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## SONOELECTROANALYSIS – APPLICATION TO LEAD DETERMINATION\*

*Ultrasound and benefits of its application in electrochemistry and electroanalysis have been explained and demonstrated. Lead determination by stripping analysis coupled with insonation was used to illustrate advantages of employing ultrasound in electroanalysis. Quantitative analysis of lead under insonation was shown to be possible in different media, including organic and biological samples where electroanalytical techniques often fail to give satisfactory results due to passivation of electrodes in these complex matrices.*

REVIEW PAPER

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The electrochemical methods of analysis have found a vast range of applications due to their several advantages. These include high sensitivities and the detection of a wide range of analytical targets down to concentrations of the order of  $10^{-10}$  M or below in favourable conditions, while still being relatively inexpensive over alternative analytical techniques such as spectroscopic methods.

Stripping analysis, a highly sensitive electroanalytical technique [1,2], involve two steps: deposition of target ion at the electrode surface, and stripping of the deposited material by sweeping the potential to induce a faradaic current arising from the oxidation or reduction of the accumulated material. Stripping voltammetry takes three variations: anodic stripping voltammetry (ASV) [3–6], cathodic stripping voltammetry (CSV) [7,8] and adsorptive stripping voltammetry (AdSV) [9–12]. Nanomolar limits of detection can be achieved using stripping analysis with optimisation of the deposition and stripping processes parameters, namely the deposition time, the deposition potential and mass transport of the analyte to the electrode. The use of some form of forced convection during the electrochemical deposition improves the stripping analysis by enhancing the mass transport of the analyte towards the electrode surface and thus allowing both higher sensitivity and reduced accumulation periods.

Application of ultrasound to electrochemical processes, among other advantages, provides higher mass

transport of the electroactive species to the electrode. Ultrasound in broad sense is defined as sound above 20 kHz and up to 100 MHz and can be subdivided into two distinct regions: power, ultrasound of 20–100 kHz frequency commonly employed in sonochemistry, and sonoelectrochemistry, and diagnostic, high frequency ultrasound from around 5 MHz generally used in medical imaging. Power ultrasound applied to a solution produces three main physical effects: acoustic streaming, cavitation and microjetting [13–18].

These effects resulting from application of ultrasounds in chemistry and electrochemistry offer significant benefits: enhanced mass transport, reduced diffusion layer which reduces depletion of electroactive species, constant degassing at the electrode surface, depassivating, *i.e.* cleaning of the electrode surface and significant increase of current. When applied to electrochemical experiments, ultrasound provides an increased mass transport regime in which the voltammetric response is qualitatively and quantitatively changed [18,19]. The effect of application of ultrasound in electrochemistry are clearly evidenced by the transformation of the electrochemically reversible cyclic voltammogram observed under silent conditions into a hydrodynamic voltammogram with no forward/back sweep hysteresis. The currents obtained under insonated conditions are significantly larger than the silent ones. Additionally, the limiting current is not flat as expected for a hydrodynamic voltammogram measured at a rotating disc electrode or channel electrode. Rather there are irregular spikes present and the magnitudes of these are related to the power of the ultrasound applied.

The above mentioned advantages of ultrasound have suggested coupling of power ultrasound to electroanalytical procedures [19,20–22]. The *in situ* cleaning of the electrode during insonation is particularly useful in anal-

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ysis of highly passivating media, namely real samples that may contain organic materials and natural surfactants [5,23,24]. In the stripping analysis, the ultrasound is applied during the deposition step to enhance the mass transport of analyte towards the electrode surface. The ultrasound parameters, the horn tip to electrode surface distance and the ultrasound amplitude, must be carefully optimised particularly to ensure that micro-jetting is not removing analyte electrodeposited during stripping analysis or even damaging the electrode surface itself [19].

In this paper, the advantages of insonation for electroanalysis are demonstrated on the example of determination of lead (II) in laboratory conditions as well as in real samples, including both aqueous and non-aqueous media.

### SONOELECTROANALYTICAL DETERMINATION OF LEAD

A significant increase in the accumulation of toxic species in the environment due to human activities has been noticed in the last few decades. Heavy metals, such as copper, cadmium, zinc and lead, are continuously introduced into the surroundings throughout numerous industrial processes and are accumulated due to their non-degradability and recirculation; they are potentially dangerous to humans and the environment, depending on the chemical form and exposure level [25]. This urged a need to develop a simple and inexpensive, but efficient, procedure for monitoring the level of heavy metal ions in the environment [26]. Electrochemical sensors for determination of heavy metals present relatively easy and low-cost method, and are also characterised by high sensitivity, high regeneration – easy stripping of accumulated heavy metal, and long-time use [27–31]. Determination of lead in different media, aqueous, organic and biological, was made possible by coupling the existing electrochemical techniques with power ultrasound.

#### Sonoelectroanalytical determination of lead in aqueous solutions

The synergy of power ultrasound with electroanalytical techniques is shown to be possible and beneficial. Proof-of-concept is shown for the anodic stripping voltammetry of lead at screen printed electrodes and screen printed calomel modified electrodes offering shorter deposition times and increased sensitivity. All measurements were conducted in a three-electrode cell. The working electrode was either carbon screen printed electrode or calomel modified carbon screen printed electrode of 0.1 cm<sup>2</sup> working area, prepared by earlier reported procedures [32–34]. The intensity of the ultrasound was determined calorimetrically [35] and it was found that a 10% amplitude corresponded to 36 W cm<sup>-2</sup>.

The working electrode was placed in a side-on arrangement to the ultrasonic horn. Different horn-to-electrode distances were used in the measurements. During experiments, the temperature was kept constant at 22 °C (±2 °C).

First, the voltammetric response of a screen printed carbon electrode was explored in a 1 mM solution of ferrocyanide in 0.1 M KCl, under silent conditions and during the application of 8 and 15% power ultrasound, where the cyclic voltammetric response was transformed into a mass transport limited current with the magnitude of the current increasing significantly. Cyclic voltammetry was run again under quiescent conditions, to explore the effect of ultrasound on the SPC electrode, where a slight increase in both the oxidative and reductive peak currents has occurred compared to that obtained for the fresh electrode without any ultrasound applied. This increase in the magnitude of the voltammetric peaks likely reflects the “roughening” of the surface of the screen printed electrode from either cavitation, or high mass transport, or a combination of both processes which likely removes ink covering carbon particles and thus increases the overall effective surface area of the electrode.

Subsequently, the benefits of power ultrasound for anodic stripping voltammetry of lead (II) at screen printed electrodes were investigated. Anodic stripping voltammetry of screen printed electrodes in a 50 μM lead acetate solution stirred by conventional method showed a sharp stripping peak for lead at *ca.* -0.6 V (*vs.* SCE). A significant increase in stripping peak current is observed after applying ultrasound during the deposition step compared to the one obtained with deposition under silent conditions (Figure 1). The limit of detection (*LOD*) was calculated using standard addition method. For 90 s deposition under silent conditions, the *LOD* was found to be 1×10<sup>-7</sup> M. The *LOD* was found to be as low as 0.5×10<sup>-8</sup> if ultrasound was applied during the deposition time of only 20 s. Noticeably, further improvement in sensitivity is possible by optimizing ultrasound parameters such as horn-to-electrode distance and ultrasound power, or deposition time.

Next, a preliminary investigation of lead (II) determination at calomel modified screen printed electrodes using sonovoltammetry was carried out. The behaviour and stability under ultrasound of the calomel modified screen printed carbon electrode was first considered as it is recognised that for some materials sustained insonation can lead to significant ablation. Comparing cyclic voltammogram of new, previously unused calomel modified screen printed electrode and cyclic voltammogram of the same electrode after ten-hour insonation, it can be noticed that the peak current has increased after the use of ultrasound. Again, this likely reflects the removal of ink from carbon particles allowing more of these particles to be accessible to the solution and consequently to be voltammetrically stripped.

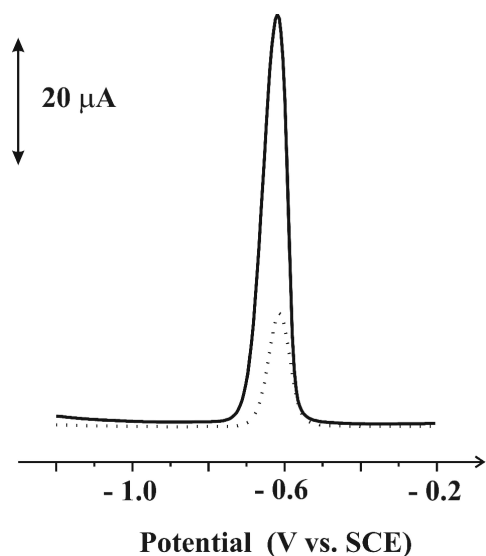


Figure 1. Anodic stripping voltammograms of determination of lead (II) at screen printed carbon electrode of  $0.1 \text{ cm}^2$  working area under quiescent conditions (.....) and with ultrasound applied (—) during deposition in  $50 \text{ } \mu\text{M}$  lead (II) solution in acetate buffer at a scan rate of  $0.25 \text{ V s}^{-1}$  [42].

The calomel modified screen printed electrode was proven to be very stable under ultrasound and subsequently lead (II) determination using this electrode with ultrasound applied was examined. The limit of detection for lead (II) using insonated calomel modified screen printed electrode was determined to be  $4 \times 10^{-9} \text{ M}$  using only a 20 s accumulation time with ultrasound applied during the deposition; again, this value can be optimised to allow lower concentrations to be determined.

Last, the effect of the ultrasound parameters was explored. Ultrasound was applied to the screen printed carbon electrodes (unmodified) at different intensities and at various horn-to-electrode distances using a constant deposition time with the magnitude of the lead stripping peak *via* anodic stripping voltammetry monitored. It was found that the optimum ultrasound power and horn-to-electrode distance in terms of the electrode stability and increase of magnitude of currents are 10% and 30 mm, respectively. Any further decrease in the horn-to-electrode separation or increasing the ultrasound power resulted in the magnitude of the lead stripping peak to decrease reflecting a turbulent regime in the sonoelectrochemical cell.

It was shown that sono-anodic stripping voltammetry could be employed for the determination of lead content in aqueous solutions and the improvements in sensitivity arising from insonation were demonstrated [31,36–42].

#### Sonoelectroanalytical determination of lead in river sediment [43]

Sono-anodic stripping voltammetry was proved to be useful for the determination of lead content in aqueous

solutions [31,36–42]. Furthermore, Saterlay *et al.* showed that cathodic stripping technique coupled with ultrasound can also be employed for quantitative analysis of lead in aqueous media. Moreover, this technique was shown to be exceptionally useful for the quantification of lead in real samples such as river sediment where other heavy metals are present as well.

Boron-doped diamond electrode was placed directly opposite to the horn tip at a 10 mm distance. Ultrasound of  $14 \text{ W cm}^{-2}$  power was employed for all cleaning, conditioning and electrodeposition steps. CSV protocol included cleaning of the electrode at 0.0 V for 120 s followed by the preconditioning of the electrode at  $-1.0 \text{ V}$  for 60 s. Lead was deposited on the electrode surface by holding the potential at 5.0 V for only 60 s. During the stripping step, lead accumulated at the electrode was stripped by sweeping the potential from 1.5 to 0.0 V at a scan rate of  $100 \text{ mV s}^{-1}$ . Limit of detection for lead in aqueous solutions using sono-CSV was found to be as low as  $3 \times 10^{-7} \text{ M}$ . Limits of detection of one order smaller ( $10^{-8}$ ) could be obtained using longer deposition time of 240 s and ultrasound of higher power. However, limits of detection of the order of  $10^{-7}$  were accepted as satisfactory since application of the ultrasound of higher powers could lead to the electrode breakage.

The interference of the other heavy metals present in real samples was noticed during the quantitative analysis of lead in real samples by ASV. Therefore, the possible interference of other heavy metals for the determination of lead content in real samples by the sono-CSV was also examined. It was observed that metals such as chromium, nickel, cadmium and zinc were not detectable by sono-CSV, even when present in higher concentrations. Copper and iron gave cathodic stripping peaks but with no effect on the lead cathodic stripping peak. A small decrease in the cathodic stripping peak of lead was only observed when mercury or selenium were present in exceptionally high concentration (100 times higher than lead concentration).

When the same procedure was applied for the determination of lead in river sediment (old channel of the River Severn at Welshpool in MidWales), the content of lead was evaluated to be 187.1 mg/kg for the sediment sample. Lead content in the same sample was found to be 206.1 mg/kg using inductive coupled plasma mass spectroscopy (ICP–MS) technique. These two results were in a good agreement, with sono-CSV procedure having a great advantage of being only *ca.* 60 min long compared to 48-hour long ICP–MS procedure.

#### Sonoelectroanalytical determination of lead in wine [44]

Anodic stripping voltammetry has been widely used for trace metal measurements in food and beverages due to its high sensitivity. Metals ions are often bound in inorganic and organic complexes in such samples and

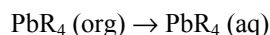
different pretreatment procedures have to be employed prior to metal determination. Application of anodic stripping voltammetry for determination of lead in wine under silent condition failed to give satisfactory results. Commonly only about 50% of lead present in wine could be detected under these conditions [45]. However, coupling ultrasound with ASV made quantitative analysis of lead in wine possible.

ASV protocol included acidification and degassing of the sample, white Italian Chardonnay wine. The working electrode was a 4 mm diameter mercury plated platinum disk electrode. The mercury film was prepared *ex situ* by the procedure previously described in the literature [37]. The working electrode was placed in a face-on arrangement relative to the horn at a 10 mm distance from the horn tip. Ultrasound of  $26 \text{ W cm}^{-2}$  power was applied only during the deposition step for 240 s, while stripping step was performed under the silent conditions. Lead ions were reduced at the electrode at  $-1.0 \text{ V}$  forming a Hg–Pb amalgam. The potential was then swept anodically at a scan rate of  $50 \text{ mV s}^{-1}$  and a well-defined peak of lead oxidation was observed. The amount of lead present in the solution could be determined from the peak area, *i.e.* charge deposited onto the electrode. For the quantitative analysis of lead in wine, additions of the standard lead solution were made with 10 min insonation of each solution for the mixing and equilibration to complete. The value of total lead in wine yielded by sono-ASV was found to be  $22 \pm 6 \text{ } \mu\text{g/l}$ . The result was found to be in excellent agreement with the results obtained with the currently accepted standard method ( $24$  and  $27 \pm 6 \text{ } \mu\text{g/l}$ ) [46]. With enhanced mass transport as well as cleaning and activation of the electrode under the ultrasound, the quantification of lead in wine is performed without the need for removal or destruction of organic matter *via* elaborate and lengthy pre-treatment processes.

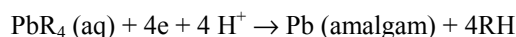
#### Sonoelectroanalytical determination of lead in petrol [47]

Determination of small quantities of lead in petrol in order to meet regulation standards has urged a need for more efficient methods for lead analysis. Blythe *et al.* suggested a sono-anodic stripping method for determination of lead in petrol. In the proposed method, 4 or 12 mm diameter Hg/Pt electrode was employed as a working electrode and positioned opposite to the ultrasonic horn at a 7 mm distance from the horn tip. Sono-ASV protocol included cleaning step at  $0.0 \text{ V}$  for 10 s to remove any residual lead, followed by the accumulation step at  $-1.0 \text{ V}$  for 240 s under the ultrasound of  $52 \text{ W cm}^{-2}$  power. The stripping step was performed under silent conditions, sweeping the potential from  $-1.0$  to  $-0.15 \text{ V}$  at a scan rate of  $100 \text{ mV s}^{-1}$ . Proposed mechanism of lead determination in petrol using sono-ASV includes following steps:

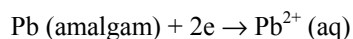
(1) Extraction of the tetraalkyl leads from the petrol phase under insonation:



(2) Reduction of lead at the surface of the electrode at a potential of  $-1.0 \text{ V}$ :



(3) Analytically sensitive stripping of Pb to form  $\text{Pb}^{2+}$  in solution:



Concentration of lead in petrol detected by sono-ASV using standard microaddition protocol was evaluated from the peak area, *i.e.* the charge deposited onto the electrode surface, and calculated to be  $308 \pm 04 \text{ mg/l}$ . Concentration of lead in the same sample detected by atomic absorption spectroscopy was  $400 \pm 20 \text{ mg/l}$ . Furthermore, it should be mentioned that the same procedure also can be applied to the determination of very small (two orders of magnitude smaller) quantities of lead present in petrol. The electroanalytical analysis allows quantitative determination of the total lead content in petrol in a total time of *ca.* 2 h without the need for sample pretreatment.

#### Sonoelectroanalytical determination of lead in saliva [48,49]

Determination of a lead levels in clinical samples are of great importance due to toxic effects of lead. It was found that concentration of lead in saliva is closely related to recent lead exposure and may provide an efficient and non-invasive method of analyzing lead level for persons who are exposed to lead, such as workers who are occupationally exposed or children who came into contact with lead paints.

In the proposed method, the working electrode was a 3mm diameter glassy carbon disc electrode (GCDE) or a Nafion coated mercury thin film glassy carbon electrode (NCMTFGCE). This electrode was positioned in face on arrangement with the ultrasonic horn during the insonation. Glassy carbon electrode proved to be unsuitable for the determination of lead in saliva under silent conditions due to simultaneous oxidation and reduction of lead at this electrode. The signal was significantly increased if ultrasound was employed during the deposition step though simultaneous oxidation and reduction of lead could still be noticed. These problems could be overcome using NCMFGCE. Square-wave anodic stripping protocol involved silent pre-treatment at  $0.6 \text{ V}$  for 60 s followed by an insonated deposition at  $-1.5 \text{ V}$  for 120 s with a horn-to-electrode distance of 5 mm. During the following, square-wave anodic stripping step the potential was swept from  $-0.725$  to  $-0.3 \text{ V}$  at  $50 \text{ mV s}^{-1}$ . Standard addition method was used to determine the limit of detection of lead in artificial sa-

liva using sono-SW-ASV. The limit of detection was evaluated to be 0.25 µg/l using 3 sigma method. It was noticed that artificial saliva, more precisely salivary glycoproteins, rapidly passivate the electrode, after 9 additions when insonation was employed.

Furthermore, similar procedure was employed for determination of lead in human saliva giving a good response for lead over the range 0.9–20 µg/l. The obtained results were in good agreement with the results evaluated from ICP–MS data. Therefore, sono-anodic stripping voltammetry could be used for determination of lead in human saliva as an alternative to conventional and costly inductively coupled plasma mass spectroscopy and atomic absorption spectroscopy.

#### Sonoelectroanalytical determination of lead in blood [50]

Normal lead levels in blood are in the range from 0 to  $2 \times 10^{-7}$  M, but it can be as high as micromolar concentrations after exposure. Several attempts of determination of lead in blood by electrochemical techniques have been reported in the literature [51–53]. Krusma *et al.* suggested the first mercury-free electroanalytical method for lead determination in blood. Determination of lead by *in situ* formed bismuth-film-modified boron doped diamond electrode with ultrasound was first investigated in nitric acid solution and limits of detection of the order of  $10^{-8}$  and  $10^{-10}$  M were found for 60 and 300 s deposition time, respectively. Analysis of lead in whole blood was performed under somewhat changed conditions (deposition at –3.1 V under ultrasound of 34.3 W cm<sup>-2</sup> power with a horn-to-electrode distance of 10 mm) and limits of detection of the order of  $10^{-8}$  M in a diluted blood solution were obtained with excellent inter- and intra-reproducibility and sensitivity. This was the first example of mercury-free lead determination in blood with advantages of also being rapid and sensitive compared to existing techniques.

#### Acknowledgment

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## IZVOD

### SONOELEKTROANALIZA – PRIMENA U ODREĐIVANJU OLOVA

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(Pregledni rad)

Ultrazvuk i korisni efekti primene ultrazvuka u elektrohemiji i elektroanalitičkoj hemiji su objašnjeni i ilustrovani. Kvalitativna i kvantitativna analiza olova pomoću elektroanalitičkih tehnika uz primenu ultrazvuka primer je na kom su ilustrovane prednosti korišćenja ultrazvuka u elektroanalitičkoj hemiji. Pokazano je da je uz primenu ultrazvuka moguća kvantitativna analiza olova u različitim medijima, uključujući organske i biološke uzorke. U ovim medijima elektroanalitičke tehnike često ne daju zadovoljavajuće rezultate usled pasivizacije electrode u ovim složenim matricama.

Ključne reči: Elektroanaliza • Elektroanalitičke tehnike • Ultrazvuk • Sonoelektroanaliza • Olovo  
Key words: Electroanalysis • Stripping analysis • Ultrasound • Sono-electroanalysis • Lead