Abstract

Charles University

Faculty of Pharmacy in Hradec Králové

Department of analytical chemistry

Candidate: Filip Bařinka

Supervisor: PharmDr. Pavel Jáč, Ph.D.

Consultant: MSc. Dmytro Kosolapov

Diploma thesis title: Optimization of selected parameters of CE-MS method for the analysis of dietary supplements containing boswellic acids.

The aim of this work was to optimize the selected parameters of the existing CE-MS method for the analysis of boswellic acids, the main biologically active substances contained in plants of the genus *Boswellia*. Boswellic acids, or *Boswellia serrata* extract, are a popular phytomedicines with anti-inflammatory and anti-rheumatic effects, available on the Czech market in the form of dietary supplements, for which the method is being developed. The method also targets the presence of substances from the group of non-steroidal anti-inflammatory drugs (NSAIDs) as potential adulterants of these dietary supplements, as their therapeutical activity overlaps with boswellic acids. The separation took place in a background electrolyte with a composition of 40 mmol/L ammonium acetate (pH 8.5), methanol, and acetonitrile (5:1:4, v/v/v). Capillary electrophoresis was coupled with MS using a sheath-liquid interface. The mass spectrometer was equipped with an Agilent JetStream ion source and a triple quadrupole for targeted analysis in the selected reaction monitoring (SRM) mode.

In this diploma thesis, the ion source settings were optimized by means of design-of-experiments (DoE). DoE was performed in two phases. The screening was carried out using fractional factorial design, during which 4 parameters that had the most significant effect on the ionization of analytes (ESI capillary voltage, nozzle voltage, high-pressure, and low-pressure iFunnel voltage) were selected for subsequent optimization. Optimization was done using a central composite design. In the next step, two procedures for the extraction of boswellic acids from dietary supplements were compared. Subsequently, experiments were carried out to study the influence of other factors affecting the repeatability and sensitivity of the method in order to achieve the lowest possible values of RSD of the corrected area while maintaining or increasing sensitivity. The composition of the sheath-liquid, its flowrate, nebulization pressure, and time and mode of sample injection were optimized. Selected dietary supplements containing *Boswellia serrata* extract were analysed using the final method. From the resulting SRM electropherograms, a quantitative difference in the proportion of individual boswellic acids in different dietary supplements was clearly visible, showing the potential of this method for the quality control of these products. None of the adulterants from the NSAID group whose SRM transitions were included in the CE-MS method were identified in the analysed dietary supplements.