DETERMINATION OF MERCURY SPECIES BY DIFFUSIVE GRADIENTS IN THIN FILMS AND LIQUID CHROMATOGRAPHY - ATOMIC FLOURESCENCE SPECTROMETRY

Faculty of Agronomy

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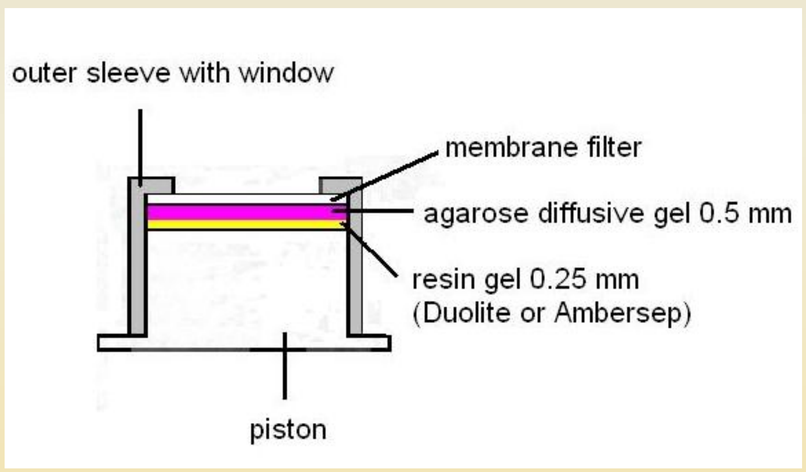
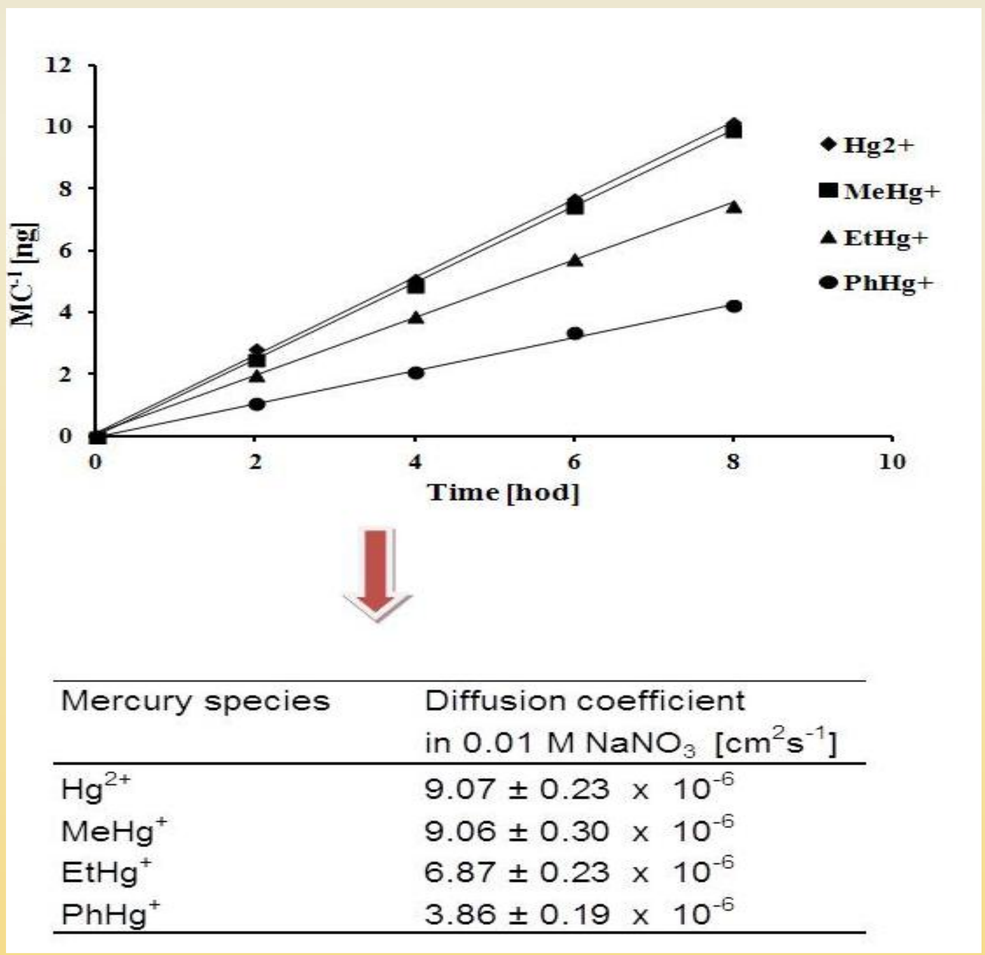
ABSTRACT

A diffusive gradient in thin films technique (DGT) was combined with high-performance liquid chromatography (HPLC) and cold vapour fluorescence spectrometry (CV-AFS) for the simultaneous quantification of four mercury species (Hg^{2+}, CH_3Hg^+, $C_2H_5Hg^+$, and $C_6H_5Hg^+$). After diffusing though an agarose diffusive layer, the mercury species were accumulated in resin gels containing thiol-functionalized ion-exchange resins (Duolite GT73, and Ambersep GT74). The diffusion coefficients at 25 °C were $9.07\pm0.23\times10^{-6}$ cm^2 s^{-1}, $9.06\pm0.30\times10^{-6}$ cm^2 s^{-1}, $6.87\pm0.23\times10^{-6}$ cm^2 s^{-1}, and $3.86\pm0.19\times10^{-6}$ cm^2 s^{-1} for Hg^{2+}, CH_3Hg^+, $C_2H_5Hg^+$, and $C_6H_5Hg^+$, respectively. A microwave-assisted extraction in presence 6M HCl and 5M HCl (60°C, 20 min) was used for isolation mercury species from Ambersep and Duolite resin gels, respectively. The extraction efficiency was better than 92.0 % (RSD 3.0%). Isolated mercury species were separated by gradient elution at a flow rate 0.8 ml min^{-1} (with a mobile phase containing 6% methanol + 0.05% 2-mercaptoethanol + 0.02 M ammonium acetate with a stepwise increase of methanol content up to 80 % in the 16th min) on a Zorbax C18 RP column.

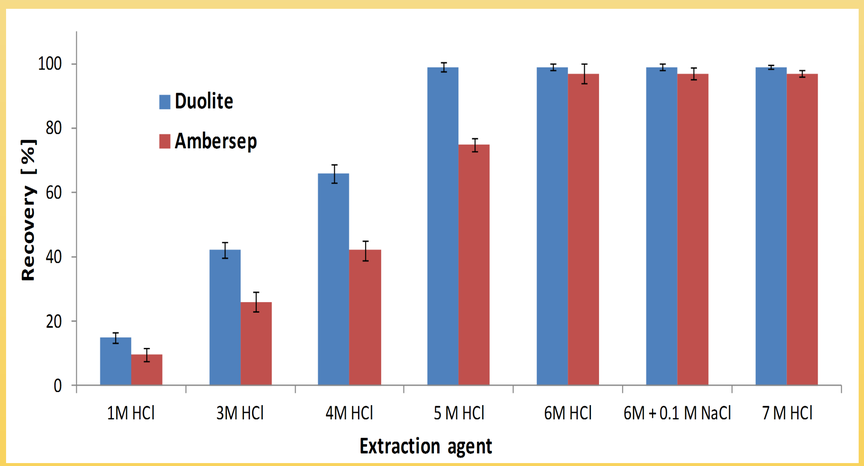


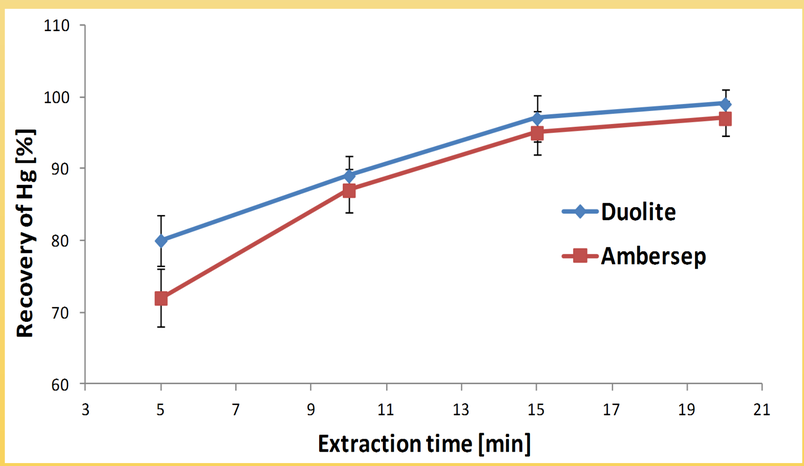


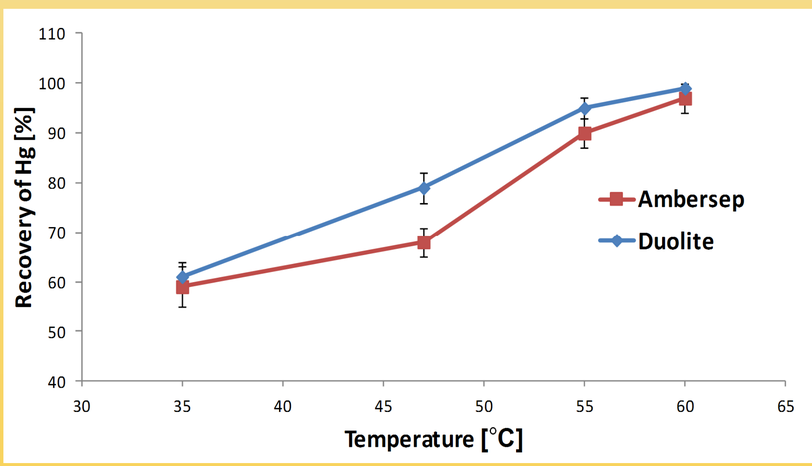
Mercury species	Diffusion coefficient in 0.01 M $NaNO_3$ [cm^2s^{-1}]
Hg^{2+}	9.07 ± 0.23 x 10^{-6}
$MeHg^+$	9.06 ± 0.30 x 10^{-6}
$EtHg^+$	6.87 ± 0.23 x 10^{-6}
$PhHg^+$	3.86 ± 0.19 x 10^{-6}

Isolation of mercury species from the resin gels

Isolation of mercury species from the resin gels is the most complicated part of the analytical procedure. Recovery, repeatability, and preservation of the initial distribution of mercury species were the most important criteria for development of isolation method. Simultaneously, eluted mercury species must be in a form that can be subsequently used for the chromatographic separation. Very rapid (20 min) and quantitative (better than 92.0 %) release of mercury species from resin gels was obtained by microwave-assisted extraction (ETHOS SEL, Millestone, Italy). Extraction reagents (6M HCl and 5M HCl) were used for isolation mercury species from Ambersep and Duolite resin gels, respectively.

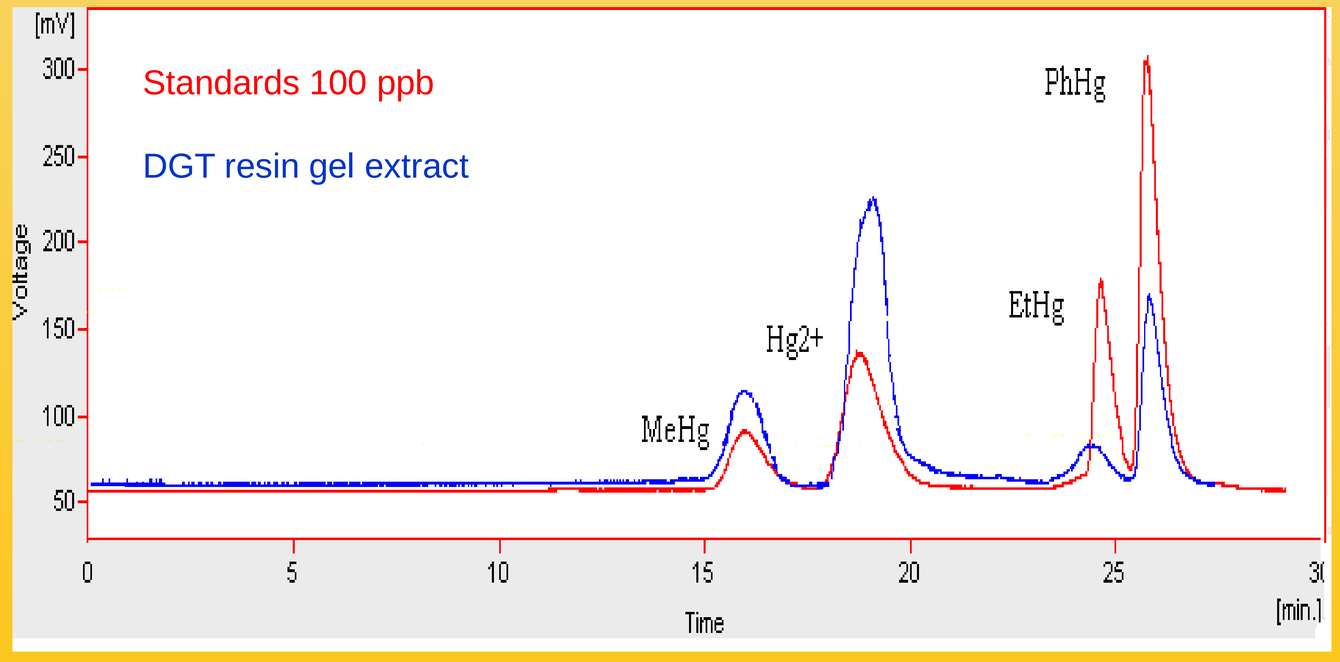






Separation and detection of mercury species by HPLC-CV-AFS

Isolated mercury species were separated on a Zorbax C18 RP column (4.6 x 150 mm, 5 μm) using a mobile phase containing 6% methanol + 0.05% 2-mercaptoethanol + 0.02 M ammonium acetate with a stepwise increase of methanol content up to 80 % in the 16th min. Separated analytes were detected CV-AFS (PSA Millenium Merlin, GB). pH of resin gel extracts was adjusted by NaOH to value 3-5.



Acknowledgement:

This work was supported financially by the project no.**CZ.1.07/2.2.00/28.0302**

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