

## THE EFFECT OF REAPING TIMES ON VOLATILE COMPONENTS OF NATURAL *PHLOMIS L. (LAMIACEAE)* TAXA IN THE LAKES DISTRICT OF TURKEY

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(Received 14<sup>th</sup> Feb 2016; accepted 23<sup>rd</sup> Jun 2016)

**Abstract.** This study performed SPME analysis in the Lakes District of Turkey between 2012-2015 and found that *Phlomis armeniaca* includes 54 volatile components, *P. bourgaei* 62, *P. grandiflora* var. *grandiflora* 60, *P. leucophracta* 70, *P. lycia* 62, *P. nissolii* 54, *P. pungens* var. *pungens* 74 and *P. samia* 49. The main components were (E)-2-hexenal (12.12%),  $\beta$ -caryophyllene (16.63%) and germacrene-D (27.22%) for *P. armeniaca*;  $\alpha$ -cubebene (16.04%),  $\beta$ -caryophyllene (21.98%) and germakren-D (15.12%) for *P. bourgaei*;  $\alpha$ -pinene (26.40%),  $\alpha$ -cedrene (28.15%) and  $\alpha$ -curcumene (13.92%) for *P. grandiflora* var. *grandiflora*; (E)-2-hexenal (8.74%), limonene (14.56%) and  $\beta$ -caryophyllene (22.45%) for *P. leucophracta*, limonene (17.68%),  $\beta$ -caryophyllene (23.66%) and germacrene-D (21.88%) for *P. lycia*; limonene (23.75%),  $\beta$ -caryophyllene (12.50%) and germacrene-D (20.73%) for *Phlomis nissolii*; (E)-2-hexenal (17.60%), vinyl amyl carbinol (20.44%) and germacrene-D (9.84%) for *P. pungens* and  $\alpha$ -copaene (10.59%),  $\beta$ -caryophyllene (15.20%) and germacrene-D (23.44%) for *P. samia*.

**Keywords:** *Phlomis*, volatile component, SPME analyses,  $\beta$ -caryophyllene, germakren-D, Turkey

### Introduction

The flora in Turkey has approximately 11.466 plant taxa. As a comparison, the European continent has approximately 12.000 (Guner et al., 2012). Turkey also has species variety that is the gene center of several plants and there are many endemic species in different geographical regions (Tan, 1992). In particular for the endemics, aromatic and medical values of hundreds of plant types grown in Turkey are naturally higher (Baydar, 2009). This is especially the case for volatile oil content in medicinal and aromatic plant groups which has a separate importance. Volatile oils (perfumes, etheric oils) and aromatic extracts are commonly used for perfume production, to enhance smell and taste, as food additives, in cleaning products, in cosmetic and drugs, and as sources of aroma chemicals or identical natural and semi-synthetic aroma chemicals for the synthesis of starting materials. Currently, there is increasing demand for volatile oils in aromatherapy applications (Weiss, 1997).

The Lamiaceae family has generally sweet smelling one or multi perennial plants that are rarely briars and with some trees. This cosmopolite family is represented by 200 genera and approximately 3000 species. *Lamiaceae* (labiate) family members that are represented by 45 genera and 546 species in Turkey are important for the pharmacology and perfumery industry due to their volatile and aromatic oils. Etheric oil is an example that is used as a spice is also grown as a decorative plant. The *Phlomis* taxa which has the most species of genera of the Lamiaceae family, has over 100 species all around the world. The taxa of this genus are

distributed in Asia, South Europe and North Europe (Matthiesen et al., 2011). In Turkey, it is represented by 39 taxon and 13 hybrids, for a total of 52 taxon (Guner et al., 2012).

The discovery of new usage areas for medical and aromatic plants, increasing demand for natural products, increases the demand for these plants each passing day. The medical plant market is estimated to currently reach approximately 60 billion dollars (Kumar, 2009). Moreover, there is growing public interest towards such plants and for use of aromatic and medical plants.

*Phlomis* taxa have an important place in the natural distribution of medical plants and one of the species that has the most types of the Lamiaceae family. It has about 100 species all around the world. The length of species can vary between 30 cm and 2 meters. The sides of leaves are jagged and opposing in alignment but are not in a bulk condition. Feathers covering the surface of plant are stellate. Flowers are purple, pink, white or yellow colored (Huber-Morath, 1982). Leaves and flowers are used to make products more appetizing, as an anti-allergic, as a diuretic, for diarrhea prevention, against stomach aches, to relieve pain, as an anti-diabetic, herbal tea and tonic. The plant is also colloquially known to be used for respiratory tract diseases and hemorrhoid problems (Harput et al., 2006).

Although it is used in many areas, there is little research about the *Phlomis* taxa of Turkey. Research about the volatile oil of leaves is also limited. For these reasons, this study aimed to research the effect of different picking periods in terms of volatile oil compounds and to determine suitable picking periods for *Phlomis armeniaca* Willd., *P. bourgaei* Boiss., *P. grandiflora* H.S. Thompson var. *grandiflora*, *P. leucophracta* P.H.Davis & Hub.-Mor., *P. lycia* D. Don., *P. nissolii* L., *P. pungens* Willd. and *P. samia* L.

## Materials and Methods

*Phlomis* samples were collected from the Lakes District of Turkey (C2, C3, C4 squares) between the years 2012 and 2015 (Figure 1). Thirty-three samples for *Phlomis* species were collected from the Lakes District in three different annual time periods: pre-bloom period, bloom period, and post-emergence period (Table 1).

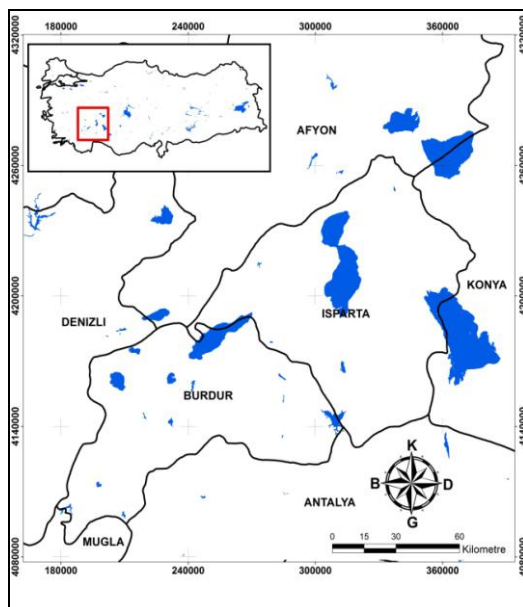


Figure 1. Research area

**Table 1.** Ecological land informations of collecting samples

Taxa	Province	Location	Altitude (m)
<i>Phlomis armeniaca</i> Willd.	Afyon-Çay county	37° 47' 08" N 30° 45' 54" E	830
	Konya-Beyşehir county, Kurucuova province	37° 50' 25" N 31° 06' 11" E	1155
	Isparta-Aksu county, Sorgun Yaylası province	37° 42' 11" N 31° 17' 38" E	1490
	Isparta- Davraz mountain, Ardiçdibi province	37° 48' 09" N 30° 45' 28" E	1604
	Isparta-Davraz mountain	37° 41' 55" N 31° 24' 18" E	1622
	Isparta-Yenişarbademli county, Melikler Yaylası province	38° 57' 02" N 31° 02' 19" E	1762
<i>Phlomis bourgaei</i> Boiss.	Isparta- Sığla ( <i>Liquidambar orientalis</i> ) Forest Nature Protected Area	37° 34' 15" N 30° 50' 06" E	379
	Isparta- Eğirdir county, Aşağıgökdere province	37° 37' 33" N 30° 43' 50" E	471
	Antalya-Isparta motoway, Dereboğazi province	37° 19' 48" N 30° 37' 22" E	590
	Isparta- Eğirdir county, Aşağıgökdere Akbelenli province	37° 33' 57" N 30° 51' 58" E	705
	Burdur- Karlık Kavaklık province	37° 44' 28" N 30° 33' 18" E	1228
	Isparta- Urban forest, Sidre province	37° 22' 31" N 30° 49' 18" E	1312
<i>Phlomis grandiflora</i> H. S. Thompson var. <i>grandiflora</i>	Antalya-Burdur motoway, Dağbeli province	37° 38' 44" N 30° 52' 09" E	855
	Antalya-Isparta motoway, Gökbel province	37° 38' 08" N 30° 42' 48" E	900
	Isparta- Kovada Lake National Park	36° 53' 03" N 29° 25' 38" E	920
	Burdur- Gölhisar county, Ballık province	37° 11' 11" N 30° 11' 02" E	1427
	Isparta-Sütçüler county, Tota mountain Böğülüuyurt province	37° 35' 01" N 31° 04' 34" E	1580
<i>Phlomis leucophracta</i> P. H. Davis & Hub.-Mor.	Burdu- Bucak county, Kargı Taşdibi province	37° 34' 05" N 30° 50' 43" E	176

Taxa	Province	Location	Altitude (m)
	Isparta- Eğirdir county, Aşağıgökdere Akbelenli province	37° 38' 16" N 30° 43' 41" E	420
	Burdur- Ağlasun county, Çamlidere village	37° 15' 30" N 30° 48' 39" E	690
<i>Phlomis lycia</i> D. Don	Burdur- Bucak county, Boğazköy province	37° 09' 15" N 30° 17' 35" E	783
	Burdur- Bucak county, Uğurlu province	37° 09' 22" N 30° 30' 05" E	823
	Burdur- Bucak county, Çubuk Beli province	37° 14' 23" N 30° 29' 50" E	908
<i>Phlomis nissolii</i> L.	Afyon- Çay county	37° 47' 08" N 30° 45' 54" E	830
	Isparta-Sav county, Yazısöğüt village	37° 46' 09" N 30° 37' 13" E	989
	Isparta-Şarkikaraağaç county, Belceğiz village	38° 00' 25" N 31° 17' 32" E	1203
	Isparta-Yalvaç county, İleği village	37° 41' 46" N 31° 22' 18" E	1224
	Isparta- Şarkikaraağaç county, Salur village	37° 58' 03" N 31° 17' 45" E	1236
	Isparta- Aksu county, Zindan cave province	38° 57' 02" N 31° 02' 19" E	1280
	Isparta- Urban forest, Sidre province	37° 44' 28" N 30° 33' 18" E	1316
<i>Phlomis pungens</i> Willd. var. <i>pungens</i> (Silvanok)	Isparta- Sorgun Yaylası province	37° 50' 12" N 31° 06' 15" E	1488
	Burdur- Gölhisar county	37° 44' 28" N 29° 24' 24" E	1649
<i>Phlomis samia</i> L.	Isparta- Kovada Lake National Park	37° 38' 53" N 30° 52' 08" E	942
	Isparta-Yenişarbademli county Pınargözü province	37° 42' 06" N 31° 18' 36" E	1529

### Preparation of plant samples for GCMS analyses

Samples belonging to three different vegetation periods, pre-bloom, bloom, and post-emergence were taken from the determined areas. Collected leaves and flower samples were transported to the laboratory after placement in paper packages and without delay or exposure to sunlight. All materials were dried.

### ***Determination of volatile compounds***

Volatile components of *Phlomis* leaves and flowers were determined using a solid phase microextraction method (SPME) (Vichy et al., 2003). For this aim, after drying the plant materials at room temperature (25 °C), two grams from each sample were bottled and heated 15 minutes in 60 °C. A proper edge injector sank to the bottom and was absorbed for 30 minutes. Compounds held to the fiber edge were injected to a GC injection block and given five minutes for desorbing. The model for the used SPME Fiber was 75UMCAR/PDMSFUSED-SILICA, Supelco, USA, PA.

Gas chromatography-mass spectrometry equipment (GC-MS, Shimadzu QP 5050, Japan) was used in order to determine volatile compounds. HP-5 MS (30 m x 0.25 mm length and 0.25  $\mu$ m film was used in device and helium was used as the column and carrier gas (10 psi flow rate). The temperature of the injection block was 240 °C and detector temperature was 250 °C.

### ***Method for statistical data***

Non-parametrical tests were used, as the ratios determined for each volatile oil compound did not meet preconditions for parametric tests. The Kruskal-Wallis test, which is a non-parametrical test, was used in determining inter-species differences. The Friedman test, a non-parametrical test, was used for determining differences in different vegetation periods. The Bonferroni-Dunn method, which is a multi-compare method, was used for determining differences between the media.

## **Results**

Leaf and flower volatile compounds of *Phlomis armeniaca*, *P. bourgaei*, *P. grandiflora* var. *grandiflora*, *P. leucophracta*, *Phlomis lycia*, *P. nissolii*, *P. pungens* var. *pungens*, *P. samia* were determined by SPME analysis (solid-based micro extraction method).

SPME analysis found 54 volatile components of *Phlomis armeniaca*, 62 of *Phlomis bourgaei*, 60 of *Phlomis grandiflora* var. *grandiflora*, 70 of *Phlomis leucophracta*, 62 of *Phlomis lycia*, 53 of *Phlomis nissolii*, 70 of *Phlomis pungens* var. *pungens* and 64 of *Phlomis samia*. These results were given in Table 2.

(E)-2-hexenal,  $\beta$ -caryophyllene and germakren-D were volatile components of *Phlomis armeniaca*. Volatile components during the pre-bloom period were: (E)-2-hexenal (11.64%),  $\beta$ -caryophyllene (15.73%) and germakren D (23.45%); during the bloom period (E)-2-hexenal (12.12%),  $\beta$ -caryophyllene (16.63%) and germakren-D (27.22%) and during the post-emergence period were: (E)-2-hexenal (10.07%),  $\beta$ -caryophyllene (11.55%) and germakren-D (25.03%).

$\alpha$ -cubebene,  $\beta$ -caryophyllene and germakren-D were volatile components of *P. bourgaei*. Specifically:  $\alpha$ -cubebene (15.55%),  $\beta$ -caryophyllene (20.80%) and germakren-D (12.41%) ratios in the pre-bloom period,  $\alpha$ -cubebene (16.04%),  $\beta$ -caryophyllene (21.98%) and germakren-D (15.12%) in the bloom period; and  $\alpha$ -cubebene (13.92%),  $\beta$ -caryophyllene (14.73%) and germakren-D (11.21%) in the post emergence period.

**Table 2.** Volatile components of leaf and flower according to different vegetation periods

Components	Volatile components																							
	<i>P. armeniaca</i>			<i>P. bourgaei</i>			<i>P. grandiflora</i>			<i>P. leucophracta</i>			<i>P. lycia</i>			<i>P. nissolii</i>			<i>P. pungens</i> var. <i>pungens</i>			<i>P. samia</i>		
	P.B.	B.P.	P.E.	P.B.	B.P.	P.E.	P.B.	B.P.	P.E.	P.B.	B.P.	P.E.	P.B.	B.P.	P.E.	P.B.	B.P.	P.E.	P.B.	B.P.	P.E.	P.B.	B.P.	P.E.
Dimethyl sulfide	0.26	1.23	1.06	0.3	0.28	-	0.3	0.13	0.1	-	-	0.26	-	-	-	-	-	1.89	0.82	0.18	-	-	-	-
2-methyl-Propanal	-	0.86	0.66	-	-	-	-	0.26	-	-	-	0.14	-	-	-	-	-	0.26	0.95	-	-	-	-	-
Isobutyl alcohol	0.3	-	-	0.59	0.43	-	0.6	-	-	-	-	-	-	-	-	-	-	-	-	0.17	-	-	-	-
2-Butenal	-	-	-	-	-	-	-	-	-	1.09	1	0.67	0.47	0.24	0.58	0.14	0.6	-	-	1.17	0.64	0.68	-	-
3-Methylbutanal	0.28	0.97	1.31	1.18	0.61	0.18	0.13	0.26	0.75	0.38	0.39	1.67	0.03	0.06	0.08	0.23	0.16	0.41	1.29	4.41	0.55	0.45	0.68	0.88
2-Methylbutanal	0.33	0.55	1.07	1.11	0.47	-	0.17	0.24	0.65	0.52	0.61	1.12	0.06	0.1	0.09	-	-	-	1.08	4.33	0.25	0.86	-	-
Methyl propyl ketone	-	1	0.55	-	-	-	-	-	-	-	-	-	-	-	-	1.06	-	-	-	-	-	-	-	-
n-Pentenal	-	-	-	-	-	-	-	-	-	0.54	0.47	0.56	0.45	0.15	0.33	-	-	-	-	1.16	0.32	0.7	-	-
Ethyl Vinly Carbinol	-	-	-	0.19	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.93	-	-	-	-
Ethyl Propyl Keton	-	-	-	-	-	0.13	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Furan, 2-ethyl-	0.39	0.75	3.11	-	0.89	0.32	0.51	0.36	0.12	0.35	0.3	2.09	-	0.23	0.32	0.88	0.39	0.59	1.42	1.1	1.11	0.95	-	-
Ethyl vinly ketone	-	-	-	-	-	-	0.11	-	-	0.34	0.12	0.66	-	-	-	-	-	-	-	0.52	-	0.62	-	-
trans-3-Penten-2-one	-	-	-	-	-	-	-	-	-	0.16	0.18	0.1	-	0.04	0.17	-	-	-	-	-	-	-	-	-
3-Methyl-1-butanol	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.28	1.57	-	-	-	-
2-Methyl-1-butanol	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.55	-	-	-	-
(E)-2-Pentenal	-	0.24	0.25	-	-	-	-	-	-	0.18	0.2	0.75	0.1	0.12	0.21	0.45	0.32	0.37	0.31	0.57	0.59	0.56	-	0.34
(Z)-2-Pentenal	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.44	0.32	0.14	-	-
3-Methyl-2-butanol	-	-	-	-	-	-	-	-	-	-	-	0.37	-	-	-	-	-	-	-	-	-	-	0.47	0.48
1-Pentanol	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.77	-	-	-	-
n-Hexanal	0.74	1.36	1.95	0.79	0.85	0	0.39	0.11	0.2	4.89	3.54	3.27	2.95	1.56	3.88	0.58	1.87	1.27	2.68	4.86	3.73	2.69	2.5	4.1
(E)-2-Hexenal	11.64	12.12	10.07	5.52	3.82	0.5	4.34	0.51	1.02	7.5	8.74	6.1	3.78	3.92	6.85	8.57	10.57	9.05	16.87	17.6	12.68	9.04	2.98	3.66
Z-3-hexenol	-	0.93	-	-	-	-	-	-	-	0.22	0.44	1.91	0.11	0.18	0.35	0.76	0.18	-	-	0.33	2.52	1.24	-	-
cis-3-Hexene-1-ol	0.8	-	1.9	0.66	-	-	-	-	-	-	-	-	-	-	-	-	-	-	2.65	3.88	-	-	-	-
cis-Hex-2-en-1-ol	0.27	0.2	1.83	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.34	1.3	-	-	-	-

2-Hexenol, (E)-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.83	-	-	-	-	0.44	-	-	-
Hexanol <n->	0.34	0.5	3	-	-	-	-	-	-	0.16	0.25	0.51	0.05	0.17	0.09	0.81	0.05	1.17	1.3	2.17	2.2	0.3	-	0.42	
Amyl methyl ketone	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.46	0.36	
n-Heptanal	-	-	-	0.29	0.29	0.29	-	-	-	0.92	0.85	2.3	0.15	0.15	0.24	-	-	-	-	0.23	0.4	1.02	-	9.75	
2,4-Hexadienal	-	-	-	-	-	-	-	-	-	0.42	0.56	0.2	0.26	0.1	0.43	-	-	-	-	0.54	-	0.19	-	-	
Sorbaldehyde, (E,E)	-	-	-	-	-	-	-	-	-	-	-	2.15	-	-	-	-	-	-	0.68	-	-	-	-	2.61	
$\alpha$ -Thujene	-	-	-	0.31	0.39	0.43	1.64	1.43	1.22	0.54	0.51	0.5	0.18	0.39	0.33	0.68	0.13	0.54	0.77	1.48	0.66	-	-	-	
$\alpha$ -Pinene	0.74	0.93	1.44	0.11	2.13	2.8	25.97	26.4	18.95	2.85	2.72	1.92	1.26	2.24	2.03	6.86	6.93	6.43	4.91	4.07	3.85	0.26	2.2	1.1	
(E)-2-Heptenal	-	-	-	-	-	-	-	-	-	1.33	1.26	1.25	0.67	0.35	0.79	-	-	-	-	-	0.23	0.39	-	-	
Benzaldehyde	2.63	3.8	0.39	0.49	0.86	-	0.88	0.3	0.11	2.5	1.98	2.77	0.8	1.99	1.9	0.58	0.24	4.03	5.42	2.88	3.12	5.36	2.51	2.08	
Methyl 2-hexenoate	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.33	-	-	-	-	-	-	
2-Hexenoic acid, methyl ester	-	1.02	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Sabinene	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.4	0.23	-	-	-	
$\beta$ -Phellandrene	-	-	-	-	-	-	0.44	0.41	0.39	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
$\beta$ -Pinene	-	-	-	-	-	-	0.79	0.13	0.66	0.15	0.22	0.1	-	-	-	-	-	-	-	0.16	0.33	-	-	-	
Amyl vinyl ketone	-	-	-	-	-	-	-	-	-	0.24	0.23	0.39	0.15	0.05	0.17	-	-	-	-	-	-	-	-	-	
Vinly amly carbinol	2.13	5.45	5.55	3.02	0.25	0.94	0.18	0.09	0.15	1.06	0.83	0.7	1.38	0.75	1.52	2.71	1.44	3.83	12.85	18.44	18.6	0.69	0.55	0.73	
4-Pentene-1-yl acetate	-	-	0.38	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
6-Methyl-5-hepten-2-one	-	-	-	0.21	0.45	0	0.38	0.31	0.17	0.42	0.44	1	0.2	0.24	0.35	-	-	-	-	-	-	0.42	0.44	0.52	
Amyl ethyl ketone	0.3	0.78	1.75	-	-	0.66	-	-	-	-	-	-	-	-	-	1.96	-	0.82	1.79	2.37	-	-	-	-	
$\beta$ -Myrcene	-	-	-	-	1.15	0.87	0.47	0.98	0.87	2.15	2.28	1.76	0.86	2.62	1.47	3.18	0.52	1.46	0.88	0.82	1.63	0.27	-	-	
(R,S)-2-Propyl-5-Oxohexanal	-	-	-	-	-	-	-	-	-	-	-	0.57	-	-	-	-	-	-	-	-	-	-	-	1.26	
Fenchone	0.32	-	-	0.16	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
3-(2-methylpropyl) Cyclohexene	-	0.6	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
4-Ethylcyclohexanol	-	-	1.54	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	

2-Octanone	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.85	-
n-Octanal	-	-	-	0.18	0.36	-	0.12	-	-	2.24	2.11	1.71	0.41	0.32	0.78	0.33	0.24	0.25	-	-	0.85	0.61	1.11	1.04	
$\alpha$ -Phellandrene	-	-	-	-	-	0.21	0.14	-	-	0.44	0.64	0.77	0.06	0.58	0.18	0.66	0.23	-	-	-	0.18	-	-	-	
2,4-Heptandienal	0.18	0.2	0.37	0.81	0.32	0	0	-	-	1.18	1.48	5.17	0.41	0.39	0.72	0.51	1.11	0.36	2.39	1.27	2.82	1.6	0	0.65	
p-Dichlorobenzene	0.56	0.6	0.57	-	0.92	0.46	0.26	0.11	0.18	-	-	-	-	-	-	1.15	-	0.86	-	-	-	0.15	0.86	1.01	
$\alpha$ -Terpinene	-	-	-	-	0.49	0.38	-	-	-	0.68	1	0.4	0.08	0.56	0.21	0.41	0.15	-	-	-	-	-	-	-	
p-Cymene	-	-	-	-	0.31	0.3	0.42	0.34	0.4	1.03	1.29	1.05	0.33	0.55	0.6	1.88	0.32	1	0.3	0.53	1.51	-	-	-	
Limonene	1.53	1.4	0.69	0.13	5.84	4.6	3.36	2.62	2.29	13.64	14.56	10.93	13.7	17.68	10.65	20.65	23.75	16.17	1.58	0.67	6.83	1.17	2.16	2.03	
(E)-3-Octen-2-one	-	-	-	-	-	-	-	-	-	0.75	0.61	-	0.71	0.19	0.72	-	-	-	-	-	-	-	-	-	
cis-OCimene	-	-	-	-	-	-	1.47	0.82	2.03	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Benzeneacetaldehyde	0.58	0.69	-	0.41	0.6	-	0.26	-	-	-	-	-	-	-	-	-	-	0.32	0.78	0.46	-	-	-	-	
Phenylacetaldehyde	-	-	-	-	-	-	-	-	-	0.63	0.71	-	0.21	0.1	0.63	-	-	-	-	-	0.51	0.76	-	-	
$\beta$ . Ocimene	-	-	-	-	-	0.1	0.18	0.15	0.14	0.24	0.39	-	0.05	0.39	0.11	0.42	0.04	-	-	-	-	-	-	-	
2 Octenal	-	-	-	-	-	-	-	-	-	0.93	0.98	0.63	0.45	0.46	0.58	-	-	-	-	-	0.41	0.2	1.14	0.88	
3,5-Octadien-2-one	-	0.21	-	-	-	-	-	-	-	0.36	0.34	-	0.26	0.1	0.48	-	-	-	-	-	0.49	0.55	-	-	
$\gamma$ -Terpinene	-	-	-	-	0.63	0.51	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Octanol	-	-	-	-	-	-	-	-	-	0.1	0.19	-	-	-	-	-	-	-	-	-	-	-	-	-	
$\alpha$ -Terpinolene	-	-	-	-	-	-	-	-	-	1.55	1.29	0.5	0.14	0.89	0.36	1.1	0.42	0.32	-	-	-	-	-	-	
Dimethylstyrene < $\alpha$ -para->	-	-	-	-	-	-	-	-	-	0.1	0.21	-	-	-	-	-	-	-	-	-	-	-	-	-	
2-Nonanone	-	-	-	-	0.78	3.72	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.22	0.93	0.44	
Methyl benzoate	-	-	-	-	-	-	-	-	-	0.26	0.27	-	0.16	0.07	0.28	-	-	-	-	-	-	-	-	-	
Linalool	0.58	2.02	-	-	-	-	-	-	0.23	-	-	1.04	-	-	0.34	-	-	-	1.17	-	-	0.7	1.55	0.44	
n-Nonanal	0.54	0.76	0.42	1.02	0.73	0.3	0.3	0.15	0.6	3.44	2.61	2.32	0.7	1.03	1.24	1.26	1.41	0.99	1	0.39	7.59	3.5	3.28	3.17	
2-Hendecanone	-	-	-	0.19	3.27	29.87	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Phenethyl alcohol	0.54	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.52	0.32	-	0.25	-	-	
$\alpha$ -Campholene aldehyde	-	-	-	-	-	-	-	0.12	0.09	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
trans-Alloocimene	-	-	-	-	-	-	-	0.08	0.2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
D-Carvone	-	-	-	-	-	-	-	-	0.11	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Trans-2-Nonenal	-	-	-	-	-	-	-	-	-	0.3	0.5	0.59	-	-	-	-	-	-	-	-	-	0.25	0.92	0.62	



Pinocarpone	-	-	-	-	-	-	-	0.09	0.09	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Methyl salicylate	1.17	2.15	0.34	-	-	-	-	-	-	-	-	0.6	-	-	-	-	-	1.29	1.36	-	-	-	-	-	-	
(E)-2-Nonenal	-	-	-	-	-	-	-	-	-	-	-	0	0.23	0.17	0.23	-	-	-	-	-	-	-	-	-	-	
n-Decanal	-	-	-	-	-	-	-	-	-	0.62	0.56	0.3	0.52	1.05	0.93	0.17	0.19	-	-	-	0.4	0.47	0.96	0.43		
(E)-2-Decenal	-	-	-	-	-	-	-	-	-	0.24	0.46	0.15	0.09	-	0.11	-	-	-	-	-	-	-	-	-	-	
Hendecanal	-	-	-	-	-	-	-	-	-	0.13	0.24	-	0.02	-	-	-	-	-	-	-	-	-	-	-	-	
Nonyl methyl ketone	0.23	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
2-Hendecanone	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.85	-
. $\alpha$ -Cubebene	1.11	1.31	1.9	15.55	16.04	13.92	-	-	-	3.55	3.3	2.50-	1.86	0.29	2.75	0.27	0.45	0.27	0.38	0.41	0.3	0.42	1.29	0.92	-	-
Ylangene	0.18	-	-	-	0.22	0.23	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
. $\alpha$ - Ylangene	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.59	-	-	-	-	-	-	0.75	-
. $\alpha$ -Copaene	0.8	1.11	1.22	3.97	3.1	3.2	0.18	0.16	0.54	0.96	0.79	2.78	0.93	1.53	1.21	1.84	1.98	0.84	0.5	0.23	0.6	9.59	10.59	9.71	-	-
. $\beta$ . Bourbonene	0.58	1.15	2.48	0.31	0.52	1.81	0.34	1.05	1.06	0.79	0.17	0.92	0.25	1.12	0.24	3.86	1.32	5.85	0.75	0.41	3.57	3.43	7.47	7.13	-	-
. $\beta$ -Cubebene	-	0.22	-	6.55	4.59	-	-	0.11	-	0.89	0.2	-	1.07	0.6	1.26	0.52	0.99	0.88	-	-	0.54	0.7	-	-	-	-
(-)-. $\beta$ -Elemene	0.66	0.66	0.52	-	-	-	-	-	-	0.19	0.14	-	0.23	0.67	0.15	-	-	-	0.66	-	0.34	0.6	0.87	0.44	-	-
Sesquithujene <7-epi->	-	-	-	-	1.67	-	0.19	0.2	0.18	0	0	-	0.34	0	0.5	-	-	-	-	-	-	-	-	-	-	-
n-Tetradecane	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.36
$\alpha$ -Gurjunene	-	-	-	2.1	1.07	1.05	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.84	-	-	-
. $\alpha$ -Cedrene	-	-	-	0.55	-	-	25.92	28.15	19.14	-	-	-	0.4	0.19	0.47	-	-	-	-	-	-	-	-	-	-	-
$\beta$ -Caryophyllene	15.73	16.63	11.55	20.8	21.98	14.73	7.78	6.4	3.91	22.32	22.45	20.12	17.63	23.66	10.63	11.28	12.5	10.37	0.74	1.4	0.65	13.79	15.2	13.75	-	-
$\beta$ -Cedrene	-	-	-	-	-	-	0.25	-	-	0.23	-	-	0.3	0.49	0.2	-	-	-	-	-	0.65	0.45	-	-	-	-
. $\gamma$ -Elemene	4.17	-	-	1.67	-	-	-	-	-	-	-	-	-	-	-	-	-	-	4.37	2.36	2.2	-	-	-	-	-
. $\alpha$ -Bergamotene	-	-	-	-	-	-	0.94	1.43	2.03	-	-	-	1.56	0.25	1.76	-	-	-	-	-	0.2	0.2	-	-	-	-
. $\alpha$ -Cedrol	-	-	-	-	-	-	-	0.52	0.45	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
. $\delta$ -Guaiene	-	-	-	0.61	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
. $\alpha$ -Amorphene	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.47	-	-	-	-	-	0.85	-	-
Alloaromadendrene	-	-	-	4.63	-	-	0.17	-	-	1.17	0.56	0.77	-	0.43	0.41	1.64	0.29	-	-	-	0.29	-	-	-	-	-
(E)- $\beta$ -Farnesene	14.87	0.21	6.87	-	-	-	1.8	-	1.28	3.57	2.96	2.4	4.76	3.71	10.23	3.86	4.05	5.86	11.32	0.43	1.94	-	-	-	-	-
Isodene	-	-	-	-	0.22	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
. $\alpha$ -Humulene	-	2.08	-	1.39	3.05	1.35	-	-	-	-	-	-	0.4	-	-	-	-	-	-	-	-	-	2.15	-	-	1.66

Cyclosativene	-	-	-	5.23	-	-	0.64	-	0.68	-	-	-	-	-	-	2.66	-	-	-	-	-	-	-	-
β-Humulene	-	-	-	-	-	-	-	-	7.52	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Farnesol	-	-	-	-	-	-	-	1.19	1.1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Cadina-l(6),4-diene<10βH>	0.51	0.71	0.37	0.55	0.63	1.07	-	-	-	0.4	0.21	0.1	0.46	0.78	0.44	-	1.62	0.47	-	-	0.63	0.77	0.63	0.59
β-Selinene	-	-	-	-	-	0.15	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Epi-Bicyclosquiphellandrene	1.09	0.75	2.09	0.26	0.46	0.67	-	-	-	-	-	0.39	-	-	-	0.26	-	0.74	0.29	-	-	-	1.74	1.66
Germacrene-D	23.45	27.22	25.03	12.41	15.12	11.21	4	5.17	5.4	6.46	8.32	7.47	15.66	21.88	11.76	12.27	20.73	10.44	7.78	9.84	8.25	21.01	23.44	18.9
Ionone	-	-	-	-	-	-	0.16	-	1.28	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
α-Curcumene	-	-	-	-	-	-	11.96	13.92	13.24	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Bicyclgermacrene	0.63	1.34	2.5	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1.14	1.22	-	0.6	-	-
Zingiberene	-	-	-	-	0.41	-	-	-	-	-	-	-	6.06	0.33	5.17	-	-	-	-	-	-	-	-	-
Valencene	-	-	-	1.59	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
α Bisabolol	-	-	-	-	-	0.81	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
α-Muurolole	0.53	0.61	0.8	0.3	0.37	0.37	-	-	-	-	-	-	-	-	-	0.19	0.91	0.27	-	-	-	0.59	1.5	0.58
β-Muurolole	-	-	-	-	-	-	-	-	-	-	-	-	0.25	0.54	0.2	-	-	-	-	-	-	-	-	-
Eremophilene	-	-	-	-	-	-	-	-	3.04	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Cedr-8-e	-	-	-	-	-	-	0.26	2	3.02	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Caran-cis-3-ol	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.61	-
β-Bisabolene	-	-	-	-	0.51	-	-	-	-	-	0.22	-	-	-	-	-	-	-	-	-	-	-	-	-
α-selinene	-	-	-	-	-	-	-	-	1.68	-	-	-	-	-	-	-	-	1.87	-	-	-	-	-	-
β- Bisabolene	-	-	-	-	-	-	-	-	-	-	-	-	4.96	-	4.33	-	-	-	-	-	-	-	0.66	-
γ-Cadinene	2.06	2	1.49	0.33	1.55	0.55	-	-	-	0.38	0.24	0.2	6.8	1.09	3.71	0.63	1.8	0.4	0.36	-	0.61	0.82	1.71	0.91
δ- Cadinene	1.39	1.44	2.46	1.31	1.07	1.27	-	0.08	0.1	0.71	0.82	1.18	0.83	1.35	0.62	0.64	1.26	0.61	0.82	-	1.02	6.6	5.29	3.55
β-Sesquiphellandrene	-	-	-	-	-	-	0.21	0.21	0.15	0.17	0.47	-	1.38	0.37	1.08	-	-	-	-	-	-	-	-	-
Germacrene B	4.48	1.24	-	2.26	-	-	-	-	-	-	-	-	-	-	-	0.55	0.22	4.69	4.11	-	1.28	-	-	-
(+)-spathulenol	0.17	-	-	-	-	-	-	-	0.37	-	-	-	-	-	-	-	-	0.39	-	-	-	-	-	-
Isolongifolene, 4,5,9,10-dehydro-	-	-	-	-	-	-	-	0.18	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

(-)-Caryophyllene oxide	0.21	-	-	-	-	0.2	0.79	1.22	1.19	-	-	-	0.71	0.16	0.43	0.37	0.62	0.98	-	-	-	-	-	0.5
(2-Ethylhexyl)Ether	-	-	-	-	-	-	-	-	-	-	-	1.32	-	-	-	-	-	-	-	-	-	-	-	0.34
p-Menthane, 2,3-dibromo-8-phenyl-	-	-	-	-	-	-	0.29	1.21	0.7	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
$\alpha$ -Elemol	-	-	-	-	-	-	0.21	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Ar-tumerone	-	-	-	-	-	-	-	0.11	0	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Androstan-17-one. 3-ethyl-3-hydroxy-(5.alpha.)-	-	-	-	-	-	-	-	0.29	0.22	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Nonadecane	-	-	-	-	-	-	-	-	-	0.44	0.59	-	1.12	0.38	1.12	-	-	-	-	-	-	0.71	0.18	-

Volatile components of *Phlomis grandiflora* var. *grandiflora* were:  $\alpha$ -pinene (25.97%),  $\alpha$ -cedrene (25.92%) and  $\alpha$ -curcumene (11.96%) in the pre-bloom period;  $\alpha$ -pinene (26.40%),  $\alpha$ -cedrene (28.15%) and  $\alpha$ -curcumene (13.92%) in the bloom period; and  $\alpha$ -pinene (18.95%),  $\alpha$ -cedrene (19.14%) and  $\alpha$ -curcumene (13.24%) in the post-emergence period.

(E)-2-hexenal, limonene and  $\beta$ -caryophyllene were volatile components of *P. leucophracta*. Specifically in the pre-bloom period: (E)-2-hexenal (7.50%), limonene (13.64%) and  $\beta$ -caryophyllene (22.32%); in the bloom period (E)-2-hexenal (8.74%), limonene (14.56%) and  $\beta$ -caryophyllene (22.45%) and in the post emergence period (E)-2-hexenal (6.10%), limonene (10.93%) and  $\beta$ -caryophyllene (20.12%) were determined.

Limonene,  $\beta$ -caryophyllene and germacrene-D were volatile components of *Phlomis lycia*. Specifically limonene (13.70%),  $\beta$ -caryophyllene (17.63%) and germacrene-D (15.66%) in pre-bloom period; limonene (17.68%),  $\beta$ -caryophyllene (23.66%) and germacrene-D (21.88%) in the bloom period and also limonene (10.65%),  $\beta$ -caryophyllene (10.63%) and germacrene-D (11.76%) in the post-emergence period.

For *Phlomis nissolii* L., the volatile components were: limonene,  $\beta$ -caryophyllene and germacrene-D. Specifically, limonene (20.65%),  $\beta$ -caryophyllene (11.28%) and germacrene-D (12.27%) in the pre-bloom period; limonene (23.75%),  $\beta$ -caryophyllene (12.50%) and germacrene D (20.73%) in the bloom period; and limonene (16.17%),  $\beta$ -caryophyllene (10.37%) and germacrene D (10.44%) in the post-emergence period.

(E)-2-hexenal, vinly amyl carbinol and germacrene-D were volatile components of *Phlomis pungens* var. *pungens*. In the pre-bloom period, (E)-2-hexenal (16.87%), vinly amyl carbinol (12.85%) and germacrene-D (7.78%); in the bloom period (E)-2-hexenal (17.60%), vinly amyl carbinol (18.44%) and germacrene-D (9.84%) and in the post-emergence period (E)-2-hexenal (12.68%), vinly amyl carbinol (18.60%) and germacrene-D (8.25%).

The volatile components of *P. samia* were  $\alpha$ -copaene,  $\beta$ -caryophyllene and germacrene-D. Specifically  $\alpha$ -copaene (9.59%),  $\beta$ -caryophyllene (13.79%) and germacrene-D (21.01%) in the pre bloom period;  $\alpha$ -copaene (10.59%),  $\beta$ -caryophyllene (15.20%) and germacrene-D (23.44%) in the bloom period and  $\alpha$ -copaene (9.71%),  $\beta$ -caryophyllene (13.75%) and germacrene-D (18.90%) in the post-emergence period.

The differences between the medians of *Phlomis* species were statistically important as found by the Kruskal-Wallis test in respect to volatile components per rates for (E)-2-hexenal,  $\alpha$ -pinene, vinly amly carbinol, limonene,  $\alpha$ -cubebene,  $\alpha$ -copaene,  $\alpha$ -cedrene,  $\beta$ -caryophyllene, germacrene-D and  $\alpha$ -curcumene (Table 3).

While there was no statistically important difference between *P. pungens* and *P. armeniaca* in respect to the ordinal method for an (E)-2-hexenal compound per the multi-comparison Bonferroni-Dunn test that was applied to determine the differences between the medians, there was a statistically significant difference between *P. pungens* and *P. bourgaei*, *P. grandiflora*, *P. leucophracta*, *P. lycia*, *P. nissolii* and *P. samia*. There was no statistically significant difference between *P. bourgaei* and *P. grandiflora*. There was no statistically significant difference between these two species and *P. lycia* and *P. samia*. There was a statistically significant difference between *P. bourgaei* and *P. grandiflora* and *P. armeniaca*, *P. leucophracta*, *P. nissolii* and *P. pungens*.

There was a statistically significant difference between *P. grandiflora* and *P. armeniaca*, *P. bourgaei*, *P. leucophracta*, *P. lycia*, *P. pungens* and *P. samia*, however,

there was no statistically significant difference for ratios of  $\alpha$ -pinene compounds according to the ordinal method between *P. grandiflora* and *P. nissolii*. While there was no statistically significant different between *P. armeniaca* and *P. samia* and *P. bourgaei* and *P. lycia*. There was a statistically significant difference between *P. armeniaca* and *P. samia* and *P. grandiflora*, *P. leucophracta*, *P. nissolii* and *P. pungens*.

**Table 3.** Kruskal-Wallis analyses results according to *Phlomis taxa*

Component	Taxa	N	N*	Mean	SEMean	StDev	Median	P	Rank
<b>(E)-2-Hexenal</b>	<i>P. armeniaca</i>	3	0	11.277	0.619	1.072	11.640	0.009	19.7ab
	<i>P. bourgaei</i>	3	0	3.28	1.47	2.55	3.82		6.0e
	<i>P. grandiflora</i> var. <i>grandiflora</i>	3	0	1.96	1.20	2.08	1.02		4.7e
	<i>P. leucophracta</i>	3	0	7.447	0.763	1.321	7.500		13.0cd
	<i>P. lycia</i>	3	0	4.85	1.00	1.73	3.92		8.7de
	<i>P. nissolii</i>	3	0	9.397	0.603	1.044	9.050		16.7bc
	<i>P. pungens</i> var. <i>pungens</i>	3	0	15.72	1.53	2.66	16.87		23.0a
	<i>P. samia</i>	3	0	5.23	1.92	3.32	3.66		8.3de
Component	Taxa	N	N*	Mean	SEMean	StDev	Median	P	Rank
<b><math>\alpha</math>-Pinene</b>	<i>P. armeniaca</i>	3	0	1.037	0.209	0.362	0.930	0.008	4.7e
	<i>P. bourgaei</i>	3	0	1.680	0.808	1.400	2.130		8.3de
	<i>P. grandiflora</i> var. <i>grandiflora</i>	3	0	23.77	2.41	4.18	25.97		23.0a
	<i>P. leucophracta</i>	3	0	2.497	0.291	0.504	2.720		12.0cd
	<i>P. lycia</i>	3	0	1.843	0.298	0.516	2.030		9.0de
	<i>P. nissolii</i>	3	0	6.740	0.156	0.271	6.860		20.0ab
	<i>P. pungens</i> var. <i>pungens</i>	3	0	4.277	0.323	0.559	4.070		17.0bc
	<i>P. samia</i>	3	0	1.187	0.562	0.973	1.100		6.0e
Component	Taxa	N	N*	Mean	SEMean	StDev	Median	P	Rank
<b>Vinly amly carbinol</b>	<i>P. armeniaca</i>	3	0	4.38	1.12	1.95	5.45	0.006	19.0ab
	<i>P. bourgaei</i>	3	0	1.403	0.833	1.442	0.940		11.0de
	<i>P. grandiflora</i> var. <i>grandiflora</i>	3	0	0.1400	0.0265	0.0458	0.1500		2.0f
	<i>P. leucophracta</i>	3	0	0.863	0.105	0.182	0.830		9.7de
	<i>P. lycia</i>	3	0	1.217	0.237	0.410	1.380		12.3cd
	<i>P. nissolii</i>	3	0	2.660	0.690	1.196	2.710		16.7bc
	<i>P. pungens</i> var. <i>pungens</i>	3	0	16.63	1.89	3.27	18.44		23.0a
	<i>P. samia</i>	3	0	0.6567	0.0546	0.0945	0.6900		6.3ef
Component	Taxa	N	N*	Mean	SEMean	StDev	Median	P	Rank
<b>Limonene</b>	<i>P. armeniaca</i>	3	0	1.207	0.261	0.452	1.400	0.011	4.7b
	<i>P. bourgaei</i>	3	0	3.52	1.73	3.00	4.60		9.3b
	<i>P. grandiflora</i> var. <i>grandiflora</i>	3	0	2.757	0.316	0.548	2.620		11.0b
	<i>P. leucophracta</i>	3	0	13.04	1.09	1.89	13.64		18.3a
	<i>P. lycia</i>	3	0	14.01	2.04	3.53	13.70		19.0a

	<i>P. nissolii</i>	3	0	20.19	2.20	3.81	20.65		22.7a
	<i>P. pungens</i> var. <i>pungens</i>	3	0	3.03	1.92	3.33	1.58		8.0b
	<i>P. samia</i>	3	0	1.787	0.311	0.538	2.030		7.0b
<b>Component</b>	<b>Taxa</b>	<b>N</b>	<b>N*</b>	<b>Mean</b>	<b>SEMean</b>	<b>StDev</b>	<b>Median</b>	<b>P</b>	<b>Rank</b>
<b><math>\alpha</math>-Cubebene</b>	<i>P. armeniaca</i>	3	0	1.440	0.237	0.411	1.310	0.008	15.0bc
	<i>P. bourgaei</i>	3	0	15.170	0.641	1.110	15.550		22.0a
	<i>P. grandiflora</i> var. <i>grandiflora</i>	3	0	0.000000	0.000000	0.000000	0.000000		2.0f
	<i>P. leucophracta</i>	3	0	3.425	0.125	0.177	3.425		19.5ab
	<i>P. lycia</i>	3	0	1.633	0.719	1.246	1.860		13.3cd
	<i>P. nissolii</i>	3	0	0.3300	0.0600	0.1039	0.2700		6.7ef
	<i>P. pungens</i> var. <i>pungens</i>	3	0	0.3633	0.0328	0.0569	0.3800		8.0de
	<i>P. samia</i>	3	0	0.877	0.252	0.437	0.920		12.0cde
<b>Component</b>	<b>Taxa</b>	<b>N</b>	<b>N*</b>	<b>Mean</b>	<b>SEMean</b>	<b>StDev</b>	<b>Median</b>	<b>P</b>	<b>Rank</b>
<b><math>\alpha</math>-Copaene</b>	<i>P. armeniaca</i>	3	0	1.043	0.126	0.218	1.110	0.006	11.3b
	<i>P. bourgaei</i>	3	0	3.423	0.275	0.476	3.200		20.0a
	<i>P. grandiflora</i> var. <i>grandiflora</i>	3	0	0.293	0.123	0.214	0.180		2.7c
	<i>P. leucophracta</i>	3	0	1.510	0.637	1.103	0.960		12.0b
	<i>P. lycia</i>	3	0	1.223	0.173	0.300	1.210		12.7b
	<i>P. nissolii</i>	3	0	1.553	0.359	0.622	1.840		14.0b
	<i>P. pungens</i> var. <i>pungens</i>	3	0	0.443	0.111	0.191	0.500		4.3c
	<i>P. samia</i>	3	0	9.963	0.315	0.546	9.710		23.0a
<b>Component</b>	<b>Taxa</b>	<b>N</b>	<b>N*</b>	<b>Mean</b>	<b>SEMean</b>	<b>StDev</b>	<b>Median</b>	<b>P</b>	<b>Rank</b>
<b><math>\alpha</math>-Cedrene</b>	<i>P. armeniaca</i>	3	0	0.000000	0.000000	0.000000	0.000000	0.006	9.0b
	<i>P. bourgaei</i>	3	0	0.183	0.183	0.318	0.000		13.0b
	<i>P. grandiflora</i> var. <i>grandiflora</i>	3	0	24.40	2.71	4.69	25.92		23.0a
	<i>P. leucophracta</i>	3	0	0.000000	0.000000	0.000000	0.000000		9.0b
	<i>P. lycia</i>	3	0	0.3533	0.0841	0.1457	0.4000		19.0a
	<i>P. nissolii</i>	3	0	0.000000	0.000000	0.000000	0.000000		9.0b
	<i>P. pungens</i> var. <i>pungens</i>	3	0	0.000000	0.000000	0.000000	0.000000		9.0b
	<i>P. samia</i>	3	0	0.000000	0.000000	0.000000	0.000000		9.0b
<b>Component</b>	<b>Taxa</b>	<b>N</b>	<b>N*</b>	<b>Mean</b>	<b>SEMean</b>	<b>StDev</b>	<b>Median</b>	<b>P</b>	<b>Rank</b>
<b><math>\beta</math>-Caryophyllene</b>	<i>P. armeniaca</i>	3	0	14.64	1.57	2.71	15.73	0.009	14.3bc
	<i>P. bourgaei</i>	3	0	19.17	2.25	3.89	20.80		18.3ab
	<i>P. grandiflora</i> var. <i>grandiflora</i>	3	0	6.03	1.13	1.96	6.40		5.0de
	<i>P. leucophracta</i>	3	0	21.630	0.756	1.309	22.320		21.3a
	<i>P. lycia</i>	3	0	17.31	3.76	6.52	17.63		16.7ab
	<i>P. nissolii</i>	3	0	11.383	0.617	1.069	11.280		9.0cd
	<i>P. pungens</i> var. <i>pungens</i>	3	0	0.930	0.236	0.410	0.740		2.0e

Component	Taxa	N	N*	Mean	SEMean	StDev	Median	P	Rank
Germacrene-D	<i>P. samia</i>	3	0	14.247	0.477	0.826	13.790		13.3bc
	<i>P. armeniaca</i>	3	0	25.23	1.09	1.89	25.03	0.004	23.0a
	<i>P. bourgaei</i>	3	0	12.91	1.16	2.00	12.41		13.3c
	<i>P. grandiflora</i> var. <i>grandiflora</i>	3	0	4.857	0.433	0.751	5.170		2.0e
	<i>P. leucophracta</i>	3	0	7.417	0.538	0.931	7.470		5.7de
	<i>P. lycia</i>	3	0	16.43	2.95	5.10	15.66		16.0bc
	<i>P. nissolii</i>	3	0	14.48	3.17	5.49	12.27		13.7c
	<i>P. pungens</i> var. <i>pungens</i>	3	0	8.623	0.623	1.080	8.250		7.3d
<i>P. samia</i>	3	0	21.12	1.31	2.27	21.01		19.0ab	
$\alpha$ -Curcumene	<i>P. armeniaca</i>	3	0	0.000000	0.000000	0.000000	0.000000	0.002	11.0b
	<i>P. bourgaei</i>	3	0	0.000000	0.000000	0.000000	0.000000		11.0b
	<i>P. grandiflora</i> var. <i>grandiflora</i>	3	0	13.040	0.575	0.995	13.240		23.0a
	<i>P. leucophracta</i>	3	0	0.000000	0.000000	0.000000	0.000000		11.0b
	<i>P. lycia</i>	3	0	0.000000	0.000000	0.000000	0.000000		11.0b
	<i>P. nissolii</i>	3	0	0.000000	0.000000	0.000000	0.000000		11.0b
	<i>P. pungens</i> var. <i>pungens</i>	3	0	0.000000	0.000000	0.000000	0.000000		11.0b
	<i>P. samia</i>	3	0	0.000000	0.000000	0.000000	0.000000		11.0b

\*a,b,c,d,e,f show differences between *Phlomis* taxa.

There was no statistically significant difference between *P. pungens* and *P. armeniaca* for vinly amly carnibol compound ratios, while there was a statistically significant difference between *P. pungens* and *P. bourgaei*, *P. grandiflora*, *P. leucophracta*, *P. nissolii* and *P. samia*. While there was no statistically significant different between *P. grandiflora* and *P. samia*. There was a statistically significant difference between *P. grandiflora* and *P. armeniaca*, *P. bourgaei*, *P. leucophracta*, *P. lycia*, *P. nissolii* and *P. pungens samia*.

There was no statistically significant difference between *P. leucophracta*, *P. nissolii* and *P. samia* for Limonene compound ratios according to the ordinal method, however there was a statistically significant difference between these three species and *P. armeniaca*, *P. bourgaei*, *P. grandiflora*, *P. lycia*, *P. nissolii* and *P. pungens*. Also there was no statistically significant difference between *P. armeniaca*, *P. bourgaei*, *P. grandiflora*, *P. lycia*, *P. nissolii* and *P. pungens*.

While there was no statistically significant difference between *P. bourgaei* and *P. leucophracta* for  $\alpha$ -cubebene compound ratios according to the ordinal method, there was a statistically significant difference between these two species and *P. armeniaca*, *P. grandiflora*, *P. lycia*, *P. nissolii*, *P. pungens* and *P. samia*. While there was no statistically significant difference between *P. grandiflora* and *P. nissolii*, there was a statistically significant difference between these two species and *P. armeniaca*, *P. bourgaei*, *P. leucophracta*, *P. lycia*, *P. pungens* and *P. samia*.

There was a statistically significant difference between these two species and *P. armeniaca*, *P. grandiflora*, *P. leucophracta*, *P. lycia*, *P. nissolii* and *P. pungens*, however, there was no statistically significant difference between *P. bourgaei* and *P. samia* for  $\alpha$ -cubebene compound ratios according to the ordinal method, while there was no statistically significant difference between *P. armeniaca*, *P. leucophracta*, *P. lycia* and *P. nissolii*, while there was a statistically significant difference between these species and *P. bourgaei*, *P. grandiflora*, *P. pungens* and *P. samia*. Also there was no statistically significant difference between *P. grandiflora* and *P. pungens*.

While there was no statistically significant difference between *P. grandiflora* and *P. lycia* for  $\alpha$ -cedrene compound ratios according to the ordinal method, there was a statistically significant difference between these two species and *P. armeniaca*, *P. bourgaei*, *P. leucophracta*, *P. nissolii*, *P. pungens* and *P. samia*. There was no statistically significant difference between *P. armeniaca*, *P. bourgaei*, *P. leucophracta*, *P. nissolii*, *P. pungens* and *P. samia*.

There was no statistically significant difference between *P. bourgaei*, *P. leucophracta* and *P. lycia* for  $\beta$ -caryophyllene compound ratios according to the ordinal method, while there was a statistically significant difference between these species and *P. armeniaca*, *P. grandiflora*, *P. nissolii*, *P. pungens* and *P. samia*. While there was no statistically significant difference between *P. grandiflora* and *P. pungens*, there was a statistically significant difference between these two species and *P. armeniaca*, *P. bourgaei*, *P. leucophracta*, *P. lycia*, *P. nissolii* and *P. samia*.

There was no statistically significant difference between, *P. armeniaca* and *P. samia* for germacrene-D compound ratios according to the ordinal method. There was a statistically significant difference between *P. armeniaca* and *P. bourgaei*, *P. grandiflora*, *P. leucophracta*, *P. lycia*, *P. nissolii*, *P. pungens* and *P. samia*. While there was no statistically significant difference between *P. bourgaei* and *P. nissolii* and *P. lycia*, statistically significant difference between *P. bourgaei* and *P. nissolii* and *P. armeniaca*, *P. grandiflora*, *P. leucophracta*, *P. pungens* and *P. samia*. While there is not statistically significant difference between *P. grandiflora* and *P. leucophracta*, there was a statistically significant difference between *P. grandiflora* and *P. armeniaca*, *P. bourgaei*, *P. lycia*, *P. nissolii*, *P. pungens* and *P. samia*.

There was a statistically significant difference between *P. grandiflora* and *P. armeniaca*, *P. bourgaei*, *P. leucophracta*, *P. lycia*, *P. nissolii*, *P. pungens* and *P. samia* for  $\alpha$ -curcumene component ratios per the ordinal method. There was no statistically significant difference between *P. armeniaca*, *P. bourgaei*, *P. leucophracta*, *P. lycia*, *P. nissolii*, *P. pungens* and *P. samia*.

Differences between the essential compounds of  $\beta$ -caryophyllene and germacrene-D were statistically important per the Friedman test in respect to volatile components by rates. The multi comparison Bonferroni-Dunn test was applied to determine the difference between the medians and found that there was a statistically significant difference between the pre bloom period, bloom period and post-emergence period for  $\beta$ -caryophyllene compound ratios according to an ordinal method. There was no statistically significant difference between the pre bloom period, bloom period and post-emergence period for germacrene-D component ratios according to an ordinal method (Table 4).



**Table 4.** Friedman testi results according to vegetation periods

Component	Period	N	N*	Mean	SEMean	StDev	Median	P	Rank
β-Caryophyllene	Before flowering	8	0	13.76	2.51	7.10	14.76	0.001	17.0b
	Flowering	8	0	15.03	2.83	7.99	15.91		23.0a
	After flowering	8	0	10.71	2.16	6.12	11.09		8.0c
Component	Period	N	N*	Mean	SEMean	StDev	Median	P	Rank
Germacrene-D	Before flowering	8	0	12.88	2.44	6.89	12.34	0.010	12.0b
	Flowering	8	0	16.47	2.84	8.03	17.93		23.0a
	After flowering	8	0	12.31	2.31	6.52	10.82		13.0b

\*a,b,c show differences between vegetation periods

## Discussion and Conclusion

Yaşar et al. (2010) determined 12 volatile components for *P. armeniaca*. The main components were germakren-D (35.68%), β-caryophyllene (18.08%), caryophyllene oksit (13.35%), (E)-β-farnesene (7.24%) and hexahydrofarnesyl aseton (6.99%). They also determined that germakren-D was the dominant distinguishing compound of *P. armeniaca*. The results supported our thesis. Germakren-D was determined as dominant distinguishing compound.

Sixty-two volatile components of *Phlomis bourgaei* were found thru SPME analysis and these components of *P. bourgaei* had the following ratios: α-cubebene (16.04%), β-caryophyllene (21.98%) and germakren-D (15.12%). Sarıkurkcu et al. (2013) determined the chemical compounds of volatile oils of *P. bourgaei* by hydro distillation and examine anti-toxicant potentials. The main components were: β-caryophyllene (37.37%), (Z)-β-farnesene (15.88%) and germakren-D (10.97%). Baser et al. (2008) determined the components of *P. bourgaei* as germakren-D (11.3%) and β-caryophyllene (112%). β-caryophyllene and germakren-D were the main compounds. These findings support our research results. The only difference was that we determined that the α-cubebene compound was the dominant compound.

In our research α-pinene (26.40%), α-cedrene (28.15%) and α-curcumene (13.92%) were volatile components of *Phlomis grandiflora* var. *grandiflora* among 60 components as determined by SPME analysis. Celik et al. (2005) determined components of *P. grandiflora* var. *grandiflora* as germakren-D (%45.4%), β-caryophyllene (22.8%) and bicycle germakren (4.9%). Demirci et al. (2008) found that β-eudesmol (42%) and α-eudesmol (16%), which are oxygenic sesquiterpenes as the most important compounds of *P. grandiflora* var. *grandiflora*. Ozcan et al. (2009) determined 32 components that represent 92.1 % oil obtained from flowers. They determined the essential components as β-eudesmol (61.48%), β-curcumene (5.81%), E-β-farnesene (2.35%), α-zingiberene (2.18%) and α-cedrene (1.94%). They found 39 compounds that represent 87.7% of oil obtained from leaves. Volatile components were β-eudesmol (62.04%), β-curcumene (3.43%), α-curcumene (2.20%) and linalool (2.03%). It has been characterized that there are high percentages of β-eudesmol in both oils. The results of our study differ from the study of Celik et al. (2005). Meanwhile, Ozcan et al. (2009) found α-cedrene and α-curcumene as the main compounds in their studies. This

result supports our study. In other research,  $\alpha$ -pinene has been found as the dominant component.

Seventy volatile components of *Phlomis leucophracta* were determined by SPME analysis and the essential compounds were (E)-2-hexenal (8.74%), limonene (14.56%) and  $\beta$ -caryophyllene (22.45%). Celik et al. (2005) found  $\beta$ -caryophyllene (20.2%),  $\alpha$ -pinen (19.2%) and limonene (11%) in *P. leucophracta*.  $\beta$ -caryophyllene and limonene were the volatile components. The result of this research supports our study. In other studies, (E)-2-hexenal was among the main component.

Sixty-two volatile components of *Phlomis lycia* were determined with SPME analysis and the main components of *P. lycia* were Limonene (17.68%),  $\beta$ -caryophyllene (23.66%) and germacrene-D (21.88%).

Fifty-three volatile components of *Phlomis nissolii* were determined with SPME analysis and the main components of *P. nissolii* were limonene (23.75%),  $\beta$ -caryophyllene (12.50%) and germacrene-D (20.73%) Kirimer et al. (2006) found 18 compounds thru GC/MS analysis. They found that the main components were germacrene-D (33.9%), bicycle germacrene (15.3%) and (Z)-  $\beta$ - farnesene (10.7%). Their results differ from our study. However, germacrene-D was found as a main component in both studies.

Seventy volatile compounds of *Phlomis pungens* var. *pungens* were determined and the main compounds were (E)-2-hexenal (17.60%), vinly amyl carbinol (18.44%) and germacrene-D (9.84%). Masoudi et al. determined 24 compounds in Iran for *P. pungens* var. *pungens* thru GC/MS analysis. The volatile components included bicycle germacrene (14.1%),  $\alpha$ -pinen (13.5%) and (E)- $\beta$ -farnese (13.5%). These results differ from our study.

A total of 64 volatile components of *Phlomis samia* were determined by the SPME analysis. The main components were  $\alpha$ -copaene (10.59%),  $\beta$ -caryophyllene (15.20%) and germacrene-D (23.44%). Aligiannis et al. (2004) determined 72 components of upper sections of *P.samia* which grow in Greece and was represented at 67.4% by  $\alpha$ -pinen, limonen,  $\beta$ - caryophyllene, linaol, (E)- $\beta$ -farnesene, germacrene -D, (Z)- $\gamma$ -bisabolene and cis- $\beta$ -ocimene. Demirci et al. (2006) determined germacrene -D (33.8%) and  $\beta$ - caryophyllene (6.4%) as the main components of *P. samia* with GC/MS analysis. The above mentioned results support our results. Additionally, the  $\alpha$ -copaene compound was found.

The result of Kruskal-Wallis test found; 2-butenal, n-pentenal, trans-3-penten-2-one, 3-methyl-1-butanol, n-hexanal, (E)-2-hexenal, cis-hex-2-en-1-ol, hexanol <n->, amyl methyl ketone, n-heptanal, 2,4-hexadienal,  $\alpha$ -thujene,  $\alpha$ -pinene, (E)-2-heptenal, sabinene,  $\beta$ -phellandrene,  $\beta$ -pinene, amyl vinyl ketone, vinly amly carbinol, 6-methyl-5-hepten-2-one,  $\beta$ -myrcene, n-octanal,  $\alpha$ -phellandrene, 2,4-heptandienal, p-dichlorobenzene,  $\alpha$ -terpinene, p-cymene, limonene, (E)-3-octen-2-one, cis-ocimene, 2 octenal,  $\gamma$ -terpinene,  $\gamma$ -terpinene, octanol,  $\alpha$ -terpinolene, dimethylstyrene < $\alpha$ -para->, 2-nonanone, methyl benzoate, n-nonanal, 2-hendecanone,  $\alpha$ -campholene aldehyde, trans-alloocimene, trans-2-nonenal, pinocarvone, (E)-2-nonenal, (E)-2-nonenal, n-decanal, (E)-2-decenal,  $\alpha$ -cubebene,  $\alpha$ -copaene, (-)- $\beta$ -elemene, sesquithujene <7-epi->,  $\alpha$ -gurjunene,  $\alpha$ -cedrene,  $\beta$ -caryophyllene,  $\gamma$ -elemene,  $\alpha$ -bergamotene, (E)- $\beta$ -farnesene, farnesol, epi-bicyclosesquiphellandrene, germacrene-D, ionone,  $\alpha$ -curcumene, bicyclogermacrene, zingiberene,  $\alpha$ -muurolene,  $\beta$ -muurolene, cedr-8-e,  $\gamma$ -cadinene,  $\delta$ -cadinene,  $\beta$ -sesquiphellandrene, (-)-caryophyllene oxide, p-menthane, 2,3-dibromo-8-

phenyl, androstan-17-one ve 3-ethyl-3-hydroxy and nonadecane components, and the differences between medians of *Phlomis* species were statistically significant.

The differences between medians of vegetation periods were statistically significant for hexanol <n->, n-heptanal,  $\beta$ -caryophyllene and germacrene-D components by Friedman test.

As a result, it is significantly important to reap samples of *Phlomis* taxa in the bloom period in respect to volatile compound productivity. These results are important in providing guidance for local growers and traders to reduce untimely plant picking and to reduce economic losses.

The leaves and flowers of *Phlomis* are used for several purposes, from making products more appetizing, as an anti-allergic, as a diuretic, for diarrhea preventive, against stomach aches, to relieve pain, anti-diabetic, and for herbal teas and tonics. Additionally, taxa that have yellowish flowers are important for food coloring. As such, research and further studies should be pursued in order to investigate how various plants can be used as a raw material for medicine or as food and cosmetic products. Importantly, future research will promote further application.

**Acknowledgments.** This study was a part of PhD thesis of first author Ayse Gul SARIKAYA. We express our sincere appreciation to Suleyman Demirel University for their financial support by project which numbered as 3360-D2-12.

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