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Determination of Volatile Oil Components in Different parts of *Lavandula angustifolia* Mill.

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Abstract

This study investigates the chemical composition of volatile oils extracted from stem, leaf and flower parts *Lavandula angustifolia* Mill. This study was carried out at the trial plots of Recep Tayyip Erdogan University Faculty of Agriculture in 2020. The active ingredient content of the lavender plant was determined by harvesting the stem, leaf and flowers during the full blooming period. The oil was extracted using the hydrodistillation method, with a NeoClevenger device. The essential oil fire in the oil harvested during the full decomposition period were determined by using SPME (Solid Phase Microextraction) method using Gas Chromatography (GC-MS) device. As a result of the analysis, nearly 50 different volatile oil components were detected in all investigated plant parts. The obtained max.-min. values were as following: Camphor (33.1-13.77 %), Eucalyptol (27.91-13.53 %), Linalool (12.69-1.61 %), γ-Cadinene (5.83-0.88 %), β-Pinene (6.16-0.32 %), α-Terpineol (5.79-1.96 %), α-Bergamotene (5.7-0.89 %), and Caryophyllene oxide (5.25-0.26 %). Different components of medicinal importance could be detected in different parts of this species. These compounds could be isolated and used for further basic investigations.

Keywords: GC-MS, Lavender, chemical composition

Introduction

Throughout the beginning of time, medicinal and aromatic plants have served a variety of purposes. More recently, they have gained popularity as profitable crops used to produce essential oils all over the world (Bajalan et al., 2016). Because they give off the idea of promoting a "well-being" lifestyle, the essential oils these plant have recently gained commercial popularity (Yang et al., 2010).

With its 39 species, multiple hybrids, and over 400 officially recognized cultivars, the genus Lavandula (Lamiaceae family) is one of the best-known medicinal plants in the world (Ben Abdelkader et al., 2011). The principal producing areas are Asia, the Middle East, and Northern Africa. Britain and France. Although they may not produce as much, Morocco, Yugoslavia, Hungary, Italy, Russia, Spain, Romania, Ukraine, and Turkey do produce in varying amounts (Zheljazkov et al., 2013).

The genus Lavandula contains short-lived, shrub-like perennials, annuals, and herbaceous perennials (Upson and Andrews 2004). In temperate areas, a large number of members of the genus are grown for their ornamental uses in gardens and landscapes as well as for their essential oils. The calming, antispasmodic, anticonvulsant, analgesic, antioxidant, and local anesthetic action of lavandula species are among their many pharmacological properties. They are also employed medically (Kovatcheva et al., 2001; Kageyama et al., 2012).

Since ancient times, the distilled volatile oil of this plant has been extremely important in aromatherapy as well as the perfume, cosmetic, and flavoring industries. Additionally, the food and pharmaceutical industries place a specific emphasis on the volatile components of lavender (Evandri et al., 2005; Cong et al., 2008).

Exogenous and endogenous variables both influence the chemical makeup of volatile oils. Exogenous or environmental-regulated elements (light, precipitation, growth site, and soil) might change the qualitative/quantitative amount of volatile oil in plants. Endogenous variables, on the other hand, are inextricably linked to plant anatomy and physiology. These are also linked to genetically-related chemical variation between different parts of the plant (Barra et al., 2009).

The structures of the characteristic components of important essential oils of *L. angustifolia* Mill are given in Fig. 1. These components are the most present volatile oils in the Lavandula genus.

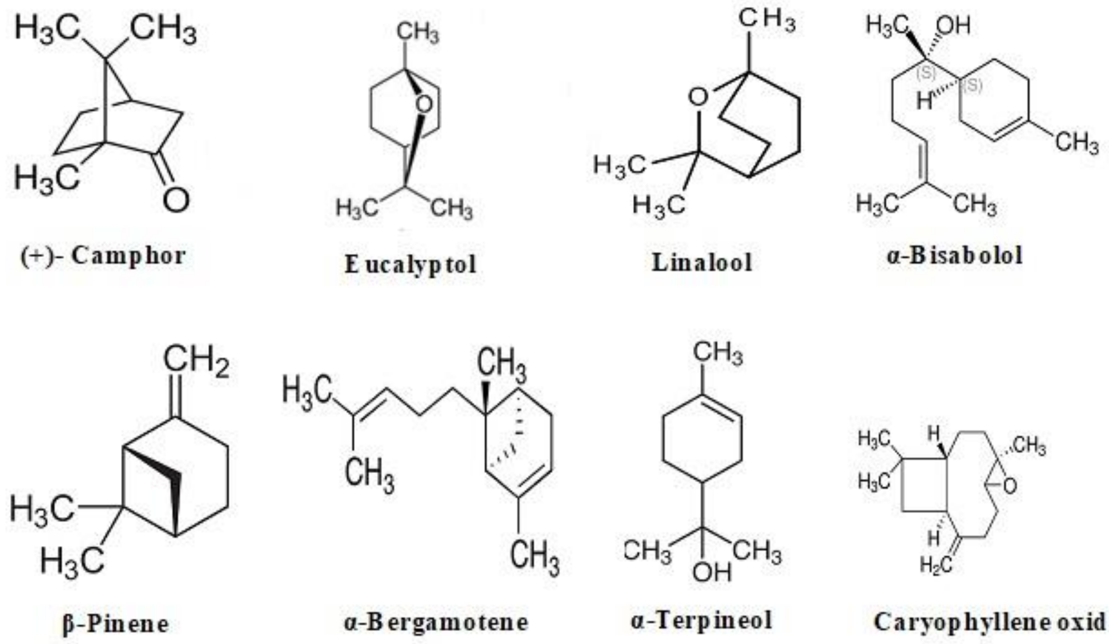

Figure 1. Structures of the characteristic components of essential oils of *L. angustifolia*.

The aim of our study is to examine the volatile oil composition of different parts (stem, laf and flower) of *L. angustifolia* Mill in Rize ecological conditions, comparing the obtained results with the literature, considering them in terms of current industry standards and to bring them to the regional economy.

Material and Methods

2.2. Sample preparation and volatile oil analysis

A GC-MS Device (Shimadzu, Japan) equipped with SPME was used for analysis. 1.00 g weighted plant samples were grounded and placed afterwards in a 10 mL vial sealed with a silicone-rubber septum cap. Manufacturer instructions were used pre-conditioning the fiber. At equilibrium, the fiber was exposed to the headspace for 1 min at room temperature. After sampling, the fiber was put into the needle and transferred afterwards to the injection port of the GC or GC–MS system. A CP 5MS (30 m x 0.25 mm i.d., film thickness 0.25 μm) column was used. Oven temperature was programmed from 40°C to 240°C at 2°C/min, then isothermal at 220 °C for 20 min. Helium was used as a carrier gas with a constant flow at 1 mL/min. The temperature of injector and detector was 240°C. The determination of essential oil components was done using the Wiley, Nist Mass Spectral and aroma method databases (Yurteri et al., 2021).

Data Analysis

Biplot Analysis were performed using the XLSTAT 2023 (Addinsoft, 2023). Statistical Program to visualize present variation in *Lavandula angustifolia* Mill. plant parts investigated for chemical variability. Scatter plot diagrams were created using current data (Backhaus et al., 1989). Based on GC-MS analysis data Separate Biplot Diagram was also created.

Results and Discussion

The volatile composition of investigated plant parts of *L. angustifolia* Mill. are given in Table 1. Based on obtained results, we can say that specific components were detected only in stem, leaf and flower parts seperately. Totally 70 volatile oil components were detected.

15 components, namely, α-Pinene, Camphene, Sabinene, Vinyl amyl carbinol, γ-Terpinene, Fenchone, Terpinen-4-ol, Dihydromyrcenyl acetate, Caryeol <trans->, Sabinene hydrate <cis-> and Carvone, were only detected in leaf parts. 10 components were identified only in flower part: Linalool oxide <trans->, Hex-3 (Z)-Enyl isobutanoate, Phytol acetate, Bornyl isovalerate, β-Bourbonene, Thujone, β-Chamigrene, β-Bergamotol, β-Cedrene and Isopulegyl acetate. Stem part contained 19 volatile oil components; Limonene, Sabinene hydrate <cis>, Pinocarveol <trans>, Theaspirane <6-hydroxydihydro, Verbenol, Lavandulyl acetate, Tridecane, Neryl acetate, Geranyl propanoate, Caryophyllene, Coumarin, Bicyclogennacloene, β- Elemene, Spathulenol, Pentadecane, β-Eudesmol, Azulene, (Z)-Jasmone and Hemianin.

15 volatile oil components could be detected in all plant parts, namely β-Pinene, Eucalyptol, Sabinene hydrate <trans->, Linalool, Camphor, Pinocarvone, α-Terpineol, 1,4 Cineole, Myrtenol, α-Bergamotene, β-Phellandrene, (E)-β-Famescene, β-Selinene, α-Muurolol and α-Bisabolol.

Highest amounts were detected with 17.47 % for Eucalyptol and with 13.77 % for Camphor in leaves, with 14.71 % for Camphor and 13.53 % for Eucalyptol in flowers and with 33.01% for Camphor and 27.91 % for Eucalyptol in stem. All plant parts varied in their volatile oil composition.

Table 1: Volatile oil composition of *L. angustifolia* Mill. plant parts

			Lavandula angustifolia Mill.		
Numeric	Retation Time	% Component	Leaf	Flower	Stem
1	9.190	α-Pinene	4.02	0	0
2	9.715	Camphene	1.21	0	0
3	10.698	Sabinene	1.91	0	0
4	10.794	β-Pinene	6.16	0.34	0.32
5	10.986	Vinyl amyl carbinol	0.62	0	0
6	11.410	Myrcene	1.00	0	0
7	12.815	Limonene	0	0	0.31
8	13.054	Eucalyptol	17.47	13.53	27.91
9	14.006	γ-Terpinene	0.39	0	0
10	14.311	Sabinene hydrate <trans->	2.45	1.09	0.55
11	14.535	Linalool oxide <trans->	0.59	1.53	0
12	15.112	Fenchone	1.46	0	0
13	15.151	Linalool oxide <cis>	0	2.91	0
14	15.153	Sabinene hydrate <cis>	0	0	0.37
15	15.679	Linalool	3.19	12.69	1.61
16	15.851	Hex-3 (Z)-Enyl isobutanoate	0	0.34	0
17	16.546	Verbenone	0.45	0.45	1.20
18	16.599	Pseudolimonene	0.35	0.45	0
19	17.071	Pinocarveol <trans>	0	0	0.38
20	17.423	Camphor	13.77	14.71	33.01
21	17.991	Pinocarvone	1.15	0.79	0.44
22	18.148	Isoborneol	0	2.25	0.99
23	18.188	α-Terpineol	5.79	3.47	1.96
24	18.573	Terpinen-4-ol	0.35	0	0
25	18.882	Dihydromyrcenyl acetate	0.63	0	0

26	18.982	Myrtenol	1.6	1.49	0.62
27	19.302	Hept-5-en-2-one <6-methyl->	0	0.99	0.30
28	19.870	Theaspirane <6-hydroxydihydro	0	0	0.53
29	20.130	Caryeol <trans->	0.30	0	0
30	20.916	Sabinene hydrate <cis->	0.31	0	0
31	20.927	Verbenol	0	0	0.41
32	21.040	Carvone	0.38	0	0
33	21.418	Piperitone	0.35	0	0
34	21.420	Lavandulyl acetate	0	0	0.67
35	22.130	Phytol acetate	0	0.39	0
36	22.230	Tridecane	0	0	0.22
37	23.822	Bornyl isovalerate	0	0.35	0
38	23.922	Neryl acetate	0	0	0.56
39	25.930	Geranyl propanoate	0	0	0.76
40	26.114	β-Bourbonene	0	1.37	0
41	26.214	Caryophyllene	0	0	1.18
42	26.261	Coumarin	0	0	2.29
43	26.361	Thujone	0	1.02	0
44	27.111	α-Bergamotene	5.7	1.75	0.89
45	27.113	β-Phellandrene	1.22	0.94	0.33
46	27.295	Aromadendrene	2.22	0	0
47	27.297	β-Chamigrene	0	2.89	0
48	27.397	(E)-β-Famescene	1.46	3.43	0.49
49	28.023	β-Selinene	0.68	0.35	0.40
50	28.451	Bicyclogenaclœne	0	0	0.48
51	28.924	β- Elemene	0	0	0.77
52	29.320	Neryl isovalerate	0	1.87	1.11
53	29.420	Germacrene D	0	3.71	1.89
54	29.894	Germacrene B	1.35	0	0
55	30.144	Bisabolene <beta->	1.39	0	0
56	30.250	β-Bergamotol	0	0.35	0
57	30.350	Spathulenol	0	0	0.34
58	30.369	γ-Cadinene	5.83	2.63	0.88
59	30.624	β-Cedrene	0	0.74	0
60	31.205	Pentadecane	0	0	0.23
61	31.207	α-Humulene	1.40	0.64	0
62	31.569	Caryophyllene oxide	2.63	5.25	0.26
63	32.551	β-Eudesmol	0	0	0.23
64	34.238	α-Muurolol	1.72	0.91	0.72
65	34.654	α-Bisabolol	2.22	2.73	1.86
66	35.121	Azulene	0	0	0.22
67	36.226	Isopulegyl acetate	0	0.41	0
68	36.326	(Z)-Jasmone	0	0	0.58
69	37.245	Hemianin	0	0	0.44
70	37.246	Lanceol <cis->	0.55	0.44	0

The Biplot Analysis of *L. angustifolia* Mill. Plant parts is given in Fig.2.

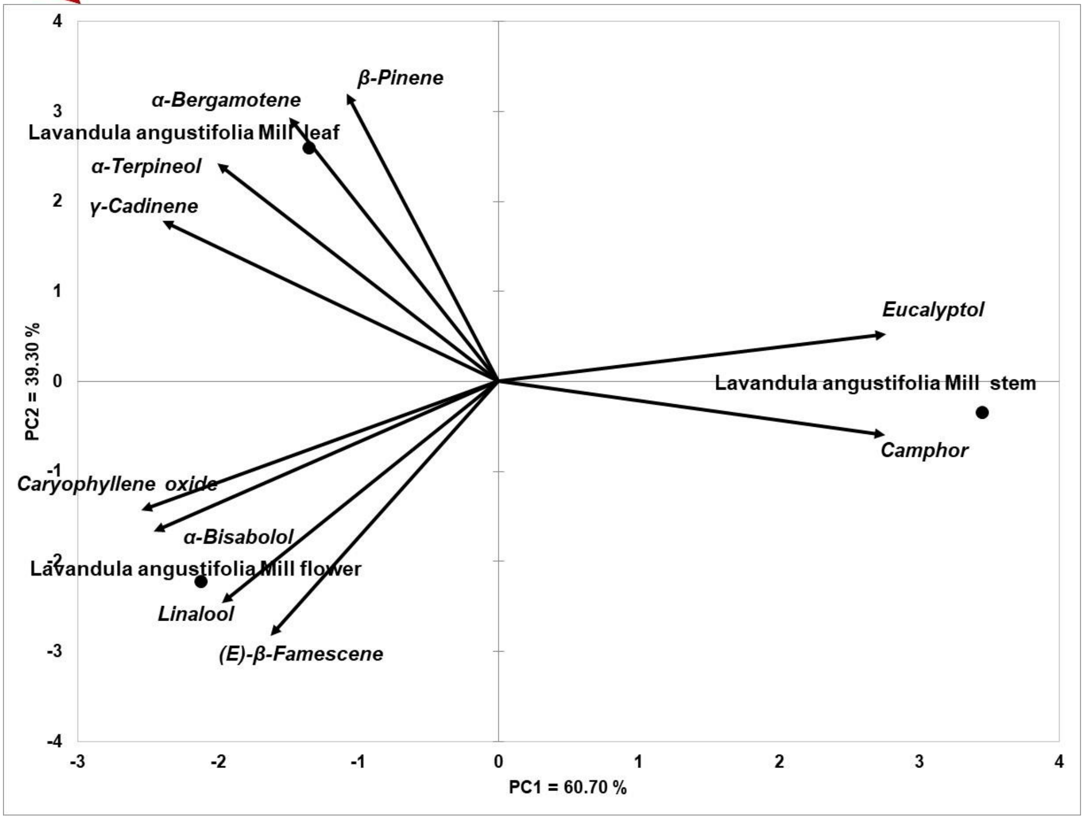

Figure 2: Biplot Analysis of *L. angustifolia* Mill. plant parts based on determined volatile oil composition

In creating Biplot the most present volatile oil components, namely Camphor, Eucalyptol, Linalool, α-Bisabolol, Caryophyllne oxide, β-Pinene, α-Temineol, α-Bergamotene, (E)-β-Famescene and γ-Cadinene were used. Total 100 % o the present variation could be expalined using the first two principal components.

Specially the stem part could clearly differentiated from the leaf and flower parts, based on Eucalyptol and Camphor. B-Pinene, α-Bergamotene, α-Terpineol and γ-Cadinene were helpful in distingushing the leaf part. Further, the flower parts displayed different volatile oil composition regarding Caryopyllene oxide, α-Bisabolol, Linalool and (E)- β-Famescene (Fig. 2).

In research conducted with Ukrainian cultivars, Pokajewicz et. al. found that Linalool (11.42–44.05%), (-)-Terpinen-4-ol (1.17–11.25%), and Linalyl acetate (15.79–35.27%) as the main chemical compounds (Pokajewicz et al., 2021). Moreover, Dong et al. (2020) reported Linalool (19.71%), Linalyl acetate (26.61%), *α*-Terpineol (3.61%), and Lavandulyl acetate (12.68%) as the main compounds. In addition, Adaszynska et al. (2013) reported that Linalool (15.85–23.88%) and Linalyl anthranilate (1.58–12.78%) are the main ingredients together with geraniol acetate (2.37–10.61%), Caryophyllene (2.78–6.24%), 1-Terpinen-4-ol (5.53–9.73%), and p-Menth-1-en-8-ol (3.98–7.94%).

According to the European Pharmacopoeia, the relative content of lavender oil should be Limonene (maximum 1.0%), Eucalyptol (maximum 2.5%), 3-Octanone (0.1–5.0%), Camphor (maximum 1.2%), Linalool (20.0–45.0%), Linalyl acetate (25.0–47.0%), Terpinen-4-ol (0.1–8.0%), Lavandulyl acetate (minimum 0.2%), Lavandulol (minimum 0.1%), and *α*-Terpineol (maximum 2.0%) (Lavender, 2019).

The obtained max.-min. values in our study were as following: Camphor (33.1-13.77 %), Eucalyptol (27.91-13.53 %), Linalool (12.69-1.61 %), γ-Cadinene (5.83-0.88 %), β-Pinene (6.16-0.32 %), α-Terpineol (5.79-1.96 %), α-Bergamotene (5.7-0.89 %), and Caryophyllene oxide (5.25-0.26 %). These results are in conform with the European Pharmacopoeia values for Lavender

The chemical composition of volatile oils is affected by environmentally-regulated factors (light, precipitation, growing site, and soil) and endogenous factors (anatomical and physiological characteristics of the plants). This leads to chemical variation between different parts of the plants (Barra, 2009). Many studies showed that chemical composition varies depending on the plant parts and the developing stage of the plant at harvest time (Amin and Sleem, 2007; Yili et al., 2006; Faber et al., 1998; Hodisan et al., 1980).

Principal Component Analysis (PCA) and Cluster Analysis tools are helpfull in genotype characterization and related grouping calculated on similarity basis (Mohammadi and Prasanna, 2003; Peeters and Martinelli, 1989). PCA analysis can be used in the differentation of plant materials, further differences of various species based on their chemical composition could be achieved (Smelcerovic et al., 2008; Bertoli et al., 2011). If these two methods are combined characters which are critically contributing for genetic variability in crops can be analysed (Rachovska et al., 2003). Biplot is a further step in PCA, where factors contributing to the differentiation of obtained variation another could be grouped and detected (Aghaee et al., 2010). Investigated *L. angustifolia* Mill. plant parts could be clearly differentiated based on their volatile oil composition. Especially the stem part of this plant species differed based on volatile oil composition from stem and leaf parts. In the present study, besides chemical content analysis statistical tools were used to evaluate the volatile oil composition of *L. angustifolia* Mill. Plant parts.

Conclusion

50 different volatile oil components were determined in investigated plant parts of *L. angustifolia*. The most detected components given with max.-min. values were Camphor (33.1-13.77 %), Eucalyptol (27.91-13.53 %), and Linalool (12.69-1.61 %), γ-Cadinene (5.83-0.88 %), β-Pinene (6.16-0.32 %), α-Terpineol (5.79-1.96 %), α-Bergamotene (5.7-0.89 %), and Caryophyllene oxide (5.25-0.26 %).

Lavender essential oils have been intensively investigated for various applications and their medicinal properties have been proved, in the present study different components of medicinal importance could be detected in different parts of *L. angustifolia*. These detected components could be isolated and used for further basic investigations in this species.

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تحديد مكونات الزيت الطيار في أجزاء مختلفة من مطحنة Lavandula angustifolia.

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الخلاصة

تبحث هذه الدراسة في التركيب الكيميائي للزيوت الطيارة المستخرجة من أجزاء الساق والأوراق والزهرة Lavandula angustifolia Mill. تم إجراء هذه الدراسة في الأراضي التجريبية لكلية الزراعة بجامعة رجب طيب أردوغان في عام ٢٠٢٠. وتم تحديد محتوى العنصر النشط لنبات الخزامى من خلال حصاد الجذع والأوراق والزهور خلال فترة الإزهار الكاملة. تم استخلاص الزيت بطريقة التقطير المائي بجهاز نيوكليفنجر. تم تحديد حريق الزيت العطري في الزيت المحصود خلال فترة التحلل الكامل باستخدام طريقة SPME (الاستخلاص الدقيق للمرحلة الصلبة) باستخدام جهاز كروماتوغرافيا الغاز (GC-MS). ونتيجة للتحليل، تم اكتشاف ما يقرب من ٥٠ مكونًا مختلفًا من الزيوت الطيارة في جميع أجزاء النبات التي تم فحصها. الحد الأقصى الذي تم الحصول عليه-دقيقة. وكانت القيم كما يلي: الكافور (٣٣.١-١٣.٧٧%)، اليوكاليبتول (٢٧.٩١-١٣.٥٣%)، لينالول (١٢.٦٩-١.٦١%)، جاما-كادينين (٥.٨٣-٠.٨٨%)، بيتا-بينين (٦.١٦-٠.٣٢%) ، ألفا-تيربينول (٥.٧٩-١.٩٦%)، ألفا-بيرجاموتين (٥.٧-٠.٨٩%)، وأكسيد كاريوفيلين (٥.٢٥-٠.٢٦%). يمكن اكتشاف مكونات مختلفة ذات أهمية طبية في أجزاء مختلفة من هذا النوع. ويمكن عزل هذه المركبات واستخدامها لإجراء مزيد من التحقيقات الأساسية.

الكلمات المفتاحية: GC-MS، الخزامى، التركيب الكيميائي