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The loss of essential oil components induced by the Purge Time in the Pressurized Liquid Extraction (PLE) procedure of *Cupressus sempervirens*

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ABSTRACT

The influence of different Purge Times on the effectiveness of Pressurized Liquid Extraction (PLE) of volatile oil components from cypress plant matrix (*Cupressus sempervirens*) was investigated, applying solvents of diverse extraction efficiencies. The obtained results show the decrease of the mass yields of essential oil components as a result of increased Purge Time. The loss of extracted components depends on the extrahent type – the greatest mass yield loss occurred in the case of non-polar solvents, whereas the smallest was found in polar extracts. Comparisons of the PLE method with Sea Sand Disruption Method (SSDM), Matrix Solid-Phase Dispersion Method (MSPD) and Steam Distillation (SD) were performed to assess the method's accuracy. Independent of the solvent and Purge Time applied in the PLE process, the total mass yield was lower than the one obtained for simple, short and relatively cheap low-temperature matrix disruption procedures – MSPD and SSDM. Thus, in the case of volatile oils analysis, the application of these methods is advisable.

1. Introduction

The first step in the qualitative and quantitative analysis of plant constituents is the sample preparation procedure, the aim of which is to effectively and rapidly remove the analyte from its matrix. Solid liquid extraction is most frequently applied for this purpose. The choice of extraction technique is frequently decided upon consideration of operating costs, simplicity of operation, amount of organic solvent required and sample throughput. The traditional extraction methods (methods recommended in medicinal plant pharmacopeia, e.g. steam and water distillation, Soxhlet extraction, maceration, percolation, expression, cold fat extraction) have several shortcomings, including long extraction time and large consumption of solvents, cooling water and electric energy [1]. With the advent of laboratory automation and more and more wide-spread application of plant products in the pharmaceutical, medical, food and perfume industries, conventional extraction technologies are increasingly overlooked in routine analysis. Instrumental extraction methods requiring minimal sample handling are thus highly desirable [2]. Hence, several approaches are continuously being attempted in search of faster, cleaner and reliable analytical methodologies. As a response to such demands a number of techniques have been developed to meet the above criteria, for example, microwave-assisted extraction (MAE), supercritical fluid extraction (SFE), and Pressurized Liquid Extraction (PLE). The similarity between these techniques is the possibility of using elevated temperatures and pressures, which drastically improves the speed of the extraction process [3]. Raising the temperature increases the diffusion rates, the solubility of the analytes and their mass transfer, and decreases the viscosity and surface tension of the solvents. These changes improve the contact of the analytes with the solvent and enhance the extraction efficiency [4]. PLE has been shown to have significant advantages over competing techniques. For example, unlike MAE, in PLE no additional filtration step is required, since the matrix components that are not dissolved in the extraction solvent may be retained inside the sample extraction cell. This is very convenient for the purpose of automation and on-line coupling of extraction and separation techniques [5] which makes it more expensive than other assisted extraction methods (e.g. MAE). The principle of PLE is simple. The sample placed in the extraction cell is extracted with a solvent at a temperature ranging from ambient to 200 °C and at a relatively high pressure (from 4 to 20 MPa). In this approach, the selected solvent is pumped to fill the cell containing the sample, which is kept for a specified time at the selected pressure and temperature. Next, the extracted solvent is transferred to a collection vial. The sample and the connective tubings are then rinsed with a pre-selected volume of solvent. The inclusion of an additional nitrogen purge to guarantee the complete removal of the solvent from the PLE system is current practice. Together, these steps constitute a cycle and can be repeated several times if necessary. The total extraction time is

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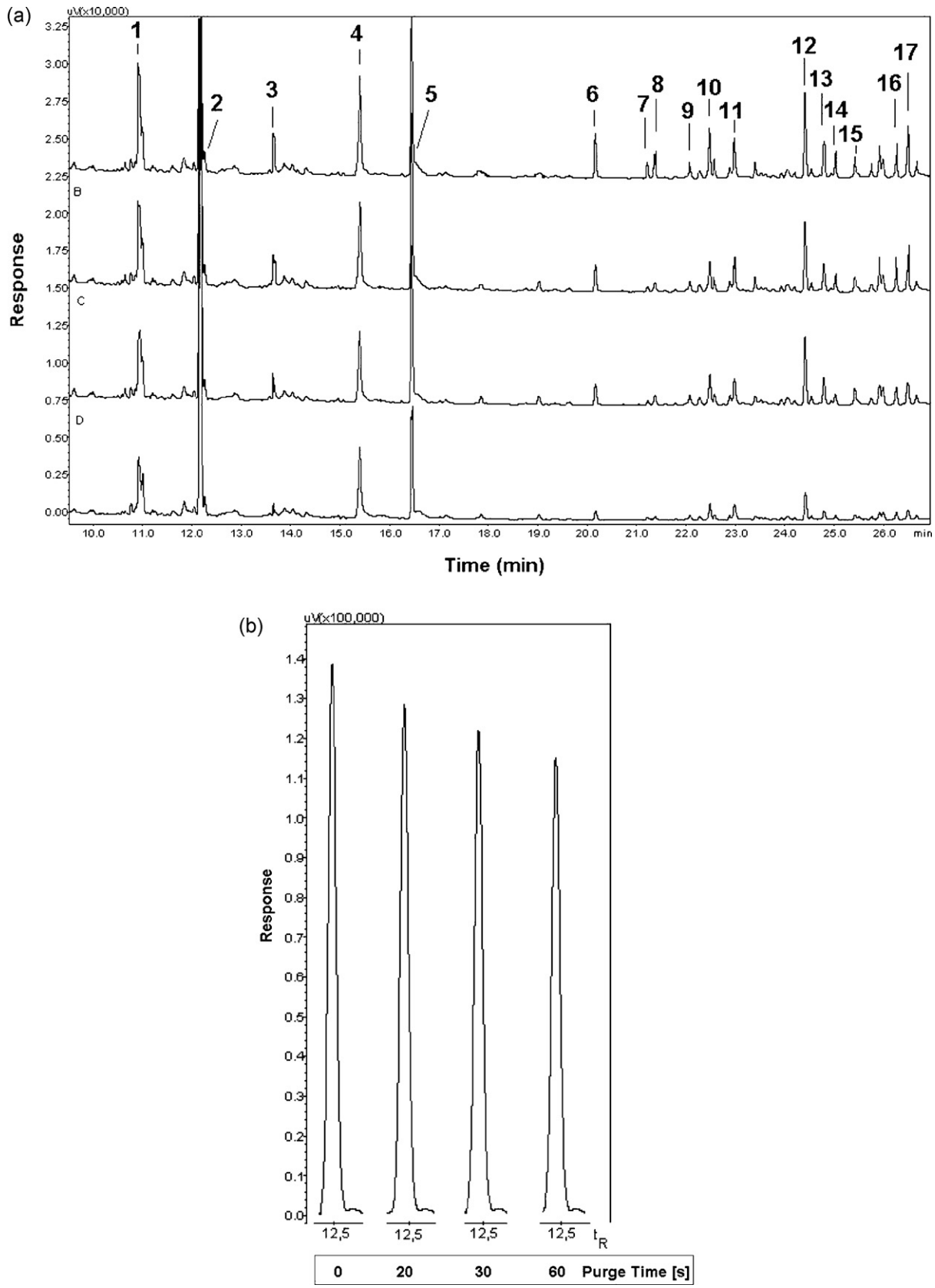

Fig. 1. (a) Exemplary GC-FID chromatograms of cypress extracts obtained by the means of PLE using ethyl acetate as an extraction solvent while applying different Purge Times 0 s (A), 20 s (B), 30 s (C) and 60 s (D). The peaks visible on chromatograms are: 1. β-Myrcene, 2. D-Limonene, 3. α-Terpinolene, 4. (−)-Terpinen-4-ol, 5. Standard, 6. Terpinyl acetate, 7. Carveol acetate, 8. Longifolene, 9. Thujopsene, 10. α-Humulene, 11. γ-Cadinene, 12. cis-Muurola-5-en-4-α-ol, 13. 1,2-epoxide-Humulene, 14. 1,10-si-epi-Cubenol, 15. 1-epi-Cubenol, 16. Hinesol, 17. epi-α-Cadinol. (b) The same as in (a) but for D-Limonene peak.