



Essential oil composition of endemic plant *Sideritis* rubriflora Hub. Mor

Hatice Ersöz¹, Ömer Kılıç^{1*}, Erkan Yılmaz¹, Miray Ege¹, Şinasi Yıldırımlı²

¹Adıyaman University, Pharmacy Faculty, Department of Pharmacy Professional Sciences Adıyaman, Türkiye ²Hacettepe University, Science Faculty, Biology Department, Ankara, Türkiye

*Correspondence to

Ömer Kılıç, Email: okilic@ adiyaman.edu.tr

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Abstract

Background: The genus *Sideritis* L.is mostly found in habitats dominated by the Mediterranean climate and is represented by approximately 150 taxa in the world. Turkey is among few countries where *Sideritis* includes about 52 species and the endemic rate of this genus goes up to almost 80%. *Sideritis* species are generally known as 'sage or ironwood worth' or as a tea plant is widely used in folk medicine in Turkey. There are important studies in the literature on *Sideritis* species due to their widespread use in ethnobotanical and related uses.

Materials and Methods: Plant samples were collected from their natural habitats, dried following the herbarium technique, and then identified and stored at the Department of Pharmaceutical Botany, Faculty of Pharmacy, Adıyaman University with the number ÖK 6854. The essential oil was obtained from 100 g of plant samples by the water distillation method. Essential oil composition was determined by gas chromatography—mass spectrometry (GC-MS).

Results: It was found that the studied plant is rich in essential oil with 0.4 ml oil content in the sample. Forty components were determined from the essential oil of the plant and the main components were found as carvacrol (33.26%), β -caryophyllene (12.5%), α -pinene (7.32%), and β -pinene (6.13%). The results were discussed in terms of natural products, renewable resources, usage areas, and culture of Sideritis taxa.

Conclusions: With this study, the economic values of the members of the genus were emphasized by referring to the characteristics of *Sideritis* members such as aromatherapeutic, phytotherapy, ethnobotanical, and biological activity. In order to better evaluate the *S. rubriflora* plant and to continue the activities related to this endemic plant of high economic value more efficiently, basic data were obtained and it was tried to contribute to the sector. It is important to take the necessary precautions and show the necessary sensitivity in order to protect and conserve *Sideritis* members, especially *S. rubriflora*. In this way, *Sideritis* members and *S. rubriflora* will have the opportunity to continue their existence and spread more in nature. This will easily make available sufficient raw materials for the sectors they are used.

Keywords: Sideritis, Sideritis rubriflora, Lamiaceae, Essential oil



Background

The genus *Sideritis* L. belongs to the family Lamiaceae (Syn. Labiatae). This family is one of the richest families in medicinal, aromatic, scented, and essential oil plants Although medicinal and aromatic plants are used as a source of tea, spice, folk medicine, and essential oil, they are also widely used in other related sectors. Members of the genus *Sideritis* are also among the plants with the high economic value used for these purposes. The occurrence of these economically valuable plants in their habitats is one of the important issues in order to make an economic contribution to the relevant sector and to reduce our foreign dependency. In addition, in order to use medicinal plants in appropriate, effective, and sufficient quantities, the amount and types of active substances in their chemical content, their properties,

and mechanisms of action must be determined. For this reason, it is important to determine the chemical contents of the economically valuable plants found in their natural habitat in our country and which are frequently used by the public and to investigate which mechanism and which properties they exhibit in order to ensure more accurate use of these plants. The emergence of many side effects of foodstuffs and artificial medicines containing chemical additives has increased the interest in natural products. It is known that Turkey and East Anatolian Region are rich in medicinal and aromatic plant species that can be used for these purposes. Species of genus Sideritis are more common especially in the Mediterranean basin and their spread is cosmopolitan. Turkey is a rich country in terms of Sideritis species, and high endemism ratio, so we can say that Turkey is the gene center of Sideritis genus. Sideritis is



derived from the word «Sideron-sword wound healing» in ancient Greek, and species of this genus are known to have antirheumatic, pain reliever, gastric and antimicrobial effects.² Although members of the genus are widely used as herbal tea in Anatolia, especially in the Aegean and Mediterranean regions due to these and similar effects, *Sideritis* members are frequently encountered in ethnobotanical studies related to their usage areas.³ The fact that there are many studies in the literature about the chemical contents of *Sideritis* taxa, the amount and species of active ingredients, their properties, and mechanisms of action are among the indicators that the usage areas and economic values of *Sideritis* species are high.^{4,5}

Sideritis species, which are widely used in ethnobotany as folk medicine and herbal tea in Turkey, are mostly referred to by different local names such as "dağ çayı, yayla çayı, sarıkız çayı, kuyruk çayı, adaçayı."6,7 The members of the Sideritis genus are widely used in the treatment of colds and gastrointestinal diseases due to their nervous system stimulant, anti-inflammatory, antispasmodic, carminative, analgesic, sedative, antitussive, stomachic, and anti-convulsant effects. Sideritis species are also widely used in aromatherapy due to their high content of fragrant oils and widely-used aromatic substances. For this reason, members of the Sideritis genus are of high economic value in terms of aromatherapy and phytotherapy. In recent years, it has been determined that some Sideritis extracts have antifeedant, antistress, analgesic, antioxidant, antibacterial, and anti-inflammatory effects.8 Sideritis rubriflora, which is an endemic and rare species, is an herbaceous perennial. It is a plant that flowers in 4-6 months and is seen mostly in the Eastern Mediterranean and South-eastern Anatolia region of Turkey. S. rubriflora especially like rocky, stony slopes and maquis habitats. S. rubriflora, is used as a pleasure tea for various purposes, especially in the treatment of flu and colds in Turkey. In the Aydıncık district of Mersin, S. rubriflora known as sage or Kazıklı tea is used in the form of an infusion, mouthwash, and pleasure tea. It has been determined that it is used in cold, flu, pharyngitis, and bronchitis by drinking a tea glass of mouthwash 3 times a day, and also a tea glass 2-3 times a day for 2-3 weeks.9

This study, it was aimed to contribute to the relevant literature by determining the qualitative and quantitative composition of the essential oil of *S. rubriflora* plant, which is an endemic and a rare species and there are very few studies in the literature.

Materials and Methods *Plant sample*

Plant sample was collected from Adıyaman (Çelikhan), southwest of Akdağ, upper parts of the forest open areas in July 2019. Plant sample was dried in accordance with the herbarium technique, and then identified and stored at the Department of Pharmaceutical Botany, Faculty of

Pharmacy, Adiyaman University with the number ÖK 6854.

Essential oil

Essential oil was obtained from 100 grams of plant samples by the water distillation method. The plant material was cut into small pieces and brought to suitable sizes and placed in the distillation container with water. Clevenger apparatus was used for this distillation process and the water was heated to the boiling point. With the boiling of water, essential oils evaporated with water vapor and condensed on the cooling surface, and were collected in the separation vessel. Thus, essential oil was obtained (Figure 1).

Analysis of the Essential Oil

Essential oil composition was determined by gas chromatography–mass spectrometry (GC-MS). The essential oil extraction and GC-MS analyzes of the plant were carried out in the Harran University (Şanlıurfa) Central Research Laboratory. Used devices: Shimadzu Gaz kromatografi Kütle Spektroskopisi (GCMSQP2010). Injection block: RTX*-5MS, Restek (30 m \times 0.25 mm \times 0.25 um), SPL. Types of equipment: AOC-20i Plus, AOC-20s autosampler. Helium gas was used as a carrier under the operating conditions of the device. The characterization of components in essential oils has been done using electronic libraries. Injection capacity: 1 μ L. Split: 1/20. Injection block temperature: 250°C. Detector temperature: 220°C. Interface: 250°C. Analysis results are shown in Table 1.

Results and Discussion

An aliquot of 0.4 mL of light yellow essential oil was obtained from 100 g of the plant by the method of water distillation. In all, 40 components were determined by analyzing the essential oil obtained by GC-MS. Major components of the essential oil of *S. rubriflora*



Figure 1. Stages of Obtaining Essential Oil by Using the Clevenger Apparatus Distillation Method.

Table 1. Essential Oil Composition of Sideritis rubriflora

Compounds	% Amount	RRI
α-Phellandrene	0.37	11.563
β-Phellandrene	0.37	11.660
α-Pinene	7.32	11.853
β-Pinene	6.13	13.890
Myrcene	0.42	14.757
Fenol <3-isopropil->	0.19	16.023
Cymene <para-></para->	0.01	17.324
Cis-ocimene	1.71	17.549
Limonene	0.81	17.895
Terpinolene	2.47	17.990
1,8-Cineole	1.93	18.522
Terpinene <gamma-></gamma->	0.50	20.320
Dimetilstiren <alfa-para-></alfa-para->	0.36	20.735
Linalool	1.72	21.287
Terpineol <gamma-></gamma->	0.57	22.203
Terpinen-4-ol	0.42	24.445
Sabinene hydrate <cis-></cis->	3.17	26.197
İzoöjenol fenilacetate	3.26	27.987
Carvacrol	33.26	34.187
Himaşelen <alfa-></alfa->	8.23	37.177
Butirat <2-metil-, feniletil->	0.66	39.120
Bisiklogermacrene	0.19	41.102
β-Caryophyllene	12.15	41.737
Copaene	4.13	43.371
Ligustral	0.53	44.050
Seychellene	0.71	44.833
Germacrene D	1.22	46.333
Chamigrene <beta-></beta->	0.20	47.517
Naphtalene	0.18	49.615
Fitalat <dietil-></dietil->	0.18	53.179
α-Cadinol	0.68	54.151
Palmitat <metil-></metil->	0.74	55.132
Pentacosane	0.38	56.403
Aromadendrene	0.84	57.820
Ionone <alfa-< td=""><td>1.56</td><td>59.861</td></alfa-<>	1.56	59.861
Cedrol	1.31	60.659
Valerenol	1.45	62.192
Geranyl nitrile	0.35	64.055
Viridiflorol	0.34	65.315
Carotol	0.24	66.314

were as follows: carvacrol (33.26%), β -caryophyllene (12.15%), α -pinene (7.32%), β -pinene (6.13%). The other components detected in essential oil are shown in Table 1. In Turkey *Sideritis* species are divided into six groups according to their volatile oil components; those rich in monoterpene hydrocarbons, rich in sesquiterpene hydrocarbons, rich in oxygenated monoterpene, rich in

oxygenated sesquiterpene, rich in diterpene, and others. About 57% of Turkish Sideritis species have essential oil rich in monoterpene hydrocarbons and contain high amounts of α -pinene, β -pinene, β -phellandrene, and myrcene. Kırımer et al have detected a rough correlation between major component groups of Sideritis species; the higher the oil yield, the higher the monoterpene hydrocarbon content, the lower the oil yield, the lower the sesquiterpene content. Kırımer et al found that the oil yields of Sideritis species vary from trace amounts (<0.01%) to 0.85%.10 Hence, obtaining a 0.4% yield in this study shows that S. rubriflora has an average essential oil yield among Sideritis species. Major compounds of S. rubriflora (which was collected from steppes in İçel-Anamur Aydıncık region in 1994) was found to contain β-pinene (13.22%) and α-pinene (9.91%)¹¹; however, in this research epi-cubebol (7.8%), germacrene D (6.4%), β-caryophyllene (6.1%) and carvacrol (4.53%) were detected as dominant compounds.11 Similarly, in this study, α -pinene and β -pinene contents were found in high amounts. However, a large difference was observed in terms of carvacrol (Table 1). In another study, β-pinene (10.7%) and germacrene D (10.7%) were found to be the main compounds of S. Rubriflora. 12 In addition, Ceylan et al reported that S. Rubriflora showed antioxidant activity and these results support that S. rubriflora can be used as a natural agent in phytopharmaceutical applications.¹² The results of this study are similar to the results obtained by Ceylan et al in terms of high β -pinene (6.13%) content; whereas germacrene D (1.22%) differs in terms of its low content (Table 1). The essential oil yield obtained from the dried aboveground parts of S. rubriflora collected from two different locations (Mollaömerli and Sütlüce town-Mersin) was found to be 0.67% and 0.46%, respectively. The main components of the essential oil obtained from plants in the Mollaömerli region are β -pinene (20.53%), α -pinene (14.88%), cubebol (15.17%), and δ -cadinene (12.99), while the volatile components of plants obtained from Sütlüce region are β-pinene (20.29%), α-pinene (14.82%), 14-hydroxy-9-epi-caryophyllene (13.99%) and β-caryophyllene (8.83%). Carvacrol was not detected in both.13

Lamiaceae family, which includes *Sideritis* species, especially *Origanum*, *Satureja*, *Thymbra*, and *Thymus* species, are rich in carvacrol.¹⁴ In this study, the rate of carvacrol was found to be 33.26% (Table 1). This is one of the highest carvacrol content values obtained in designated *Sideritis* species in Turkey. Carvacrol is a monoterpene phenol produced by a large number of aromatic plants. Currently, carvacrol is used at low concentrations in the food industry as a preservative, flavoring, and fragrance component in cosmetic formulations. In recent years, many studies have been carried out on carvacrol. Results from *in vitro* and *in vivo* studies show that carvacrol has a variety of biological and pharmacological properties,

including antioxidant, antibacterial, antifungal, hepatoprotective, anticancer, anti-inflammatory, spasmolytic, and vasorelaxant properties. 15 S. rubriflora may be a good source of carvacrol compared to Thymus and Satureja species, which are known to be a good source of carvacrol. Low water consumption, as well as the high component amount, can support the use of S. rubriflora in regions with hot and dry desert climates where the annual average rainfall is very low. It supports the evaluation of S. rubriflora cultivation as a new and important source of carvacrol. More studies on S. rubriflora are needed. This may also indicate that S. rubriflora can be used to improve food quality and flavor. Kırımer et al have determined the main components of S. curvidens as bicyclogermacrene (20.60%) and caryophyllene (8.93%). In this research, bicyclogermacrene was detected in a low amount (0.19%) in S. rubriflora (Table 1). In S. montana subsp. montana and S. montana subsp. remota bicyclogermacrene (10.8%, 13.86%) and germacrene D (24.59%, 10.33%) were found to be the main components, respectively.¹⁶ In this study, a low percentage of germacrene D (1.22%) was detected in S. rubriflora essential oil (Table 1). Major constituents of S. argyrea, S. armeniaca, S. hololeuca and S. stricta species are β-pinene (20%, 39%, 35%, and 30% respectively) and α-pinene (14%, 17%, 16%, and 13%) respectively.¹⁷ α -Pinene (16.3-19.5%) and sabinene (6.1%-10.4%) were detected as the main compounds of S. erythrantha var. erythrantha; myrcene (21.9%-24.3%) and α -pinene (11.4-12.4%) were detected as the main components of S. erythrantha var. Cedretorum. 18 In the essential oil of S. dichitoma 39 components were detected and the main compounds were found to be α -pinene (22.5%), β -pinene (28.5%), limonene (4.6%) ve α -terpinene (4.5%).⁵ The results of this study are parallel with the above results, especially in terms of high α -pinene and β -pinene contents (Table 1). Different biological activities of plants under different conditions may be closely related to the fact that their essential oil compositions differ under these conditions.¹⁹ In three samples collected from the same locality at different vegetation periods, the major compounds of *Sideritis ozturkii* were detected as α-pinene (31.1%, 16.0%, 6.2%) and β-pinene (20.2%, 14.2%, 7.3%).²⁰ The main compounds of *S. bilgerana* were β -pinene (48%) and α -pinene(32%); β -pinene (39%), α -pinene (28%), and β-phellandrene (20%) were detected as the major components of S. cilicica.21 Evaluating the antimicrobial effects of oils using the microdilution broth method, İşcan et al reported that both oils showed good inhibitory effects on Candida albicans.21 It has been concluded that S. ozturkii and Sideritis caesarea extracts can be used as a natural antimicrobial and antioxidant agents in food preservation and human health.22 Gonzales-Burgos et al reported that there are differences in quality and quantity between the essential oil components of the same species in the literatüre. ²³ The differences in both essential oil ratio

and essential oil components may result from genetic and ecological reasons, as well as morphogenetic, ontogenetic, and diurnal effects. Composition and amounts of essential oils can also vary depending on many factors such as plant species, ecological conditions, plant part, edaphic conditions, production method, climate, and the geographical structure of the region where it is grown. The differences observed in the chemical properties of the essential oil of *Sideritis* species can be explained in this context.

Conclusions

In this study, it was found that S. rubriflora was rich in essential oil content by obtaining 0.4 mL of essential oil from 100 g plant samples. As a result, forty components were determined from the essential oil of the plant, the main components were carvacrol (33.26%), β-caryophyllene (12.5%), α -pinene (7.32%), and β -pinene (6.13%). In this study, the chemotypes of S. rubriflora were determined as carvacrol, β -caryophyllene, α -pinene, and β -pinene. From the analysis results and the literature records, it is understood that the S. rubriflora plant is rich in essential oil amount and various chemical content and has a high economic value. In addition, with this study, the economic values of the members of the genus were emphasized by referring to the characteristics of Sideritis members such as aromatherapeutic, phytotherapy, ethnobotanical, and biological activity. In order to better evaluate the S. rubriflora plant and to continue the activities related to this endemic plant of high economic value, more basic data were obtained and it was tried to contribute to the sector. It is important to take the necessary precautions and show the necessary sensitivity to know the Sideritis members and especially the S. rubriflora plant better, to be cultivated and to prevent damage. In this way, Sideritis members and S. rubriflora will have the opportunity to continue their existence and spread more in nature, and easy and sufficient raw materials will be obtained for the sectors they are used.

Competing Interests

None.

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References

- Duman H. Sideritis L. Flora of Turkey and Aegean Islands Supplement II. Edinburg: Edinburg University Press; 1988:201-204.
- Piozzi F, Bruno M, Rosselli S, Maggio A. The diterpenoids from the genus *Sideritis*. *Stud Nat Prod Chem*. 2006;33:493-540. doi:10.1016/s1572-5995(06)80033-5
- 3. Kılıç Ö. Essential oil composition of two *Sideritis* L. taxa from Turkey: a chemotaxonomic approach. *Asian J Chem.*

- 2014;26(8):2466-2470.
- 4. Kılıç Ö, Bağci E, Doğan G, et al. Essential oil composition of endemic *Sideritis dichotoma* Huter (Lamiaceae) from Turkey. *Bilecik Şeyh Edebali Univ Fen Bilim Derg*. 2014;1(2):55-58.
- Schulz H, Özkan G, Baranska M, Krüger H, Özcan M. Characterisation of essential oil plants from Turkey by IR and Raman spectroscopy Author links open overlay panel. *Vibrational Spectroscopy*. 2005;39(2):249-256. doi: 10.1016/j.vibspec.2005.04.009
- Kök M, Kılıç Ö. Kök M, Kılıç Ö. Osmaniye İli (Türkiye) Etnobotaniğine Katkılar. Munzur Zirvesi. 2. In: Uluslararası Uygulamalı Bilimler Kongresi; 31 Mayıs 2020; Tunceli; Tam metin Kitabi.
- 7. Kılıç Ö, Bağci E. An ethnobotanical survey of some medicinal plants in Keban (Elazığ-Turkey). *J Med Plants Res.* 2013;7(23):1675-1684. doi:10.5897/jmpr2013.4451
- 8. Yeşlada E, Ezer N. The antiinflammatory activity of some *Sideritis* species growing in Turkey. *Int J Crude Drug Res*. 1989;27(1):38-40. doi:10.3109/13880208909053936
- 9. Sargin SA, Selvi S, Büyükcengiz M. Ethnomedicinal plants of Aydıncık district of Mersin, Turkey. *J Ethnopharmacol*. 2015;174:200-216. doi:10.1016/j.jep.2015.08.008
- 10. Kirimer N, Başer KH, Demirci B, Duman H. Essential oils of *Sideritis* species of Turkey belonging to the section *Empedoclia*. *Chem Nat Compd*. 2004;40(1):19-23. doi:10.1023/B:CONC.0000025458.00475.cf
- Kirimer N, Tabanca N, Özek T, Başer KH, Tümen G. Composition of essential oils from two endemic *Sideritis* species of Turkey. *Chem Nat Compd.* 1999;35(1):61-64. doi:10.1007/BF02238211
- 12. Ceylan R, Zengin G, Aktümsek A. GC-MS analysis and antioxidant potential of essential oil from endemic *Sideritis rubriflora* Hub.-Mor. *Proceedings*. 2019;40(1):24. doi:10.3390/proceedings2019040024
- 13. Chalchat JC, Özcan MM, Figueredo G. The composition of essential oils of different parts of laurel, mountain tea, sage and ajowan. *J Food Biochem*. 2011;35(2):484-499. doi:10.1111/j.1745-4514.2010.00397.x

- 14. Başer KH, Kirimer N. Essential oils of Anatolian Lamiaceae-an update. *Nat Volatiles Essent Oils*. 2018;5(4):1-28.
- Suntres ZE, Coccimiglio J, Alipour M. The bioactivity and toxicological actions of carvacrol. *Crit Rev Food Sci Nutr.* 2015;55(3):304-318. doi:10.1080/10408398.2011.653458
- Kirimer N, Tabanca N, Özek T, Tümen G, Başer KH.
 Essential oils of annual *Sideritis* species growing in Turkey.
 Pharm Biol. 2000;38(2):106-111. doi:10.1076/1388-0209(200004)3821-1ft106
- 17. Kirimer N, Tabanca N, Özek T, Başer KH, Tümen G, Duman H. Composition of essential oils from five endemic *Sideritis* species. *J Essent Oil Res.* 2003;15(4):221-225. doi:10.1080/10412905.2003.9712125
- 18. Tabanca N, Kirimer N, Başer KH. The composition of essential oils from two varieties of *Sideritis erythrantha* var. *erythrantha* and var. *cedretorum*. *Turk J Chem*. 2001;25(2):201-208.
- Iscan G, Ozek T, Ozek G, Duran A, Başer KH. Essential oils of three species of *Heracleum*. Anticandidal activity. *Chem Nat Compd*. 2004;40(6):544-547. doi:10.1007/ s10600-005-0032-z
- Kirimer N, Tabanca N, Demirci B, Başer KH, Duman H, Aytac Z. The essential oil of a new *Sideritis* species: *Sideritis ozturkii* Aytac and Aksoy. *Chem Nat Compd*. 2001;37(3):234-237. doi:10.1023/a:1012561806033
- 21. Iscan G, Kirimer N, Kurkcuoglu M, Başer KH.
 Composition and antimicrobial activity of the essential oils of two endemic species from Turkey: *Sideritis cilicica* and *Sideritis bilgerana*. *Chem Nat Compd*. 2005;41(6):679-682. doi:10.1007/s10600-006-0010-0
- Sagdic O, Aksoy A, Ozkan G, Ekici L, Albayrak S. Biological activities of the extracts of two endemic Sideritis species in Turkey. Innov Food Sci Emerg Technol. 2008;9(1):80-84. doi:10.1016/j.ifset.2007.06.001
- 23. González-Burgos E, Carretero ME, Gómez-Serranillos MP. *Sideritis* spp.: uses, chemical composition and pharmacological activities--a review. *J Ethnopharmacol*. 2011;135(2):209-225. doi:10.1016/j.jep.2011.03.014

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