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Determination of volatile oil compounds and antioxidant activities of some *Cirsium* taxa grown in Türkiye

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Abstract. *Saral Ö, Karaköse M. 2024. Determination of volatile oil compounds and antioxidant activities of some* Cirsium *taxa grown in Türkiye. Nusantara Bioscience 16: 62-67.* Several *Cirsium* taxa are commonly used as a folk remedy or food in Anatolia and some countries. The study aims to determine the volatile oil profile and antioxidant activity of endemic *Cirsium trachylepis* Boiss., *Cirsium echinus* Hand.-Mazz., and *Cirsium osseticum* Petr. subsp. *osseticum*. The volatile oil compounds of three *Cirsium* species were analyzed by Gas Chromatography-Mass Spectrometry (GC-MS) using Solid Phase Microextraction (SPME). Additionally, total phenolic content was measured, and antioxidant activity capacity was determined by FRAP and DPPH• analyses in three *Cirsium* taxa methanolic extracts. A total of 32 (87.89%), 25 (88.69%), and 27 (80.68%) volatile compounds were identified from *C. trachylepis*, *C. osseticum* subsp. *osseticum*, and *C. echinus*, respectively. Pentadecanolide was the major volatile oil in three *Cirsium* species and was first reported in *Cirsium* taxa in this study. When the fatty acid content was examined, palmitic acid was determined as the common and main fatty acid for the three *Cirsium* species. A comparison of the antioxidant activity of three species showed that *C. echinus* had the highest antioxidant activity. The total phenolic content of *C. echinus* was found to be 250.84±0.46 mg GAE/100 g sample. While the FRAP value of *C. echinus* was 36.47±1.04 µmol Fe/g sample, the SC_{50} value was 1.89±0.05 mg/mL. This study may pave the way for the determination of volatile oils by SPME and the further development of research on *Cirsium* taxa.

Keywords: Antioxidant, *Cirsium trachylepis*, GC/MS, SPME, volatile oil

INTRODUCTION

Cirsium Mill., generally known as thistles, belongs to the Asteraceae family. It spreads worldwide, including Europe, North Africa, Siberia, Central Asia, West and East Africa, and Central America (Özcan et al. 2016). *Cirsium* taxa are known in Türkiye as "*körkenger, çakır dikeni,* and *eşek dikeni*" (Orhan et al. 2007). In Türkiye, this genus is represented by 73 taxa, and 28 taxa of these are found in North-east Anatolia. *Cirsium echinus* Hand.-Mazz. grows on rocky slopes, rarely on shores at 1,200-1,600 m asl (Özcan et al. 2008). *Cirsium trachylepis* Boiss. grows in woods at 500-1,760 m asl and is endemic (Özcan et al. 2008; Karaköse 2019). *Cirsium osseticum* Petr. subsp. *osseticum* grows at 700-1,132 m asl (Özcan 2017). Due to the uncontrolled proliferation of *Cirsium* taxa in Türkiye, it is considered a harmful weed in agricultural areas. In addition, local people use the root and stem of the genus *Cirsium* as a food and alternative medicinal plant (Demirtaş et al. 2017). Not only in Türkiye, but also in different world cultures, *Cirsium* taxa are used for medical purposes (Akbulut et al. 2022; Karaköse 2022a; Şen et al. 2022). *Cirsium* leaves are used to relieve abdominal pain and intestinal disorders in Italy (Guarrera 2005). Root or whole plant relieves bleeding, jaundice, and gastrointestinal disorders in China (He et al. 2014). *Cirsium* taxa are rich in silibinin and silymarin, which have biological activity. These two metabolites have a hepatoprotective effect (Yıldız et al. 2013; Ma et al. 2016). In addition, researchers have shown that *Cirsium* taxa have antioxidant (Sabudak et al. 2017), antifungal, antibacterial (Gulen et al. 2019), antidiabetic (Perez et al. 2001), and hepatoprotective effects (D'Andrea et al. 2005).

Although oxygen molecules are indispensable for the continuity of biological life, they also constitute the source of free radicals, which are highly reactive in the natural functioning of metabolism. These Reactive Oxygen Species (ROS) are a natural byproduct of metabolic functioning and are harmful substances (Chaudhary et al. 2023). Excessive ROS production in the body causes disorders such as DNA damage, lipid peroxidation, or cancer. Antioxidants protect cells by counteracting the damaging effects of the physiological process of oxidation (Kumar et al. 2017). Recently, we have witnessed increased interest in using natural substances from plant sources. Plants have valuable bioactive compounds such as phenolics, vitamins, carotenoids, and volatile oils. These compounds are responsible for significant antioxidant, anti-inflammatory, and antimicrobial activities (Pavela and Benelli 2016; Che and Zhang 2019).

Plant-based volatile oils are natural compounds with biological activity (Zeng et al. 2016). Therefore, volatile oils are used as additives in the food and cosmetics industry and pharmaceuticals, as well as their use in the field of health. The widespread use of volatile oils in different areas adds economic importance to the plant (Xing et al. 2019; Sadiq et al. 2021). The plant type and extraction method affect the composition and amount of volatile oils (Amiri et

al. 2018). The most widely used method to obtain volatile oil from plants is hydrodistillation. The use of the SPME (solid phase microextraction) method, which does not require solvent and is faster, has been increasing recently. SPME has been used in various fields, including determining volatile composition and screening flavors and taints (Kim et al. 2020).

There are many studies on volatile oil analysis by hydrodistillation in different *Cirsium* taxa (Özcan et al. 2016; Tüfekçi et al. 2018; Gulen et al. 2019; Kim et al. 2020), but the number of studies with SPME is limited in *Cirsium* species (Nazaruk et al. 2012; Amiri et al. 2018). For this reason, it was aimed to determine the volatile oil contents of *C. echinus*, and *C. osseticum* subsp. *osseticum*, and *C. trachylepis* (endemic to Türkiye) grown in Türkiye using the SPME method. In addition, the antioxidant activities of these plants were examined in the study.

MATERIALS AND METHODS

Plant material

The *C. echinus* and *C. trachylepis* were collected from Akpınar Village in Giresun (Türkiye) in July 2019. The *C. osseticum* subsp. *osseticum* was collected from Güllüce Village in Giresun (Türkiye) in July 2019 (Karaköse 2022b); Dr. Mustafa Karaköse identified these plants. Aerial parts of plants were dried at room temperature and powdered in an electric grinder (Waring Commercial, USA). The dry plant samples were divided into two. Five (5) g of the dry samples were weighed, and 25 mL methanol was added. Then, it was stirred at room temperature for 24 hours and filtered. Methanolic extracts were used for antioxidant activity assay. The remaining dry samples were used for volatile oil analysis. All the samples were stored at -20°C until analysis.

SPME procedure and GC/MS analysis

The dry plant (one gram) was placed in a vial of 10 mL, and then fiber coating was placed in the headspace. An SPME fiber (A polydimethylsiloxane/divinyl-benzene, Supelco, USA) was firstly conditioned for 5 min at 250°C in a gas chromatography (GC) injector. SPME analysis was done at 50°C with incubation time of 5 min, and extraction time of 10 min. Volatile oil analysis was performed on a Shimadzu QP2010 plus gas (connected to a Shimadzu QP2010 Ultra mass selector detector) chromatography using a TRB-5MS capillary column (30 mx 0.25 mm, film thickness, 0.25 µm). SPME fiber was inserted into the injection port of the GC-MS. The oven temperature was programmed to hold at 60°C for 2 min and then to increase to 240°C at 3°C/min, finally holding at 250°C for 4 min. The column flow rate was 1.0 mL/min, and transporter gas was utilized as Helium (99.999%). The MS was scanned from 40 m/z to 400 m/z at 70 eV. The volatile compounds were detected by comparing the mass spectra of the two libraries (FFNSC1.2 and W9N11) (Renda et al. 2016).

Total phenolic content analysis

The plant's Total Phenolic Content (TPC) was obtained using the Folin-Ciocalteu assessment (Slinkard and Singleton 1977). Initially, 400 µL Folin-Ciocalteu solution (0.5 N), 20 µL methanolic extract or standard (Gallic acid, 1-0.125 mg/mL), 680 µL of distilled water were added in a test tube, and the solution was vortexed. After 3 minutes of waiting, 400 µL of Na_2CO_3 (10%) was added, and the solution was vortexed again. After incubation for about 2 h, absorbance was measured at 760 nm. All measurements were made in triplicate.

Determination of antioxidant activity

The FRAP assay was made utilizing the technique of Benzie and Szeto (1999). The 100 µL of sample solution or standard ($FeSO_4$) and daily prepared 3 mL of FRAP solution (including TPTZ, iron (III) chloride, and acetate buffer) were added and vortexed. The absorbance on 593 nm was determined to be about 4 min at 25°C. All measurements were made in triplicate.

The scavenging capacity of DPPH• radical (2,2-diphenyl-1-picrylhydrazyl) of methanolic extraction was defined using the method of Molyneux (2004). 0.75 mL of methanolic extract or standard (various concentrations) and 0.75 ml of methanolic DPPH• solution (0.1mM) were added to the test tube, and the mixture was vortexed. Then, the mixture was left at room temperature for 50 min in the dark. Absorbance was monitored at 517 nm. Trolox was utilized as standard, and amounts were explained as SC_{50} (mg sample per mL). All measurements were made in triplicate.

RESULTS AND DISCUSSION

Volatile oils are a mixture of several bioactive chemical components, and their content varies from plant to plant, according to the growing season and environmental conditions (Zeng et al. 2016; Elshibani et al. 2020). SPME is one of the techniques for identifying volatile oil components and is based on the adsorption of these compounds on silica phase-coated fiber. It also determines the volatile oil composition of substances with different properties, such as fruit, vegetables, meat, or biological fluids (Bentivenga et al. 2004). SPME is a solvent-free, simple, inexpensive, rapid, and selective method for evaluating volatile compounds (Zhao et al. 2007; Renda et al. 2017). SPME fibers determine volatile oils based on their polarity and the thickness of the selected fibers (Pripdeevech et al. 2011).

GC-MS determined the volatile oil composition of Cirsium taxa with SPME. The volatile oil contents of the plant samples are given in Table 1. A total of 32 (87.89%), 25 (88.69%), and 27 (80.68%) constituents were identified from *C. trachylepis, C. osseticum* subsp. *osseticum,* and *C. echinus,* respectively. Of these, 14 volatile oils were the shared components, imparting the same aroma to all samples. While most of its volatile oil consists of

hydrocarbons and lactones, it contains small amounts of terpenes, esters, carboxylic acids, aldehydes, and ketones. As we all know, alcohols and aldehydes are the main aromatic substances.

Pentadecanolide was the major volatile oil in all samples, even though this was not reported previously in *Cirsium* taxa. Pentadecanolide, a macrocyclic lactone, is mostly used in the polymer, perfumery, and pharmaceutical industries (Emel'yanenko et al. 2011). 2-Hexenal, which has antimicrobial activity against food spoilage and pathogenic microbial species (Patrignani et al. 2008), was only detected in the *C. osseticum* subsp. *osseticum*. The lowest volatile l oil in it was found to be 2-ethyl hexanol. As far as we know, no study in the literature investigates the volatile content of *C. osseticum* subsp. *osseticum.* However, the fatty acid content of *C. osseticum* subsp. *osseticum* was investigated by hydrodistillation (Özcan et al. 2016). Linalool, ethyl propionate, and 2-phenylethanol were found only in *C. echinus*. In addition, 2-phenylethanol has the lowest percentage of volatile oil. This is the second study in which the volatile oil content of *C. echinus* was determined. In the first study, Rasulov et al. (1989) reported that β-sitosterol, stearic aldehyde, and triacontane were found in *C. echinus*. The *C. trachylepis* is endemic (Özcan 2017; Karaköse 2019), and only fatty acid content was investigated by hydrodistillation (Özcan et al. 2016). The volatile oil content of *C. trachylepis* was examined for the first time in this study. Among the three *Cirsium* samples, isobutyl angelate, heptanoic acid, undecylenic acid, and ethylene brassylate were found only in *C. trachylepis.*

There is limited research in the literature where volatile oil analysis in *Cirsium* has been performed using SPME. Zeng et al. (2016) compared the volatile oil content of *C. japonicum* Fisch. ex DC. and *C. setosum* (Willd.) Besser ex M.Bieb. obtained by hydrodistillation and SPME and found only 13 common components. It has been reported that 2,6,6-trimethyl-1-cyclohexene-1-carboxaldehyde, hexyl alcohol, 1-pentanol, 1-aziridineethanamine, and hexanal were detected in volatile oil analysis with SPME in *C. japonicum* and *C. setosum*. Notably, in the Zeng et al. (2016) study, 39 hydrocarbons were detected by hydrodistillation, while SPME detected 1 hydrocarbon. In the present study, 14 hydrocarbons were detected. These results indicate that volatile oil content varies depending on the extraction method and plant species. Kim et al. (2020) determined the volatile compounds in *C. setidens* (Dunn) Nakai in a study using four different SPME fibers (CWR/PDMS, DVB/PDMS, PDMS, PA). It was stated in the study that CWR/PDMS and DVB/PDMS coated SPME fibers had better results for *Cirsium* taxa. In the present study, DVB/PDMS-coated SPME fiber was used. They also reported that the main components were 2-pentylfuran, 1-methylcycloheptanol, 1-penten-3-ol, and 2,2,4,6,6,6-pentamethylheptane, regardless of SMPE fibers. However, benzaldehyde, β-ionone, and acetoin were detected, similar to our results. In another study, volatile oil analysis was examined by SPME in the fruits of *C. palustre* (L.) Scop. and *C. rivulare* (Jacq.) All.. Limonene was determined as the main component in both samples. In the same study, in which hexane extract was performed with Soxhlet apparatus, the main component was determined as β-sitosterol, but limonene could not be detected (Nazaruk et al. 2012). The present study found small amounts of limonene in *C. osseticum* subsp. *osseticum* and *C. echinus.*

As for the fatty acid content, palmitic acid stands out in all *Cirsium* samples. In addition, lower amounts of oleic and linoleic acids were detected compared to palmitic acid. However, unlike the literature, butyric acid was found in *C. osseticum* subsp. *osseticum.* Özcan et al. (2016) found that, unlike the present study, seeds of *C. trachylepis* contained high levels of linoleic and oleic acids, while palmitic acid levels were low. Similar to the previous study, Nazaruk et al. (2012) reported that linoleic and oleic acid were dominant in fruits of *C. palustre* and *C. rivulare*. Unlike the current study, extraction was performed with hexane in the Soxhlet apparatus in these studies. On the other hand, in previous studies on different *Cirsium* species, the main fatty acid was found as palmitic acid in *C. arvense* (L.) Scop. (Tüfekçi et al. 2018), *C. creticum* (Lam.) d'Urv. (Gulen et al. 2019), and *C. setidens* (Choi 2015). In addition, previous studies reported that it was detected in myristic acid, unlike other studies (Nazaruk et al. 2012; Zeng et al. 2016). It can be thought that factors such as the differences in the extraction method, plant species, and the plants' harvest time affect the results being so different.

Although oxygen molecules are indispensable for the continuity of biological life, they also constitute the source of free radicals, which are highly reactive in the natural functioning of metabolism (Kunwar and Priyadarsini 2011). Phenolics are also known to defend against free radicals due to their high antioxidant activity (Pietta et al. 2003). In this study, total phenolic content, FRAP, and DPPH$^{\bullet}$ analyses of methanolic extracts were performed to determine the antioxidant activities of the *Cirsium* samples. The results of total phenolic content and antioxidant activity are given in Table 2. In all analyses, *C. echinus* showed the highest antioxidant activity. The total phenolic content of *C. echinus* was 250.84±0.46 mg/100 g GAE sample. FRAP value was determined as 36.47±1.04 µmol Fe/g sample, and DPPH$^{\bullet}$ activities were determined as 1.89±0.05 mg/mL in *C. echinus*. There is a correlation between the total phenolic content and antioxidant activity. The FRAP value and DPPH$^{\bullet}$ scavenging activity of *C. echinus*, which has a high total phenolic content, was also high. To the best of our knowledge, our findings are the first results of antioxidant activity for *C. trachylepis*, *C. echinus*, and *C. osseticum* subsp. *osseticum*; previous studies in Türkiye and different countries have shown that other *Cirsium* taxa have antioxidant activity. While the total phenolic content in the methanolic extracts of *C. yildizianum* Arabaci & Dirmenci collected from Bingöl in Türkiye was found to be 37.10 mg GAE/g, the FRAP value was 89.95 mg TE/g, and the DPPH radical scavenging activity was found to be 40.76 mg TE/g (Llorent-Martínez et al. 2020). In another study, total phenolic of 61.21 ± 0.37µg catechol Eq/mg and SC_{50} of 0.22 mg/ml was determined in methanolic extracts of *C. vulgare* (Savi) Ten. (Thrace Region, Türkiye) (Sabudak et al. 2017). The total phenolic content was found to be 174.7±21.7 mg gallic

acid/g dw, and the DPPH· inhibition value was found to be 38.34±1.87% in the study in the methanolic extracts of the leaves of *C. palustren* in Poland (Malejko et al. 2014). It should be noted that the results of the antioxidant activity of plants could vary depending on the location, plant species, harvest time, and the extraction solvent or process.

Table 1. Volatile oil composition of *Cirsium trachylepis*, *C. osseticum* subsp. *osseticum* and *C. echinus*

Compound	**RI Exp.**[a]	**RI Lit.**[b]	***C. trachylepis* (%)**[c]	***C. osseticum* (%)**[c]	***C. echinus* (%)**[c]
Aldehydes					
Caproaldehyde	804	806	2.36	2.01	2.63
2-Hexenal	826	827	-	2.29	-
Benzaldehyde	950	962	1.48	2.64	1.68
Octanal	986	998	0.68	-	-
Nonanal	1109	1083	-	0.98	0.71
Phellandral	1250	1274	1.24	-	-
Cyclamal	1451	1459	-	4.10	-
Tridecanal	1490	1491	-	0.88	-
Ketone					
Acetoin	711	713	2.09	1.86	1.72
Hydrocarbons					
Hendecane	1065	1100	1.09	-	1.22
Dodecane	1207	1200	-	0.73	1.33
Tridecane	1291	1299	1.13	2.38	1.21
Tetradecane	1380	1399	3.63	9.44	4.55
Pentadecane	1507	1499	0.62	-	5.12
Hexadecane	1599	1600	1.43	-	-
Heptadecane	1705	1701	3.51	7.71	4.12
Octadecane	1792	1799	4.51	4.34	4.79
Nonadecane	1897	1899	-	1.61	0.56
Eicosane	1998	2000	3.28	3.09	-
Heneicosane	2093	2099	2.41	-	-
Docosane	2197	2200	1.01	-	-
Tetracosane	2397	2400	-	-	0.51
Pentacosane	2499	2500	1.30	-	0.56
Alcohols					
2-ethyl hexanol	1030	1030	-	0.75	0.53
2-Phenylethanol	1117	1114	-	-	0.48
Esters					
Ethyl propanoate	716	716	-	-	4.85
İsobutyl angelate	1053	1051	0.93	-	-
Lactones					
γ-Butyrolactone	916	915	2.76	2.32	2.36
γ-Hexalactone	1058	1047	0.45	-	-
Pentadecanolide	1841	1839	15.16	12.06	17.76
Ethylene brassylate	2017	2015	4.24	4.42	-
Terpens					
Limonene	1027	1029	-	1.81	1.17
Linalool	1165	1095	-	-	1.46
Isopulegol	1141	1145	0.65	-	-
Anethole	1281	1282	0.67	-	-
Anethofuran	1187	1186	0.67	-	-
β-ionone	1481	1487	2.55	3.62	2.89
Fatty Acids					
Butyric acid	804	808	-	3.24	-
Isovaleric acid	844	835	1.76	-	2.63
Valeric acid	887	882	3.86	3.58	3.75
Palmitic acid	1925	1921	7.52	7.07	6.25
Linoleic acid	2139	2133	6.02	3.65	2.98
Oleic acid	2144	2141	5.22	2.11	2.86
Carboxylic acids					
Heptanoic acid	1080	1076	1.55	-	-
Undecylenic acid	1453	1458	2.11	-	-
Total			87.89	88.69	80.68

Note: [a]: RI calculated from retention times relative to that of n-alkanes (C7-C30) on the nonpolar TRB-5MS column, [b]: RI lit. Literature value (Adams 2007), [c]:%Area obtained by FID peak-area normalization

Table 2. Results of TPC, FRAP and DPPH analyses

Samples	TPC (mg GAE /100 g sample)	FRAP (µmol Fe/g sample)	DPPH:SC50 (mg/mL)
C. osseticum	171.63±0.18	19.38±0.58	2.53±0.98
C. trachylepis	195.64±0.20	22.23±0.22	3.91±0.85
C. echinus	250.84±0.46	36.47±1.04	1.89±0.05
Trolox	-	-	0.02±0.00

In summary, this study aimed to determine the volatile oil content of endemic *C. trachylepis*, *C. echinus*, and *C. osseticum* subsp. *osseticum* grown in Türkiye by SPME with GC-MS. Pentadecanolide was found as the major volatile oil in all *Cirsium* species. Pentadecanolide was reported for the first time in *Cirsium* taxa in this study. Palmitic acid was detected as the main fatty acid in all samples. Additionally, in this study, the antioxidant activity of *Cirsium* species could be compared, and the highest activity was found in *C. echinus.* As far as we know, there is limited research in the literature in which volatile oil analysis has been performed in the *Cirsium* species using SPME (Nazaruk et al. 2012; Zeng et al. 2016; Kim et al. 2020). This is the first study to determine the volatile oil content of *C. trachylepis*, *C. echinus*, and *C. osseticum* subsp. *osseticum* using SPME. Fewer studies using SPME for volatile oil and fatty acid analysis in *Cirsium* taxa make it difficult to compare studies. Increasing the number of studies in which volatile oil contents are determined using SPME in different *Cirsium* taxa will allow comparisons between studies. The presence of various volatile oils of pharmaceutical or industrial importance can also be detected.

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