## **Abstract**

The presented work is focused on the investigation of novel approaches applied to bismuth and antimony based electrodes together with further improvements in their electroanalytical performance for measuring trace metal analytes. A suitable protocol for measuring trace concentration levels of Hg(II) using anodic stripping voltammetry at the in-situ prepared SbFE in the presence of Cu(II), is presented. The results obtained allowed its favorable comparison with several gold-based electrodes, which is one of the most frequently used materials for measuring trace amounts of Hg(II). The broader applicability of the in-situ BiFE under more acidic conditions (pH = 2.0) was investigated associated with the addition of tartrate ions in the plating/measurement solution. This new approach revealed auspicious results also for measuring trace Zn(II) in the presence or absence of metal ions, i.e. Cd(II) and Pb(II). For the first time, bismuth was deposited on ensembles of gold nanoelectrodes (Au-NEEs) and satisfactorily applied for anodic stripping voltammetric measurement of trace Pb(II) both at the in-situ and ex-situ prepared bismuth film. Another interesting application was provided following the in-situ bismuth deposition on a conventional gold bulk electrode by applying the AdCSV protocol in alkaline media. Tartrate ions were included in the plating/measurement solution in order to prevent bismuth hydrolysis. The subsequent Ni(II) and Co(II) dimethylglyoximate stripping signals were measured with or without the addition of several potentially interfering ions and surfactants in order to get more insight into the electrode behavior. Successful measurements of the same analytes previously mentioned were carried out by employing a macro-porous bismuth film carbon screen printed electrode. With the beneficial increment of the active surface area, the electrode provided very low detection limits for both Ni(II) and Co(II). Finally, a precise cutting procedure for carbon fibre in order to obtain a desired disk-shaped microelectrode was investigated using the commercial laser ablation instrument.

## Keywords

Anodic stripping voltammetry, adsorptive cathodic stripping voltammetry, antimony electrode, bismuth electrode, nanoelectrodes, electroanalytical measurements, trace analysis, real water sample.