JOURNAL of THE TURKISH CHEMICAL SOCIETY
SECTION: A
CHEMISTRY
TURKISH CHEMICAL SOCIETY

Volatile compounds of the *Lavandula angustifolia* Mill. (Lamiaceae) Species Cultured in Turkey

Sevim Küçük[1]*, Esra Çetintaş[1], Mine Kürkçüoğlu[2]

[1]Department of Pharmaceutical Botany, Faculty of Pharmacy, Anadolu University, Eskişehir, Turkey
[2]Department of Pharmacognosy, Faculty of Pharmacy, Anadolu University, Eskişehir, Turkey

Abstract: Within this family there are many medical species such *as Lavandula* L.*, Lavandula angustifolia* Mill. known as medical lavender, is conducting culture and research studies in many places in the world. It is also known as the "English lavender" or *L. officinalis*. Lavender is used in aromatherapy. Its sedative nature, on inhalation has been shown both in animals and man. Experimental studies in humans and animals have shown that the sedative effect comes from linalool and linalyl acetate. Tanins contained in flowers of lavender is shown antidiarrheal effect. Essential oil of lavender is used in various skin diseases and wound healing. Essential oil of lavender shortens the sleeping period, prolongs the sleeping period. In addition, it also shows antimicrobial, antiinflammatory, fungicidal, insecticidal and acaricidal effects (Zeybek and Haksel, 2010). In this study, we were made to create sources Lavandula agriculture in Turkey. We collected four different provinces in the *Lavandula (*5 samples and 1 commercial oil). The results of a total five local and commercial are given below. The flowers of Lavandula were water distilled for 3 h using a Clevenger type apparatus. The essential oils were analyzed by GC and GC-MS simultaneously. The main constituents were identified as linalool 31.9–50.0 % and linalyl acetate 15.4–42.0 %.

Keywords: *Lavandula,* Lamiaceae, Essential oils, Medicinal and aromatical plant, Cultivation, GC-MS.

Submitted: September 25, 2018. **Accepted:** November 08, 2018.

Cite this: Küçük S, Çetintaş E, Kürkçüoğlu M. Volatile compounds of the Lavandula angustifolia Mill. (Lamiaceae) Species Cultured in Turkey. JOTCSA. 2018;5(3):1303–8.

DOI: http://dx.doi.org/10.18596/jotcsa.463689.

***Corresponding author.** E-mail: (salan@anadolu.edu.tr), Tel: +90 545 299 72 14.

INTRODUCTION

The Lamiaceae family spans all regions of the earth, especially the Mediterranean region. It is one of the largest families with approximately 224 genera and 5600 species in the world. (1) In Turkey, 45 genera, 565 species and with 735 taxa is one of the important families. (2). The genus *Lavandula* L. (Lamiaceae) is represented in Turkey by 3 taxa (*Lavandula angustifolia* Mill. subsp. *angustifolia*, *L. pedunculata* (Mill.) subsp. *cariensis* (Boiss.), *L. stoechas* L. subsp. *stoechas*). *L. angustifolia* is locally known as "lavanta", *L. pedunculata* subsp. *cariensis* is locally known as "karan", *L. stoechas* subsp. *stoechas*. is locally known as "karabaş" (3,4).

Within this family there are many medical species such *as Lavandula* L., *Lavandula angustifolia* Mill. known as medical lavender, is conducting culture and research studies in many places in the world. Lavender grows very well at the arid field and then this plant is an economical. It is also known as the English lavender or *L. officinalis*. Lavender is used in aromatherapy. Its sedative nature, on inhalation has been shown both in animals and man. Experimental studies in humans and animals have shown that the sedative effect comes from linalool and linalyl acetate. Tanins contained in flowers of lavender is shown antidiarrheal effect. Essential oil of lavender is used in various skin diseases and wound healing. Essential oil of lavender shortens the sleeping period, prolongs the sleeping period. In addition, it also shows antimicrobial, antiinflammatory, fungicidal, insecticidal and acaricidal effects (5).

We collected four different Lavandula species (5 samples and 1 commercial oil). We aimed to identify and compare the compounds in essential oils. Lavender adapts very well to the arid area, so it is an economical plant. In Turkey, the results of a total five local and commercial are given below.

MATERIAL AND METHODS

Plant material

L. angustifolia were collected in 26 June, 2017 in Edirne, 8 July 2017 in Burdur, 10 July 2017 in Yalova, Turkey. Collected plant samples were identified and prepared voucher specimens are kept at the Herbarium of Faculty of Pharmacy of Anadolu University, Turkey. ESSE No: A: Yalova-Greenhouse 15485, B: Yalova 15422, C: Edirne 15421, D: Burdur-Jubileuo 15486, E: Burdur-Sevtapolis 15487.

A: Yalova- Greenhouse (yield of essential oil: 4.1%)
B: Yalova (yield of essential oil: 5.0%)
C: Edirne (yield of essential oil: 4.0%)
D: Burdur-Jubileuo (yield of essential oil: 3.5%)
E: Burdur-Sevtapolis (yield of essential oil: 6.0%)
F: Commercial oil

Isolation of essential oil

The flowers of Lavandula were water distilled for 3 h using a Clevenger type apparatus. The essential oils were analyzed by GC and GC-MS simultaneously. The essential oils were stored at 4°C in the dark until analyzed.

GC and GC-MS conditions

The oils were analyzed by Gas Chromatography (GC) and Gas Chromatography-Mass Spectrometry (GC-MS) using an Agilent GC-MSD system (Mass Selective Dedector-MSD).

GC-MS analysis

The GC-MS analysis was carried out with an Agilent 5975 GC-MSD system (Agilent, USA; SEM Ltd., Istanbul, Turkey). Innowax FSC column (60m x 0.25mm, 0.25μm film thickness) was used with helium as carrier gas (0.8 mL/min.). GC oven temperature was kept at 60 °C for 10 min and programmed to 220 °C at a rate of 4 °C/min, and kept constant at 220 °C for 10 min and then programmed to 240 °C at a rate of 1 °C/min. Split ratio was adjusted 40:1. The injector temperature was at 250 °C. The interphase temperature was at 280 °C. MS were taken at 70 eV. Mass range was from m/z 35 to 450.

GC analysis

The GC analysis was carried out using an Agilent 6890N GC system. In order to obtain the same elution order with GC/MS, simultaneous injection was performed by using the same column and appropriate operational conditions. The FID temperature was 300°C.

Identification of Compounds

The components of essential oils were identified by comparison of their mass spectra with those in the Baser Library of Essential Oil Constituents, Adams Library (6), MassFinder Library (7), Wiley GC/MS Library (8) and confirmed by comparison of their retention indices. These identifications were accomplished by comparison of retention times with authentic samples or by comparison of their relative retention index (RRI) to a series of n-alkanes. Alkanes were used as reference points in the calculation of relative retention indices (RRI) (9). Relative percentage amounts of the separated compounds were calculated from FID chromatograms. The results of analysis are shown in Table I.

Table I. The Composition of the Essential Oils of *Lavandula angustifolia* Mill.

RRI	Compounds	A	B	C	D	E	F	IM
1014	Tricyclene	tr	tr	-	tr	tr	-	MS
1032	α-Pinene	0.2	0.9	0.3	0.2	0.2	0.2	t_R, MS
1035	α -Thujene	tr	0.1	0.2	0.1	0.1	-	MS
1076	Camphene	0.2	0.4	0.1	0.1	0.2	0.2	t_R, MS
1118	*β*-Pinene	0.1	1.1	tr	tr	0.1	0.1	t_R, MS
1132	Sabinene	tr	0.4	tr	tr	0.1	0.1	t_R, MS
1159	δ-3-Carene	tr	0.2	tr	0.1	tr	tr	t_R
1174	Myrcene	1.7	0.9	1.1	1.1	1.0	0.6	t_R, MS
1176	α -Phellandrene	-	0.1	tr	0.1	0.1	-	t_R, MS
1188	α-Terpinene	tr	0.1	0.1	tr	tr	tr	t_R, MS
1203	Limonene	0.8	1.5	0.4	0.3	0.6	0.5	t_R, MS
1213	**1,8-Cineole**	1.4	**13.3**	0.7	0.7	1.9	3.0	t_R, MS
1230	n-Butyl n-butyrate	-	-	-	0.1	0.1	tr	MS
1246	**(*Z*)-β-Ocimene**	1.7	4.4	**8.0**	**6.1**	3.1	1.1	t_R, MS
1255	γ-Terpinene	tr	0.2	0.4	0.2	0.1	tr	t_R, MS
1266	(E)-β-Ocimene	2.8	1.7	2.4	3.1	2.3	1.2	t_R, MS
1267	3-Octanone	0.3	tr	0.6	0.7	1.3	0.4	t_R, MS
1280	*p*-Cymene	tr	0.1	0.2	0.1	0.1	tr	t_R, MS
1282	Hexyl acetate	0.5	0.2	0.4	0.5	0.8	0.4	t_R, MS

1290	Terpinolene	0.5	0.5	0.2	0.2	0.2	0.2	t_R, MS
1345	3-Octyl acetate	0.1	-	tr	0.1	0.2	0.1	t_R, MS
1350	Hexyl propionate	0.1	tr	tr	tr	tr	0.1	MS
1353	Hexyl isobutyrate	0.2	tr	0.1	0.1	0.1	0.1	MS
1360	Hexanol	0.1	0.3	-	-	tr	tr	t_R, MS
1382	*cis*-Alloocimene	tr	tr	tr	tr	tr	tr	MS
1386	Octenyl acetate	0.2	-	1.0	0.9	0.4	0.1	MS
1393	3-Octanol	tr	-	-	0.2	0.4	tr	MS
1424	Hexyl butyrate	1.1	0.6	0.4	0.4	0.6	0.8	MS
1438	Hexyl 2-methyl butyrate	0.1	0.2	-	-	tr	-	MS
1450	*trans*-Linalool oxide (*Furanoid*)	tr	tr	0.1	tr	tr	-	MS
1457	Hexyl isovalerate	0.1	-	-	-	-	-	MS
1459	1-Octen-3-ol	tr	0.5	0.2	0.1	tr	-	t_R, MS
1474	*trans*-Sabinene hydrate	-	0.2	0.2	0.2	0.1	-	t_R, MS
1479	*cis*-Linalool oxide (*Furanoid*)	0.1	tr	tr	tr	tr	-	MS
1483	1-Octyl acetate	0.1	-	-	-	-	-	t_R, MS
1532	**Camphor**	4.7	**6.9**	0.2	0.2	0.3	4.8	t_R, MS
1544	7-epi- Sesquithujene	-	-	-	tr	-	-	MS
1553	**Linalool**	**50.0**	**45.9**	**31.9**	**38.9**	**43.0**	**32.3**	t_R, MS
1556	*cis*-Sabinene hydrate	-	tr	0.3	0.2	0.2	-	t_R, MS
1562	Octanol	-	tr	-	-	-	-	t_R, MS
1565	**Linalyl acetate**	**6.0**	1.8	**17.5**	**16.3**	**15.4**	**42.0**	t_R, MS
1583	α-Santalene	-	-	0.2	0.3	0.2	-	MS
1590	Bornyl acetate	-	-	0.2	0.2	0.1	-	t_R, MS
1611	**Terpinen-4-ol**	0.2	4.2	**14.9**	**8.3**	3.0	tr	t_R, MS
1612	β-Caryophyllene	0.4	0.3	2.5	3.3	2.4	1.8	t_R, MS
1617	**Lavandulyl acetate**	2.0	0.7	2.2	3.0	**5.2**	1.8	t_R, MS
1618	Hexyl hexanoate	tr	0.1	-	tr	0.1	tr	MS
1631	Hexyl tiglate	0.3	tr	-	0.1	0.1	0.3	t_R, MS
1661	Sesquisabinene	-	-	-	tr	-	-	t_R, MS
1668	(Z)-β-Farnesene	0.1	1.3	1.8	2.7	3.2	1.1	MS
1684	(E)-Ocimenol	0.2	1.0	-	-	-	-	t_R
1686	Lavandulol			0.6	0.7	1.2	tr	t_R, MS
1687	α-Humulene	-	-	-	tr	tr	-	t_R, MS
1690	Cryptone	-	0.1	-	-	0.3	tr	MS
1706	**α-Terpineol**	**8.7**	1.5	3.6	3.6	3.3	1.1	t_R, MS
1719	**Borneol**	2.2	**5.7**	0.4	0.4	0.9	1.9	t_R, MS
1726	Germacrene D	0.1	0.2	0.1	0.2	0.3	0.5	MS
1733	Neryl acetate	1.8	0.1	1.0	1.0	1.0	0.4	t_R, MS
1765	Geranyl acetate	3.6	0.2	1.9	1.8	1.7	0.6	t_R, MS
1776	Cumin aldehyde	0.1	0.1	0.1	tr	0.2	0.1	t_R, MS
1808	Nerol	1.6	0.1	0.7	0.7	0.6	0.2	t_R, MS
1830	2,6-Dimethyl-3(E),5 (E),7-octatriene-2-ol	-	-	0.1	tr	-	-	t_R, MS
1856	Geraniol	4.6	0.3	2.0	2.0	1.8	0.5	t_R, MS
1864	*p*-Cymen-8-ol	0.1	tr	-	-	tr	0.1	t_R, MS
2008	Caryophyllene oxide	0.1	tr	0.7	0.4	0.8	0.1	t_R, MS
2191	T-Cadinol	0.1	-	tr	-	0.5	tr	MS
2232	α-Bisabolol	0.9	1.5	-	-	-	0.7	MS

RRI :Relative retention indices.

(Relative retention indices) calculated against n-alkanes, %: calculated from FID (Flame ionization detection) data; tr : Trace (< 0.1 %); IM, identification method: t_R, identification based on the retention times (t_R) of genuine compounds on the HP Innowax column; MS, identified on the basis of computer matching of the mass spectra with those of the Wiley and MassFinder libraries and comparison with literature data; A: Yalova-Greenhouse, B: Yalova, C: Edirne, D: Burdur-Jubileuo, E: Burdur-Sevtapolis, F: Commercial oil

RESULTS AND DISCUSSION

The flowers of Lavandula were water distilled using a Clevenger type apparatus. The essential oils were analyzed by GC and GC-MS. The oil yields obtained from flowers are between 3.5 and 6%. Forty eight – fifty nine compounds constituting about 99.5-100.0 % of the essential oils of *Lavandula angustifolia* Mill. were characterized.

Oxygenated monoterpenes (44.0-79.2 %) were the main group of constituents of the oil of *L. angustifolia* followed by monoterpene hydrocarbons (4.2-13.4 %). The oils of *L. angustifolia* is contained linalool (31.9-50.0 %) and linalyl acetate (1.8-17.5%) as main constituents. Commercial oil is contained linalool (32.3 %) and linalyl acetate (42.0%) as main constituents.

The quality of *Lavandula* essential oils are regulated by ISO standards (The International Organization for Standardization). Also various international standarts such as European Pharmacopoeia (PhEur) contain monographs on various *Lavandula* sp. preparations securing pharmaceutical grade quality. (EP; linalool 20.0-45.0% and linalyl acetate 25.0-47.0%, limonen: max.% 1.0, 1,8-cineole: max. % 2.5, 3-octanone: % 0.1- 5.0, camphor: max % 1.2, terpinen-4-ol: % 0.1- 8.0, lavandulyl acetate: min % 0.2, lavandulol: min % 0.1, α-terpineol: max. % 2.0) (10,11)

The lavender essential oil composition determined by the International Organization for Standardization (ISO 3515:2002) (12) according to quality standards. Linalool, linalyl acetate, and camphor must be between 25.0-38.0 %, 25.0-45.0 % and 0-0.5 % resp. for Australian sample. (12-14). Only the linalyl acetate content (42.0%) of the F sample is within the limits (ISO 3515:2002, linalyl acetate 25.0–47.0%).
ISO for camphor content max. The limit is 1.5% (12).

Camphor content is 0.2–0.3% for samples C, D and E. However; sample A (4.7%), B (6.9%) and F (4.8%) contain camphor content higher than 1.5%.

For camphor, the company and Yalova samples (A, B and F) are not in the standard of oil quality, but it is suited to the quality standards of samples C, D, and E.

Lavender (*L. angustifolia*) attracts attention due to the active compounds in the essential oil composition.
Kivrak studied lavender and lavandin cultures samples (*L. angustifola* and *L.* x *intermedia*) cultivars in Turkey. These samples are compatible with the legislation of international standard. Lavender and lavandin samples have high antioxidant activity (14).

Low camphor plants tend also to have higher levels of terpenes. *L. angustifolia* is used in the perfumery and cosmetic industries while the high camphor plants are used as insect repellents and for other non-perfumery uses (16).

ACKNOWLEDGEMENTS

This article contains a part of the master thesis work.

REFERENCES

1. Hickey, M and King, C. Common Families of Flowering Plants. Cambridge Univ, 1997, pp 119-27.

2. Guner, A., Ozhatay, N., Ekim, T and Baser, K.H.C. Flora of Turkey and East Aegean Islands. Supplement II. Edinburgh Univ, 2000, Vol. 11.

3. Mill, R.R. *Lavandula* L. In: Davis P.H. (Ed.) Flora of Turkey and the East Aegean Islands, vol. 7, Edinburgh University Press, 1982, 76-8.

4. Dirmenci, T. *Lavandula* L. In: Guner, A., Aslan, S., Ekim, T., Vural, M. and Babac M.T. (eds.). A Checklist of the Flora of Turkey -Vascular Plants (Türkiye Bitkileri Listesi -Damarlı Bitkiler), Nezahat Gokyigit Botanik Bahcesi ve Flora Araştırmaları Dernegi Yayını, Istanbul, 2012, 558.

5. Zeybek U. and Haksel M., Türkiye'de ve Dünyada Önemli Tıbbi Bitkiler ve Kullanım Alanları, İzmir, Zade Sağlık Yayınları, 2010, 131-2.

6. Adams, R.P. Identification of Essential Oil Components by Gas Chromatography/Mass Spectrometry. Allured Publ. Corp, Carol Stream, IL. 2007.

7. Hochmuth, D. H. MassFinder-4, Hochmuth Scientific Consulting, Hamburg, Germany. 2008.

8. McLafferty, F.W., Stauffer, D.B. The Wiley/NBS Registry of Mass Spectral Data, J.Wiley and Sons: New York. 1989.

9. Curvers, J., Rijks, J., Cramers, C., Knauss, K., Larson, P. Temperature programmed retention indexes: calculation from isothermal data. Part 1: Theory.Journal of High Resolution Chromatography 8, 1985, 607–10.

10. Council of Europe European Pharmacopoeia (PhEur), vol. 8.0. Council of Europe, Strasbourg. 2014.

11. Kirimer, N., Mokhtarzadeh S., Demirci B., Goger F., Khawar K.M. and Demirci F. Phytochemical profiling of volatile components of *Lavandula angustifolia* Miller propagated under in vitro

conditions, Industrial Crops and Products 96, 2017, 120–5.

12. ISO 3515:, Oil of Lavender (*Lavandula angustifolia* Mill.) Standard. 1985. http://www.iso.org.

13. Kara, N., Baydar H. F. Determinaton of Lavender and Lavandin Cultivars (*Lavandula* sp.) Containing High Quality Essential Oil in Isparta, Turkey, Turkish Journal of Field Crops, 2013, 18(1), 58-65.

14. Kivrak, S. Essential oil composition and antioxidant activities of eight cultivars of Lavender and Lavandin from western Anatolia, Industrial Crops & Products, 2018, 117, 88–96.

15. Sharifi-Rad J., Sureda A., Tenore G.C., Daglia M., Sharifi-Rad M., Valussi M., Tundis R., Sharifi-Rad M., Loizzo M.R., Ademiluyi A.O., Sharifi-Rad R., Ayatollahi S.A. and Iriti M. Biological Activities of Essential Oils: From Plant Chemoecology to Traditional Healing Systems, Molecules, 2017, 22, 70; doi:10.3390/molecules22010070.

16. Cavanagh, H.M.A. and Wilkinson, J.M. Biological Activities of Lavender Essential Oil, Phytother. Res. 16. 2012, 301–8.