Research Article/*Araştırma Makalesi*

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AKDENİZ ÜNİVERSİTESİ
ZİRAAT FAKÜLTESİ DERGİSİ
(2014) 27(2): 113-117

Changes in the essential oil content and composition of lavandin (*Lavandula x intermedia* Emeric ex Loisel.) under the natural and artificial drying conditions

Doğal ve yapay kurutma koşullarında lavantanın (*Lavandula x intermedia* Emeric ex Loisel.) uçucu yağ oranı ve kompozisyonundaki değişim

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ARTICLE INFO

Received 20 March 2014
Received in revised form 31 August 2014
Accepted 05 September 2014

***Keywords*:**

Lavandin
Essential oil content
Natural and artificial drying

ABSTRACT

The research was conducted with the aim to investigate effects on the essential oil content and composition of lavandin (*Lavandula x intermedia* Emeric ex Loisel.) of drying in natural (in the shade and the sun) and artificial drying conditions (30, 35, 40, 45, 50, 55, 60, 65 and 70 ^{0}C in drying machine). Essential oil content of fresh stem and dry stemless flower harvested in full blooming period of lavandin was obtained by water distillation method, and composition of essential oil was identified with GC/MS. The effect of the drying method on essential oil content of lavandin was statistically significant ($p<0.01$), and the highest essential oil content was obtained from in 35 ^{0}C (9.48%), the lowest essential oil content from the 70 ^{0}C (9.48%). The essential oil content of lavandin was decreased in the higher drying temperatures (50-70 ^{0}C). Linalool, linalyl acetate, camphor, borneol, neryl acetate, α-terpineol and geranyl acetate in essential oil of fresh stem and dry stemless flower lavandin were determined as the main components. The main components ratio of lavandin varied according to drying methods, and the highest linalool, linalyl acetate, camphor, borneol, neryl acetate, α-terpineol and geranyl acetate content were determined in 30 ^{0}C, 65 ^{0}C, 45 ^{0}C, 50 ^{0}C, 55 ^{0}C, 60 ^{0}C and 60 ^{0}C, respectively, in drying machine. The optimal drying temperature in term of both essential oil content and their quality standards was determined as 35-40 ^{0}C, and both essential oil content and components were decreased in the higher drying temperature and natural drying. Generally, natural flower color of lavandin in all the drying temperature was to be discoloration compare to fresh stem flower.

MAKALE BİLGİSİ

Alınış tarihi 20 Mart 2014
Düzeltilme tarihi 31 Ağustos 2014
Kabul tarihi 05 Eylül 2014

***Anahtar Kelimeler*:**

Lavanta
Uçucu yağ oranı
Doğal ve suni kurutma

ÖZ

Bu araştırma, doğal (gölgede ve güneşte) ve yapay kurutma (kurutma makinesinde 30, 35, 40, 45, 50, 55, 60, 65 ve 70 ^{0}C) koşullarının lavantanın (*Lavandula x intermedia* Emeric ex Loisel.) uçucu yağ oranı ve kompozisyonu üzerine etkisini araştırmak amacıyla yürütülmüştür. Tam çiçeklenme döneminde hasat edilen lavantanın taze saplı ve kuru sapsız çiçek uçucu yağ oranları Clevenger cihazında su distilasyonu yöntemiyle ve uçucu yağ bileşenleri GC/MS cihazında belirlenmiştir. Araştırmada kurutmanın lavantanın uçucu yağ oranına etkisi istatistiksel olarak önemli ($p\leq0.01$) olmuş ve en yüksek uçucu yağ oranı 35 ^{0}C (%12.63) ve en düşük 70 ^{0}C (%9.48) kurutma sıcaklığında tespit edilmiştir. Yüksek kurutma sıcaklıklarında (50-70 ^{0}C) lavantanın uçucu yağ oranında azalma olmuştur. Lavantanın taze saplı çiçek ve kuru sapsız çiçek uçucu yağında linalool, linalil asetat, kafur, borneol, α-terpinol, neril asetat ve geranil asetat ana bileşenler olarak tespit edilmiştir. Uçucu yağ ana bileşenlerin oranları kurutma metotlarına göre farklılık göstermiş, en yüksek linalool 30 ^{0}C'de, linalil asetat 65 ^{0}C'de, kafur 45 ^{0}C'de, borneol 50 ^{0}C'de, neril asetat 55 ^{0}C'de, α-terpinol ve geranil asetat oranı 60 ^{0}C'de tespit edilmiştir. Lavantanın hem uçucu yağ oranı hem de kalite standartları bakımından uçucu yağ bileşenlerini değerlendirdiğimizde en uygun kurutma sıcaklığının 35-40 ^{0}C olduğu, daha yüksek yapay kurutma sıcaklıklarında ve doğal kurutma koşullarında hem uçucu yağ oranı hem de uçucu yağ kompozsiyonunda az da olsa düşüş olmuştur. Genel olarak, taze saplı çiçek rengi ile karşılaştırıldığında tüm kurutma sıcaklıklarında lavantanın doğal çiçek renginde değişimler ortaya çıkmıştır.

1. Introduction

In agricultural production there is a constant search for alternative crops in order to enhance product range and the present production systems. Being resistant to drought and temperature (Weiss 1997), lavender is an essential oil plant that has potential for being considered for arid agricultural areas. Essential oil obtained from lavender flowers is important for pharmaceutical, perfumery and cosmetics industries. While part of fresh stem flower lavender is used for essential oil production, the rest is dried and used for producing lavender buds. The most effective way of preserving freshly harvested plants with high moisture content for utilizing and conveniently transporting plants are drying. Drying on the other hand is a method employed for preventing some chemical reactions and prolonging crops' shelf life by slowing down the growth of microorganisms (Diaz-Maroto et al. 2003). Natural drying is a widely utilized method due to its characteristics of being practical and cost effective. However, natural drying has its disadvantages, particularly in terms of its inadequacy in meeting quality standards (Soysal and Öztekin 2001). Since natural drying is established through the natural circulation of air, adequate amount of air does not go through the dried material, contaminants (such as dust, insects, etc.) are introduced to the crops, relative humidity varies within the day and accordingly homogenous and fast drying cannot be achieved and product quality decreases (Öztekin et al. 1999). High temperature and long term drying process on the other hand cause heat damage on plants, impair crops' texture, color, scent and nutritional value, and reduce their quality and accordingly market value (Yongsawatdigul and Gunasekaran 1996). In addition, during the drying process of aromatic plants, moisture is moved on leaf surfaces with diffusion and causes loss of essential oil by dragging it along (Cremasco 2003). In order to prevent quality and essential oil losses during drying of medicinal and aromatic plants, the optimum balance between quality and production cost can be achieved by determining the most suitable drying method and temperature in terms of shortening drying process and reducing energy consumption.

The present study was carried out for the purpose of determining the most suitable drying method and temperature by comparing the essential oil content, composition and discoloration of lavandin dried under natural and artificial drying conditions.

2. Materials and Methods

The material used in the research was the fresh stem flowers of the 'Super' lavandin variety belonging to the species of *Lavandula x intermedia* Emeric ex Lois founded in 2007 in fields of Suleyman Demirel University's Agricultural Research area.

2.1. Drying methods

1 kg fresh stem flowers harvested at the full blooming stage was dried according to follows methods. The laboratory study was laid out in a randomized plots design with four replications.

a- Natural Drying (Shade and Sun Drying): After the harvest while part of the plants were preserved at room temperature in a covered yet airy environment for shade drying, for sun drying the plants were thinly laid on wire shelves and exposed to sunlight. The plants were turned upside down on a daily basis. Also, in addition to prevent lavandin flowers from falling off wire shelves were covered with gauze tissues.

b- Artificial Drying (in drying machine): 1 kg lavandin samples were dried at 30, 35, 40, 45, 50, 55, 60, 65 and 70 ^{0}C in the drying system.

2.2. Artificial drying system

System consists of an electrical resistance dryer, 4 caged and continuously weighting and data accusation. There are 4 drying shelves having 1 m^2 capacity and located in each of the separate drying cage for experimental repetition purposes. Air heated by the electrical resistance is forced to move to the shelves from bottom by the fan. Drying shelves have independent air outlets and dryer connected to the mixing room which is located behind of the dryer system. In this outlet air capacity and velocity of the dryer are controlled manually by changing diameter of the outlet pipe with the help of clap. Moreover, dryer has property of mixing fresh air with the interior drying air.

Micro-data loggers are located in different places in the dryer system. These data-loggers record the temperature, relative humidity and light luminance in chosen time interval continuously. Electronic drying temperature control system has the accuracy of 0.1 ^{0}C between 30-60 ^{0}C. Inlet air velocity is controlled by single and common clap, but outlet air velocities are controlled by four different claps for each outlet pipe, and also these four pipes attached to a main outlet pipe which has another clap. In this way; air velocity in the system can be controlled separately for each shelf or four of them together. In order to calculate drying curve exactly, each shelves weighted continuously with load-cell having accuracy as 1g. All these recorded data inputted and processed by MATLAB software (Bayhan et al. 2011).

2.3. Determination of moisture

For the purpose of determining moisture rates, 100 g fresh stem lavandin samples were wrapped in blotting papers with three repetitions right after the harvest, were kept for 24 hours at 105 ^{0}C in drying oven, moisture contents were determined in % and after the drying process in the system also the dried moisture contents were determined.

2.4. Flower colors

The colors 'L (brightness), +a (red) and -b (blue), -a (green) +b (yellow)' of fresh stem flowers and each dried flowers of lavandin identified by using Minolta CR-400.

2.5. Essential oils distillation

100 g fresh stem flower in 1 L water and 50 g dry stemless flower samples in 500 ml water from each dying method and temperatures were extracted by hydro-distillation for 3 hours using Clevenger apparatus for determining the essential oil content (v/w %). The essential oil samples were stored at +4 ^{0}C until the basic essential oil compounds were determined.

2.6. GC-MS analysis

Essential oil components were identified by GC-MS (Gas Chromatography-Mass Spectrometry) under the following conditions: capillary column, CP-Wax 52 CB (50 m x 0.32 mm; film thickness = 0.25 μm); oven temperature program, 60^{o}C raised to 220^{o}C at a rate of 2^{o}C/min and than kept at 220^{o}C for 10 min); total run time 60 min; injector temperature, 240^{o}C; detector temperatures, 250^{o}C; carrier gas, helium at flow rate of

20 ml/min. Identification of constituents was carried out with the help of retention times of standard substances by composition of mass spectra with the data given in the NIST library (Stein 1990). After lavandin products were obtained according to the methods explained above, they were stored at 4°C until GC-MS analyses. 1 µL of essential oil diluted with *n*-hexane was injected into the GC-MS system.

The data were analyzed using the JUMP 5.0.1 statistical package program; significant differences between the means were separated using the DUNCAN test.

3. Results

3.1. Essential oil contents

Effects of varying drying temperatures on lavandin essential oil content were found out to be statistically significant at $p \leq 0.01$ level of significance. Significant differences occurred between the fresh stem flowers and dried flowers in terms of essential oil contents. While the essential oil contents in fresh stem lavandin, and the shade dried and sun dried lavandin were determined to be 1.73%, 10.0% and 9.15% respectively, for the flowers dried in the drying system the highest essential oil contents were found out to be in plants dried at 35 and 40 ^{0}C with 12.63% and 11.38%, and the lowest essential oil content of 9.48% was determined to be in the plant dried at 70 ^{0}C (Table 1).

The moisture content during the full bloom period as the most convenient harvest time for fresh lavandin is about 50%. After the harvested lavandin plant were dried with different methods and temperatures, moisture content fell below 10% and varied between 5.59% and 9.88%. As the moisture content in dried lavandin diminishes down to zero, a slight decrease in essential oil content can be observed. For instance, while in dried lavandin samples with 5.58%, 5.98% and 6.37% moisture content the respective essential oil content were determined to be 9.15%, 9.65% and 9.48%, in samples with 9.88%, 8.37% and 8.95% moisture content, the essential oils were 12.63%, 11.83% and 11.48% respectively (Table 1). Essential oil compositions, on the other hand, varied on the basis of moisture content. It was determined that the most suitable moisture content in dried lavandin was between 8.37% and 9.88% where the highest essential oil rates were obtained.

3.2. Chemical composition of the essential oil

Essential oil components and their contents obtained with GC-MS in fresh stem lavandin flower, each dying method and temperatures were shown in Table 1. Twenty-two essential oil components in lavandin essential oil were determined. Linalool, linalyl acetate, camphor, α-terpineol, borneol, neryl acetate and geranyl acetate were determined as the main constituents in the fresh stem flower and dry stemless flower essential oil of lavandin.

The rates of essential oil components in lavandin essential oil varied according to drying methods and temperatures. In the research, while the highest linalool content (50.92%), linalyl acetate (33.76%), camphor (2.30%), α-terpineol (3.10%), borneol (2.90%), neryl acetate (2.7%) and geranyl acetate (1.2%) were determined in 30 ^{0}C, 65 ^{0}C, 70^{0}C, 60 ^{0}C, 50 ^{0}C, 55 ^{0}C and 60 ^{0}C, respectively, the lowest linalool content (39.95%), linalyl acetate (26.46%), camphor (1.10%), α-terpineol (1.20%), borneol (1.10%), neryl acetate (1.10%) and geranyl acetate (0.65%) were obtained from 65 ^{0}C, 55 ^{0}C, 45 ^{0}C, 30-40-45 ^{0}C, 35-45 ^{0}C, 30-45 ^{0}C and 40 ^{0}C, respectively (Table 1).

Table 1. Essential oil content (%) and composition (%) of lavandin in different drying methods and temperatures.

Çizelge 1. Farklı kurutma metotları ve sıcaklıklarında lavandinin uçucu yağ oranı (%) ve kompozsiyonu (%).

		Drying methods and temperatures (^{0}C)											
RT	Components	Fresh stem flower	Drying in the shade	Drying in the sun	30	35	40	45	50	55	60	65	70
13.9	β-myrcene	0.65	0.53	0.28	0.58	0.33	0.44	0.60	-	0.81	0.68	0.84	0.57
16.1	D-Limonene	0.42	0.57	0.23	0.34	-	0.27	0.32	-	0.51	0.09	0.58	0.34
16.7	1,8-Cineole	1.30	1.20	1.10	1.10	1.10	1.10	1.10	2.12	1.20	0.27	1.10	1.10
17.9	β- Ocimene	0.77	0.59	0.31	0.71	0.45	0.51	0.64	1.20	0.67	1.10	0.69	0.62
18.9	γ- terpinene	1.10	1.50	0.63	1.10	1.30	1.10	1.10	0.56	1.10	0.58	1.10	1.10
19.3	3-Octanone	0.41	0.60	0.34	0.57	0.35	0.46	0.56	-	0.67	1.10	0.46	0.35
20.2	Hexyl actetate	0.42	0.62	0.43	0.76	0.47	0.61	0.87	-	0.75	0.47	0.53	0.49
29.2	Hexyl butan.	0.69	0.53	0.57	1.5	0.91	0.91	0.95	-	0.73	0.68	0.66	0.84
31.3	1-octen-3-ol	-	-	-	-	-	-	-	-	-	-	-	0.18
36.6	camphor	1.40	1.40	1.20	1.30	1.30	1.20	1.10	1.30	1.30	2.10	2.20	2.30
37.6	L-linalool	43.83	42.63	44.18	50.92	45.40	47.49	49.74	50.10	49.22	44.37	39.95	44.19
38.3	Linayl acetate	26.86	31.60	31.75	28.3	31.92	31.14	30.84	29.76	26.46	31.44	33.76	33.01
41.4	Neryl acetate	1.20	1.20	1.20	1.10	1.20	2.20	1.10	1.20	2.70	1.20	1.20	1.20
45.0	Farnesene	-	-	0.23	-	0.37	0.29	0.26	-	0.19	-	0.27	0.33
45.9	Lavandulol	-	-	0.15	-	0.20	-	-	-	0.19	-	-	0.11
47.4	α-Terpineol	1.30	1.30	1.30	1.20	1.30	1.20	1.20	1.30	3.20	3.10	1.30	1.30
47.8	Borneol	1.30	1.20	1.40	1.10	1.10	1.20	1.10	2.90	2.20	1.40	1.30	2.60
50.7	Geranyl acetate	1.10	1.10	1.10	1.10	1.10	0.65	0.98	1.10	1.10	1.20	1.00	1.10
53.0	Cuminal	0.55	-	0.53	0.37	0.60	0.47	0.36	-	0.51	-	0.47	0.51
55.9	Geraniol	2.10	1.10	1.10	1.10	2.60	1.10	1.10	1.10	1.10	1.20	1.10	1.10
73.7	Cadinol	-	-	-	-	-	-	-	-	-	-	0.24	-
75.6	Bisabolol	1.10	1.10	1.60	0.88	1.10	1.12	0.67	0.52	0.90	1.10	1.10	1.00
Essential oil content (%)		1.73 d	10.00 c	9.15 c	10.45 c	12.63a	11.83 ab	11.48 b	10.33 c	10.00 c	10.00 c	9.65 c	9.48 c
		CV (%): 5.19				P value: 0.001							

Means in the same letters are not significantly different at the 0.001 level as statistically

3.3. Lavandin flower and stem colors

The effect of the drying methods and temperatures on flower L, +a and –b colors and stem L, –a and +b colors of lavandin were statistically significant (p<0.01) (Table 2). Comparing the L, +a, –b values, which give lavandin its color, of processed samples with fresh flowers and stems (respectively 13.42, 14.05, 11.47 h^0 and 10.40, 12.20, 6.75 h^0) showed that in all drying systems discoloration (fading) occurred in flowers and stems. In comparison to natural drying (shade and sun drying methods) discoloration occurred at a lower rate with the application of drying heat, and the minimum level of discoloration was found out to occur at 70 ^{0}C where the samples nearly retained their initial (fresh) color. This may be explained with the fact that at 70 ^{0}C the drying process takes a short time and therefore the samples are exposed to heat for a shorter duration. While no significant loss of essential oil was observed at 70 ^{0}C drying temperature in comparison to natural (shade and sun) drying, when compared to the highest essential oil content found at 35 ^{0}C the loss was 3.15 %.

4. Discussion

The essential oil content and moisture content of lavandin varied drying methods and temperatures. Although essential oil contents decrease in the shade dried and sun dried samples and the samples dried in the system at 50 ^{0}C and higher temperatures (55, 60, 65 and 70 ^{0}C), no significance was found statistically. Essential oil contents obtained from dried lavandin flowers were found out to be higher than those found in fresh stem flowers due to higher amount of dried material content, lower moisture content and the fact that they did not have stems. However, at drying temperatures higher than 35 ^{0}C it was observed that the essential oil content of lavandin decreases. This decrease is due to the essential oil present in the surface tissue being easily dragged along with water vapor due to the effect of high temperature. Similarly to the findings of the present study, it was reported in previously conducted studies that the essential oil contents and compositions of medicinal and aromatic plants are significantly affected by drying methods (Raghavan et al. 1997). While Buggle *et al.* (1999) reported that the essential oil contents of *Cymbopogon citrates* decrease as the temperature rises from 30 ^{0}C to 90 ^{0}C, Braga et al. (2005) determined that the highest contents of essential oil in *Piper hispidinervium* exhibit variance at temperatures up to 50 ^{0}C and start to decrease at higher temperatures. Pinto et al. (2007) reported that the essential oil content of lavandin is significantly affected from both sun drying and shade drying. Soares et al. (2007) on the other hand reported that essential oil loss of basil significantly increases in cases where the air temperature exceeds 40 °C.

The essential oil composition of lavandin varied drying temperatures. Preventing microbial development and certain chemical modifications, drying process in plants may affect plant quality through changes in the view and the aroma of the plant by causing essential oil loss or formation of new components due to oxidation or esterification reactions (Hossain et al. 2010). Similarly to our findings, in the drying process Soares et al. (2007) carried out at 4 different temperatures (40, 50, 60 and 70 ^{0}C) and with 2 different air flow rates (0.9 and 1.9 $m\ s^{-1}$) for basil (*O. basilicum* L.), the authors determined the highest essential oil content at 40 ^{0}C temperature with 1.9 $m\ s^{-1}$ air flow rate and the highest linalool content in the samples dried at 50 and 60 ^{0}C with 1.9 $m\ s^{-1}$ air flow rate. In the study Radunz et al. (2002) conducted on *Lippia sidoides* plant with 5 different drying temperatures (natural, 40, 50, 60 and 70 ^{0}C), it was determined that the thymol and ρ-cymene contents were insignificant in terms of quality, yet caryophyllene values increase significantly at 50, 60 and 70 ^{0}C drying temperatures in comparison to fresh plants.

The flower and stem colors of lavandin varied drying methods and temperatures. In previous studies conducted with drying methods, Yousif et al. (2006) examined the effects of several drying methods (freeze drying, hot air drying and vacuum microwave drying) on Mexican thyme (*Lippia berlandieri* Schauer) and determined that the thyme samples dried via hot air were darker, less greener and had lower rehydration values. In the study Argyropoulos and Muller (2011) conducted on lemon balm (*Melissa officinalis*), it was reported that although both the color and medical quality of the plant can be preserved at 30 ^{0}C the process takes too long, and on the other hand higher temperatures cause discoloration, and loss of essential oil and rosmarinic acid.

Table 2. Flower and stem colors (h^0) of lavandin in different drying methods and temperatures.

Çizelge 2. Farklı kurutma metotları ve sıcaklıklarında lavandinin çiçek ve sap rengi (h^0).

Drying methods and temperatures ^{0}C)	Flower colors			Stem colors		
	L	+a	-b	L	-a	+b
Fresh stem flower	13.42 a*	14.05 a**	11.47 a*	10.40 a*	12.20 a*	6.75 a*
Drying in the shade	9.37 de	9.72 e	9.31 d	7.67 ef	8.50 e	5.12 def
Drying in the sun	9.80 de	10.25 de	9.35 d	7.70 ef	8.40 e	5.20 def
30	9.50 de	10.17 de	8.42 ef	8.47 cde	9.67 cd	5.50 de
35	13.15 a	14.05 a	11.47 a	9.80 a	11.17 b	6.22 ab
40	12.57 a	13.75 a	11.12 a	9.60 ab	10.82 b	6.07 bc
45	11.40 b	12.22 b	9.92 d	8.72 cd	9.75 c	5.55 cd
50	10.85 bc	11.40 bc	10.30 bc	7.45 f	8.17 e	4.85 f
55	10.07 cd	10.67 cd	9.30 d	8.07 cdef	8.87 cde	5.30 def
60	9.75 de	10.25 de	9.15 de	7.62 ef	8.35 e	4.95 ef
65	9.07 e	9.55 e	8.32 f	7.97 def	8.75 de	5.12 def
70	12.70 a	13.40 a	11.00 ab	8.85 bc	9.57 cd	5.47 de
CV (%)	5.37	5.56	5.50	6.94	7.03	7.07
P value	0.012	0.001	0.001	0.024	0.001	0.001

L: brightness, +a: red, -b: blue, –a: green ve +b: yellow

*: Means in the same columns followed by the same letters are not significantly different

5. Conclusions

The essential oil content of dry stemless lavandin flowers dried in both natural and artificial drying conditions were significantly higher than fresh stem flowers. While the highest essential oil content was obtained from 35-40 ^{0}C drying temperatures, between essential oil content of the natural drying and 50-70 ^{o}C drying temperatures was not statistically significantly different. Linalool, linalyl acetate, camphor, α-terpineol, borneol, neryl acetate and geranyl acetate were identified as major compounds of lavandin's essential oil, and their rates varied according to drying methods and temperatures. The drying flower and stem colors of lavandin in all drying methods and temperatures were discoloration, and optimal drying temperature was determined as 35 ^{0}C.

As a result of this study, in term of the essential oil content, composition and their quality, it is possible to say that optimal drying in lavandin was 35-40 ^{0}C.

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