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Determination of Volatile Organic Compounds in Different Parts of *Hibiscus syriacus* L. by SPME/GC-MS Method

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Abstract

Aromatic plants are plants with a wide range of traditional uses. One of the areas of medical use is their consumption for nutritional purposes. Aromatic plants have been used for thousands of years for their preservative and medicinal properties and for enhancing the aroma and flavor of foods. *Hibiscus species* L. are among the aromatic plants widely used in Türkiye. Studies have shown that *Hibiscus species* L. have anti-inflammatory, antibacterial, antifertility, antifungal, antioxidative, antihypertensive, and hypoglycaemic effects. The use of aromatic plants for therapeutic purposes for centuries has made their compounds the research subject. Gas chromatography-mass spectroscopy (GC-MS) was combined with solid phase microextraction (SPME) to determine volatile organic compounds. Twenty-nine volatile organic compounds were determined from different parts of *H. syriacus* L. species. The compounds with the highest content are carvacrol, thymol, eucalyptol, and linalool. The compounds with the highest ratios determined in the flower, seeds, leaves, and buds of *Hibiscus syriacus* L. were carvacrol (57.93 %), carvacrol (31.50 %), thymol (44.53 %) and carvacrol (54.54 %), respectively.

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1. Introduction

Aromatic plants have been used for centuries for pharmaceutical, cosmetic, and perfumery purposes. Essential oils, medicines, herbal health products, dyes and colorants, personal care products, plant protection products, and intermediate components of these products are obtained from aromatic plants (Lubbe and Verpoorte, 2011; Tetik et al., 2013). Aromatic plants have been used for thousands of years for their preservative and medicinal properties and for enhancing the aroma and flavor of foods (Christaki et al., 2020). According to the World Health Organization, traditional medicines are used by more than 80 % of the world's population for primary health care (Balkrishna et al., 2022). Türkiye is one of the countries where aromatic plants are used most intensively with its rich plant diversity (Tetik et al., 2013). Hibiscus species are among the aromatic plants widely used in our country.

Hibiscus species, including those from Asia, the Middle East, Europe, and North America, are widely distributed. More than 300 species of the genus Hibiscus, *Hibiscus sabdariffa,* and *Hibiscus rosa-sinensis* are the main species investigated for pharmacological activities. Various parts of *Hibiscus species* L., including flowers, stem and stem bark, roots, leaves, and seeds, have been used in oriental medicine (Balkrishna et al., 2022; Yang et al., 2020). *H. syriacus* L. is known for its medicinal properties in traditional treatments. The biological structure of the tree classifies it as part of the Malvaceae plant family, and it is widely cultivated in more than 50 countries worldwide, including Europe and Asia. The color of the petals of *H. syriacus* L. cultivars varies between white, pink, red, and purple, and the shape of the flower is described as a single or double flower (Park et al., 2022).

Bioactive compounds such as flavonoids, anthocyanins, phenolic acids, lignans, coumarins, hydroxybiscone A, nonanoic acid, triterpene derivatives, and polysaccharides have been isolated from Hibiscus species. In the last 20 years, the antioxidant activities of anthocyanins obtained from Hibiscus flower pigments have been investigated (Yang et al., 2020). Recently, antiproliferative activity of the root bark of *H. syriacus* L. against cancer cells has been reported, and several triterpenoids have been isolated as active constituents. There are many published studies on the pharmacological activity of the root and bark (Park et al., 2022). In addition, *Hibiscus* species are known to contain a large number of secondary metabolites with biological activities such as anti-inflammatory, antibacterial, antifertility, antifungal, antioxidative, antihypertensive, and hypoglycaemic (Balkrishna et al., 2022). It is reported that *Hibiscus* species show antioxidant, antibacterial, diuretic, antipyretic, antihypertensive, anticholesterol, and antidiabetic properties depending on the bioactive substances they contain. Hibiscus is used in the food industry to prepare herbal tea, hot and cold drinks, jams, confectionery, ice cream, chocolate, flavorings, puddings, and cakes (Dinçer et al. 2020).

The use of aromatic plants for therapeutic purposes for centuries has made their compounds the research subject. Characterization of the volatile organic compound profile is an important tool for authenticity assessment, food quality, and determination of potential benefits of the compounds (Koyuncu, 2022). The study aimed to determine the volatile organic compound characteristics of steam extracts obtained from different parts of *Hibiscus syriacus* L..

2. Materials and Methods

2.1. Plant material

The species identification of *Hibiscus syriacus* L. plant used in the study was carried out by Prof. Dr. Hasan Akan from Harran University. The flowers, seeds, leaves, and buds of this plant were used for the study.

2.2. Essential oil extraction

30 g from each part of Hibiscus dried in shadow at air conditions and powdered by a blender added to 200 mL of distilled water (1/10: w v^{-1}) separately, and extraction was performed in a Neo-Clevenger then filtered. The filtrate was used for chromatographic

analysis. Karadağ et al. (2021)'s method was used with modifications.

2.3. Analysis of volatile organic compounds

Volatile organic compounds were analyzed using the SPME/GC-MS technique. 1.00 ml of plant extract was weighed into 30 ml vials (Supelco, USA), and 5 ml of ultrapure water was added. The vials were kept at 40 ºC for 30 min to allow the volatile components to equilibrate in the headspace. SPME fiber (Supelco Co., Bellefonte, PA, USA) was used for the adsorption of volatiles. Desorption of the extracted volatiles was carried out using the Thermo Fisher Trace ISQ GC-MS gas chromatography-mass spectrometry system. The device was operated in split (1:8 ratio) mode. During desorption, helium was used as carrier gas (at a flow rate of 1.0 mL min^{-1}), and the SPME fiber was kept in the injection port at 250 ºC for 2 min. Volatile compounds were separated using a DB-5MS column (30 m x 0.25 mm x 0.25 μm; Agilent, USA). The oven temperature was kept at 40 ºC for 1 minute, then increased to 120 ºC by heating at 5 ºC per minute and kept at this value for 2 minutes, then increased to 240 ºC by heating at 10 ºC per minute and kept at this temperature for three minutes. The mass spectrometer was set to scan between 45 and 450 amu (threshold 1000) at a sampling rate of 1.11 scans s^{-1} (Koyuncu, 2021). Volatile components were identified by comparison with MS libraries.

3. Results and Discussion

Volatile organic compounds are emitted as gases and contain different structures of chemicals with health effects (EPA, 2017). Essential oils from plants have been reported to have broad antimicrobial activity against various bacterial and fungal pathogens (Zhang et al., 2017). Characterization of the volatile profile is essential for authenticity assessment and food quality (Oliveira-Alves et al., 2020). Volatile organic compounds such as ketones, terpene esters, and alcohols are volatile compounds obtained from medicinal plants. Some of these compounds are considered inhibitory agents of pathogens and are widely used for food preservation (Karadağ et al., 2021).

As a result of the chromatographic analyses (Figure 1), 29 volatile organic compounds were determined from *Hibiscus syriacus* L. plant (Table 1). Among the volatile organic compounds obtained from flowers, seeds, leaves, and buds of *H. syriacus* L., the highest ratios are carvacrol, thymol, eucalyptol, and linalool compounds, respectively. The compounds with the highest ratios determined in the flower part of *H. syriacus* L. were carvacrol (57.93 %), eucalyptol (12.09 %), and γ-terpinene (10.62 %). The compounds with the highest ratios determined from the seeds of *Hibiscus syriacus* L. were carvacrol (31.50 %), eucalyptol (21.40 %), and thymol (9.71 %). Among the volatiles determined from the leaves of this plant, 44.53 % thymol, 12.36 % eucalyptol, and 8.15 % linalool were the compounds with the highest ratios. In comparison, 54.54 % carvacrol, 10.54 % linalool, and 4.59 % eucalyptol were the compounds with the highest ratios from the buds of this plant.

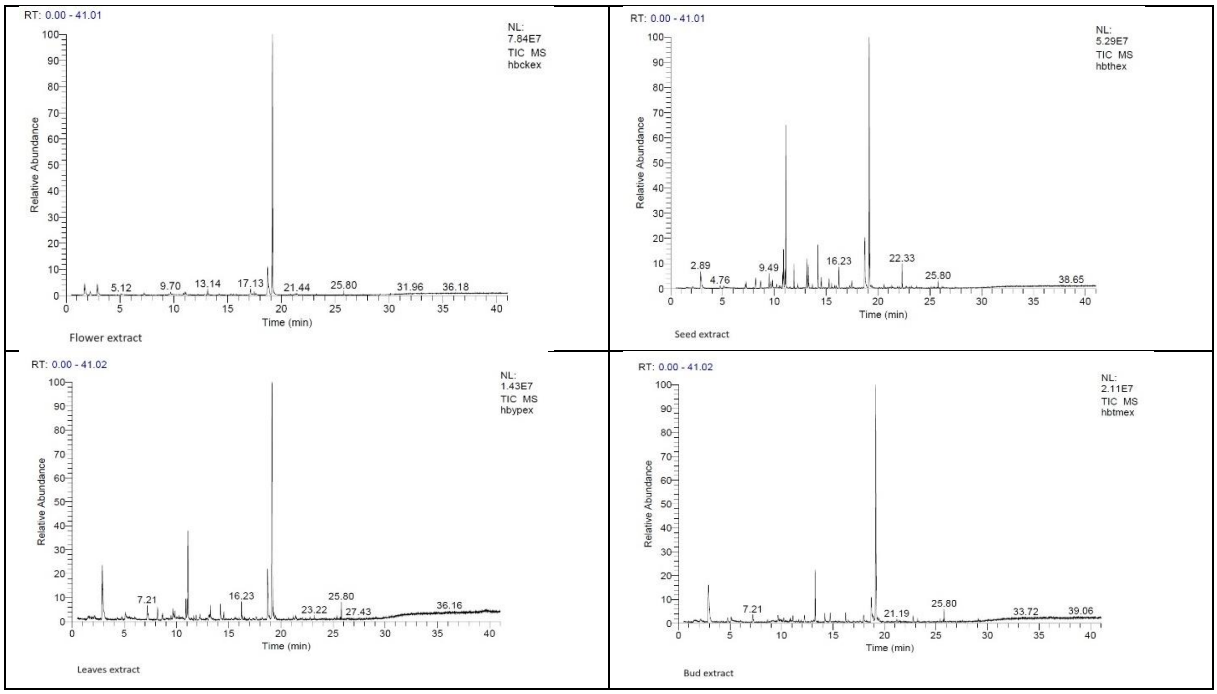

Figure 1. Chromatographs of different parts of *H. syriacus* L.

The bioactive properties of aromatic plants are determined by their ingredients. In vitro and in vivo studies show that carvacrol, which has the highest rate of volatile components in *Hibiscus syriacus* L., possesses a variety of biological and pharmacological properties including antioxidant, antibacterial, antifungal, anticancer, anti-inflammatory, hepatoprotective, spasmolytic, and vasorelaxant (Can Baser, 2008; Sharifi-Rad et al., 2018; Suntres et al., 2015). Carvacrol has been reported to be found in many aromatic plants like oregano, thyme, white thyme, summer savory, winter savory, pepperwort, and wild bergamot (Sharifi-Rad et al., 2018; Suntres et al., 2015). The second most abundant volatile organic compound in *H. syriacus* L. was thymol. Thymol has been observed its antioxidant, antispasmodic, antimicrobial, anti-inflammatory, and anticancer properties and is also identified for its antiseptic and antimicrobial effects (Memar et al., 2017). Carvacrol and thymol are isomers and are monoterpenic phenols and biosynthesized from γ-terpinene (Can Baser, 2008). In our study, we detected γ-terpinene in all parts of *H. syriacus L.*. To determined thymol and carvacrol as the highest volatile organic compounds in *Thymus vulgaris* in their study (Gavaric et al., 2015). The same survey found carvacrol to be the most volatile organic compound in the Origanum vulgare plant. Fachini-Queiroz et al. (2012) reported carvacrol, alpha-terpineol, and endo-berneol as the dominant volatile aromatic compounds in *Thymus vulgaris* L.

The compound with the third highest ratio among the volatile organic compounds detected in *H. syriacus* L. is eucalyptol. Eucalyptol displays therapeutic effects in respiratory illnesses. Several clinical trials have been conducted in people with respiratory diseases such as rhinosinusitis, bronchitis, asthma, and chronic obstructive pulmonary disease (COPD), with positive results (Galan et al., 2020; Boukhatem et al., 2020). reported a lavander species containing more than 60 % eucalyptol. In a study in which the *Lavandula angustifolia* plant was examined, eucalyptol (15.10 %) and linalool (11.98 %) were reported as the volatile components with the highest ratio (Karadağ et al., 2021). Linalool, the fourth one of volatiles, is a monoterpene alcohol that occurs naturally in many aromatic plants and is widely used in the flavor and fragrance industry. The biological activities of linalool can be listed as antimicrobial, anti-inflammatory, anticancer, antioxidant

properties, hepatoprotective, renal protective, and lung protective activity, and it has been confirmed that linalool has various effects on the central nervous system (An et al., 2021; Kamatou and Viljoen, 2008; Mitic-Culafic et al. 2009) reported that linalool and eucalyptol have a strong protective effect against oxidant-induced genotoxicity, mainly due to their radical scavenging activity.

Table 1. Volatile organic compounds of different parts of *H. syriacus* L.

			Flowers	**Seeds**	**Leaves**	**Buds**
RT	**Compound Name**	**Cas Number**	Area %			
1.73	2-Propanol	67-63-0	10.17	nd	nd	nd
2.13	Hexane	110-54-3	nd	0.80	nd	nd
4.76	Hexanal	66-25-1	0.58	0.35	0.92	0.94
6.00	2-Hexanal	505-57-7	nd	nd	2.20	0.17
7.28	Heptanal	111-71-7	0.49	1.22	0,56	0.19
8.19	α-Pinene	80-56-8	0.20	0.89	nd	nd
8.67	Camphene	79-92-5	nd	0.78	0,84	nd
9.49	2-α-Pinene	127-91-3	1.09	1.41	2.91	1.03
9.83	α-Myrcene	123-35-3	1.31	1.73	4.40	0.15
10.23	Octanal	124-13-0	nd	0.41	nd	0.29
10.65	α-Terpinene	99-86-5	2.08	0,43	nd	0.29
11.11	Eucalyptol	470-82-6	12.09	21.40	12.36	4.59
11.93	γ-Terpinene	99-85-4	10.62	2.12	1.43	1,64
12.26	Cis-Sabinen hydrate	15826-82-1	0.47	0.46	nd	0,63
13.15	Linalool	78-70-6	4.71	5.42	8.15	10.54
13.67	α-Thujone	546-80-5	nd	0.27	nd	nd
14.54	Camphor	76-22-2	nd	1.00	2.07	nd
15.29	Borneol	507-70-0	0.83	1.00	1.95	1.06
15.53	4-Terpineol	562-74-3	2.56	0.45	1.43	1,40
15.94	α-Terpineol	98-55-5	nd	0.55	1.12	nd
16.23	Decanal	112-31-2	nd	1.91	1.81	1.69
16.50	Chavicol	501-92-8	nd	nd	0.47	nd
17.48	Carvone	99490	nd	1.12	0.85	0.15
17.98	Nonanoic acid	112-05-0	nd	nd	nd	1.77
18.55	Thymol	89-83-8	nd	9.71	44.53	0.36
19.15	Carvacrol	499-75-2	57.93	31.50	nd	54.54
21.39	β-Damascenone	23726-93-4	nd	nd	0.76	nd
22.34	trans-Caryophylene	87-44-5	1.94	1.89	nd	0.25
23.03	α-Humulene	6753-98-6	nd	0.15	nd	nd

RT: Retention time**. nd:** not determined.

4. Conclusion

In our study, the characterization of volatile organic compounds in flowers, seeds, leaves, and buds of *H. syriacus* L. species, which are very important in human nutrition due to their bioactive properties, was carried out. The study was performed by chromatographic analysis method. Twenty-nine volatile organic compounds were identified from different

parts of *H. syriacus* L. Among these compounds, the highest ratios are carvacrol, thymol, eucalyptol, and linalool. In the studies on these compounds, their positive effects on human nutrition and health have been determined, so it has been understood that *H. syriacus* L, which contains these compounds, is a plant that should be included in human nutrition.

Declaration of Author Contributions

The authors declare that they have contributed equally to the article. All authors declare that they have seen/read and approved the final version of the article ready for publication.

Declaration of Conflicts of Interest

All authors declare that there is no conflict of interest related to this article.

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