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The effect of different drying temperatures on the essential oil content and chemical composition of *Lavandula angustifolia* Mill.

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Abstract

Drying temperatures affect the content and composition of essential oils in plants containing essential oils due to the organs where essential oils are synthesized and stored. For this reason, many studies have been carried out to determine the appropriate drying temperature to obtain the highest amount and the best quality essential oil. In present study, it is aimed to determine the effects of different drying temperatures on essential oil content and components in lavender (*Lavandula angustufolia* Mill.). The samples dried at four different temperatures (35°C, 45°C, 55°C and 65°C) were isolated for 3 hours using Clevenger type apparatus and the obtained oils were analyzed by GC-MS. The essential oil contents obtained at 25°C, 35°C, 45°C, 55°C, and 65°C were 1.17%, 0.96%, 0.94%, 0.65%, and 0.18% respectively. It was determined that the major components of essential oils obtained at different drying temperatures were 1.8-cineole (17.88-50.15%), camphor (32.60-48.86) and borneol (3.46-9.45%). The highest 1,8-cineole ratio was found in samples dried at 55°C (50.15%) but the lowest in samples dried at 65°C (17.88%). The highest and lowest camphor ratios were determined in samples dried at 65°C (48.86%) and samples dried at 55°C (32.60%) respectively. The highest (9.45%) borneol ratio was obtained in samples dried at 65°C, while the lowest (3.46%) ratio was obtained in samples dried at 55°C. The results obtained in the present study showed that Lavender essential oil content and composition were affected by drying temperatures and the optimum drying temperature was 35 ºC.

Keywords: drying temperatures, essential oil, GC-MS, *Lamiaceae*, *Lavandula angustifolia* Mill.

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Farklı kurutma sıcaklıklarının *Lavandula angustifolia* Mill uçucu yağ içeriği ve kimyasal bileşimi üzerine etkisi

Özet

Kurutma sıcaklıkları, uçucu yağların sentezlendiği ve depolandığı organlar nedeniyle uçucu yağ içeren bitkilerdeki uçucu yağların içeriğini ve bileşimini etkiler. Bu nedenle en yüksek miktarda ve en kaliteli uçucu yağı elde etmek için uygun kurutma sıcaklığını belirlemek üzere birçok çalışma yapılmıştır. Bu çalışmada, farklı kurutma sıcaklıklarının lavantanın (*Lavandula angustufolia* Mill.) uçucu yağ içeriği ve bileşenleri üzerindeki etkilerinin belirlenmesi amaçlanmıştır. Dört farklı sıcaklıkta (35°C, 45°C, 55°C ve 65°C) kurutulan örnekler Clevenger tipi aparat kullanılarak 3 saat izole edilmiş ve elde edilen yağlar GC-MS ile analiz edilmiştir. 25°C, 35°C, 45°C, 55°C ve 65°C'de elde edilen uçucu yağ içerikleri sırasıyla %1,17, %0,96, %0,94, %0,65 ve %0,18'dir. Farklı kurutma sıcaklıklarında elde edilen uçucu yağların ana bileşenlerinin 1.8-cineole (%17.88-50.15), camphor (32.60-48.86) ve borneol (%3.46-9.45) olduğu belirlenmiştir. En yüksek 1,8-sineol oranı 55°C'de kurutulan örneklerde (%50.15), en düşük ise 65°C'de kurutulan örneklerde (%17.88) bulunmuştur. En yüksek ve en düşük kafur oranları sırasıyla 65°C'de (%48,86) ve 55°C'de (%32,60) kurutulan örneklerde tespit edilmiştir. En yüksek (%9,45) borneol oranı 65°C'de kurutulan örneklerde elde edilirken, en düşük (%3,46) oran 55°C'de kurutulan örneklerde elde edilmiştir. Bu çalışmada elde edilen sonuçlar, Lavanta uçucu yağ içeriği ve bileşiminin kurutma sıcaklıklarından etkilendiğini ve optimum kurutma sıcaklığının 35 ºC olduğunu göstermiştir.

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Anahtar kelimeler: kurutma sıcaklıkları, uçucu yağ, GC-MS, *Lamiaceae*, *Lavandula angustifolia* Mill.

1. Introduction

Lavender (*Lavandula angustifolia*), which is one of the characteristic plants of the Western Mediterranean Region, is a perennial and shrub form and is a valuable aromatic and medicinal plant that naturally spreads in Southern France, Central Italy, Spain and Greece. Although there are two different species of the genus (*Lavandula stoechas* subsp. *stoechas* and subsp. *cariensis*) in the flora of Türkiye, this species does not show a natural distribution. It is extensively cultivated in France, Bulgaria, Spain, Italy, Greece, England, Russia, USA, Austria and North African countries [1-3]. Three important and highly commercially valuable lavender species are cultivated: Lavander (*Lavandula angustifolia = L. officinalis = L. vera*), Lavandin (*Lavandula x intermedia = L. hybrida*) and Spike lavander (*Lavandula spica*). Best quality lavender oil from lavender, also called "English lavender" is obtained. The so-called "hybrid lavender" lavandin has a higher volatile content than lavender oil content, but lower essential oil quality has [4]. Lavender flower: It is a drug that has been used in folk medicine for a long time due to its effects such as relieving rheumatic pains, increasing urine, antiseptic, expectorant, healing eczema wounds, strengthening nerve and heart [1, 5, 6]. The essential oil obtained from the flowers of the lavender plant is important for the perfume, cosmetics, flavor and fragrance industries. In addition, it is used to give fragrance to some preparations in the pharmacy, and by leaving it in the wardrobe at home, it is ensured that the clothes smell good [5, 7]. Linanyl acetate and linalool, which are found in the essential oil of *Lavandula angustifolia* L., are important essential oil components and are desired to be found at high rates in the perfumery industry. These components, which determine the quality of the essential oil, are quality lavender oil with a camphor ratio of less than 0.5% in the essential oil [8].

Medicinal and aromatic plants have an important place in world markets. Medicinal and aromatic plants are used as raw materials in the food, pharmaceutical, cosmetic, cleaning and natural dye industries. Türkiye is the homeland of a significant part of these plants and has a flora where some of them can grow naturally. Also the parts of these plants can be used for herbal teas which is preferred to treat several illnesses especially in folk medicine [9, 10, 11]. Medicinal and aromatic plants should be processed immediately after harvesting in order to preserve the active substances contained in the process until they reach the consumer. Drying is the process of reducing the moisture content of medicinal and aromatic plants from the high moisture content (70-85%) after harvesting to a moisture level (10-15%) suitable for safe storage. The aim of the drying process is to reduce the product moisture to the final moisture value in the shortest time and with the least energy consumption without any deterioration in product quality. Selection of the appropriate drying method for medicinal and aromatic plants is the most important step for successful drying [12, 13].

Most of the harvested medicinal and aromatic plants are dried by natural drying methods. In shade and sun drying method, variations in temperature and air humidity make it difficult to obtain homogenous drying of the products. In some cases, natural drying methods can lead to the development of fungi in the products and the formation of harmful chemicals such as aflatoxin. Medicinal and aromatic plants with a high water content should be dried as soon as possible after harvest. The most important factor in drying these plants with heated air is the drying air temperature. Since high drying temperatures (> 40 °C) adversely affect the secondary metabolites and natural color of the plants, lower drying temperatures (~ 35 °C) are mostly used. Drying at low drying temperatures is completed in longer periods and energy consumption is high. Different studies investigating suitable drying conditions for drying medicinal and aromatic plants have reported that variable drying air temperature applications can better maintain product quality while shortening drying time [14]. Therefore, there is a need to determine this change in medicinal and aromatic plants by separate studies. In this study, it was aimed to determine the effect of drying at different temperatures on the essential oil yield and quality of *Lavandula angustifolia* and to determine the optimum drying temperature.

2. Materials and methods

In the research, plants harvested from Hatay Mustafa Kemal University campus were used as plant material. To determine the effect of different drying temperatures on essential oil content and components, the leaves of the harvested plants were dried in an oven at four different temperatures (35 °C, 45 °C, 55 °C and 65 °C) for 48 hours.

2.1. Isolation of the essential oils

Essential oil was obtained from dried leaves and flowers. A total of 50 g of each of the ground plant samples was used for the separate hydro distillation experiment. A weighed sample was individually and carefully placed into a 2 L flask. Distilled water was then added until it covered the sample completely. Essential oils were obtained by water distillation for 3 hours by using a Clevenger type apparatus according to the European Pharmacopoeia method. The trial was repeated three times. Essential oil yield was calculated according to dry weight of plant materials and amount of

essential oils obtained. The essential oils were dried over anhydrous sodium sulfate and stored in dark vial bottles at +4°C until analysis [15-16].

2.2. GC-MS Analysis

The components of the essential oils of the plants were determined by gas-chromatographic (GC-MS) method. Determination of essential oil components was carried out with Thermo Scientific ISQ Single Quadrupole model gas chromatographic device under the following conditions. TR-FAME MS model, 5% Phenyl Polysilphenylene-siloxane, 0.25 mm inner diameter x 60 m length, 0.25 µm film thickness column was used. Helium (99.9%) was used as the carrier gas for Peer Review only at a flow rate of 1 ml/min. Mass spectra were recorded at 70eV, the mass range was from 1.2-1200 m/z. Scan Mode was used for data collection. The MS transfer line temperature was 250°C, the MS ionization temperature was 220°C, the injection port temperature was 220°C, the column temperature was initially 50°C and the temperature was raised to 220°C with a rate of heat increase of 3°C/min. The structure of each compound was identified using mass spectra with the Xcalibur program (Wiley 9) [17].

3. Results

The essential oil ratio and components of medicinal and aromatic plants are largely determined by the genotype of the plant used in production. On the other hand, production location, production techniques, harvesting time, harvesting method, drying methods and temperatures, extraction/isolation methods used to obtain effective substances, product processing techniques and storage conditions are effective in determining the essential oil ratio and components of aromatic plants [8, 18-20]. The essential oil contents obtained by drying the leaves of the lavender plant at different temperatures are as given in Table 1. The highest essential oil content was obtained at 25 °C with 1.17%, followed by 0.96% and 0.94% at 35 °C and 45 °C, respectively. The least essential oil content was obtained from plants dried at 65 °C with 0.18% (Table 1).

When the main components of the plant, whose essential oils were obtained at different temperatures, were examined, it was determined that the main components were eucalyptol, and camphor (Table 1). Eucalyptol, which is an important oxygenated monoterpene, is one of the important components determining the utilization of medicinal plants, and a high ratio is desired for use as spice or herbal tea [21]. The eucalyptol ratio varied depending on different drying temperatures and the highest (50.15%) ratio was obtained in plants dried at 55 °C, followed by plants dried at 45 °C with 39.85% and 35 °C with 39.15%. The lowest eucalyptol ratio was found in plants dried at 65 °C with 17.88% (Table 1). While it was determined that the eucalyptol ratio increased with increasing temperature, it was determined that this ratio decreased after 55°C.

The camphor content in the oxygenated monoterpene structure, which is one of the most important main components of the essential oil, varied between 32.60-48.86 % depending on different drying temperatures. The highest camphor ratio (48.86 %) was obtained from samples dried at 65 °C, while the lowest ratio (32.60 %) was obtained at 55 °C (Table 1).

Since the organs of essential oil-containing plants where these oils are synthesized and stored are close to the outer surface of the plant, the drying methods and temperatures used have an effect on the ratio and composition of the essential oil [8, 18-20]. For this reason, many studies have been carried out to determine the appropriate drying temperature to obtain the highest quantity and quality of essential oil. In these studies, it has been reported that many ecological factors and cultivation practices, especially the genotypic characteristics of the plant material used in production, have an effect on the determination of yield and quality of medicinal aromatic plants. In addition, post-harvest practices (drying methods, drying time, distillation/extraction methods and time, storage conditions, etc.) are also effective in determining the yield and quality of the product [20, 22].

Table 1. Essential oil content and components of *L. angustifolia* plant dried at different temperatures.

RT	Compound Name	SI	RSI	Area %			
Temperatures (°C)				35 °C	45 °C	55 °C	65 °C
6.58	α-Pinene	717	730	0.58	0.61	0.81	nd
7.59	Camphene	991	994	0.73	0.72	1.23	nd
8.35	Cyclohexane	900	984	nd	nd	nd	0.97
8.4	β-Pinene	977	993	0.61	0.6	0.79	nd
8.72	β-Phellandrene	976	980	0.18	nd	nd	nd
10.1	Limonene	987	990	0.51	0.38	0.31	nd
10.8	Dehydrocineole	820	899	0.10	0.16	0.14	0.17
11.2	γ-Terpinene	868	923	0.09	0.22	0.14	0.45
12	Eucalyptol	988	992	39.15	39.85	50.15	17.88
12.7	o-Cymene	941	975	1.25	1.15	1.01	nd
17.3	1-Octen-3-ol	760	848	0.07	nd	nd	nd
18.2	Anon	925	961	0.08	nd	nd	0.41
18.9	n-Hexyl butanoate	961	973	0.13	0.1	0.07	nd
19.7	cis Sabinene hydrate	969	986	0.32	0.21	0.16	nd
20.7	Linalool	985	990	0.89	0.8	0.49	0.64
21.1	α-Santalene	904	957	0.11	nd	0.07	nd
22.3	Linalyl acetate	819	841	0.09	nd	nd	nd
22.9	trans Sabinene hydrate	955	972	0.19	0.12	0.08	nd
23.3	α-Campholene aldehyde	893	909	0.13	0.12	0.12	nd
24.2	Terpinen-4-ol	983	984	0.78	0.58	0.38	0.3
25	Bornyl formate	916	932	0.13	0.08	0.07	0.51
25.2	Isopinocarveol	967	970	0.43	0.47	0.33	0.61
25.4	Hexyl tiglate	954	974	0.12	nd	nd	nd
25.9	Camphor	988	991	38.78	36.38	32.6	48.86
26.2	Verbenol	888	898	0.23	0.33	0.28	0.39
27	α-Terpineol	889	957	1.33	1.22	0.93	0.32
27.5	Borneol	993	993	5.04	4.55	3.46	9.45
28.6	Myrtenal	974	983	0.37	0.54	0.39	0.56
28.8	Nopinone	849	914	0.15	0.36	0.16	nd
29.5	Myrtenol	964	974	0.17	0.22	0.17	0.37
30.7	trans-Carveol	952	971	0.18	0.19	0.16	0.17
31.5	Cumic alcohol	893	976	0.33	0.47	0.28	0.35
31.7	Carvacrol	797	809	0.76	0.87	0.53	0.67
31.9	Cryptone	949	973	0.89	0.8	0.54	0.54
32.5	p-Cumic aldehyde	943	967	0.84	0.72	0.56	1.28
32.7	Piperitone	862	938	0.09	0.08	nd	nd
33.1	Ethanone. 1-(methylphenyl)	883	915	0.08	0.12	nd	nd
33.5	Verbenone	936	963	0.14	3.31	0.17	0.28
36.1	Teresantalol	863	906	0.11	nd	0.09	0.44
37.8	Junipene	777	810	nd	nd	nd	0.18
38	3-Carene. 4-acetyl-	797	845	0.10	nd	nd	nd
39.7	Santalol	814	863	nd	0.22	0.12	0.61
40.1	Caryophyllene oxide	979	993	1.53	1.38	1.14	4.93
40.3	α-Cadinol	943	945	0.49	0.44	0.35	1.34
40.9	o-Allylguaiacol	771	823	0.13	0.15	0.1	0.36
41.3	α-Bisabolol	956	966	0.18	0.17	0.1	0.73
46.5	Methyl 6-octadecenoate	796	839	nd	nd	nd	0.19
49.5	Hexadecamethylheptasiloxane	774	841	nd	nd	nd	2.52
51	Vitamin A alcohol	766	766	0.67	0.13	0.1	0.47
53	Testosterone	793	798	0.21	0.08	0.07	nd
54.6	γ-Sitosterol	530	572	nd	nd	nd	0.23
56.2	Eremanthin	877	949	0.10	0.46	0.14	nd
Total identified (%)				46	39	39	32
Number of compounds				99.57	98.64	99.33	97.18
Essential Oil Content (%)				1.17	0.96	0.94	0.18

4. Conclusions and discussion

Katar et al., [23] in their study associated *Hyssopus officinalis*, reported that different drying times had a significant ($p<0.01$) effect on essential oil yield, and the optimum drying time should be 24 hours at 35 ºC. Aydın et al., [24] examined the effects of different drying temperatures on the chemical composition of *Salvia fruticosa* and determined that the optimum drying temperature was 35. Müller et al. [25] in the study in which they examined the effects of drying temperature on the essential oil of medicinal sage; They dried the products at drying temperatures ranging from 30-90 ºC until they dropped to 11% humidity. No loss of essential oil was observed at 60 ºC and they found that after this temperature the loss of essential oil increased.

Verma et al. [26] reported that the essential oil ratios obtained from the lavender plant varied between 0.80-1.30%. In the same study, they reported that they determined the main components as linalyl acetate (47.56%), linalool (28.06%), lavandulyl acetate (4.34%), α-terpineol (3.75%). Jianu et al., [27] reported the main components of essential oil obtained from *L. angustifolia* Miller as 24.12% caryophyllene, 16% beta-phelandrene and 15.69% eucalyptol (1,8-cineol). Maral et al., [28] reported the major components of essential oil obtained from *L. angustifolia* Miller as linalyl acetate (34.50%), linalool (33.68%), camphor (5.04%), and 1.8-cineole (4.3%). Arabaci and Bayram [29] reported in their study that the linalool ratio varied between 34.3–54.6%, and the linalyl acetate ratio varied between 24.0–29.0%. Kara and Baydar [30] reported that the ratio of linalool ranged between 28.5–43.9%, the ratio of linalyl acetate between 3.76–42.5%, and the ratio of camphor between 4.11-19.8%.

When the results obtained from the study were evaluated, it was determined that different drying temperatures were effective on both the content and components of the essential oil. To obtain the drug with the highest essential oil content, 35°C can be recommended as the most suitable drying temperature. Likewise, different drying temperatures were also effective on the components of the essential oil, and it was determined that it should be dried at 55 °C to obtain eucalyptol-rich essential oil in production. It can be determined that drying temperatures above 55°C should not be used as they reduce both the essential oil content and the ratio of main components.

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