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Sensitive speciation of arsenic compounds with capillary electrophoresis

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Arsenic is naturally abundant in the crust of the earth and introduced into the aquatic system through dissolution and weathering of minerals. Chronic ingestion of arsenic in water may cause various diseases, including cancer and keratosis. A guideline for arsenic in drinking water has been set at 10 ppb of total arsenic by the World Health Organization (WHO). However, inorganic forms of arsenic, such as arsenites [As (III)] and arsenates [As (V)], are much more toxic than the organic forms as the monomethylarsonic acid (MMA) and dimethylarsinic acid (DMA). Hence the quantitation of specific arsenic species may be more meaningful than the total arsenic determination for the evaluation of the health risks from arsenic-contaminated drinking water. We report a highly sensitive way of arsenic speciation using a commercial Capillary Electrophoresis (CE) instrument equipped with a UV absorbance detector. We used a counter-flow electrokinetic supercharging technique to enhance the detection sensitivity. Electrokinetic supercharging is one of the most powerful sample stacking methods that combine field amplified sample injection and transient isotachophoresis. In counter flow electrokinetic supercharging, a constant counter pressure is applied during sample injection in order to counterbalance the movement of the injected sample zone, obtaining a pronounced increase in the amount of sample injected and the portion of the capillary available for electrophoresis.

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Solvent system selectivities in countercurrent chromatography using *Salicornia gaudichaudiana* metabolites as practical example with off-line electrospray mass-spectrometry injection profiling

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Salicornia gaudichaudiana (Chenopodiaceae) is a halophyte plant that grows in high-level salt soil. Plant material is used as 'green salt' in food preparations for people with high blood pressure and kidney / heart diseases. Countercurrent chromatography (CCC) is a form of liquid-liquid partition chromatography in which the stationary liquid phase is retained in the apparatus without the use of a solid support. A large variety of solvent systems (SoSy) have been proposed and employed in CCC which, despite being an efficient technique, will not separate compounds of a complex mixture without the appropriated system. This study describes the influence of SoSy selectivity on the separation of *S. gaudichaudiana* metabolites. HEMW at 0.5:6:0.5:6 and HBuWat 1:1:2, medium polarity SoSy, gave good distribution of compounds between the two phases. Two CCC runs were performed at identical experimental conditions using the two SoSy. Odd fractions were analyzed by decoupled ESI-MS/MS for metabolite monitoring. Selected ion traces in the two reconstituted CCC-ESI-MS/MS allowed the visualization of the SoSy selectivity for major compounds. In general, HEMW at was more selective and distributed all compounds along the CCC separation with isolation of flavonol glycosides, although co-elution of dicaffeoylquinic acids occurred. HBuWat with less general compound selectivity fractionated isomeric caffeoyl-quinic acids. The SoSy selectivity in CCC is very important when having a target compound in a complex mixture. The strategic use of different SoSy in a specific sequence can influence the results. From the results of our study we could plan to isolate the flavonoids with HEMW at SoSy and then re-fractionate the sample with HBuWat for the isolation of caffeoyl-quinic acid isomers.

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