KSÜ Tarım ve Doğa Derg 22(6): 972-975, 2019
KSU J. Agric Nat 22(6): 972-975, 2019
DOI:10.18016/ksutarimdoga.vi.517345

An Investigation on the Determination of Diurnal and Ontogenetic Variations of Essential Oil Composition in Sideritis trojana Growing in Kazdağı (Edremit-Balıkesir)

Cenk PAŞA[1], Selami SELVİ[2], Züleyha ÖZER[3], Turgut KILIÇ[4]

[1]Balıkesir University, Altinoluk Vocational School, Department of Plant and Animal Production, Medicinal and Aromatical Plants Programme, Altinoluk, Edremit, Balikesir, [2]Balikesir University, Faculty of Necatibey Education, Balikesir, Turkey
[1]https://orcid.org/0000-0002-6125-9767, [2]https://orcid.org/0000-0002-9959-6945, [3]https://orcid.org/0000-0003-4957-5756,
[4]https://orcid.org/0000-0002-6842-3160
✉: cpasa@balikesir.edu.tr

ABSTRACT

This study was conducted to determine the essential oil composition and diurnal variations (9.00 am, 12.00 am and 16.00 pm) of *Sideritis trojana* Bornm. which was distributed naturally at the peak of Kazdağı (Edremit-Balıkesir), west of Turkey during the growing season. The oil composition was determined with GC-MS. The highest quantity of essential oil content (0.25-0.30%) was found at the full plant flowering stage and the lowest was at fresh fruiting stage (0.07-0.1%). Diurnal fluctuation in essential oils of whole plant was also observed. The highest and lowest essential oil levels were observed at the afternoon (16.00 pm) and in the morning (08.00 am), respectively. The oils were consisted of mainly α-bisabolol (27.8%), valeranone (13.4%), 4-terpineol (10.3%), germacrene-D (8.8%) and spathulenol (5.8%) during vegetative stage, 4-terpineol (30.3%), caryophyllene (21.0%) and 3-methyl nonane (9.3%) during flowering stage, 4-terpineol (16.1%), copaene (15.4%), caryophyllene (10.6%), 3-methyl nonane (9.8%) and valeranone (7.9%) during fresh fruiting stage.

Short Communication

Article History
Received : 24.01.2019
Accepted : 18.04.2019

Keywords
Volatile oil components,
Sideritis trojana,
GC-MS,
Edremit,
Balıkesir

Kazdağları'nda (Edremit-Balıkesir) yayılış gösteren *Sideritis trojana* türünün Uçucu Yağ Oranı ve Bileşenlerinin Diurnal ve Ontogenetik Varyasyonunun Belirlenmesi Üzerine Bir Araştırma

ÖZET

Bu araştırmada, Kazdağları'nda (Edremit-Balıkesir) yayılış gösteren *Sideritis trojana* Bornm türünün uçucu yağ oranı ve bileşenlerinin diurnal ve ontogenetik varyasyonunun belirlenmesi amaçlanmıştır. Uçucu yağ bileşenleri GC-MS analizi ile yapılmıştır. En yüksek uçucu yağ oranı çiçeklenme döneminde (%0.25-30), en düşük uçucu yağ oranı ise meyve döneminde %0.07-0.10) tespit edilmiştir.
Uçucu yağ bileşenleri vejetatif dönemde esas olarak; α-bisabolol (%27.8), valeranone (%13.4), 4-terpineol (% 0.3), germacrene-D (%8.8) ve spathulenol (%5.8); çiçeklenme döneminde ise 4-terpineol (% 30.3), caryophyllene (%21.0) ve 3-methyl nonane (9.3%); meyve oluşumu döneminde 4-terpineol (%16.1), copaene (% 15.4), caryophyllene (%10.6), 3-methyl nonane (%9.8) ve valeranone (%7.9) olarak belirlenmiştir.

Kısa Not

Makale Tarihçesi
Geliş Tarihi : 24.01.2019
Kabul Tarihi : 18.04.2019

Anahtar Kelimeler
Uçucu yağ bileşenleri,
Sideritis trojana,
GC-MS,
Edremit,
Balıkesir

To Cite : Paşa C, Selvi S, Özer Z, Kılıç T 2019. An Investigation on the Determination of Diurnal and Ontogenetic Variations of Essential Oil Composition in Sideritis trojana Growing in Kazdağı (Edremit-Balıkesir). KSU J. Agric Nat 22(6): 972-975. DOI: 10.18016/ksutarimdoga.vi.517345.

INTRODUCTION

A derivate of the Greek word "Sideron" was used to name the genus *Sideritis*, also known as ironwort, shepherd's tea and mountain tea is a genus of flowering plants widely known for their use in herbal medicine as well as an herbal tea (Piozzi et al, 2006).

Sideritis L., which is one of the important genera of the Lamiaceae family, has species that are of medical and economic value (Davis, 1982; Çarıkçı et al, 2012). More than 150 taxa of the genus *Sideritis* are found mainly in the Mediterranean region (Obon and Nunez 1994). *Sideritis* genus are represented by 46 species (53 taxa) in Turkey and 39 of these taxa are endemic (endemism ration is about 80%) for our country

(Aytaç and Aksoy, 2000; Güvenç and Duman, 2010). *Sideritis* species are generally known namely "adacayi, dagcayi" and are widely used as herbal tea and folk medicine in Turkey (Kirimier et al, 2004). *Sideritis* species are also widely used in the treatment of some diseases such as cough, colds, gastrointestinal disorders, antimicrobial and anti-rheumatic (Baytop 1999; Topcu and Gören, 2007; Kilic et al, 2009; Pasa and Selvi, 2011; Selvi et al. 2012; Polat et al. 2012).

The purpose of the study was to determine the essential oil components content and ontogenetic variations of *Sideritis trojana* Bornm., cultivated in Mount Ida, Edremit-Balikesir, Turkey during a growing season.

MATERIALS and METHODS

Object of the Study

The object of this study was to determine the essential oil components content and ontogenetic variations of *S. trojana,* which was collected at different developing stages from Edremit district of Balikesir province, Turkey between April and August of 2014. The soil of the trial area was sandy with pH value (6.9), organic matter (6.8%), sand (68%), silt (24 %) and clay (8%). The trial location sustained a mean temperature of 22.1 ºC, mean rainfall of 27.4 mm and relative humidity of 61.2 % during the year of 2014. Samples were collected three times a day (9.00 am; 12.00 am and 16.00 pm) for each development stage.

Sampling and sample preparation

Ontogenetic sampling corresponded with different date for *S. trojana* shoots with leaves were harvested at the vegetative stage. At the full flowering stage, only shoots with fully opened flowers and at the fresh fruiting stage, the shoots with green capsules were harvested. The plant materials were dried at room temperature (20 ºC) and 50 g of each sample was subjected to hydro distillation for 6 h using a Clevenger type apparatus for determining the oil content.

Oil Composition and Analyses

The oil composition was determined with GC-MS. GC-MS analyses were conducted in the TUBITAK (MAM). GC-MS conditions; helium was used as carrier gas at a constant flow rate of 1 mL/min. 1µL of sample was injected. The GC temperature program was set as follows; 50 ºC hold for 5 min, ramp to 250 ºC at 5 ºC/min and hold for 10 min. The temperature of the MS transfer line was set at 220 ºC. Using scan mode a mass range from 50 to 650 *m/z*. Used column, DB-5 30 m x 0.25 mmID x0.25 µm. The Thermo Scientific TSQ GC-MS/MS was used in this study.

Statistic

Data was subjected to Analysis of Variance using MSTAT software program. Means were separated by the Duncan Multiple Range Test.

RESULTS and DISCUSSION

Total essential oils content in *S. trojana* during ontogenetic development was the highest in the full flowering stage (0.25-0.30%), followed by vegetative (0.23-0.27%) and fresh fruiting stage (0.07-0.10%) (Table 1). The differences among essential oils values during the different developmental stages were significant (P<0.01). One-way tendency of change in the essential oils content during the different hours of the day indicated that the oils values were higher in the afternoon (16.00 pm)- 0.10-0.30 % compared with at noon (12.00 am)-0.07-0.26 and in the morning (08.00 am)-0.08-0.25% in all developmental stages.

Table 1. Total essential oils content (%) and changes during collecting and development stages of *S. trojana*

Diurnal Collecting Times	Developmental Stages			
	Vej.Stage	Full Flow.	Fresh Fruit.	Mean
09:00 am	0.24 cd	0.25 c	0.08 f	0.19 b
12:00 am	0.23 d	0.26 bc	**0.07 g**	0.19 b
16:00 pm	0.27 b	**0.30 a**	0.10 e	**0.22 a**
Mean	0.25 b	**0.27 a**	0.08 c	0.20

There is no statistical (p<0.01) differences between values with the same letters in the same columns.

Several studies on ontogenetic variation of secondary metabolites were conducted in different plants. Alkaloid changes during fruit development were observed by Miriam and Pfeifer (1959) in *Papaver somniferum* and by Fairbairn and Challen (1959) in *Conium maculatum.*

Schwob et al., (2004) investigated essential oil changes during the course of ontogenesis in *Hypericum perforatum.* In another study, Gupta et al. (2002) discussed the changes of artemisinin during phonological cycle of *Artemisia annua* and foliar monoterpenoid variation in *Umbellularia californica* in seedlings and tree stages. Chemical concentrations varied considerably during the course of ontogenesis in a medicinal plant. Not only the concentrations of plant chemicals fluctuated through the season, but also the plant experienced short-lived and rapid turnovers (Smith et al., 1996).

The identity, the retention time and percent composition of the essential oils content from *S. trojana* is presented Table 2.

Table 2. Variation of volatile oils content of *S. trojana* within a one day during the course of ontogenetic (%)

			Vegetative stages			Full flowering			Fresh fruiting		
KI*	RT**	Compounds	09.00 am	12.00 am	16:00 pm	09:00 am	12:00 am	16:00 pm	09.00 am	12.00 am	16.00 pm
930	5.25	α-Thujene	0.4	0.2	0.7	1.1	0.7	0.7	0.3	0.2	0.2
939	5.44	α-Pinene	1.8	2.3	1.7	1.4	2.8	1.8	4.2	4.7	5.1
971	6.87	3-Methyl nonane	3.2	3.7	4.3	9.3	9.4	8.7	9.8	9.2	9.4
979	7.16	1-Octen-3-ol	-	-	0.1	0.7	0.2	0.5	-	-	-
979	7.55	ß-Pinene	-	0.1	0.3	0.9	0.4	0.8	0.7	0.5	0.6
1003	7.90	α-Phallandrene	-	0.1	0.1	0.6	0.5	0.6	0.9	1.1	1.2
1031	8.11	Carene-3-delta	0.1	0.1	0.2	0.9	0.6	1.1	1.2	1.4	1.8
1017	8.33	Terpinene	0.1	0.3	0.5	1.4	1.3	0.8	0.8	0.7	0.9
1025	8.60	Cymene	-	0.3	0.1	0.9	0.8	1.2	1.3	1.5	1.3
1029	8.75	Limonene	1.6	2.4	2.4	1.3	1.8	1.9	3.1	2.2	3.3
1037	9.20	ß-Ocimene	0.7	0.8	1.2	0.9	0.8	0.8	0.9	0.9	1.0
1060	9.82	τ-Terpinene	0.4	0.6	0.7	1.1	1.3	1.8	1.2	1.0	1.0
1086	10.39	Nononal	0.2	0.2	0.3	0.4	0.2	0.3	0.4	0.2	0.2
1089	10.81	Terpinolene	0.4	0.3	0.3	0.5	0.3	0.3	0.4	0.2	0.2
1100	11.28	Undecane	0.2	0.4	0.4	0.8	0.9	0.6	0.4	0.2	0.3
1157	11.38	Nonen-1-ol	0.6	0.7	0.7	0.9	1.0	0.7	0.7	0.9	0.7
1177	13.51	4-Terpineol	9.4	10.3	10.2	30.3	25.4	25.1	16.1	14.3	12.4
1189	13.89	α-Terpineol	0.5	0.4	0.5	0.4	0.5	0.7	0.5	0.3	0.6
1243	15.42	Carvone	0.5	0.4	0.4	1.0	1.2	1.4	0.5	0.2	0.2
1377	19.10	Copaene	1.7	1.3	1.9	0.9	0.8	1.1	12.2	11.4	15.4
1388	19.27	ß-Bourbene	0.1	0.2	0.5	0.2	0.4	0.3	0.7	0.5	0.6
1391	19.60	ß-Elemene	-	0.1	0.2	0.3	0.5	0.4	0.4	0.2	0.2
1409	20.10	Caryophyllene	3.6	5.1	4.2	21.0	17.1	16.7	10.6	9.7	9.5
1455	21.00	α-Humulene	0.1	0.1	0.3	0.2	0.5	0.4	0.4	0.4	0.3
1447	21.22	Aromadendrene	0.3	0.1	0.6	0.5	0.1	0.5	0.4	0.4	0.3
1462	21.56	1.8-cineole	4.3	3.9	4.2	6.8	8.2	7.6	3.4	4.9	4.7
1485	21.70	Germacrene-D	8.8	6.4	7.1	4.2	5.4	6.9	4.7	4.2	4.8
1480	22.00	τ-Muurolene	2.8	3.3	2.9	0.7	1.4	1.4	4.0	3.5	3.8
1514	22.52	y-Gamma-cadinene	0.8	1.2	0.8	0.9	1.5	1.1	0.4	0.5	0.6
1539	22.69	α-Cadinene	0.4	0.7	0.5	0.4	0.8	0.8	0.6	0.4	0.7
1572	23.90	Spathulenol	5.6	5.4	5.8	1.0	1.5	1.1	0.7	0.6	0.4
1583	24.03	Caryohyllene oxide	1.4	1.9	2.4	0.3	0.9	0.7	0.6	1.0	1.1
1593	24.22	Viridifloral	-	0.2	0.2	0.2	0.3	0.3	0.2	0.3	0.3
1636	25.10	σ-Cadinol	1.1	0.7	1.2	0.5	0.6	0.6	1.3	1.6	1.2
1660	25.61	α-Cadinol	3.1	3.8	3.6	2.3	3.3	2.7	2.9	3.1	2.8
1675	26.02	Valeranone	13.4	11.8	10.7	1.7	2.1	2.2	6.1	7.9	7.3
1686	26.30	α-Bisabolol	27.8	22.4	21.9	1.5	2.4	2.0	2.4	2.9	2.6
1943	29.70	Phytol	1.7	2.2	1.5	0.5	0.5	0.7	1.4	1.6	1.7

*KI: Kovats Retention Index; **RT: Retention Time

During vegetative stage, the oils consisted mainly of α-bisabolol (27.8 %), valeranone (13.4 %), 4-terpineol (10.3 %), germacrene-D (8.8 %) and spathulenol (5.8 %). During flowering stage the oils consisted mainly of 4-terpineol (30.3 %), caryophyllene (21.0 %) and 3-methyl nonane (9.3 %). During fresh fruiting stage flowering stage, the oils consisted mainly of 4-terpineol (16.1 %), copaene (15.4 %), caryophyllene (10.6 %), 3-methyl nonane (9.8 %) and valeranone (7.9 %).

The major constituents of the oil were ß-pinene (18.4 %), α-pinene (13.2 %), germacrene-D (5.3 %), ß-caryophyllene (4.9 %), limonene (3.7 %), and *p*-cymene (3.4 %) (Kirimer et al, 2008).

The effects of the diurnal variation on the essential oil's composition of *S. trojana* have not been reported previously. Nevertheless, differences in the essentials composition of developmental stages have been described for the closely related species of *S. trojana* (Kirimer et al, 2004).

CONCLUSIONS

In this study, diurnal and ontogenetic variability of essential oil and components of S. trojana has been determined. The highest essential oil content of the plant has been identified during full plant flowering, before flowering and fresh fruit stages, respectively.

As a result of this study, 38 different essential oil components have been detected in three different development stages of the plant. The main components of the essential oil are α-bisabolol (27.8%) and valeranone (13.4%) during vegetative stage; caryohyllene (21.0%) and 4-terpineol (30.3%) during flowering stage; 4-terpineol (16.1%) and copaene (15.4 %) during fresh fruiting stage.

In addition to the above, the most suitable collection time and collection period has been obtained in terms of the essential oil ratios and components of the investigated species and the most effective utilization method of the species was determined in this study. Furthermore, we believe that S. trojana will contribute to various upcoming scientific studies, especially in phytochemical and pharmaceutical arena.

REFERENCES

Aytaç Z, Aksoy A 2000. A new Sideritis species (Labiatae) from Turkey. Flora Mediterranea, 10: 181-184.

Baytop T 1999. Therapy with medicinal plants in Turkey (Past and Present). Nobel Medicine Publications, 2nd edition, Istanbul, 118-119.

Çarıkçı S, Kılıç T, Azizoğlu A, Topçu G 2012. Chemical Constituents of Two Endemic *Sideritis* Species from Turkey with Antioxidant Activity. Rec. Nat. Prod., 6(2): 101-109.

Davis PH 1982. Flora of Turkey and the East Aegean Island, Edinburgh University Press. 7: 178-199.

Fairbairn JW, Challen SB 1959. Alkaloids of Hemlock (*Conium maculatum* L.) distribution in relation to the development of the fruit. Biochem, 72: 556-562.

Gupta SK, Singh P, Bajpai P, Ram G, Singh D, Gupta MN, Gupta DJ, Jain SP, Khanuja SP, Kumar S 2002. Morphogenetic variation for artemisinin and volatile oil in *Artemisia annua*. Ind. Crop Prod., 16: 217-224.

Güvenç A, Duman H 2010. Morephological and anatomical studies of annual taxa of *Sideritis* L. (Lamiaceae), with notes on chorology in Turkey. Turk J. Bot., 34: 83-104.

Kılıç T, Çarıkçı S, Topcu G, Aslan I, Gören AC 2009. Diterpenoids from *Sideritis* condensate evaluation of chemotaxonomy of *Sideritis* species and insecticidal activity. Chem. Nat. Comp., 45: 918-920.

Kırımer N, Baser KHC, Demirci B, Duman H 2004. Essential oils of *Sideritis* species of Turkey Belonging to the section Empedoclia. Chemistry of Natural Compunds, 40(1): 19-23.

Kırımer N, Demirci B, İşcan G, Baser KHC, Duman H 2008. Composition of The Essential Oils of Two Sideritis Species From Turkey and Antimicrobial Activity. Chemistry of Natural Compounds, 44(1): 121-123.

Miriam R, Pfeifer S 1994. Über die Veranderungen im Alkaloidhaushalt der Mohnpfanze warend einer Vegatationsperiode. Sci Pharm., 27: 34-40.

Obon de Castro C, Rivera Nunez D 1994. A taxonomic revision of the section Sideritis. (Berlin-Stuttgart, Ed. Cramer, J) 86.

Paşa C, Selvi S 2011. Kazdağları'nda (Balıkesir-Edremit) yöre halkı tarafından toplanan ve ticareti yapılan tıbbi bitkiler üzerine bir araştırma, IX. Tarla Bitkileri Kongresi, 12-15 Eylül 2011, Bursa, s:1409-1412.

Piozzi F, Bruno M, Rosselli S, Maggio 2006. The diterpenoids from the genus Sideritis. S.Nat. Prod. Chem., 33: 493-540.

Polat R, Selvi S, Çakılcıoğlu U, Açar M 2012. Investigations of ethnobotanical aspect of wild plants sold in Bingöl (Turkey) local markets, Biological Diversity and Conservation, 5(3):155-161.

Selvi S, Satıl F, Polat R, Çakılcıoğlu, U 2012. Kazdağlarından (Balıkesir-Edremit) Toplanan Ve Bölgedeki Aktarlarda Satılan Tıbbi Bitkiler Üzerine Bir Araştırma, Kazdağları III. Ulusal Sempozyumu, 24-26 Mayıs 2012, Edremit Balıkesir, s: 505-513.

Schwob L, Bessiere JM, Masotti V, Viano J 2004. Changes in essential oil composition in Saint John's wort (*Hypericum perforatum* L.) aerial parts during its phonological cycle. Biochem.Syst.Ecol, 32: 735-745.

Smith R, Caswell D, Carriere A, Zielke B 1996. Variation in the ginsenoside content of American gingseng, Panax quinquefolius L. roots. Can. J. Bot., 74: 1616-1620.

Topçu G, Gören AC 2007. Biological activity of diterpenoids isolated from Anatolian Lamiaceae Plants. Rec.Nat.Prod., 1: 1-16.