysis and identification of components

A SPME (SUPELCO) device consisting of a fused silica fiber, coated with 65 µm Polydimethylsiloxane/ Divinylbenzene (PDMS/DVB) adsorbent (Blue) was used. The air dried and crushed aerial parts volatile components of *F. aucheri* were captured with HS-SPME fiber and analyzed by GC/MS [23].

Analysis of GC and GC/MS

GC/MS: The GC/MS analysis was performed with an Agilent 5975 GC-MSD system. Innowax FSC column (60m x 0.25mm, 0.25mm film thickness) was used with helium as carrier gas (0.8 mL/min.). The temperature of GC oven 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 40:1 (splitless for HS-SPME). The temperature of the injector was at 250°C. MS were taken at 70 eV. Mass range was from m/z 35 to 450.

GC: The GC analysis were performed with Agilent 6890N GC system equipped with a FID detector set at 300 °C. To obtain the same elution order with GC-MS, simultaneous auto-injection was performed on a duplicate of the same column applying the same operational conditions. Relative percentage amounts of the separated substances were calculated from FID chromatograms.

Table 1. Essential oil (A) and HS-SPME (B) components of *F. aucheri*

RRI	Components	A %	B %
1032	**α-Pinene**	**59.8**	**47.8**
1076	Camphene	0.3	-
1118	β-Pinene	1.9	0.8
1132	Sabinene	1.1	2.1
1138	Thuja-2,4(10)-dien	0.4	1.1
1159	**δ-3-Carene**	**5.4**	**11.4**
1174	Myrcene	1.8	2.0
1187	*o*-Cymene	0.2	-
1203	**Limonene**	**18.8**	**21.9**
1246	(*Z*)-β-Ocimene	1.9	1.3
1255	γ-Terpinene	0.1	-
1266	(E)-β-Ocimene	0.2	-
1278	*m*-Cymene	0.2	-
1280	*p*-Cymene	1.5	3.5
1286	Isoterpinolene	0.1	-
1290	Terpinolene	0.1	-
1382	*cis*-Alloocimene	-	1.8
1435	γ-Campholene aldehyde	-	0.1
1443	2,5- Dimethylstyrene	-	0.2
1452	*p*-Cymenene	-	0.2
1466	α-Cubebene	-	0.2
1499	α-Campholene aldehyde	0.3	0.6
1535	Pinocamphone	-	0.2
1586	Pinocarvone	0.1	0.2
1591	Bornyl acetate	0.1	-
1611	Terpinen-4-ol	0.1	-
1617	Lavandulyl acetate	0.1	-
1638	*trans*-p-Menth-2,8-dien-1-ol	0.1	0.1
1645	*cis*-Verbenyl acetate	-	0.6
1648	Myrtenal	0.2	0.3
1670	*trans*-Pinocarveol	0.7	0.5
1678	*cis*-p-Mentha-2,8-dien-1-ol	0.1	-
1684	*trans*-Verbenol	1.7	0.5
1725	Verbenone	-	0.5
1726	Germacrene D	0.2	-
1747	*p*-Mentha-1,5-dien-8-ol	0.4	-
1754	Carvone	0.3	0.2
1797	Myrtenol	0.2	0.1
1811	*trans*-*p*-Mentha-1(7),8-dien-2-ol	-	tr
1845	*trans*-carveol	0.5	0.3
1856	*m*-Cymen-8-ol	0.1	0.1
1864	*p*-Cymen-8-ol	0.1	0.1
1867	*cis*-Carveol	0.1	tr
2144	Spathulenol	0.4	-

RRI: Relative retention indices calculated against n-alkanes; % calculated from FID data; tr; Trace (<0.1 %).

Table 2. Previously reported volatile components of *F. aucheri* and this study

Localities	Methods	Plant parts	Main Components	References
Muğla	Microdistillation	Crushed fruits	α-Pinene (35.9%)	Başer et al. 2002
Antalya	Hydrodistillation	Crushed fruits	Limonene (43.1%), α-pinene (18.3%), myrcene (7.0%)	Başer et al. 2008
Manisa	Hydrodistillation	Crushed fruits	Germacrene D (25.7%), (2E, 6E)-farnesol (8.0%)	Başer et al. 2008
Ankara (Mülk)	Hydrodistillation	Root	α-Pinene (80.3%)	Cumhur 2019
Ankara (Mülk)	Hydrodistillation	Aerial parts	α-Pinene (28.7%), 2,5-dimethoxy-*p*-cymene (15.3%), limonene (10.9%) and bornyl acetate (6.1%)	Cumhur 2019
Ankara (Nallıhan)	Hydrodistillation	Aerial parts	α-Pinene (59.8%), limonene (18.8%) and δ-3-carene (5.4%)	This study
Ankara (Nallıhan)	HS-SPME	Aerial parts	α-Pinene (47.8%), limonene (21.9%) and δ-3-carene (11.4%)	This study

Identification of substances

The components of essential oils were detected by comparison of their mass spectra with those in the Baser Library of Essential Oil Constituents, Wiley GC/ MS Library, Adams Library, Mass Finder Library and confirmed by comparison of their retention indices. Alkanes were used as the reference agents while calculating relative retention indices (RRI). Relative percentage amounts of the separated substances were calculated from FID chromatograms. Table 1 shows the data of the analysis.

3. RESULTS AND DISCUSSION

In the present study, volatile components of *F. aucheri* were investigated. Although the amount of oil obtained in the hydrodistillation process is high, the high temperature applied during boiling of water causes some thermal reactions. As a result, artifact formation, hydrolysis and isomerization events occur. In the SPME method, the sample preparation, extraction and concentration stages are combined in a single solvent-free step [24]. The yield of essential oil was 0.5%. Thirty-five components representing 99.6% and thirty components representing 98.7% were characterized by essential oil and HS-SPME from the aerial parts, respectively.

The main components of the essential oil and HS-SPME were identified as α-pinene (59.8%, 47.8%), limonene (18.8%, 21.9%) and δ-3-carene (5.4%, 11.4%) respectively.

Table 3. Essential oil (A) and HS-SPME (B) components groups

Grouped substances	A %	B %
Monoterpene hydrocarbons	93.8	93.9
Oxygenated monoterpenes	5.0	4.0
Sesquiterpenes hydrocarbons	0.2	0.2
Oxygenated sesquiterpenes	0.4	-
Others	0.2	0.6
Total %	99.6	98.7

Monoterpene hydrocarbons (93.8%, 93.9%) and oxygenated monoterpenes (5.0%, 4.0%) were the main groups present in the oil and HS-SPME, respectively (Table 3).

Some components were identified only in the oil (such as *p*-mentha-1,5-dien-8-ol, spathulenol, camphene) and some in the HS-SPME technique (such as *cis*-alloocimene, *cis*-verbenyl acetate, verbenone) (Table 1). Major components of the volatiles of *F. aucheri* previously reported (Table 2). Studies on pinene, one of the main components obtained from *F. aucheri*, show that its biological activity is high and it is promising as a therapeutic agent [25]. There are also studies showing that it has antitumor and antiviral activity [25,26].

4. CONCLUSION

In conclusion, the volatile components of *F. aucheri* was investigated and it was found that the main components were quite consistent for the two

techniques. However, it is thought that the main components of this plant may vary according to the region. Biological activity studies can be performed due to the availability of major components.

Ethical approval

Not applicable, because this article does not contain any studies with human or animal subjects.

Author contribution

Concept: EO, FEK, MK, SK; Design: EO, FEK, MK, SK; Supervision: EO, FEK, MK, SK; Materials: EO, FEK, MK, SK; Data Collection and/or Processing: EO, FEK, MK, SK; Analysis and/or Interpretation: EO, FEK, MK, SK; Literature Search: EO, FEK, MK, SK; Writing: EO, FEK, MK, SK; Critical Reviews: EO, FEK, MK, SK.

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Conflict of interest

The authors declared that there is no conflict of interest.

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