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# COMPARATIVE CHEMICAL ANALYSIS OF ESSENTIAL OILS FROM LAVENDER OF DIFFERENT GEOGRAPHIC ORIGINS

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**ABSTRACT.** The purpose of this study was to determine and analyse the chemical composition of essential oils of dry lavender flowers from the area of Budva and Rovinj and to compare to the results obtained in the commercially available sample in Montenegro. Based on gas chromatography (GC and GC/MS) analysis approximately 100 components were identified in the analysed samples, which makes 96.95% to 99.74% of the components present in essential oils. The analysis has shown that oxygenated monoterpenes were the dominant group of compounds. The significant difference in oxygenated monoterpene content between the samples from the area of Budva (71.74%) and Rovinj (91.79%) and the commercially available sample (87.63%) was noted. The most abundant oxygenated monoterpenes in the lavender oil sample from Budva region were linalool and borneol (27.32% and 20.24% respectively); the sample from Rovinj contained linalool and camphor (47.67% and 11.82% respectively) and commercial sample camphor and linalool (21.23% and 19.92% respectively). Monoterpene hydrocarbons were present in a significantly lower percentage in the commercial sample (2.64%) and in a sample from Rovinj (3.57%), while the high content occurred in the sample from the climate of Montenegrin seaside (Budva) (23.74%) with the dominant constituent β-phellandrene (19.1%). The oxygenated sesquiterpenes were slightly less present in the sample from Rovinj (1.14%) and Budva (1.88%), and almost twice as much present in the commercial sample (4.37%) represented with caryophyllene oxide as the dominant ingredient in all three samples; the samples from Rovinj, Budva and commercial ones contained 0.36%, 0.43% and 2.78% respectively. Hydrocarbon sesquiterpenes were insignificantly present in the analysed samples. Hydrocarbon sesquiterpenes were insignificantly presented in the commercial sample (0.83%); the sample from Budva (1.38%) and in a sample from Rovinj (1.66%). As the dominant constituent was sesquisabinen (1.0%) in the sample from Budva and *trans*-(*E*)-caryophyllene in a sample from Rovinj (0.58%) and commercial sample (0.22%).

***Keywords:*** *lavender, essential oil, isolation, identification, chemical composition.*

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## INTRODUCTION

*Lavandula angustifolia* Mill. syn. *Lavandula officinalis* Chaix was commonly known as lavender is a species of the genus *Lavandula* from *Lamiaceae* family [1]. Lavender species are grown worldwide primarily for their essential oils, which are used in the food processing, aromatherapy products, cosmetics and perfumes [2, 3].

*L. angustifolia* is a perennial herbaceous plant being one of the most loved aromatic and medicinal plants in the world. Lavender is the term, which comes from Latin word "lavando" derived as a part of the verb "lavare" which means “to wash”. The Romans used lavender for obtaining a pleasant smell in their bath, as well as for its beneficial effect on health [1].

The plant grows as a small shrub up to 60 cm high with the blue-violet flowers, which contain essential oil, responsible for the plant’s beautiful scent. It is native to the Mediterranean areas [1, 4]. The dried incompletely developed blossoms of the lavender plant have been used, as an official drug [5].

According to the European Pharmacopoeia, 8$^{th}$ Edition (Ph. Eur. 8.0), lavender flowers should contain at least 13 ml / kg of anhydrous essential oil [6]. The main constituents of lavender dried blossoms essential oil are linalool, linalyl acetate, cineole, camphor and β-ocimene. The plant also contains a high percentage of tannins up to 12 %. The essential oil extracted from the fresh blossoms of lavender contains in high percentage linalool and linalyl acetate [5].

Lavender essential oil can be used externally and internally for various purposes. Externally the essential oil is used in salves, balms, cosmetics, perfumes and topical skin preparations. Taken internally essential oil of lavender is believed to have a beneficial effect on many health impairments, including anxiety, stress, depression, irritability, nervousness, exhaustion, insomnia, headaches, migraines, colds, flatulence, upset stomach, digestion, loss of appetite, liver and gallbladder problems. In addition, it has been known as a breath freshener and mouthwash [7].

Dried lavender flowers have been used for tea-like preparation for improving the mood disorder, insomnia and lassitude, also with beneficial effects on abdominal complaints. Lavender essential oil has its application as a corrector of the taste and odour for pharmaceutical preparations, also as a rubefacient and insecticide [8].

According to the U.S. Food and Drug Administration, *L. officinalis* has been classified as safe and has been included on the safe substances list, commonly known as “Generally Recognised as Safe” (GRAS) [5].

## RESULTS AND DISCUSSION

The yield of essential oils (% v/w) amounted to approximately 3 % of dry lavender flowers.

According to the Ph. Eur. 8.0 requirements, dried lavender flowers should contain at least 13 ml / kg of anhydrous essential oil [6]. Essential oil yields found in our samples were in accordance with literature data.

The qualitative and quantitative chemical composition assessment were done using both gas chromatography (GC) and gas chromatography/ mass spectrometry (GC/MS) analysis. Using the GC/MS analysis approximately 100 components were identified in the analysed samples, which makes 96.95% to 99.74% of the components present in essential oils (Table 1).

**Table 1.** The chemical composition of essential oils from *L. angustifolia* Mill. of different geographic origins

| GC Peak No. | Compound | Budva % | Rovinj % | Commercial sample % |
|---|---|---|---|---|
| 1. | tricyclene | 0.15 | - | 0.05 |
| 2. | thujene | 0.16 | 0.12 | - |
| 3. | α-pinene | 0.72 | 0.35 | 0.26 |
| 4. | camphen | 0.70 | 0.43 | 0.64 |
| 5. | thuja-2-4(10)-diene | 0.28 | - | - |
| 6. | sabinene | 0.50 | 0.09 | 0.06 |
| 7. | β-pinene | 0.11 | 0.29 | 0.26 |
| 8. | 3-octen-1-ol | 0.42 | 0.41 | - |
| 9. | 3-octanone | - | 0.13 | - |
| 10. | myrcene | 0.62 | 0.75 | 0.53 |
| 11. | dehydroxy-*trans*-linalool oxide | | | 0.15 |
| 12. | α-phellandrene | 0.03 | 0.05 | - |
| 13. | δ-3-carene | 0.13 | 0.18 | - |
| 14. | α-terpinene | 0.09 | 0.04 | - |
| 15. | *p*-cymene | 0.08 | 0.04 | 0.19 |
| 16. | limonene | - | - | 0.28 |
| 17. | β-phellandrene | 19.1 | 0.16 | - |
| 18. | 1-8-cineole | | 9.60 | 14.89 |
| 19. | *(Z)*-β-ocimene | 0.38 | 0.47 | 0.19 |
| 20. | *(E)*-β-ocimene | 0.18 | 0.46 | 0.08 |
| 21. | γ-terpinene | 0.14 | 0.09 | 0.10 |
| 22. | *cis*-sabinene hydrate | 0.39 | 0.22 | 0.11 |
| 23. | *cis*-linalool oxide | 0.80 | 0.61 | 4.20 |
| 24. | camphenylon | - | - | t |
| 25. | *trans*-linalool oxide | 0.73 | 0.66 | 3.09 |
| 26. | terpinolene | 0.37 | 0.05 | |
| 27. | linalool | 27.32 | 47.67 | 19.92 |
| 28. | 3-*(Z)*-hexenyl isobutanoate | | | 0.64 |
| 29. | 1-octen-3-yl acetate | - | - | 0.55 |
| 30. | *(Z)*-*p*-menth-2-en-1-ol | 0.39 | - | - |
| 31. | α–campholenal | 0.13 | - | - |
| 32. | *(Z)*-*p*-menth-2,8-dien-1-ol | t | - | - |
| 33. | nopinon | | | 0.14 |

| GC Peak No. | Compound | Budva % | Rovinj % | Commercial sample % |
|---|---|---|---|---|
| 34. | camphor | 7.52 | 11.82 | 21.23 |
| 35. | *trans*-verbenol | 0.15 | | 0.31 |
| 36. | nerol oxide | - | - | 0.10 |
| 37. | hexyl isobutanoat | t | 0.03 | t |
| 38. | pinocarvone | t | 0.05 | 0.14 |
| 39. | borneol | 20.24 | 8.11 | 5.33 |
| 40. | lavandulol | - | - | 0.48 |
| 41. | terpinene-4-ol | 4.60 | 3.08 | 0.74 |
| 42. | *p*-cymene-8-ol | t | - | - |
| 43. | cryptone | 2.69 | 0.85 | - |
| 44. | α–terpineol | 0.85 | 1.86 | 2.01 |
| 45. | dihydrocarveol | - | - | 0.35 |
| 46. | myrtenol | 0.24 | - | t |
| 47. | 4-*cis*-caranone | - | - | t |
| 48. | verbenone | 0.20 | - | 0.32 |
| 49. | *trans*-carveol | 0.44 | - | - |
| 50. | β–cyclocitral | - | - | 0.09 |
| 51. | nerol | 0.33 | 0.40 | 0.52 |
| 52. | hexyl-2-methyl-butanoate | 0.17 | - | 0.09 |
| 53. | isobornyl formate | - | - | 0.10 |
| 54. | cymine aldehyde | 0.64 | - | - |
| 55. | carvone | 0.31 | - | 0.14 |
| 56. | hexyl isovalerate | - | 0.42 | 0.13 |
| 57. | carvotanacetone | - | 0.20 | - |
| 58. | linalool acetate | 1.50 | 4.56 | 9.73 |
| 59. | geraniol | - | - | t |
| 60. | bornyl acetate | 0.06 | t | 0.05 |
| 61. | lavandulyl acetate | 1.26 | 0.49 | 1.83 |
| 62. | *p*-cymene-7-ol | 0.20 | - | - |
| 63. | hexyl tiglate | 0.08 | 0.18 | 0.07 |
| 64. | neryl acetate | 0.06 | 0.45 | 0.46 |
| 65. | α-copaene | - | t | - |
| 66. | geranyl acetate | - | 0.98 | 0.95 |
| 67. | hexyl hexanoate | 0.33 | - | - |
| 68. | sesquithujene | 0.05 | 0.09 | - |
| 69. | α-*cis*-bergamotene | - | 0.15 | - |
| 70. | *trans*-(*E*)-caryophyllene | 0.04 | 0.58 | 0.22 |
| 71. | santalene | 0.05 | | 0.33 |
| 72. | lavandulyl isobutanoate | 0.07 | 0.05 | t |
| 73. | linalool butanoate | | | t |
| 74. | α–*trans*-bergamoten | 0.14 | 0.02 | t |
| 75. | β-*(Z)*-farnesene | - | t | - |
| 76. | α-humulene | - | t | - |
| 77. | β-*(E)*-farnesene | 0.05 | | t |
| 78. | sesquisabinene | 1.0 | 0.44 | - |
| 79. | 7-epi-1,2-dehydro-sesquicineol | 0.04 | - | - |
| 80. | geranyl propanoate | 0.10 | - | - |

| GC Peak No. | Compound | Budva % | Rovinj % | Commercial sample % |
|---|---|---|---|---|
| 81. | neryl isobutanoate | - | 0.13 | t |
| 82. | *trans*-muurola-4(14),5-diene | 0.05 | 0.38 | 0.07 |
| 83. | γ-amorphene | - | t | - |
| 84. | isodaucene | - | t | - |
| 85. | cuparene | t | - | - |
| 86. | lavandyl isovalerate | 0.52 | - | 0.25 |
| 87. | γ-cadinene | - | - | 0.21 |
| 88. | longipinalol | - | - | 0.19 |
| 89. | caryophyllene oxide | 0.43 | 0.36 | 2.78 |
| 90. | davanone | - | t | - |
| 91. | Fokienol | - | - | t |
| 92. | humulene epoxide II | - | - | 0.06 |
| 93. | 1,10-di-epi-cubenol | - | - | 0.08 |
| 94. | curcumen | - | - | t |
| 95. | davanol | - | t | - |
| 96. | caryophylla-4(12),8(13)-dien-5α -ol | - | - | t |
| 97. | epi-α-cadinol | 0.20 | t | 0.55 |
| 98. | α-bisabolol oxide B | 0.17 | 0.05 | - |
| 99. | *(E)*-14-hydroxy-9-epi-caryophyllene | - | t | 0.16 |
| 100. | *(Z)*-14-hydroxy-caryophyllene | - | - | 0.40 |
| 101. | epi-α-bisabolol | 1.04 | 0.73 | - |
| 102. | α-germacra-4(15),5,10(14)-trien-1-ol | - | - | 0.08 |
| 103. | *(Z,Z)*-2,6-farnesol | - | t | - |
| 104. | *(R,R)*-6,7-bisabolone | t | - | - |
| 105. | cyclocolorenone | - | - | 0.07 |
| | **% of total** | **99.74** | **99.33** | **96.95** |
| | * % - Percentage<br>* t – Compounds with percentage less of 0.05% | | | |

The analysis showed that oxygenated monoterpenes were the dominant group of compounds. The significant difference between the quantitative and qualitative chemical patterns in the investigated samples from the area of Budva (71.74%), Rovinj (91.79%) and the commercially available sample (87.63%) was noted. The most abundant oxygenated monoterpenes in the lavender oil sample from Budva region were linalool and borneol (27.32% and 20.24% respectively); the sample from Rovinj contained linalool and camphor (47.67% and 11.82% respectively) and commercial sample camphor and linalool (21.23% and 19.92% respectively).

Monoterpene hydrocarbons were present in a significantly lower percentage in the commercial sample (2.64%) and in a sample from Rovinj (3.57%), while the high content occurred in the sample from the climate of Montenegrin seaside (Budva) (23.74%) with the dominant constituent being β-phellandrene (19.1%).

The investigated samples contained oxygenated sesquiterpenes in small quantity, with the percentage of 1.14%, 1.88% and 4.37% (the samples from Rovinj, Budva and the commercial sample respectively). Caryophyllene oxide was found to be the dominant ingredient in all three investigated samples (samples from Rovinj, Budva and commercial sample, representing 0.36, 0.43 and 2.78%, respectively).

Hydrocarbon sesquiterpenes were insignificantly present in the commercial sample and the samples from natural origin from Budva and Rovinj (0.83%, 1.38% and 1.66% respectively) with sesquisabinen (1.0%) in the sample from Budva, *trans-(E)*-caryophyllene in the sample from Rovinj (0.58%) and commercial sample (0.22%) as the dominant constituent within this group of compounds.

The percentage distribution of different classes of chemical compounds present in the essential oils of lavender *L. angustifolia* Mill. of different geographic origins was presented in Table 2 below.

**Table 2.** The chemical composition of essential oils from lavender of different geographic origins

| **The different classes of the compounds usually present in essential oils** | **Budva** | **Rovinj** | **Commercial sample** |
|---|---|---|---|
| Monoterpene hydrocarbons | 23.74 | 3.57 | 2.64 |
| Oxygenated monoterpenes | 71.74 | 91.79 | 87.63 |
| Hydrocarbon sesquiterpenes | 1.38 | 1.66 | 0.83 |
| Oxygenated sesquiterpenes | 1.88 | 1.14 | 4.37 |
| Other compounds | 1.0 | 1.17 | 1.48 |
| % of total | **99.74** | **99.33** | **96.95** |

The major constituent of lavender oils from the samples from Budva and Rovinj was linalool (27.32% and 47.67% respectively). The commercial sample has been shown to be abundant in camphor, as the major constituent (21.23%) (Table 1).

As presented in Table 3, comparing the obtained results to the Ph. Eur. 8.0 requirements for the representative and chosen components (limonene, 1,8-cineole, camphor, linalool, terpinen-4-ol, lavandulyl-acetate, lavandulol and α-terpineol), the certain undesirable difference might be observed. For example, the percentage of camphor and 1,8-cineole (known

as eucalyptol), deviated from the requirements of Ph. Eur. 8.0 in all investigated samples. On the other side, the content of linalool conformed to the requirements of Ph. Eur. 8.0 in the sample from Budva and commercial sample, but did not meet the corresponding requirements in the sample from Rovinj.

**Table 3.** Comparison of the obtained chemical analysis to Ph. Eur. 8.0 monograph requirements

| Terpenoid Components | Ph. Eur. 8.0 [6] | Budva | Rovinj | Commercial sample |
|---|---|---|---|---|
| **Limonene** | Maximum 1.0 % | - | - | 0.28 % |
| **1,8-cineole** | Maximum 2.5 % | - | 9.6 % | 14.89 % |
| **Camphor** | Maximum 1.2% | 7.52 % | 11.82 % | 21.23 % |
| **Linalool*** | From 20 to 45 % | 27.32 % | 47.67 % | 19.92 % |
| **Terpinen-4-ol** | From 0.1 to 8.0 % | 4.6 % | 3.08 % | 0.74 % |
| **Lavandulyl-acetate*** | Minimum 0.2 % | 1.26 % | 0.49 % | 1.83 % |
| **Lavandulol** | Minimum 0.1 % | - | - | 0.48 % |
| **α-terpineol** | Maximum 2.0 % | 0.85 % | 1.86 % | 2.01 % |

* The components responsible for antibacterial and antifungal properties [1,11].

Numerous previous studies and results obtained in this study indicated the difference in the chemical composition of terpenoid compounds.

Variability in the chemical composition was most probably dependent on numbers of factors such as genetics (species or variety) of the plant, environmental conditions (geographical, climatic or seasonal) and processing of plant material [9].

In the literature, the major components of the lavender essential oil were reported to be linalool and linalyl acetate. It was stated that it also contained α-thujene, camphene, α- and β-pinene, sabinene, p-cymene, eucalyptol (1,8-cineole), myrcene, limonene, camphor, lavandulol, β-caryophyllene, lavandulyl acetate, γ-terpinene, terpinen-4-ol, *(Z)*- and *(E)*- β-ocimene etc. [1].

The chemical composition of the essential oils in this study indicated the presence of linalool and linalyl acetate in all analysed samples. Percentage of these compounds corresponded to the literature data. Limonene and lavandulol were not found in the samples from Budva and Rovinj. In commercial sample limonene and lavandulol were present in a percentage of 0.28% and 0.48%, respectively, being in accordance with the requirements of Ph. Eur. 8.0. In the samples from Budva, Rovinj and commercial sample, camphor was found to represent 7.52%, 11.82% and 21.23%, respectively, in investigated samples, being the most abundant component in the commercial sample. Taking into account the Ph. Eur. 8.0 requirements for this compound for lavender essential

oil (maximum 1.2%) it is obvious that the determined concentration extended the limits. Comparing the content of 1,8-cineole (found in the quantity of 9.6% and 14.9% in the sample from Rovinj and commercial sample, respectively, and being not present in the sample from Budva) to the Ph. Eur. 8.0 requirements (maximum 2.5%), the statement could be made that the sample from Budva met this requirement. The concentration of terpinen-4-ol in all investigated samples conformed to the Ph. Eur. 8.0 requirements (from 0.1 to 8.0%) (Table 3).

## CONCLUSIONS

Lavender essential oils were analysed using gas chromatography techniques (GC and GC/MS). The yield of essential oils (% v/w) amounted to approximately 3 % of dry lavender flowers. Essential oils yields were in accordance with literature data.

Based on GC/MS analysis approximately 100 components were identified in the analysed samples, which makes 96.95% to 99.74% of the components present in essential oils. Linalool was the main constituent of lavender essential oil in the samples from Budva (27.32%) and Rovinj (47.67%), followed by borneol, β-phellandrene, camphor and 1,8-cineole. In the commercial sample, camphor was dominant compound (21.23%) followed by linalool, 1,8-cineole, linalool acetate and borneol.

Previous studies and obtained results in this study indicated the existence of differences in the terpenoid chemical composition. Variability in the qualitative and quantitative composition most probably depended on the genotype of the plant and the influence of different environmental factors. The application of lavender essential oil for therapeutic purposes depends on its qualitative properties. The obtained results might be significant in order to evaluate the quality of the plant raw material.

## EXPERIMENTAL SECTION

### Plant material and extraction procedure

Plants' material was collected in the initial flowering stage in Budva and Rovinj during summer 2014. Dried plant material was packed in paper bags and stored in a cool and dry place. The commercially available sample was purchased in a community pharmacy in typified package in Podgorica.

The essential oil of lavender was obtained by applying the method of hydro-distillation in the Clevenger type apparatus, according to Procedure I of the Yugoslav Pharmacopoeia IV. Hydro-distillation was performed for a period of 150 minutes (2,5 h) [10].

Each hydro-distillation was performed in 5 replicates. Lavender oil was separated by decantation from the remaining water and then dried on anhydrous sodium sulphate ($Na_2SO_4$). The essential oil was packaged and stored in dark glass phials until gas chromatography analyses.

### Qualitative and Quantitative analysis of essential oils (GC and GC/MS)

**GC analysis.** Oil samples were analysed by gas chromatography technique (GC) on Hewlett-Packard (Model 5890 II) with a split-splitless injector connected to an HP-5 column (25 m x 0.32 mm, 0.52 μm) and flame ionising detector (GC/FID). Operated was using the following conditions: injector temperature, 250 °C; column temperature changes in column temperature programmed linearly from 40 to 260 °C with a rate of change of the 4 °C / min; carrier gas, $H_2$; injection volume, 1 ml / min, with a split ratio of 1:30; temperature detector 300 °C, while the isothermal conditions were bracketed at 260 °C for 10 minutes. The amount of the injected sample, dissolved in methylene chloride ($CH_2Cl_2$), was 1 μl. Surface percentages obtained in the range as a result of the determination of standard chromatogram are used for quantitative analysis.

**GC/MS analysis.** The analysis was performed on HP 1800 C II, GCD system (Hewlett-Packard, Palo Alto, CA, USA), equipped with a column HP-SMS (30 m x 0.25 mm x 0.25 μm). As the carrier gas used helium. Transfer line was heated to 260 °C. The mass spectra were obtained in the Ei mode (70 eV), in the range of from 40 to 450 m/z. The amount of the injected sample, dissolved in methylene chloride ($CH_2Cl_2$) was 0.2 μl. The components of the oil were identified by comparing the mass spectra with those in the database Wiley 275 and NIST/NBS (NIST–National Institute of Standards and Technology/ NBS-National Bureau of Standards) libraries, without using the various search engines. Identification of the components was achieved by comparing their retention indices and mass spectra with those found in the literature and compared with those obtained with the help of an automatic mass spectral analysis and software system for the identification (The Automated Mass Spectral Deconvolution and Identification System - AMDIS ver. 2.1), GC/MS library. The percentage of the constituents of the essential oil was obtained by normalising the surface of the corresponding peak and all the relative response factors were entered as 1.

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