

Determination of trace elements in the Lavender inflorescence (*Lavandula angustifolia* Mill.) — Lavender oil system

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Abstract

Bulgaria is a famous world producer of essential oils. Quality control of the lavender oils produced is an important analytical task in view of their wide use in perfumery and cosmetics, the food industry and aromatherapy. The present paper studies the relationship between the trace elements content in lavender inflorescence (*Lavandula angustifolia* Mill.) and in lavender oil derived from this inflorescence. Three model regions were investigated: two of them are situated far from industrial areas — the village of Zelenikovo and the town of Pavel Banya, and one region is located near the Plovdiv Non-Ferrous Metallurgical Plant. The content of the elements Cd, Cr, Cu, Fe, Mn and Pb in plants was determined after acid digestion by flame and electrothermal atomic absorption spectrometry (ETAAS). Lavender oils were analyzed directly by ETAAS. The results undoubtedly show that there is no strong correlation between the trace elements content in inflorescence and in essential oils. It might be assumed that the trace elements present and accumulated in the inflorescence do not pass in lavender oil during the production by steam distillation.

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1. Introduction

High quality lavender oil is produced through steam distillation of lavender inflorescence (*Lavandula angustifolia* Mill.). One kilogram of lavender oil is derived from about 120–150 kg of lavender inflorescence, depending on the climatic conditions. Essential lavender oils are very complex matrices consisting of various classes of organic compounds — terpenes, hydrocarbons, esters, alcohols, aldehydes, ketones, etc. [1,2]. Essential oils are widely used in perfumery and cosmetics and in the food industry [3–5]. Their therapeutic and bactericidal properties are well-known [5–9]. The wide use of essential oils requires strict control with respect to various undesirable impurities, such as artificially added substances [10,11], pesticide residues [12,13], toxic elements, etc.

No specific requirements and standards (Bulgarian or European) were found in literature regarding the trace metal content in essential oils. However, elements like Fe, Cr, Cu and Mn could most probably affect oil stability during storage due to

their catalytic properties, and the Cd and Pb content is subject to restrictions due to the high toxicity of these elements. In general, depending on the use of the oils, the standards for the respective product are observed — foodstuffs, cosmetic products, etc. There are only several papers related to the determination of trace elements in essential oils [14–16]. One of them is based on preliminary dry ashing of oil samples followed by ETAAS determination of trace elements [14]. Cd, Cu, Pb, Zn, Mn have been determined in citrus oils potentiometrically after hydrochloric acid extraction and the results have been compared with those obtained by ETAAS, after wet digestion of the sample [15,16]. Direct ETAAS has been used for the determination of As in rose and lavender oils and a very low content of As was found [17]. Our survey of literature showed that there is no systematic data on the levels of trace elements in essential lavender oil produced from different regions in Bulgaria. In this respect, no data has been published on the potentials of instrumental methods for the determination of trace elements in essential oils.

The aim of the present study was to perform an analysis of lavender inflorescence and lavender essential oils produced from these inflorescences in order to delineate the migration of

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Fig. 1. The map of Bulgaria and sampling areas.

trace elements (Cd, Cr, Cu, Fe, Mn and Pb) in the lavender inflorescence — essential oil system. As a first step, it was necessary to elaborate a set of methods for ETAAS determination of trace elements in lavender oil. As a second step, lavender inflorescences originating from three model regions and lavender oils produced from them were analyzed. Two of the regions are far from any industrial activities, in relatively unpolluted areas — the village of Zelenikovo (in the southern part of Sredna Gora mountain, one locality) and the town of Pavel Banya (in the northern part of Sredna Gora mountain, with four separate localities-Atyovo, Kara, Rosena and Chaliyata). One region is located very close to Plovdiv Non-Ferrous Metallurgical Plant (some 2 km away) (see Fig. 1). A sample of laboratory-produced lavender oil (Clavenger equipment) was also analysed in order to establish the impact of the processing equipment on the trace element content in the final product. Samples of lavender inflorescence were taken from a distillery immediately prior to oil production and they were acid-digested without any other preliminary treatment (without prior washing or air blowing). Lavender oils were analyzed directly by ETAAS.

2. Experimental

2.1. Instrumentation

Flame atomic absorption spectrometric measurements were carried out on a Perkin Elmer Zeeman 1100 B spectrometer with an air/acetylene flame. The instrumental parameters were optimized in order to obtain maximum signal-to-noise ratio. Electrothermal atomic absorption spectrometric measurements were carried out on a Perkin Elmer Zeeman 3030 spectrometer with an HGA-600 atomizer. The light sources used were hollow cathode lamps for Cr, Cu, Fe, Mn and Pb and an electrodeless discharge lamp for Cd (Perkin Elmer). The spectral band-pass was 0.7 nm. Pyrolytic graphite-coated graphite tubes coated with Pt as permanent modifier and tungsten impregnated tubes were used as atomizers. Permanent modification with Pt was achieved by triple injection of 40 μL $(\text{NH}_4)_2\text{PtCl}_6$ modifier solution, 1000 $\mu\text{g L}^{-1}$ in 5% v/v HCl with 1000 °C ashing temperature and 2100 °C atomization temperature. Tungsten impregnation was

Table 1

The optimized HGA-600 temperature program for ETAAS measurements of oil samples

Step	Temperature, °C	Ramp, s	Hold, s	Ar flow, mL min ⁻¹
1	50	10	20	300
2	150	10	20	300
3	900 ^a , 1200 ^b , 1300 ^c	20	60	300
4	1400 ^a , 2200 ^b , 2500 ^c	0	3	0

^a Cd and Pb.

^b Cu, Mn and Fe.

^c Cr.

performed as follows: an uncoated graphite tube was left in 10% m/V Na_2WO_4 solution for about 24 h. After drying at room temperature, the tube was heated according to the following temperature programme: 1. 100 °C for 30 s, 2. 1400 °C for 30 s, 3. 2650 °C for 10 s [18]. Sample solutions (20 μL) were introduced into the graphite atomizer using an AS-60 auto-sampler. Atomic absorption signals were recorded on an Anadex printer. Only peak areas were used for quantification. The optimal instrumental parameters for the ETAAS measurements were investigated and are presented in Table 1.

2.2. Reagents

All reagents were analytical reagent grade. The stock standard solutions of Cd, Cr, Cu, Fe, Mn and Pb 1000 $\mu\text{g mL}^{-1}$ were Titrisol, Merck in 2% v/v HNO_3 . The working standard solutions were prepared daily by proper dilution with organic solvent (p.a. Merck). Nitric acid was additionally purified by sub-boiling distillation.

Stock standard solution of Pt 980 $\mu\text{g mL}^{-1}$ in 5% v/v HCl (Aldrich) was injected (40 μL) into the graphite furnace to obtain permanent modifier coatings. Doubly distilled water was used in all experiments.

2.3. Sample preparation

2.3.1. Mineralization of the plant samples

The plant samples were taken from the distillation installations immediately before feeding the plant mass into the equipment and were subjected to mineralization without prior washing or air blowing.

A plant sample, ca. 0.5 g, was placed in a 25 mL beaker, 10 mL of concentrated nitric acid were added, the beaker was covered with a watch glass and left to stay for one night. Then the sample was heated (120 °C) on a sand bath, still covered with the watch glass, until the volume of the solution was reduced to 1–2 mL. After cooling down, 2 mL of H_2O_2 were added and the sample was heated again for further 10 min. Finally, the sample solution was transferred to a 25 mL measuring flask and doubly distilled water was added up to the 25 mL mark.

2.3.2. Preparation of the lavender oil samples

The lavender oil samples were taken from the producer after the end of the distillation process. The samples were diluted

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