



Effect of Post-Harvest Drying Times on Chemical Components of Essential Oils of *Lavandula angustifolia* and *L. intermedia* Species

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Abstract

This study reported the chemical composition of essential oils of Lavender (*L. angustifolia* subs. *angustifolia*) and Lavandin (*Lavandula x intermedia* var. Super A). GC and GC/MS analyses were used for the identification of essential oils. The essential oils from fresh and air-dried aerial parts of Lavender and Lavandin were obtained by hydrodistillation method. In total, 49, 51, 50 and 40 constituents were identified and quantified in the essential oils of fresh and air-dried aerial parts of *L. angustifolia* and *L. intermedia* (fresh and air-dried aerial parts), respectively. The major components of essential oils of *L. angustifolia* (fresh), *L. angustifolia* (air-dried aerial parts), *L. intermedia* (fresh) and *L. intermedia* (air-dried aerial parts) were 1,8-cineole (12.65%, 10.15%, 12.74%, 2.28%), linalool (38.31%, 37.94%, 38.10%, 47.47%), camphor (13.74%, 12.52%, 7.61%, 6.67%), borneol (6.82%, 7.16%, 6.84%, 4.64%). While the 1,8 cineole (12.65%, 10.15%) concentration decreased with the post-harvest drying process, the linalyl acetate (1.02%, 2.80%) concentration increased in the essential oils of fresh and air-dried aerial parts of *L. angustifolia*. This study provides evidence that there is a change in essential oil components with post-harvest drying. Thus, it was concluded that the essential oil of the studied *Lavandula* species can be obtained by distillation of fresh and air-dried aerial plants after the harvest, considering the desired compound ratio.

Keywords: *Lavandula angustifolia*, *Lavanta intermedia*, Essential oil, Hydrodistillation

Hasat Sonrası Kuruma Sürelerinin *Lavandula angustifolia* ve *L. intermedia* Türlerinin Uçucu Yağlarının Kimyasal Bileşenleri Üzerindeki Etkileri

Öz

Bu çalışma, Lavanta (*L. angustifolia* subs. *angustifolia*) ve Lavandin (*Lavandula x intermedia* var. Super A) uçucu yağlarının kimyasal bileşimini rapor etmektedir. Uçucu yağların tanımlanmasında GC ve GC/MS analiz yöntemleri kullanıldı. Lavanta ve Lavandin'in taze ve hava ile kurutulmuş toprak üstü kısımlarından hidrodistilasyon yöntemi ile uçucu yağlar elde edildi. Toplamda, *L. angustifolia* (taze ve hava ile kurutulmuş toprak üstü kısımları) ve *L. intermedia*'nin (taze ve hava ile kurutulmuş toprak üstü kısımları) uçucu yağlarında sırasıyla 49, 51, 50 ve 40 bileşen tanımlandı ve miktarı belirlendi. *L. angustifolia* (taze), *L. angustifolia* (hava ile kurutulmuş toprak üstü kısımları), *L. intermedia* (taze) ve *L. intermedia*'nin (hava ile kurutulmuş toprak üstü kısımları) uçucu yağlarının ana bileşenleri 1,8-sineol (%12,65, %10,15, %12,74, %2,28), linalool (%38,31, %37,94, %38,10, %47,47), kafur (%13,74, %12,52, %7,61, %6,67), borneol (%6,82, %7,16, %6,84, %4,64)'dür. Hasat sonrası kurutma işlemi ile *L. angustifolia* 'nin taze ve hava ile kurutulmuş toprak üstü kısımlarının uçucu yağlarında 1,8 sineol (%12,65, %10,15) konsantrasyonu azalırken, linalil asetat (%1,02, %2,80) konsantrasyonu artmıştır. Bu çalışma, hasat sonrası kurutmayla birlikte uçucu yağ bileşenlerinde bir değişiklik olduğuna dair kanıt sağlamaktadır. Bu nedenle, bitkinin uçucu yağının, istenen bileşik oranı dikkate alınarak, hasattan sonra toprak üstü kısımlarının taze veya hava ile kurutulmuş şekilde damıtılmasıyla elde edilebileceği sonucuna varılmıştır.

Anahtar Kelimeler: *Lavandula angustifolia*, *Lavandula intermedia*, Uçucu yağ, Hidrodistilasyon

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1. Introduction

Medicinal and aromatic plants have been used for a variety of purposes worldwide since ancient times [1,2,3]. Nowadays, essential oils are primarily used in cosmetic, pharmaceutical and food industries [4,5,6]. For this reason, plants that can produce essential oils have a strong position as economic products worldwide so the market of essential oils is growing in time [7]. The *Lavandula* genus, which belongs to the Labiatae (Lamiaceae) family and is one of the medicinal and aromatic plants, consists of over 39 species. Lavender flowers, leaves, and branches are used in widely herbal medicine, the cosmetic industry, and for culinary purposes [8,9]. Although *Lavandula* grows naturally in the Mediterranean region, it is grown commercially in several countries (e.g. France, Turkey, China, Bulgaria, Spain, the United States, United Kingdom, Australia, Portugal) mainly for essential oils. They are drought-resistant and need sunny, warm growing locations [7,10,11,12].

Essential oils obtained from most *Lavandula* species can be used in the cosmetic, pharmaceutical, and food industries [7,8,12,13]. Lavender (*Lavandula angustifolia*) and Lavandin (*Lavandula intermedia*) are the most important medicinal and aromatic plants. The lavender essential oil which contains more than a hundred compounds has two major compounds that are linalool and linalyl acetate. Other constituents include 1,8-cineole, α -pinene, *p*-cymene, camphor, lavandulol, camphene, lavandulyl acetate, *p*-caryophyllene, myrcene etc. According to the ISO 8902:2009a and ISO 3515:2002, constituents of Lavender and Lavandin have different standard values. These are linalool (25–38%), camphor (0.5–1.0%), linalyl acetate (25–45%) in lavender essential oil; linalool (24–35%), camphor (6–8%), linalyl acetate (28–38%), lavandin essential oil. While Lavandin yield more than Lavender, it has less commercial value [9,14]. Lavender essential oil is preferred by industrial fields such as perfumery, alternative medicine and cosmetics because of the low level of camphor and high linalool, linalyl acetate content, while Lavandin is used by cleaning industry in household cleaner products, detergents and pest control products because of the high level of camphor [8,15,16]. Essential oils are produced from different techniques [17,18] and the most preferred extraction methods are hydro- and steam distillation, which are inexpensive and easy to use. In this study, hydrodistillation was used to extract the essential oil from *L. angustifolia*, *L. intermedia*. *Lavandula* essential oil is also an important product due to its pharmacological properties and rich compounds, and it is promoted to be grown for oil production. In Turkey, the cultivation of *Lavandula* species has expanded considerably in recent years.

In this study, it was aimed to investigate the fresh and air-dried aerial parts of lavandin and lavender in terms of both essential oil yields and components. Components of essential oils obtained from fresh and dried lavender were compared qualitatively and quantitatively.

2. Material and Method

2.1. Plant Material

Plant materials of *Lavandula angustifolia* spp *angustifolia* “*Lavandula vera*” and *Lavandula x intermedia* var. Super A were added from Ula, Armutçuk/ Muğla, Turkey in July 2020. The spices were identified in Muğla Sıtkı Koçman University, Faculty of Science, Department of Molecular Biology and Genetics e-ISSN: 2148-2683

(voucher no MU1151 (for *L. Angustifolia* subs. *Angustifolia*) and MU1152 (for *Lavandula x intermedia* var. Super A). The plant materials were studied as fresh and air-dried aerial parts.

2.2. Drying Method

The aerial parts of *L. angustifolia* and *L. intermedia* harvested from the same field were air-dried in the shade for 7 days (average temperature 30 °C).

2.3. The Moisture Content of Herbal Drogs

The moisture content of the fresh and air-dried aerial parts of *L. angustifolia* and *L. intermedia* was determined in triplicate using a laboratory oven at 110 °C.

2.4. Analysis of Essential Oils

2.4.1. Gaz Chromatography (GC-FID)

GC-FID analysis was carried out using a DB-5 fused silica capillary non-polar column (30 m×0.25 ID., film thickness 0.25 µm). The detector (270 °C) and injector temperatures (250 °C) were adjusted. The Helium was used as carrier gas at a flow rate of 1.4 mL/min volume. The sample injection volume was 0.2 µL, split ratio of 20:1. Firstly oven temperature was programmed at 60 °C for 5 min. After that it was scaled up to 240 °C with 4 °C/min. The final temperature was maintained for 10 min. Components of essential oils were identified by comparing retention times (RT) and mass spectra with data from NIST 2008 and Willey libraries.

2.4.2. Gaz Chromatography/mass spectrometry (GC-MS).

GC/MS analyzes were performed using an ion trap MS spectrometer and a DB-5 MS fused silica non-polar capillary column (30 m × 0.25 mm ID, film thickness 0.25 µm). Helium was used as carrier gas at a flow rate of 1.4 mL/min. The injector (220 °C) and MS (290 °C) transfer line temperatures were set. The ion source temperature was 200 °C, the injection volume was 0.2 µL with a 1:20 partition ratio. EI-MS, was performed with electrons of 70 eV. The oven temperature was programmed to rise from 60 to 240 at a rate of 4 °C/min and held at this temperature for 10 min. The identification of the constituents of essential oils depends on their GC retention index and computer matching with Wiley, NIST-2008 and the TRILIB Library. The library, as well as the fragmentation patterns of mass spectra, compared with those reported in the literature [19,20,21].

3. Results and Discussion

In this study, the yield of essential oil of fresh and air-dried aerial parts of *L. intermedia* were 4.70% and 5.68% while *L. angustifolia* were 3.83% and 4.37% (Table 1.) Among the lavandin and lavender cultivars, the highest essential oil yield was obtained from *L. intermedia* in both fresh and air-dried aerial parts. It has been reported that different methods change the essential oil ratio and composition during production and after harvest [22]. In addition to the distillation method, it has been stated that the type of plant, the soil, the climatic conditions, the age of the plant, the harvest date and storage conditions affect the quality of essential oil [23,24,25]. According to previous researches by Kara and Baydar, the essential oil yield was determined 5.07% for dry stemless flower and 1.34% for fresh stem flower of *L. vera*. Moreover, it was determined essential oil ratio 1.49% for fresh of *L. intermedia* and 7.75% for dry stemless flower of *L. intermedia* [16]. While the yield of dry, stemless

flower essential oil was 7.1-9.9% in Lavandin species, it has been reported to vary between 2.8-5.0% in Lavander species [26]. In other study, essential oil yields from the different lavender samples were determined to have changed between 0.8-4 mL [27].

Table 1. The yield and moisture of essential oil in fresh and air-dried aerial parts of lavender and lavandin cultivars (%)

	Air-dried % (w/w) (dry weight)	Moisture %	Fresh % (w/w) (dry weight)	Moisture %
<i>L. angustifolia</i>	4.38	10.6	3.81	62.0
<i>L. intermedia</i>	5.68	10.4	4.53	57.6

The essential oils obtained from fresh and air-dried aerial parts of *L. angustifolia* and *L. intermedia* were analyzed using the GC and GC-MS techniques. The results are shown as relative percentages (%) and Kovats index of compounds in the Table 2. In total, 49, 51, 50 and 40 constituents were identified and quantified in LAF (*L. angustifolia*, fresh), LAD (*L. angustifolia*, air-dried), LIF (*L. intermedia* fresh), LID (*L. intermedia*, air-dried) respectively. It was determined that LAF and LAD contain 1,8-cineole (12.65%, 10.15%), linalool (38.31%, 37.94%), camphor

(13.74%, 12.52%), borneol (6.82%, 7.16%), terpinen-4-ol (10.20%, 8.65%) as main compounds while LIF and LID comprise 1,8-cineole (12.74%, 2.28%), linalool (38.10%, 47.47%), camphor (7.61%, 6.67%), borneol (6.84%, 4.64%), *a*-terpineol (6.50%, 8.28%), linalyl acetate (8.87%, 13.65%). When the essential oil components of fresh and dry drogs of both types are examined; While the amount of *a*-pinene, 1,8-cineole, terpinen-4-ol and camphor decreased in dry drogs, the amount of *a*-terpineol, nerol, linalyl acetate, geranyl acetate and β -terpenyl acetate increased. The same major compounds were reported in different studies. While linalool, camphor and 1,8-cineole were identified as the most important compounds in essential oil of LAF. Linalool, camphor, borneol, *a*-terpineol, linalyl acetate were stated in essential oil of LIF. The same major compounds have been determined in the essential oil of *L. angustifolia* [11,18,28] and *L. intermedia* [29,30].

Our results showed that the essential oil fresh and air-dried parts of *L. angustifolia* contains monoterpene hydrocarbons (5.4;6.19%), monoterpenoids (89.4;88.65%), sesquiterpene hydrocarbons (1.63;1.10%), sesquiterpenoids (0.1;0.52%) while *L. intermedia* contains monoterpene hydrocarbons (3.83;1.56%), monoterpenoids (89.85;95.02%), sesquiterpene hydrocarbons (1.32;0.77%), sesquiterpenoids (2.39;1.28%) (Table 2).

Table 2. Chemical composition of the essential oils of fresh and, air-dried aerial parts of *L. angustifolia* and *L. intermedia*

Nu	Compounds ^a	LAF (% ^b)	LAD (% ^b)	LIF (% ^b)	LID (% ^b)	RI ^c	Identification Methods ^d
1	Isohexanol	0.20	0.18	0.06	tr	842	MS, RI
2	α -Thujene	0.20	0.17	Nd	Nd	924	Co-I; MS, RI
3	α -Pinene	0.98	0.8	0.43	0.08	928	Co-I; MS, RI
4	Camphene	0.61	0.57	0.38	0.13	940	Co-I; MS, RI
5	Sabinene	tr	0.53	0.48	Nd	956	Co-I; MS, RI
6	Fenchene	0.22	0.17	0.22	Nd	958	Co-I; MS, RI
7	1-Octen-3-ol	tr	0.12	Nd	Nd	961	Co-I; MS, RI
8	3-Octanone	0.28	0.32	0.63	0.41	964	Co-I; MS, RI
9	β -Pinene	0.57	0.68	1.33	0.59	969	Co-I; MS, RI
10	Butyl butyrate	0.07	0.1	0.11	tr	979	MS, RI
11	α -Phellandrene	0.04	0.06	0.04	Nd	996	Co-I; MS, RI
12	3-Carene	0.21	0.16	0.2	Nd	1000	Co-I; MS, RI
13	Hexyl acetate	0.43	0.45	0.22	0.43	1007	MS, RI
14	α -Terpinene	0.12	0.13	0.05	Nd	1012	Co-I; MS, RI
15	<i>p</i> -Cymene	0.48	0.33	0.2	tr	1015	Co-I; MS, RI
16	Limonen	1.03	1.51	Nd	0.35	1019	Co-I; MS, RI
17	1,8-Cineole	12.65	10.15	12.74	2.28	1023	Co-I; MS, RI
18	<i>trans</i> - β -Ocimene	1.5	1.6	1.25	0.62	1027	Co-I; MS, RI
19	<i>Cis</i> - β -Ocimen	0.36	0.74	tr	1.14	1035	Co-I; MS, RI
20	γ -Terpinene	0.35	0.34	0.1	Nd	1041	Co-I; MS, RI
21	<i>Cis</i> -linalool oxide	0.26	0.3	0.02	0.28	1060	Co-I; MS, RI
22	Terpinolen	0.59	0.74	0.6	0.41	1080	Co-I; MS, RI
23	Linalool	38.31	37.94	38.1	47.47	1092	Co-I; MS, RI
24	6-Camphenenol	0.07	tr	0.05	tr	1108	MS, RI
25	Allo-Ocimene	0.08	0.08	0.06	0.03	1110	Co-I; MS, RI
26	Camphor	13.74	12.52	7.61	6.67	1113	Co-I; MS, RI
27	Hexyl isobutyrate	0.24	0.23	0.17	0.13	1131	MS, RI
28	Lavandulol	1.33	1.22	0.21	0.22	1140	MS, RI
29	Borneol	6.82	7.16	6.84	4.64	1147	Co-I; MS, RI
30	Terpinen-4-ol	10.2	8.65	0.34	0.17	1159	Co-I; MS, RI

Nu	Compounds ^a	LAF (% ^b)	LAD (% ^b)	LIF (% ^b)	LID (% ^b)	RI ^c	Identification Methods ^d
31	Cryptone	0.1	0.15	0.65	0.3	1161	MS, RI
32	Hexyl butyrate	1.46	1.19	Nd	Nd	1171	MS, RI
33	<i>a</i> -Terpineol	1.42	2.48	6.5	8.28	1177	Co-I; MS, RI
34	Nerol	0.08	0.22	0.75	1.16	1208	Co-I; MS, RI
35	Bornyl formate	0.1	0.13	0.23	0.18	1211	MS, RI
36	Hexyl valerate	0.37	0.4	0.18	0.12	1235	MS, RI
37	<i>p</i> -Cumic aldehyde	0.07	0.1	0.22	0.11	1237	MS, RI
38	Linalyl acetate	1.02	2.8	8.87	13.65	1242	Co-I; MS, RI
39	Neryl acetate	0.78	1.21	2.19	2.84	1336	Co-I; MS, RI
40	Undecanol	0.21	0.24	0.3	0.26	1351	Co-I; MS, RI
41	Geranyl acetate	0.08	0.3	1.08	1.65	1358	Co-I; MS, RI
42	β -Terpenyl acetate	0.16	0.54	1.93	2.89	1367	MS, RI
43	Hexyl caproate	0.2	0.25	0.04	Nd	1373	MS, RI
44	β -Caryophyllene	0.36	0.32	0.82	0.57	1415	Co-I; MS, RI
45	β -Farnesene	1.13	0.67	0.3	Nd	1428	Co-I; MS, RI
46	Linalyl butyrate	0.06	0.07	0.09	0.27	1432	
47	Germacrene D	0.14	0.11	0.2	0.2	1472	Co-I; MS, RI
48	Geranyl propionate	0.28	0.29	0.17	0.17	1481	MS, RI
49	α -Cadinene	Nd	Nd	0.24	Nd	1504	MS, RI
50	δ -Cadinene	Nd	Nd	0.19	Nd	1513	Co-I; MS, RI
51	Caryophyllene oxide	0.1	0.17	0.26	0.13	1561	Co-I; MS, RI
52	<i>tau</i> -Cadinol	Nd	0.11	0.88	0.07	1616	Co-I; MS, RI
53	α -Cadinol	Nd	Nd	0.23	Nd	1631	MS, RI
54	<i>a</i> -Bisabolol	Nd	0.24	1.25	1.08	1649	Co-I; MS, RI
	Monoterpene hydrocarbons	5.4	6.2	4.0	1.6		
	Monoterpenoids	89.4	88.7	89.9	95.0		
	Sesquiterpene hydrocarbons	1.6	1.1	1.8	0.8		
	Sesquiterpenoids	0.1	0.5	2.6	1.3		
	Others	3.5	3.5	1.7	1.4		
	Total identified (%)	100.0	100.0	100.0	100.0		
	Total number of compounds	49	51	50	40		

^a Compounds are listed in the order of their elution from a DB-5 fused silica column. ^b Percentage concentration. ^c Retention index on DB-5 fused silica column. ^d Identification methods: Co-I; Co-injection: based on comparison with authentic compounds; MS: based on comparison with WILEY, ADAMS and NIST 08 MS databases; RI: based on comparison of calculated with those reported in ADAMS and NIST 08, Nd: not detected, tr: trace

4. Conclusions and Recommendations

In this study, the changes in the chemical components of essential oils obtained in fresh and dry form from two *Lavandula* species cultured in the same field and under the same ecological conditions were investigated. It was determined that some compounds of the essential oil changed quantitatively depending on whether the herbal drug was fresh or dried. It was also revealed that the way of harvesting affects the essential oil yield. With this study, the qualitative and quantitative results of essential oils of lavender varieties will guide scientific studies in the industrial field.

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