

The effect of harvest time on the bioactive properties and volatile components of lavender (*Lavandula officinalis*)

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Abstract

This study was designed to determine the effect of harvest time on the bioactive properties and volatile components of flowers, leaves and stems of *Lavandula officinalis*. The bioactive properties were examined in terms of total phenolic content, antiradical activity and antioxidant capacity by the Folin-Ciocalteu, 1,1-diphenyl-2-picrylhydrazyl scavenging and phosphomolybdenum methods, respectively. Volatile components were detected with gas chromatography-mass spectrometry. Water and methanol were used for extraction solvent and three different parts of the day (morning, midday and evening) were selected for harvest time. Water extracts had higher total phenolic contents (337.2-771.5 mg gallic acid equivalents (GAE)/g dry extract) than methanolic extracts (170.3-320.8 mg GAE/g dry extract), and in general, midday values were less than morning and evening rates and had a significant difference depending on harvest time. A similar feature was observed in respect to antioxidant properties. The inhibition rates (%) of flowers and leaves were higher than those of the stems for either extract. In terms of the volatile components linalool, camphor, 1,8-cineole, terpinen-4-ol and isoborneol were the most representative compounds in the samples.

Keywords: *Lavandula officinalis*, lavender, harvest, antioxidant, volatile component

1. Introduction

Lavender, a crucial medicinal plant, contains many important essential oils which are used in the food and fragrance industries for example in soaps, colognes, perfumes, skin lotions and other cosmetics (Palá-Paúl *et al.*, 2004). Lavender essential oil contains several components, such as linalool, linalyl acetate, lavandulyl acetate, camphor and 1,8-cineole (Şerban *et al.*, 2011). In the food industry, it is used as a natural flavouring agent for beverages, ice cream, baked goods, and chewing gum (Kim and Lee, 2002). Also, it has been used in aromatherapy and alternative medicine because of its useful effects on the central nervous system. In addition, essential oil in lavender can be used in pharmaceutical applications for its therapeutic properties such as antiseptic, antidepressant, and antiviral (Haas, 2001). Moreover, antioxidants are used in many functional foods to contribute additional physiological benefits over normal nutritional requirements (Krishnaiah *et al.*, 2007).

Furthermore, researchers have recently focused on the antioxidant activity of lavender essential oil. That antioxidant activity has also been used to protect food products from oxidative rancidity, loss of labile compounds, and the formation of off-flavours in the food industry (Danh *et al.*, 2012). Presently, majority of antioxidants used in food manufacturing are artificial compounds. For example, butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT) are commonly used as antioxidants in the food industry (Gulcin *et al.*, 2002). However, some studies have reported that these antioxidants cause liver damage and carcinogenesis (Grice, 1986; Wichi, 1988). Because consumers' dislike towards artificial food additives, food producers demand more natural antioxidants to protect human health and their markets (Jayaprakasha *et al.*, 2001; Loliger, 1991; Oktay *et al.*, 2003).

Phenolic compounds are crucial natural antioxidants which are provided by plants such as lavender. These compounds

not only provide protection for plants for example against stress, UV and radiation, but they also preserve food against oxidation (Torras-Claveria *et al.*, 2007). Also, they add nutritional value to foods for a healthy diet and provide several beneficial effects to the human body because of their anti-microbial, cardio protective, anti-allergenic, and anti-inflammatory activities (Kamatou *et al.*, 2008). In addition, several studies have determined that phenolic compounds reduce the risk of chronic diseases (Figueiredo *et al.*, 2008; Quave *et al.*, 2008; Takaki *et al.*, 2008). These compounds contain primarily simple phenols, phenolic acids, coumarins, and flavonoids which occur in the form of glycosides or esters in lavender (Torras-Claveria *et al.*, 2007).

The leaves, flowers and rarely the stems of the lavender are used for mentioned purposes in several countries. But the bioactive properties of these parts of the plant may change by the harvest times. Tulukcu *et al.* (2012) detected significant effect on the bioactive properties of the *Salvia sclarea* leaves collected in different time of the day. Ozkan *et al.* (2010) studied the influence of harvest time on essential oil composition, phenolic constituents and antioxidant properties of Turkish oregano (*Origanum onites* L.) and expressed that these properties of Turkish oregano changed importantly depending on vegetative periods of growing season. Tavarini and Angelini (2013) stated the following conclusion as a result of their work: the harvest time played a key role in determining the stevia quality, influencing the rebaudioside A/stevioside ratio.

The main purpose of this study is to determine antioxidant properties and volatile compounds of flowers, leaves and stems of lavender with a special focus on variations in harvesting period. This is the first report that investigate the effect of the collection time of the lavender in a day.

2. Experimental

Plant material

Lavender (*Lavandula officinalis*) was obtained from plants cultivated at the Experimental Horticulture area of Cumra Agricultural Vocational High School, Konya, Turkey. This plant belongs to the *L. officinalis* germplasm bank of this school. Lavender was collected at three different periods of the day; morning (07.00-08.00 am), midday (00.00-01.00 pm) and evening (07.00-08.00 pm) and was then divided into three parts (stems, flowers, leaves). They were harvested in June and July 2010.

Sample extraction for antioxidant activity tests

Samples were dried at room conditions for one week and powdered and then extracted with methanol and water (1:20, w/w) for 24 h at 25 °C in the dark. The extracts

were centrifuged at 4,100 rpm for 15 min then filtered and the supernatants were collected. After filtration, the clear supernatants were evaporated with rotary evaporator (Rotavapor R-200; Büchi, Flawil, Switzerland) under vacuum at 50 °C. After these treatments, the dry extracts were preserved at +4 °C.

Determination of total phenolic content

Total phenol estimation in extract was determined by the Folin-Ciocalteu colourimetric method. To each tube, 2,400 µl distilled water, 40 µl extracts (solvent for control) and 200 µl Folin-Ciocalteu reagent were added. After 5 min, 600 µl sodium carbonate (20%) and finally 760 µl distilled water were added. The solution was homogenised by a mixer and incubated at room temperature for 2 h in the dark. After incubation, absorbance of the reaction mixture was measured at 765 nm (Yalcin and Kavucuoglu, 2014). The amount of total phenolic compounds was expressed as gallic acid equivalents (GAE) in milligrams per gram dry plant extract.

Determination of antiradical activity

The 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical scavenging method was used for the determination of antiradical activity of the samples. This method was adapted by some modifications from the method used by Tulukcu *et al.* (2012). A 100 µl solution of extracts (solvents for control) was added to each tube and then mixed with 3,500 µl DPPH methanol solution (10 mg/kg) and homogenised with a mixer. Then, after 30 min incubation at room temperature in the dark, the absorbance was measured at 517 nm. Methanol was used as a blank to zero the absorbance. The radical scavenging activities were calculated by the equation:

$$\text{Radical scavenging activity (\%)} = [(A_0 - A_1) / A_0] \times 100$$

Where, A_0 = the absorbance of the control, and A_1 = the absorbance of the sample.

Evaluation of total antioxidant capacity

Total antioxidant capacity by the phosphomolybdenum method was measured spectrophotometrically using the procedure of Polat *et al.* (2014). To each tube, 400 µl solution of extracts (solvents for control) was added and mixed with 4 ml of the reagent solution (0.6 M sulphuric acid, 28 mM sodium phosphate and 4 mM ammonium molybdate). After homogenisation with a mixer, the reaction mixture was incubated at 95 °C for 90 min and absorbance was measured at 695 nm. Results were expressed as ascorbic acid equivalents (AAE) in milligrams per gram dry plant extract.

Analysis of volatile composition

Analysis was performed according to the procedure described by Yalçin *et al.* (2007) by gas chromatography-mass spectrometry using a mass selective detector (Agilent 7890A gas chromatography system; Agilent Technologies, Avondale, AZ, USA) and a HP-5MS column (60 m × 0.250 mm internal diameter; film thickness, 0.25 µm; Supelco, Palo Alto, CA, USA). The oven temperature was held at 40 °C for 10 min, heated to 95 °C at 3 °C/min, heated from 95 to 210 °C at 10 °C/min, and finally increased to 210 °C/min and held for 10 min. The carrier gas was helium with a flow rate of 0.5 ml/min. The voltage of the electron ionization detector was 70 eV. The compounds adsorbed by the fibres were desorbed from the injection port for 15 min at 50 °C in the splitless mode. The compounds were identified by comparison with spectra from the Flavor 2, NIST 05a, and Wiley7Nist05 libraries and by using internal standards.

Statistical analysis

Experimental results were the means ± standard deviations of four parallel measurements. Analysis of variance was performed by ANOVA procedures (SPSS 18.0 for Windows; SPSS Inc., Chicago, IL, USA). Significant differences between means were determined by Duncan's Multiple Range tests. *P*-values <0.05 were regarded as significant.

3. Results and discussion

Bioactive properties

The total phenolic concentration of the flowers harvested at different times periods, morning, midday and evening, and extracted with water were significantly different. Although the total phenolic concentration of leaves and stems harvested at midday was significantly different, that of leaves harvested in the morning and evening was not significantly different. Table 1 shows that the phenolic concentration of flowers and stems was reduced at midday, but that this concentration was increased at evening. However, the phenolic concentration of leaves gave the opposite results. Furthermore, the phenolic concentration of the methanolic extract of leaves, flowers, and stems was not significantly different. The highest phenolic concentration was found in leaves harvested at midday using the water extraction method.

In a similar study, the total phenolic concentration of *Lavandula angustifolia* was found to be 50.6 mg GAE/g (Spiridon *et al.*, 2011). Gonçalves *et al.* (2013) investigated the effect of heat on extraction of the phenolic component. In that study, which used the hot extraction method, they evaluated 1,325.09 µmol_{GAE}/g_{dw} phenolic concentration and it decreased to 308.81 µmol_{GAE}/g_{dw} with the cold extraction method. The phenolic content of the plant may change according to species, different parts of the plant and location. For example, in a study on three wild species of lavender and the phenolic concentrations were as follows:

Table 1. Total phenolic content, inhibition (%) and antioxidant capacity of *Lavandula officinalis* flowers, leaves and stems depending on harvest time and solvent of extraction.^{1,2}

Harvest time	Water extract			Methanol extract		
	Flower	Leaf	Stem	Flower	Leaf	Stem
Total phenolic content (mg GAE/g dry extract)						
Morning	585.7±42.53 A,a	450.2±38.54 B,b	601.7±76.51 A,a	291.7±6.54 A,a	181.4±14.62 B,a	287.9±5.40 A,a
Midday	337.2±13.40 C,b	771.5±54.49 A,a	430.2±1.27 B,b	320.8±54.55 A,a	180.7±23.84 C,a	253.4±5.48 B,a
Evening	583.9±26.60 A,a	442.7±60.89 B,b	545.8±12.03 A,a	277.5±7.79 A,a	170.3±5.30 B,a	279.7±36.73 A,a
% Inhibition						
Morning	90.1±0.09 B,a	91.6±0.08 A,a	25.8±0.69 C,c	92.1±0.18 A,a	88.2±0.28 B,b	50.1±3.62 C,a
Midday	81.7±1.12 B,b	91.1±0.21 A,b	33.5±0.82 C,a	54.3±2.56 B,c	88.0±0.24 A,b	28.0±2.08 C,c
Evening	53.1±1.24 B,c	91.4±0.13 A,a	29.1±1.94 C,b	64.4±1.71 B,b	88.7±0.11 A,a	41.6±4.85 C,b
Antioxidant capacity (mg AAE/g dry extract)						
Morning	5,142.0±182.11 B,a	5,268.2±77.97 B,b	6,997.7±310.35 A,a	4,746.7±115.93 A,c	3,183.4±323.83 B,a	3,390.3±83.33 B,b
Midday	3,455.8±185.40 C,b	8,733.3±197.61 A,a	5,187.7±179.28 B,b	12,029.7±525.62 A,a	2,836.3±63.02 C,b	3,529.3±47.86 B,a
Evening	5,393.7±388.63 B,a	4,905.0±205.62 B,c	6,787.6±1,085.57 A,a	9,443.3±395.27 A,b	3,453.0±168.81 B,a	3,079.0±64.27 B,c

¹ Mean ± standard deviation. Different capital letters indicate the statistical differences in the same column (*P*<0.05). Different small letters indicate the statistical differences in the same row (*P*<0.05).

² AAE = ascorbic acid equivalents; GAE = gallic acid equivalents.

31.3 mg GAE/g in *Lavandula coronopifolia*, 30.8 mg GAE/g in *Lavandula multifida* and 25.2 mg GA/g in *Lavandula stoechas* (Messaoud *et al.*, 2012). Another study on this subject found the following phenolic concentrations: 92.4 µg GAE/mg (*L. stoechas*), 71.3 µg GAE/mg (*Lavandula hybrida*), 97.4 µg GAE/mg (*Lavandula intermedia* 'impress purple'), 79.8 µg GAE/mg (*L. intermedia* 'Provence'), 58.4 µg GAE/mg (*Lavandula latifolia*), 70 µg GAE/mg (*Lavandula allardii* 'rly') and 61.8 µg GAE/mg (*Lavandula dentata*) (Lee *et al.*, 2011). The phenolic concentration of *L. officinalis* leaves was evaluated at 16.2 mg GAE/g_{dw} (Bouayed *et al.*, 2007). Two different extraction method were used to determine the total phenolic concentration of lavender, it was found as 153.92 µg GAE/mg with water extraction while it was equal 226.74 µg GAE/mg with methanol extraction (Gülçin *et al.*, 2004).

The phenolic concentration of *L. stoechas* species of lavender changed in the flowers and leaves. In flowers phenolic concentration was observed as 56.65 mg GAE/g_{dw}, this increased to 63.50 mg GAE/g_{dw} in the leaves. Different parts of the plant (stem, flower, leave, seed) may show changeable phenolic activity. When comparing these differences flowers and leaves are richer than other parts of the plant. The phenolic concentration of *Salvia officinalis* a member of Lamiaceae family was measured as 43.50 mg GAE/g_{dw} in the leaves and 22.30 mg GAE/g_{dw} in the stem (Kırca and Arslan, 2008).

The phenolic concentration of *S. sclarea* (clary sage) which is a member of Lamiaceae, changed according to harvesting time and harvesting year (Tulukcu *et al.*, 2012). The phenolic concentration of clary sage was the highest in that collected in the morning of July 2005 and at midday of June 2005, as 97.84 mg GAE/g dry extract. On the other hand, the lowest phenolic concentration of 38.34 mg GAE/g dry extract was measured in sage collected in the evening in July 2005.

Based on the DPPH radical scavenging method, the highest result was found in leaves harvested in the morning by using the methanol extraction. However, the lowest antioxidant activity was observed in stems harvested in the morning with the water extraction. The antioxidant activity of leaves, stems and flowers of lavender harvested at different times (morning, midday, evening) was significantly different ($P<0.05$).

Danh *et al.* (2012) evaluated the DPPH inhibition of lavender flowers (*L. angustifolia*) extracted by supercritical extraction at the highest value of 73.8%, while the lowest value was 21.9%. In that study the efficiency of the supercritical extraction method changed by temperature, pressure, extraction time and lavender species. Different extraction methods changed the DPPH radical scavenging activity of plants. Miliauskas *et al.* (2004) used ethyl acetate, acetone

and methanol for investigation of DPPH inhibition of *L. angustifolia*. They evaluated DPPH inhibition as 2.5, 7.4 and 35.4%, respectively. While water extract of lavender exhibited 45% DPPH inhibition, this increased to 50% DPPH activity by using ethanol extraction (Gülçin *et al.*, 2004). In our study, DPPH inhibition values were found to be higher than those of other researchers. This may have been caused by the used lavender species. Different parts of the lavender (flowers, stems, leaves) exhibited different DPPH inhibition. The DPPH inhibition of *L. stoechas* was increased by rise in plant concentration. At 10 µl/ml the DPPH radical scavenging activity was evaluated as 63.05% (Cherrat *et al.*, 2014). Natural antioxidants can be used as food additives for their antioxidative effects. For this purpose Taha Nejad *et al.* (2012) used the extract of *L. angustifolia* instead of BHA in crude soybean oil. They found that the effect of 0.8 mg/ml extracts of *L. angustifolia* was equal to the effect of 0.2 mg/ml BHA. The highest antioxidant activity (54.4%) was found by using 10 mg/ml extract of *L. angustifolia*.

According to the phosphomolybdenum antioxidant capacity measurement method, lavender leaves and flowers were not significantly different in the samples of harvested in the morning and evening ($P<0.05$). On the other hand, the samples of stem showed opposite results. Lavender flowers and leaves displayed more antioxidant capacity than stems as shown in Table 1. At the harvesting time of midday, the water extract of leaves, flowers and stems had a statistically important difference in terms of antioxidant capacity ($P<0.05$). The antioxidant capacity of leaves, stems and flowers of lavender harvested at different times (morning, midday, evening) are significantly different ($P<0.05$). The antioxidant capacity of flower samples harvested in the morning and evening seemed similar but samples harvested in midday varied from the others. Phosphomolybdenum antioxidant capacity changed according to the harvesting time of the leaf samples. The methanol extract values of leaves and stems obtained by the phosphomolybdenum method were not significantly different ($P>0.05$) for samples harvested in the morning and evening but the flower samples were different from other pieces of lavender. The antioxidant values of lavender samples harvested in midday (flowers, stems, leaves) by the phosphomolybdenum method statistically different ($P<0.05$). No study has been conducted in lavender by using phosphomolybdenum total antioxidant capacity method. In one research about the Lamiaceae family *O. onites L.* was studied according to harvesting time by using the phosphomolybdenum method. In that study antioxidant capacity results change according to harvesting time and the results were evaluated as follows: for June 108.31 mg AAE/g, for July 180.41 mg AAE/g, for August 159.96 AAE/g, and for September 151.87 mg AAE/g (Ozkan *et al.*, 2010). In another research, antioxidant capacity was measured in different plants and the results were as follows: in *Calamintha clinopodium* water extract

it was 13.76 mg AAE/g, in *Teucrium flavum* water extract it was 25.93 mg AAE/g, and in *Thymus algeriensis* water extract it was 218.37 mg AAE/g (Boulekache-Makhlouf and Madani, 2014). The antioxidant capacity of *Origanum syriacum* L. methanolic extract was found to be 335.33 mg AAE/g extract (Ozkan *et al.*, 2007).

Tulukcu *et al.* (2012) studied the effect of harvesting time (morning, midday, evening) and harvesting year of *S. sclarea*. In that research the antioxidant capacity of the methanolic extract of the plant evaluated and the highest value (217.11 mg AAE/g dry extract) was observed in the samples harvested at midday in July 2006. The lowest antioxidant value of 106.40 mg AAE/g dry extract was measured in samples harvested in the morning in June 2006.

Volatile components

The volatile compounds were characterised in the flowers, stems and leaves of *L. officinalis* harvested in different periods of day and are listed in Table 2. Both quantitative and qualitative differences were found in the essential oil composition of the analysed samples depending on the harvest time. Linalool, camphor, 1,8-cineole, terpinen-4-ol and isoborneol were the most representative compounds in the samples. Linalool was determined in all parts of *L. officinalis*. The amount of linalool in the stems was high in

midday (27.88%) harvested samples than morning (25.23%) and evening (24.73%) harvested samples. In the flowers, the highest amount of linalool was found in the evening (26.09%) harvest, the followings were midday (18.38%) and morning (17.59%) harvests. The amounts in the leaves (1.32-5.09%) were less than the stems and flowers and the largest amount was found in the morning harvest. It was proposed that a good lavender includes a high rate of linalool because it has antiviral and antibacterial properties. Moreover, this compound is used in the production of vitamin E and has a protective effect against convulsions (Kim and Lee, 2002). Besides linalool, *cis*-linalool oxide and *trans*-linalool oxide were detected in the samples. Linalool (35.96-36.51%) and linalyl acetate (21.74-14.42%) were found as the principal components in essential oils of *L. angustifolia* (Da Porto *et al.*, 2009). Unfortunately there is no study on the volatile component of lavender harvested in different periods. For this reason we cannot compare the obtained values to other studies in terms of harvest periods. But there are studies related with the composition of volatile component of lavender. In 2012, Danh *et al.* (2012) found that linalool (<43%), linalyl acetate (<23%), camphor (<8%) and borneol were the major compounds of *L. angustifolia*. In their study, the amounts of linalool were higher than ours.

The other major component, 1,8-cineole (also named eucalyptol), is a monocyclic monoterpeno containing

Table 2. Volatile components of *Lavandula officinalis* flowers, leaves and stems depending on harvest time.

Compounds	Flower			Leaf			Stem		
	Morning	Midday	Evening	Morning	Midday	Evening	Morning	Midday	Evening
Limonene	—	—	—	0.15	—	1.41	—	—	—
Myrcene	1.31	0.68	0.78	—	—	—	1.39	1.37	1.28
<i>p</i> -cymene	0.05	1.42	1.19	0.16	0.21	2.77	—	—	—
α -santalene	—	—	—	6.37	4.21	13.24	0.98	0.77	1.14
1,8-cineole	1.3	9.18	3.36	0.62	0.75	12.32	4.89	0.59	5.94
Hexyl butyrate	0.39	0.67	1.21	—	—	—	—	—	—
Butanoic acid	0.86	1.06	1.19	—	—	—	—	—	—
<i>cis</i> -linalool oxide	5.9	7.75	—	1.16	—	0.51	6.04	1.58	4.94
<i>trans</i> -linalool oxide	5.17	7.35	3.11	1.04	0.71	0.31	—	—	—
Camphor	13.5	18.86	15.21	2.28	2.58	0.71	13.55	3.96	10.62
Linalool	17.59	18.38	26.09	5.09	3.81	1.32	25.23	27.88	24.73
Linalyl butyrate	11.36	—	0.06	—	—	1.53	—	—	—
β -pinene	0.75	4.97	4.92	4.68	2.74	—	7.14	9.05	7.31
Tricycloheptane	1.93	0.62	0.53	—	—	—	—	—	—
2-furanone	1.91	1.85	—	—	—	—	—	—	—
Geranyl propionate	—	0.91	1.23	—	—	—	—	—	—
Epoxylinalol 2H-pyran-3-ol	1.57	0.76	0.39	—	—	—	—	—	—
β -caryophyllene	2.77	2.25	—	—	—	—	—	—	—
Acetic acid	—	—	—	—	1.15	—	—	—	—

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Table 2. Continued.

Compounds	Flower			Leaf			Stem		
	Morning	Midday	Evening	Morning	Midday	Evening	Morning	Midday	Evening
4-carvomenthenol	—	—	—	1.17	1.01	0.14	—	—	—
δ-3-carene	—	—	—	0.25	0.23	2.2	—	—	—
trans-pinocarveol	—	—	—	—	1.78	1.07	—	—	—
Eucarvone	—	—	—	—	0.99	3.1	—	—	—
D-verbonene	—	—	—	1.19	1.33	0.33	—	—	—
Carvone	—	—	—	1.42	2.03	2.73	—	—	—
Valencene	—	—	—	5.36	2.63	0.87	—	—	—
α-pinene-10-ol	—	—	—	0.55	1.78	1.32	—	—	—
Thymol	—	—	—	3.82	—	3.76	—	—	—
p-cymen-8-ol	—	—	—	1.73	5.52	—	—	—	—
p-benzoquinone	—	—	—	3.53	—	—	—	—	—
Caryophyllene oxide	—	—	—	0.3	2.26	0.13	—	—	—
Norpinane	—	—	—	3.79	0.85	0.91	—	—	—
Camphepane	—	—	—	0.4	0.23	2.02	—	—	—
Dimethyl-3,6-dihydrobenzothiophene	—	—	—	—	3.77	2.25	—	—	—
α-cadinol	—	—	—	1.44	0.71	0.17	—	—	—
Caryophyllene-5-ol	—	—	—	3.14	1.14	0.93	—	—	—
Ocimene	—	—	—	—	—	1.45	—	—	—
Fenchyl alcohol	—	—	—	—	—	—	1.25	—	0.63
α-fenchyl acetate	—	—	—	—	—	—	—	1.03	—
Geranyl acetate	—	—	—	—	—	—	—	2.01	1.08
terpinen-4-ol	7.16	3.78	10.12	—	—	—	6.95	10.55	5.46
Farnesol	—	0.02	6.68	1.31	0.2	0.38	—	—	—
β-farnesene	—	—	—	—	—	—	1.19	—	—
Nerolidol	—	—	—	—	—	—	—	0.18	1.15
Farnesol	—	—	—	—	—	—	—	3.83	—
Geraniol	—	—	—	—	—	—	1.4	—	1.36
Lavandulol	—	—	—	—	—	—	—	1.99	—
2-cyclohexen-1,4-(1-methylethyl)	1.61	0.64	0.47	1.8	6.32	7.71	0.75	1.01	1.57
Isoborneol	3.61	3.34	3.85	14.19	19.27	1.05	10.25	10.55	4.77
Nerol	—	—	—	2.84	1.15	1.94	0.98	0.45	0.39
Neryl acetate	1.35	0.03	—	—	—	—	—	1.93	—
2,4-dimethylbenzaldehyde	—	—	—	—	—	—	—	1.57	—
4-propyl-benzaldehyde	—	—	—	—	—	—	—	1.35	—
Carvacrol	—	—	—	—	—	—	1.05	0.75	0.76
Coumarin	—	—	—	—	—	—	2.03	0.93	1.93
Cuminaldehyde	1.18	0.48	0.33	0.81	3.58	5.46	—	—	—
Total % identified	81.27	85	80.72	70.59	72.94	74.04	85.07	83.33	75.06

an epoxide group. 1,8-cineole is used as a fragrance in perfumes and for flavouring in the food industry. The highest amounts of 1,8 cineole were in the leaves harvested at midday accounting 12.32% and the least amount was found in stems harvested at midday with a concentration of 0.59%. Sanz *et al.* (2004) studied flowers and leaves from the 51 individual plants of *Lavandula luisieri* 1,8-cineole (0.00-12.32%), fenchone (0.00-1.16%) and camphor (0.0-14.23%) were detected in leaf volatiles. 1,8-cineole (0.00-5.96%),

fenchone (0.00-2.45%) and camphor (0.0-7.22%) in flower volatiles. The major constituents of the essential oil of *L. latifolia* were the oxygenated monoterpenes 1,8-cineole (6.6-57.1%), linalool (3.7-61.1%) and camphor (1.1-46.7%) (Herraiz-Peñalver *et al.*, 2013). Karamanolli *et al.* (2000) reported that *L. angustifolia* essential oil had 1,8-cineole (44.9%), camphor (14.3%), β-phellandrene (5.0%) and α-pinene (4.7%).

Camphor which is a terpenoid, is detected less in leaves (0.71-2.58%) than in stems and flowers. The highest amount was observed in flower harvested at midday (18.86%). A significant decrease occurred in terms of quantity in the midday harvest (3.96%) in stem samples. The essential oils from dried leaves of *Lavandula bipinnata* (Roth) Kuntze (Lamiaceae) were determined by Hanamanthgouda *et al.* (2010); transcarveol (18.93%), pulegone (8.45%), camphor (7.09%) and menthol (5.89%) were found as the major constituents. Camphor is also found in high concentrations in some *Lavandula* species, especially in *L. stoechas*, *Lavandula pedunculata*, *L. sampaioana*, and *L. luisieri*. Camphor is used as an antimicrobial material and for improving sleep quality. It is also used as a natural flavouring agent and as an ingredient in food processing (Danh *et al.*, 2012).

The essential oil composition of *Lavandula canariensis* which grows in Australia was examined (Palá-Paúl *et al.*, 2004). The principal compound was found to be carvacrol (23.6%). However, in our study, this component was observed only stems of *L. officinalis* in a small amount (0.75-1.05%). When previous studies were examined, carvacrol was not generally found to be dominant in the essential oils of other lavender species.

In 2010, Hassiotis (2010), who conducted research on *L. stoechas* (leaves and flowers) essential oil, found that fenchone (45.2%), 1,8-cineole (16.3%), and camphor (9.9%) were the major components and subsequently *p*-cymene (4.9%), α -cadinol (4.2%), lavandulyl acetate (3.2%) and α -pinene (2.5%) were reported as predominant compounds. The essential oils of Spanish lavender *L. stoechas* were studied and the major constituents were detected as fenchone (55.79%), camphor (18.18%), 1,8-cineole (8.03%), and myrtenyl acetate (6.25%) (Dadalioglu and Evrendilek, 2004). Essential oils from the stems, leaves and flowers of *L. stoechas* ssp. *stoechas* which grows wild in southern Sardinia (Italy) were examined. Fenchone was found to be the main compound in both leaves and flowers accounting, during the experiment, for average amounts of 52.60 and 66.20%, followed by camphor (27.08 and 13.13% respectively) (Angioni *et al.*, 2006). In a study by Costa *et al.* (2013) camphor (40.6%) and fenchone (38.0%) were found as the major constituents in *L. pedunculata* subsp. *lusitanica* in south Portugal. Zuzarte *et al.* (2013) determined that *L. stoechas* essential oil was rich in fenchone (37.0%) and camphor (27.3%). Fenchone was expressed as a major component in these studies but this compound not observed in the present study.

A terpene, terpinen-4-ol, was not determined in the leaves but this component was observed in large amounts in the stems and flowers. Isoborneol had the highest amount in leaves collected at midday (19.27%). β -Pinene, a monoterpenene, was detected only in leaves harvested in

the evening. In stems the amounts of this component found in morning and evening harvests were close to each other (7.14 and 7.31%, respectively). However the amount was higher in samples harvested at midday (9.05%). Myrcene, a monoterpenene, was detected in the stems and flowers.

As can be seen above, a lot of study reported that the essential oil composition of lavender may vary according to genotype, species, cultivar alteration, etc. Furthermore we determined by the present study that the harvest period in a day may affect the volatile composition of lavender.

4. Conclusions

It was revealed that *L. officinalis* has high total phenolic content, antioxidant and antiradical activity and that levels varied depending on parts (stem, flower, leave), harvest time (morning, midday, evening) and extraction solvent (methanol, water). Lavender can be utilised as an easily accessible source of natural antioxidants, as a possible food supplement or for pharmaceutical applications. Moreover, linalool, camphor, 1,8-cineole, terpinen-4-ol and isoborneol were the most representative compounds in the samples. These constituents are used as flavouring agents in the cosmetics and food industries.

References

- Angioni, A., Barra, A., Coroneo, V., Dessi, S. and Cabras, P., 2006. Chemical composition, seasonal variability, and antifungal activity of *Lavandula stoechas* L. ssp. *stoechas* essential oils from stem/leaves and flowers. *Journal of Agricultural and Food Chemistry* 54: 4364-4370.
- Bouayed, J., Piri, K., Rammal, H., Dicko, A., Desor, F., Younos, C. and Soulimani, R., 2007. Comparative evaluation of the antioxidant potential of some Iranian medicinal plants. *Food Chemistry* 104: 364-368.
- Boulekache-Makhlouf, L. and Madani, K., 2014. Antioxidant capacity of crude extracts and their solvent fractions of selected Algerian Lamiaceae. *Industrial Crops and Products* 52: 177-182.
- Cherrat, L., Espina, L., Bakkali, M., Pagán, R. and Laglaoui, A., 2014. Chemical composition, antioxidant and antimicrobial properties of *Mentha pulegium*, *Lavandula stoechas* and *Satureja calamintha* Scheele essential oils and an evaluation of their bactericidal effect in combined processes. *Innovative Food Science and Emerging Technologies* 22: 221-229.
- Costa, P., Gonçalves, S., Valentão, P., Andrade, P.B., Almeida, C., Nogueira, J.M. and Romano, A., 2013. Metabolic profile and biological activities of *Lavandula pedunculata* subsp. *lusitanica* (Chaytor) Franco: studies on the essential oil and polar extracts. *Food Chemistry* 141: 2501-2506.
- Da Porto, C., Decorti, D. and Kikic, I., 2009. Flavour compounds of *Lavandula angustifolia* L. to use in food manufacturing: comparison of three different extraction methods. *Food Chemistry* 112: 1072-1078.

Dadalioglu, I. and Evrendilek, G.A., 2004. Chemical compositions and antibacterial effects of essential oils of Turkish oregano (*Origanum minutiflorum*), bay laurel (*Laurus nobilis*), Spanish lavender (*Lavandula stoechas* L.), and fennel (*Foeniculum vulgare*) on common foodborne pathogens. *Journal of Agricultural and Food Chemistry* 52: 8255-8260.

Danh, L.T., Triet, N.D.A., Zhao, J., Mammucari, R. and Foster, N., 2012. Antioxidant activity, yield and chemical composition of lavender essential oil extracted by supercritical CO₂. *Journal of Supercritical Fluids* 70: 27-34.

Figueiredo, A., Barroso, J., Pedro, L., Salgueiro, L., Miguel, M. and Faleiro, M., 2008. Portuguese Thymbra and Thymus species volatiles: chemical composition and biological activities. *Current Pharmaceutical Design* 14: 3120-3140.

Gonçalves, S., Gomes, D., Costa, P. and Romano, A., 2013. The phenolic content and antioxidant activity of infusions from Mediterranean medicinal plants. *Industrial Crops and Products* 43: 465-471.

Grice, H., 1986. Safety evaluation of butylated hydroxytoluene (BHT) in the liver, lung and gastrointestinal tract. *Food and Chemical Toxicology* 24: 1127-1130.

Gulcin, I., Buyukokuroglu, M.E., Oktay, M. and Kufrevioglu, O.I., 2002. On the *in vitro* antioxidative properties of melatonin. *Journal of Pineal Research* 33: 167-171.

Gülçin, İ., Şat, İ.G., Beydemir, Ş., Elmastaş, M. and Küfrevioğlu, Ö.İ., 2004. Comparison of antioxidant activity of clove (*Eugenia caryophyllata* Thunb) buds and lavender (*Lavandula stoechas* L.). *Food Chemistry* 87: 393-400.

Haas, C., 2001. *Lavender – the most essential oil*. Pennon Publishing, Essendon North, Australia.

Hanamanthagouda, M.S., Kakkalami, S.B., Naik, P.M., Nagella, P., Seetharamareddy, H.R. and Murthy, H.N., 2010. Essential oils of *Lavandula bipinnata* and their antimicrobial activities. *Food Chemistry* 118: 836-839.

Hassiotis, C.N., 2010. Chemical compounds and essential oil release through decomposition process from *Lavandula stoechas* in Mediterranean region. *Biochemical Systematics and Ecology* 38: 493-501.

Herraiz-Peña, D., Cases, M.Á., Varela, F., Navarrete, P., Sánchez-Vioque, R. and Usano-Alemany, J., 2013. Chemical characterization of *Lavandula latifolia* Medik. essential oil from Spanish wild populations. *Biochemical Systematics and Ecology* 46: 59-68.

Jayaprakash, G., Singh, R. and Sakariah, K., 2001. Antioxidant activity of grape seed (*Vitis vinifera*) extracts on peroxidation models *in vitro*. *Food Chemistry* 73: 285-290.

Kamatou, G., Makunga, N., Ramogola, W. and Viljoen, A., 2008. South African *Salvia* species: a review of biological activities and phytochemistry. *Journal of Ethnopharmacology* 119: 664-672.

Karamanolis, K., Vokou, D., Menkissoglu, U. and Constantinidou, H.-I., 2000. Bacterial colonization of phyllosphere of Mediterranean aromatic plants. *Journal of Chemical Ecology* 26: 2035-2048.

Kim, N.-S. and Lee, D.-S., 2002. Comparison of different extraction methods for the analysis of fragrances from *Lavandula* species by gas chromatography – mass spectrometry. *Journal of Chromatography A* 982: 31-47.

Kirca, A. and Arslan, E., 2008. Antioxidant capacity and total phenolic content of selected plants from Turkey. *International Journal of Food Science and Technology* 43: 2038-2046.

Krishnaiah, D., Sarbatly, R. and Bono, A., 2007. Phytochemical antioxidants for health and medicine – a move towards nature. *Biotechnology and Molecular Biology Reviews* 1: 97-104.

Lee, C.-J., Chen, L.-G., Chang, T.-L., Ke, W.-M., Lo, Y.-F. and Wang, C.-C., 2011. The correlation between skin-care effects and phytochemical contents in Lamiaceae plants. *Food Chemistry* 124: 833-841.

Loliger, J., 1991. The use of antioxidants in foods. In: Aruoma, O.I. and Halliwell, B. (eds.) *Free radicals and food additives*. Taylor Francis, London, UK, pp. 121-150.

Messaoud, C., Chograni, H. and Boussaid, M., 2012. Chemical composition and antioxidant activities of essential oils and methanol extracts of three wild *Lavandula* L. species. *Natural Product Research* 26: 1976-1984.

Miliauskas, G., Venskutonis, P. and Van Beek, T., 2004. Screening of radical scavenging activity of some medicinal and aromatic plant extracts. *Food Chemistry* 85: 231-237.

Oktay, M., Gülçin, İ. and Küfrevioğlu, Ö.İ., 2003. Determination of *in vitro* antioxidant activity of fennel (*Foeniculum vulgare*) seed extracts. *LWT – Food Science and Technology* 36: 263-271.

Ozkan, G., Baydar, H. and Erbas, S., 2010. The influence of harvest time on essential oil composition, phenolic constituents and antioxidant properties of Turkish oregano (*Origanum onites* L.). *Journal of the Science of Food and Agriculture* 90: 205-209.

Ozkan, G., Sagdic, O., Ekici, L., Ozturk, I. and Ozcan, M., 2007. Phenolic compounds of *Origanum sspyleum* L. extract, and its antioxidant and antibacterial activities. *Journal of Food Lipids* 14: 157-169.

Palá-Paúl, J., Brophy, J., Goldsack, R. and Fontaniella, B., 2004. Analysis of the volatile components of *Lavandula canariensis* (L.) Mill., a Canary Islands endemic species, growing in Australia. *Biochemical Systematics and Ecology* 32: 55-62.

Polat, B., Oba, S., Karaman, K., Arici, M. and Sagdic, O., 2014. Comparison of different solvent types for determination biological activities of myrtle berries collected from Turkey. *Quality Assurance and Safety of Crops and Foods* 6: 221-227.

Quave, C.L., Plano, L.R., Pantuso, T. and Bennett, B.C., 2008. Effects of extracts from Italian medicinal plants on planktonic growth, biofilm formation and adherence of methicillin-resistant *Staphylococcus aureus*. *Journal of Ethnopharmacology* 118: 418-428.

Sanz, J., Soria, A. and García-Vallejo, M., 2004. Analysis of volatile components of *Lavandula luisieri* L. by direct thermal desorption – gas chromatography – mass spectrometry. *Journal of Chromatography A* 1024: 139-146.

Spiridon, I., Colceru, S., Anghel, N., Teaca, C.A., Bodirlau, R. and Armatu, A., 2011. Antioxidant capacity and total phenolic contents of oregano (*Origanum vulgare*), lavender (*Lavandula angustifolia*) and lemon balm (*Melissa officinalis*) from Romania. *Natural Product Research* 25: 1657-1661.

Serban, E.S., Ionescu, M., Matinca, D., Maier, C.S. and Bojiță, M.T., 2011. Screening of the antibacterial and antifungal activity of eight volatile essential oils. *Farmacia* 59: 440-446.

Taha Nejad, M., Barzegar, M., Sahari, M. and Naghdi Badi, H., 2012. Evaluation of Antioxidant activity of *Lavandula angustifolia* essential oil in crude soybean oil system. *Journal of Medicinal Plants* 1: 127-140.

Takaki, I., Bersani-Amado, L., Vendruscolo, A., Sartoretto, S., Diniz, S., Bersani-Amado, C. and Cuman, R., 2008. Anti-inflammatory and antinociceptive effects of *Rosmarinus officinalis* L. essential oil in experimental animal models. *Journal of Medicinal Food* 11: 741-746.

Tavarini, S. and Angelini, L. G., 2013. *Stevia rebaudiana* Bertoni as a source of bioactive compounds: the effect of harvest time, experimental site and crop age on steviol glycoside content and antioxidant properties. *Journal of the Science of Food and Agriculture* 93: 2121-2129.

Torras-Claveria, L., Jauregui, O., Bastida, J., Codina, C. and Viladomat, F., 2007. Antioxidant activity and phenolic composition of lavandin (*Lavandula* × *intermedia* Emeric ex Loiseleur) waste. *Journal of Agricultural and Food Chemistry* 55: 8436-8443.

Tulukcu, E., Sagdic, O., Albayrak, A., Ekici, L. and Yetim, H., 2012. Effect of collection time on biological activity of clary sage (*Salvia sclarea*). *Journal of Applied Botany and Food Quality* 83: 44-49.

Wichi, H., 1988. Enhanced tumor development by butylated hydroxyanisole (BHA) from the prospective of effect on forestomach and oesophageal squamous epithelium. *Food and Chemical Toxicology* 26: 717-723.

Yalcin, H. and Kavucuoglu, H., 2014. Physical, chemical and bioactive properties of onion (*Allium cepa* L.) seed and seed oil. *Journal of Applied Botany and Food Quality* 87: 87-92.

Yalçin, H., Anik, M., Sanda, M.A. and Çakir, A., 2007. Gas chromatography/mass spectrometry analysis of *Laurus nobilis* essential oil composition of northern Cyprus. *Journal of Medicinal Food* 10: 715-719.

Zuzarte, M., Gonçalves, M., Cavaleiro, C., Cruz, M., Benzarti, A., Marongiu, B., Maxia, A., Piras, A. and Salgueiro, L., 2013. Antifungal and anti-inflammatory potential of *Lavandula stoechas* and *Thymus herba-barona* essential oils. *Industrial Crops and Products* 44: 97-103.

