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Topiař, Martin
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Fractionation of volatile compounds from eucalyptus and lippia using sorbents together with supercritical fluid extraction

M. Topiar, M. Sajfrtova, H. Sovova, J. Karban

Institute of Chemical Process Fundamentals of the ASCR, v.v.i., Rozvojova 135, 165 02, Prague 6, Czech Republic; tel. +420 220 390 241, e-mail: topiar@icpf.cas.cz

In the recent years, natural products from plants became more often used in many fields. The traditional techniques for isolation of active compounds from plants are steam distillation and extraction by organic solvents. However, these processes have significant drawbacks, including the thermal degradation of unstable substances in case of distillation or a high consumption of organic solvents with their traces in product at conventional extraction. These problems can be solved by adopting supercritical fluid extraction (SFE) using carbon dioxide as a solvent, which is a gentle technology with particular interest for the extraction of biologically active substances. On the top of that, their composition in extract is almost the same such as that of plant. Extracts from plants are mostly mixtures of many chemical compounds with different characteristics, which can be applied in different branches. Certain groups of chemical species extracted by SFE can be concentrated directly by using series of separators or in combination with sorption [1]. The aim of this work was to separate and concentrate the groups of terpenes and terpenoids from eucalyptus (*Eucalyptus grandis* L.) and lippia (*Lippia javanica* L.) to several fractions using SFE in one step with adsorption. Extracts of both plants contain significant amount of biologically active compounds and therefore they can find application in medicine or as botanical insecticide in agriculture [2].

The fractionation of volatile oils was achieved by changing the density of carbon dioxide during adsorption and desorption step. The first fraction of extract was obtained by the SFE coupled with adsorption. The extractor with plant material was then disconnected and the second fraction was obtained by desorption with supercritical CO₂. The regeneration of adsorbent by pressurized solvent extraction with hexane yielded the third fraction of non-volatile compounds. The efficiency of adsorption and desorption steps was tested with two adsorbents, silica gel and charcoal. The extraction kinetics was evaluated by collecting several samples during the experimental run. The experiments were carried out with 40 g milled plant material at temperatures from 40 to 60 °C and pressures from 9 to 30 MPa. The flow rate of CO₂ was adjusted from 0.9 to 1.8 g.min⁻¹. The chemical composition of volatile fractions and their yield were determined by GC-MS and GC-FID. Terpenes and terpenoids were the major volatile compounds found in the extracts from eucalyptus (globulol, α-pinene, 1,8-cineole, and aromadendrene) and lippia (perillaldehyde, limonene and germacrene D). The adsorption and desorption conditions (pressure, temperature, flow rate and kind of adsorbent) were optimized to maximize the yields of volatile fractions.

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References:

1. Z. Shen et al. (2002) J. Agric. Food Chem. 50: 154
2. J. Madzimure et al. (2011) Trop. Anim. Health. Prod. 43: 481.