Introduction

Relatively high levels of Pb can be readily consumed as part of our diet from ceramic glazed tableware containing Pb as part of the decorative pattern or glaze. Awareness of such problems has generated a demand for methods that are rapid, inexpensive, reproducible, sensitive and accurate. Electrochemical techniques such as anodic stripping voltammetry (ASV) offer a number of advantages both in terms of economics, sensitivity, portability and ease of use, requiring little more than an appropriate power supply for such applications. Coupled to these advantages, the technique of ASV can also be tailored to give a wide dynamic range from these extremely low levels to the ppm range, or higher.

However, previously, ASV has suffered from the common use of Hg working electrodes, the use and subsequent disposal of which have lead to its lack of market penetration compared to other techniques. Therefore, there are a growing number of reports in which Hg free SPECS with working electrodes of the micrometer (µm) range have been utilised, the radial diffusion inherent with these devices allows for analysis to be made without the need for forced convection.

In this present study we have investigated the possibility of determining trace Pb concentrations at a screen-printed microband electrode (µBSPCE). This to our knowledge is the first report on using these electrodes for anodic stripping voltammetry (ASV) for the determination of Pb in the leachates from glazed ceramic plates.

Experimental

Voltammetric Conditions

Cyclic voltammograms were recorded in plain solutions (0.1 M) of the supporting electrolyte and then in the same solution containing Pb. Voltammetric conditions were as follows: starting and final potential, 0.0 V with a scanning rate of 100 mV/sec using a scan rate of 5 mV/sec in a 3 mm x 3 mm Ag/AgCl pseudo reference/counter electrode. Each voltammetric run was followed by a delay of 30 s to allow the cell to equilibrate. All ASV experiments were performed in a 25 ml Pyrex beaker containing 2 ml of 0.1 M pH 4.1 acetate buffer. A working electrode of 38 µm in diameter of printed carbone microband electrode (µBSPCE) was employed as the working electrode. All ASV experiments were conducted at room temperature.

Results & Discussion

Effect of Chloride Ion Concentration

In order to explore the voltammetric behaviour and optimise the conditions for the ASV determination of Pb at a microband SPECS, studies were made to ascertain the optimum supporting electrolyte. For a 0.1 mM Pb acetate buffer, it can be seen from Figure 4 that with increasing NaCl concentration a noticable increase in the resulting ip for the Pb stripping peak is obtained, with a maximum seen between 10 mM and 33 mM NaCl. Consequently, a 0.1 M pH 4.1 acetate buffer containing 13 mM NaCl was used in further investigations.

Conclusions

We have successfully demonstrated a rapid and quick economic approach for the determination of microband screen printed carbon electrodes.

Acknowledgements

The authors would like to thank the HEFCE for funding. They are grateful to Gwent Electronic Materials Ltd, for supplying the screen printed carbon electrodes. Senter Research is thanked for his assistance with preliminary studies.

The Voltammetric Behaviour of Lead at a Microband Screen-Printed Carbon Electrode and its Determination in Acetate Leachates from Glazed Ceramic Plates

Kevin C. Honeychurch and John P. Hart*

Centre for Research in Analytical, Material and Sensors Sciences, University of the West of England, Bristol, Coldharbour Lane, Filton, Bristol, BS16 1QY, UK.

*Corresponding Author: Tel: +44 117 3282469, Fax: +44 117 3282904, email john.hart@uwe.ac.uk

Centre for Research in Analytical, Material and Sensors Sciences, University of the West of England, Bristol, Coldharbour Lane, Filton, Bristol, BS16 1QY, UK.

*Corresponding Author: Tel: +44 117 3282469, Fax: +44 117 3282904, email john.hart@uwe.ac.uk

Figure 1. Schematic diagram demonstrating the method for the manufacture of microband SPECS from C16839P14. (a) 3x3 mm SPECS working electrode, with screen printed Ag/AgCl pseudo-reference/counter electrode. (b) Microband SPECS plan view. (c) Microband SPECS cross section.

Figure 2. Typical cyclic voltammograms obtained with a microband SPECS for 0.1 mM Pb acetate buffer/0.1 M NaCl, (a) in the absence of and (b) the presence of 0.1 mM Pb. Initial and final potential, 0.0 V, scanning potential, 100 mV/sec, scan rate 5 mV/sec.

Figure 3. Typical cyclic voltammograms obtained with a microband SPECS for 0.1 mM Pb acetate buffer/0.1 M NaCl, (a) in the absence of and (b) the presence of 0.1 mM Pb. Initial and final potential, 0.0 V, scanning potential, 100 mV/sec, scan rate 5 mV/sec.

The effect of applied potential was studied for a 0.1 mM Pb solution over the range -0.7 V to 1.7 V (vs. Ag/AgCl) using an accumulation time of 60 s (Figure 5). Under these conditions, the magnitude of the stripping peak was found to increase with increasing negative potential, until forming a plateau between -1.0 V and -1.6 V (vs. Ag/AgCl). Consequently, further investigations were made using an applied potential of -1.3 V (vs. Ag/AgCl).

Figure 4. Effect of Cl ions on the LSASV Peak current for 0.1 mM Pb acetate buffer/0.1 M solution containing 0.1 M Pb. Voltammetric conditions: accumulation time 80 s, accumulation potential -1.3 V

Figure 5. Effect of accumulation time on the current density gained at a microband SPECS and a 3x3 mm Ag/AgCl pseudo-reference/counter electrode.

Figure 6. LSASV determination of 0.605 mg/l Pb in the presence of 250 mg/l Sn.

Figure 7. LSASVs of plate sample leachate at µBSPCE (a) and (b) example voltammograms for added concentrations of Pb. Accumulation time: 15 s for plate A and 1500 s for plate B, deposition potential -1.2 V (vs. Ag/AgCl).

Plate A

Plate B

Analytical Application

Using a shorter accumulation time of 15 s, a linear range of between 2 mg/l and 50 mg/l Pb was obtained.