

Short Assessment on Thin film Electrodes

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Abstract

Anodic stripping voltammetry (ASV) is a strong electrochemical analytical method for detecting and quantifying a wide range of metal ion species in aqueous fluids at extremely low concentrations. While early ASV measurements used macroscopic electrodes such as Hg drop electrodes to create surfaces appropriate for plating/stripping, more recent work on the method has used thin film metal electrodes manufactured in situ to replace these electrodes. Such electrodes are plated onto the surface of a primary electrode with the analyte species, resulting in a composite metal electrode from which the analyte(s) may be removed, identified, and measured. In this article, we'll look at how these unusual electrodes were created and how they're used in a range of applications. In both acidic and alkaline environments, a variety of metals (e.g., Hg, Bi, Sn, etc.) have showed promise as thin film ASV electrodes, and numerous metals, in addition to the analyte of interest, are typically deposited simultaneously to optimize plating/stripping behavior and improve sensitivity. It has been widely used to test dangerous metals in the environment, assess battery materials, and enable biological assays, among other uses, due to the comparatively simple nature of the measurement and its applicability for a wide range of pH. We'll go through these applications in further depth, as well as providing some insight into the thin film electrodes' future development and applications in ASV measurements.

Keywords: Anodic stripping voltammetry; electrochemical analytical method; analyte species; metals

Introduction

Anodic stripping voltammetry (ASV) is an essential electrochemical method with roots dating back to the beginnings of the field [1]. It is a simple approach that enables for the identification and measurement of electro active species in solution at nanomolar (nM) concentrations (10s of ppb) and lower, with sensitivity comparable to inductively coupled plasma mass spectrometry (ICP-MS). A more extensive description of the procedure may be found elsewhere. ASV is made up of two main steps:

- Cathodic electrodeposition to concentrate dissolved analytes at an electrode,
- Anodic electrode stripping to re-dissolve the species into solution

Each electrodeposited species should produce a stripping peak, whose voltage may be used to identify the species being oxidized and whose peak current/integrated charge is proportional to the species concentration in solution [2-5]. Traditionally, ASV has been conducted with metallic Hg electrodes, such as Hanging Mercury Drop Electrodes (HMDE) or Mercury Film Electrodes (MFE). Hg's strong conductivity, ability to create easily-stripped amalgams with a variety of metals, and weak Hydrogen Evolution Reaction (HER) kinetics on Hg enable for higher reductive potentials to be given during electrodeposition benefit both electrodes [6-7]. MFEs are usually favored over HMDEs because they have a smaller electrode volume and thickness, which increases the measurement's sensitivity. MFEs are commonly co-deposited onto an inert electrode, typically glassy carbon, from the analyte solution after adding modest amounts of Hg^{2+} to obtain low electrode thickness [8]. Co-deposition also makes sample preparation easier and minimizes the amount of mercury needed. Although these features make MFEs appealing for use in ASV studies because they lower the amount of Hg required, the inherent toxicity of Hg and the severe health hazards it poses have pushed researchers to

seek for more benign alternatives.

As a result, contemporary research in the sector has concentrated on using metals like Bi, Sn, Au, and others to replace Hg. Although these metals do not create the same amalgams as Hg, they are an intriguing addition to the field due to their low toxicity and compatibility with non-acidic electrolytes. Furthermore, most of them, like Hg, may be co-deposited to generate Metal Thin Film Electrodes (MTFEs) that are quite comparable to more typical MFEs.

Electrode Types

Mercury Film Electrodes: During the 1950s and 1960s, ASV utilizing MFEs became popular as a precise electrochemical method for assessing metal content in aqueous samples. While MFEs provide high sensitivity ASV measurements, the high concentration of analyte metal in the electrode might lead to intermetallic phase interference [9]. MFEs are frequently placed onto glass carbon (GC) electrodes to increase measurement reliability. GC electrodes are very conductive and may be polished to a very flat surface that is impervious to and chemically inert to Hg, enabling for the construction of a consistent electrode surface throughout several experiments. MFEs are typically deposited in situ with the analyte species during the pre-concentration process, despite the fact that they may be deposited from a separate Hg^{2+} solution and subsequently employed for ASV analysis (*ex-situ* electrode). The outcome is a tens of micron-thick heterogeneous film made up of the MFE and the analyte(s) of interest that can be easily peeled and has a large amount of analyte to aid detection [10]. MFEs can be utilized for ASV analysis of non-aqueous samples as well as for assessing metal content in aqueous samples.

Non-Hg MTFEs: Despite the fact that MFEs are virtually perfect electrodes for ASV analysis, worries about Hg toxicity and a desire to enhance the technique have fueled the creation of MTFEs made of other metals. Bi is one of the most widely utilized non-Hg MTFEs, and it may be employed in the same way as *in-situ* and *ex-situ* Hg analogues. It was discovered that adding Bi^{3+} to the deposition solution was necessary for plating of Zn, Cd, and Pb, and that it could be employed in conjunction with an MFE to increase Tl and Cd sensitivity. Bi MTFEs, like MFEs, may be plated *ex situ* and employed for ASV detection of Pb content, according to later publications [11].

Bi electrodes might also be utilized to do ASV analysis in very alkaline liquids, according to further study. Typical ASV analysis takes performed in acidic media with solvated M^+ metal ions, and should follow well-established methods for metal electrodeposition in acidic media. However, in solutions with a pH greater than 11, Bi^{3+} can produce soluble, readily stripped/plated $\text{Bi}(\text{OH})_2^+$ species due to the high concentration of OH in the solution. Other metals, such as Pb and Zn, have this hydroxide complication capability; however Hg generates insoluble hydrated mercury oxides, which limits the usage of MFEs.

Conclusion

MTFEs provide a number of advantages over HMDE and bulk electrodes for ASV analysis. MTFEs may be deposited in situ with the analyte species, resulting in highly concentrated electrode films with exceptional sensitivity over a wide range of concentrations. Many metals, including the conventional (but poisonous) Hg and less toxic alternatives like Bi or Sn, can be used to make MTFEs. This versatility allows the MTFE to be tailored for a specific test, resulting in electrochemically different bimetallic alloys with the species of interest, even when additional species of interest are present.

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