

ABSTRACT

Development and testing of novel non-toxic electrode materials, detection arrangements and analytical methods applicable in determination of selected agrochemicals is the main aim of this Ph.D. Thesis.

New working electrodes based on silver solid amalgam paste (AgSA-PE) with organic pasting liquid and other based on crystalline silver amalgam (CAgAE) were developed, their electrochemical behaviour investigated and further used in voltammetric determination of widespread and toxic environmental pollutant 4-nitrophenol (4-NP). This analyte could be determined by DPV at AgSA-PE with limit of detection (L_D) $1 \times 10^{-6} \text{ mol l}^{-1}$ and using CAgAE with L_D $4 \times 10^{-7} \text{ mol l}^{-1}$, both in 0.2 mol l^{-1} acetate buffer pH 4.8. Attempts to decrease L_D s by utilization of adsorptive stripping voltammetry were not successful in either case. Crystalline silver amalgam was also successfully used for construction of microcylindric flow-through cell and tested for amperometric determination of nitrophenol mixture in HPLC-ED system. Both novel electrodes were found to be suitable alternatives to toxic mercury electrodes and the CAgAE seems to be promising working electrode for flowing systems.

Method for sample preparation and voltammetric determination of broad-spectrum herbicide Glyphosate in contaminated soil samples was also observed. However, it was unfortunately found out that application of the silver solid amalgam electrode can not be used in this case. Its narrower potential window prevented its use and hanging mercury drop electrode had to be utilized. This fact still demonstrated usefulness of the mercury electrodes and its irreplaceability in some special cases. Sample extraction, cleaning and pre-concentration steps were proceeded before the determination using DPV of derivatised Glyphosate to N-nitrosoglyphosate with L_D about $1 \times 10^{-5} \text{ mol l}^{-1}$ in the soil samples with three different organic matter contents. Apparently, higher content of the organic matter facilitated the recovery of the GP from the soils, supposing being easier to remove the GP associate with organics than adsorbed in inorganic soil matter.

Furthermore, application of the silver solid amalgam for construction of amperometric detectors was verified. Polished silver solid amalgam electrodes were used in thin layer and wall jet arrangements utilizable in detection of selected nitrophenol derivatives using HPLC-ED. Both detection systems were successfully used for determination of selected nitrophenols, mainly contained in commercially available plant growth stimulators, with L_D s from 5 to $10 \times 10^{-6} \text{ mol l}^{-1}$ using thin layer detector and from 10 to $25 \times 10^{-6} \text{ mol l}^{-1}$ using wall jet arrangement coupled with HPLC.