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Original article

Diurnal Variations of Essential Oil Content and Composition of *Satureja cilicica* P.H. Davis under Culture Conditions

Hasan Maral *

Ermenek Vocational School, Karamanoğlu Mehmetbey University, Karaman, Turkey

Abstract

Satureja (Lamiaceae) species are aromatic plants used to produce essential oil and aromatic water in the Mediterranean region of Turkey, and *Satureja cilicica* P.H. Davis is an endemic species for Turkey. This study was conducted to determine the effects of diurnal variability on essential oil (EO) content and components of *Satureja cilicica* in the Ermenek district/Turkey in 2021. During the day, four different harvest times were considered as follows: 7:30 and 10:30 a.m., 01:30 and 4:30 p.m. at the flowering stage. The aerial parts of harvested plants were dried in the shade. The greatest and least EO contents of *S. cilicica* aerial parts were obtained at 4:30 p.m. (0.61%) and 1:30 p.m. (0.32%), respectively. The results of the analysis revealed that the major components of essential oils of *S. cilicica* are p-cymene (19.24-40.04%), carvacrol (16.42-29.59%), thymol (10.04-19.34%), γ-terpinene (4.92-12.94 %) and linalool (4-42-6.53%). In this study, it was determined that the best harvest time for optimum essential oil content and chemical properties was the cool hours of the day.

Keywords: Diurnal variations, Endemic, Essential oil, GC/MS, *Lamiaceae, Satureja cilicica.*

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* **Corresponding author:**

Hasan Maral, Ermenek Vocational School, Karamanoğlu Mehmetbey University, Karaman, Turkey.
Email: hasmaral@gmail.com

INTRODUCTION

The flora of Turkey hosts 11707 plant taxa among which 3649 are endemic (endemism rate 31.82%) (Güner et al., 2012; Şirin and Ertuğrul, 2015). Turkey is a rich genetic resource of the *Lamiaceae* family. While some of the plants belonging to the *Lamiaceae* family are used as folk remedies in the treatment of various diseases, some are also used in medicine, food industry, perfumery and cosmetics. In addition, most preparations used in phytotherapy are obtained from plants belonging to this family (Saleem, 2000). In Turkey, the plants belonging to the *Lamiaceae* family, whose essential oil main components are thymol and carvacrol, are known as thyme. These genera are *Origanum*, *Thymus*, *Thymbra*, *Satureja* and *Coridothymus* (Maral and Kirici, 2018). The common features of these genera are that the main components of their essential oils are usually carvacrol and/or thymol (Lukas et al., 2013). Some of these genera are collected naturally from nature, while others can be cultivated. Some of these genera exported as thyme are *Satureja* species. There are 15 species of *Satureja*, which has an important place among the thyme species traded in Turkey. The trade of *Satureja* species in Turkey is more common, especially in the Aegean and Mediterranean Regions. However, there is not enough information about the traded *Satureja* species, their distribution areas and the nature of their populations. In addition, the commercial capacities, export and production amounts of these plants on the basis of genus have not been clearly determined until now (Satýl et al., 2004).

The *Satureja* genus is widely used in the herbal beverage, spice, food additive and flavoring, perfume and cosmetics industries around the world. *Satureja* species are traditionally used in folk medicine to treat a variety of ailments such as cramps, muscle aches, nausea, indigestion, diarrhea, and infectious diseases. *Satureja* species are generally subjected to antibacterial, antifungal, antioxidant, cytotoxicity, antidiabetic, anti-HIV, anti-hyperlipidemic, reproductive stimulant, expectorant and vasodilator activity studies (Arabacý et al., 2017).

Satureja cilicica L. is an aromatic and endemic medicinal plant. The aerial material has a distinctive flavor and can be added as a seasoning to meat pies, sausages and some dairy products. Fresh twigs of *Satureja cilicica* can be boiled with legumes such as peas, beans and lentils as a flavoring or alternatively used as a garnish instead of parsley and chives (Ozkan et al., 2007). Medicinal and aromatic plants and their derivatives have long been used for culinary and medicinal purposes and are added to foods to prevent the formation of unwanted oxidation products. Aromatic herbs are not only distinctly pleasant in taste and aroma, but they can also be used to mask bad tastes and odors. Research on the antioxidant and antimicrobial properties of plants has generally focused on their essential oils in the last 15-20 years (Tainter and Grenis, 1993; Ozkan et al., 2007).

The biosynthesis of secondary metabolites, although controlled genetically, is strongly affected by environmental factors, agronomic practices, harvesting time, and post-harvesting management (Miguel et al., 2004; Zafar et al., 2017; Safa et al., 2019). In addition, several studies have shown that the concentration of secondary metabolites can change daily. Environmental conditions such as light (quality, intensity and duration), temperature, irrigation, altitude and edaphic factors greatly affect the amount and quality of secondary metabolites in plants (Ramezani et al. 2009; Safa et al., 2019). In addition, temperature changes during the day have a greater effect on essential oil accumulation. The variations depending on different day times and growth stages can affect the active substance and agronomic production of medical plants (Gumuscu et al., 2008; Safa et al., 2019).

In addition, essential oil production is not only dependent on plant genetics or developmental stage. Environmental changes can significantly affect biochemical pathways and physiological processes that alter plant metabolism and essential oil biosynthesis. For these reasons, essential oil content and composition are very variable according to seasonal and daily temperature (Maral and Kırıcı, 2019). Therefore, the study was carried out to determine the change of essential oil content and composition of *Satureja cilicica* plant during the day and the most suitable harvesting hours for essential oil production and oil quality.

MATERIALS and METHODS

Plant Material

The plant material was wild *Satureja cilicica* species collected from Ermenek district flora in 2014 (36° 69' 82" N, 32° 96' 69" E and 1515 m elevation). The obtained cuttings were planted in the Karamanoğlu Mehmetbey University, Ermenek Vocational School research farm (36° 37' 30" N, 32° 55' 11" E and 1303 m elevation). The essential oils used were obtained from 7-year-old plants. Plants described by Prof. Dr. Necattin Turkmen.

Methods

The plants in the trial area were harvested in 4 times in 2021 during the flowering period (18.07.2021) with 3 hour intervals (07:30 and 10:30 a.m., 01:30, 04:30 p.m.). The essential oil was obtained from the dried aerial parts. For isolation, 25 g of sample was subjected to hydro distillation with distilled water with a neo-Clevenger type apparatus for three hours in 2 repetitions. The oils were dried over anhydrous sodium sulfate and stored in dark bottles at 4°C.

A gas chromatography (GC) system (Agilent Technologies, 7890B) equipped with a flame ionization detector (FID) and connected to a mass spectrometer detector (MSD) (Agilent Technologies, 5977A) was used to identify the chemical components of EOs. The column for separation of the compounds was HP-Innowax (Agilent 19091N-116: 60 m × 0.320 mm inner diameter and 0.25 μm film thickness). The carrier gas was Helium (99.999%) with a flow rate of 1.3 mL min-1. The injection volume was set to 1 μl (20 μL EO dissolved in 1 mL n-Hexane). The solvent latency was 8.20 minutes. The injection was carried out in split mode (40:1). Samples were analyzed with the column initially kept at 70°C after being injected with a 5 minutes retention time. Then the temperature was raised to 160°C with a 3°C min-1 heating ramp and 5 min holding time. Eventually, the temperature reached 250°C with a 6°C min-1 heating ramp and 5 min hold time. Detector, injector, and ion source temperatures were 270°C, 250°C, and 230°C, respectively. MS scan range was (mz-1): 50-550 atomic mass units (AMU) under electron impact (EI) ionization of 70 eV (Maral et al., 2022).

RESULTS AND DISCUSSION

The essential oil contents obtained and the resulting major components are given in Table 1. The essential oil content of *S. cilicica* varied between 0.32% and 0.61% during the daytime. The highest essential oil content of 0.61% was obtained at 4:30 p.m., and the lowest content of 0.32% was obtained at 1:30 p.m. The essential oil contents of the plants harvested at 7:30 a.m. and 10:30 a.m. were 0.38% and 0.48%, respectively (Table 1). According to the results, essential oil production was higher during the cold hours of the day. Previous studies have also reported that early morning and afternoon are more suitable for essential oil harvesting (Kaya et al., 2013; Maral and Kırıcı, 2019).

High content of oxygenated monoterpene (41.98-67.47%) and monoterpene hydrocarbon (27.91-54.35%) were characterized in *S. cilicica* essential oil. In addition, sesquiterpene hydrocarbon (2.99-4.39%) was detected as the other minor chemical group. The highest oxygenated monoterpene ratio was obtained from the plants harvested at 07:30 a.m. In contrast, the lowest rate of this group was achieved at 01:30 p.m. The highest and lowest ratios of monoterpene hydrocarbons were obtained at 01:30 p.m. and 07:30 a.m., respectively. The amount of monoterpene hydrocarbons decreased with the increase of oxygenated monoterpenes (Table 1).

As a result of the research, sixteen components were determined in the essential oils of the plants harvested at 07:30 a.m., constituting 99.77% of the total oil. The main components of the essential oil of the plants harvested at 07:30 in the morning were determined as carvacrol (29.59%), thymol (19.34%), p-cymene (19.24%), linalool (6.53%), caryophyllene oxide (5.60%), γ-terpinene (4.92%) and genariol (3.12%). It was determined that there were 15 components in the essential oils of the plants harvested at 10:30, and they constituted 99.42% of the total components. The highest component was p-cymene with 40.04%, followed by carvacrol with 22.14%, thymol with 10.04%, γ-terpinene with 8.22% and linalool with 4.42%. The number of compounds identified in the essential oils of the plants harvested at 1:30 p.m. 15, representing 99.54% of the oil. The major components of the essential oil were determined as p-cymene (34.05%), carvacrol (16.42%), thymol (15.94%), γ-terpinene (12.94%) and linalool (4.98%). Eighteen components were identified, constituting 99.19% of the total oil, in the essential oils of the plants harvested at 4.30 p.m. The main components of the essential oil are p-cymene (23.80%), carvacrol (21.91%), thymol (17.97%), γ- terpinene (11.33%) and linalool (5.18%) were determined (Table 1).

Carvacrol and thymol ratios, which are the main components obtained in this study, reached higher levels in the first and last hours of the day when the air temperature was low. Because carvacrol and thymol levels were at minimum levels in the middle of the day. γ-terpinene and p-cimen were the other main compounds of *S. cilicica* essential oil. Interestingly, the amount of carvacrol and thymol was highest at the same time as the lowest levels of γ-terpinene and p-cymene. γ-terpinene and p-cymene are precursors of thymol and carvacrol. It has also been reported that there is a negative relationship between γ-terpinene and carvacrol. At high temperatures, phenolic compounds rise to the detriment of their predecessors. It can be explained by the effect of environmental factors, especially temperature and seasonal changes. These conditions accelerate the conversion of γ-terpinene and p-cymene components to phenolic components (Said et al., 2009; Maral and Kırıcı, 2019).

Tumen et al., (1993), reported that the main component of *S. cilicica* essential oil is 37.58% carvacrol. Schulz et al., (2005), reported that the main components of the essential oil of *S. cilicica* were found as thymol (22.76%), carvacrol (18.90%), p-cymene (19.52%) and c-terpinene (13.40%). Ozkan et al., (2007), reported the main components of *S. cilicica* essential oil as thymol (22.76%), carvacrol (18.90%), p-cymene (19.52%) and γ-terpinene (13.40%). Arabacı et al., (2017), reported the main components of *S. cilicica* essential oil as p-cymene (17.68%), carvacrol (14.02%), γ-terpinene (11.23%) and thymol (8.76%). Maral and Kırıcı, (2018), reported the main components of *S. cilicica* essential oil as carvacrol (21.56%), γ-terpinene (11.20%), cyclohexane (6.16%) and α-methylcyclopentanone (5.51%). In previous studies on satureja essential oil, it is seen that the main components of the essential oil generally consist of four main components (carvacrol, thymol, p-cymene and γ-terpinene). In this study, it was determined that the main components of the essential oil were carvacrol, thymol, p-cymene and γ-terpinene.

CONCLUSION

In previous studies, it has been reported that there are some qualitative and quantitative differences between the components of essential oil. These differences vary depending on different factors such as climatic, genetic factors, applied agricultural practices or plant chemotype. One of these factors investigated in this study on *S. cilicica* chemical properties is diurnal variability. The largest and least essential oil content of *S. cilicica* was obtained at 04:30 p.m. (0.61%) and 01:30 p.m. (0.32%). Carvacrol was found at the highest rate in plants harvested at 07:30, while p-cimen reached higher levels at other harvest hours (10:30 a.m., 01:30 and 04:30 p.m.). As a conclusion, in this study, it was determined that the best harvest time for optimum essential oil content and chemical properties was the cool hours of the day.

Table 1. Effects of diurnal variation on essential oil content and components of endemic *S. cilicica*

No	RT	RI	Components	07:30 a.m.	10:30 a.m.	01:30 p.m.	04:30 p.m.
1	3.38	1027	á-Pinene	0.85	0.96	1.18	1.20
2	4.73	1114	â-Pinene	1.22	0.64	1.14	1.12
3	5.98	1161	â-Myrcene	ns	ns	0.90	0.85
4	6.33	1184	á-Terpinene	1.44	1.01	1.59	1.46
5	6.81	1204	dl-Limonene	0.24	1.46	0.22	1.47
6	7.02	1214	1.8-Cineole	ns	ns	ns	1.48
7	7.90	1234	cis-Ocimene	ns	1.72	2.33	2.68
8	8.14	1249	ã-Terpinene	4.92	8.22	12.94	11.33
9	8.92	1275	ñ-Cymene	19.24	40.04	34.05	23.80
10	19.01	1545	Linalool	6.53	4.42	4.98	5.18
11	20.26	1548	trans-Sabinene hydrate	0.17	0.32	ns	ns
12	20.79	1602	Caryophyllene	2.32	2.99	1.87	1.90
13	24.37	1701	Borneol	1.31	1.48	ns	1.66
14	25.26	1727	â-Bisabolene	2.07	ns	1.55	1.30
15	29.80	1840	Geraniol	3.12	ns	1.71	1.07
16	33.63	1980	Caryophyllene oxide	5.60	2.77	2.73	2.19
17	38.42	2125	spathulenol	1.81	1.21	ns	0.64
18	40.82	2181	Thymol	19.34	10.04	15.94	17.97
19	41.58	2210	Carvacrol	29.59	22.14	16.42	21.91
Chemical grouped compounds (%)							
Oxygenated monoterpenes				67.47	42.38	41.98	50.62
Monoterpene hydrocarbons				27.91	54.05	54.35	45.37
Sesquiterpene hydrocarbon				4.39	2.99	3.42	3.20
Total (%)				99.72	99.42	99.75	99.19
Number of identified compounds				16	15	15	18
Essential oil content (%)				0.38	0.48	0.32	0.61

RT: Retention time; RI: Retention indices calculated against n-alkanes (C7-C30) on HP-Innowax column

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