



Stainless steel electrode for simultaneous stripping analysis of Cd(II), Pb(II), Cu(II) and Hg(II)

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ABSTRACT

Traditional electrodes for stripping analysis generally have narrow electrochemical window, require the modification of electrode or the addition of additional ions. To solve these problems, stainless steel has been used as the electrode for electrochemical stripping analysis for the first time. Square wave anodic stripping voltammetry (SWASV) has been used for the detection of Cd²⁺, Pb²⁺, Cu²⁺, and Hg²⁺. Type 304 stainless steel electrode gives well-defined, sharp, and separated stripping peaks for these metal ions. The electrode, best operated at + 0.3 V (Hg²⁺), - 0.05 V (Cu²⁺), - 0.41 V (Pb²⁺), and - 0.7 V (Cd²⁺) and after a 300 s deposition at - 1.0 V, has linear responses in the concentration ranges of 0.075–5 μM for Pb²⁺ and Cu²⁺, 0.5–5 μM for Cd²⁺, and 0.1–5 μM for Hg²⁺. The limits of detection (at S/N = 3) are 0.033 μM for Pb²⁺, 0.0073 μM for Cu²⁺, 0.23 μM for Cd²⁺, and 0.028 μM for Hg²⁺. The reproducibility, expressed as relative standard deviation, is 3.2% for Pb²⁺, 2.6% for Cu²⁺, 5.1% for Cd²⁺, and 2.5% for Hg²⁺ (each 1 μM levels; for n = 6). The electrode was successfully applied to the determination of the ions in spiked groundwater samples. This study shows that stainless steel is a better alternative to mercury electrode for stripping analysis because of its well-defined and sharp stripping peaks, high sensitivity, low background, low toxicity, good reproducibility, and much wider electrochemical window.

1. Introduction

Heavy metal ions are the main contaminants of aqueous systems due to their solubility in water and non-biodegradable characteristics. The presence of heavy metal ions in drinking water is a worldwide concern, getting the paramount attention due to their adverse effects on humans, animals and species living in water [1–4]. Different acute and chronic diseases are the worse effects associated with heavy metal ions poisoning even at low level exposure. Therefore, sensitive and selective analysis of heavy metal ions in different environmental samples, such as drinking water, is of utmost importance [5–7].

The simultaneous detection of trace metal ions at very low concentration level by using inexpensive, fast and easily operative instrumentation is interesting topic because these methods can save time; reduce the costs of analysis and ability to analyze multi-elements.

Stripping analysis has been demonstrated to show significant sensitivity toward heavy metal ions detection [8–11]. Anodic stripping voltammetry (ASV) is most effective and widely used technique, in which the heavy metal ions were predeposited onto the electrode surface and afterward detected by stripping step [12–16]. Mercury-based electrodes have been widely used for stripping voltammetric analysis due to their outstanding sensitivity and reproducibility [2,17,18]. However, due to the toxicity of mercury, many efforts have been made to replace mercury with more environmentally friendly electrodes for the simultaneous determination of trace heavy metals [19–21]. As a result, electrode materials, such as carbon material [22], gold [23,24], bismuth based [25–28], antimony based [10,29], nanocomposite [30], tantalum [14] electrodes have been developed. In particular, bismuth based electrodes have been received much attention [25–28].

Stainless steel is a widely used iron alloy which contains alloying

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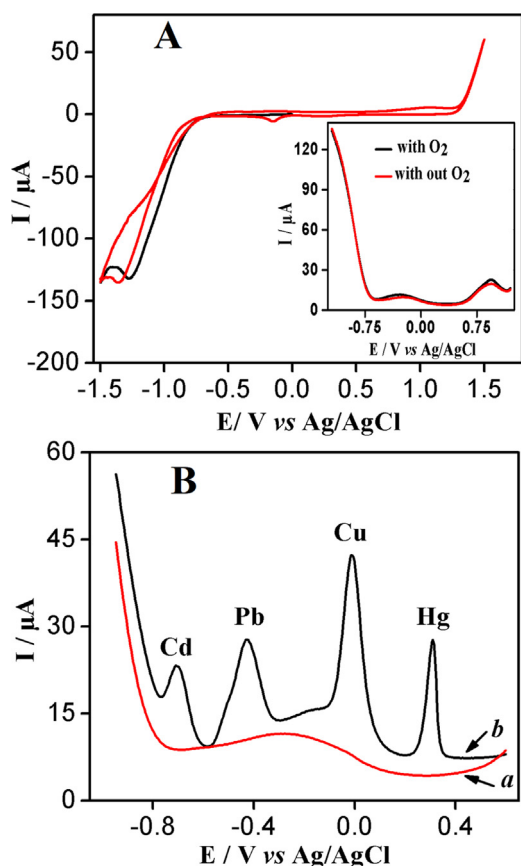


Fig. 1. (A) CV in 0.1 M pH 4.5 acetate buffer solution (Inset: effect of oxygen on SWASV in 0.1 M pH 4.5 acetate buffer solution); (B) Stripping voltammograms: (a) 0.1 M pH 4.5 acetate buffer solution; (b) after the addition of $1 \mu\text{M Cd}^{2+}$, Pb^{2+} , Cu^{2+} , and Hg^{2+} at type 304 stainless steel electrode; SWASV settings: frequency 15 Hz, potential step 4 mV, and pulse amplitude 25 mV.

elements such as, chromium, nickel, molybdenum, copper, carbon, nitrogen and some other elements to enhance the properties of steel. The surface oxide film of stainless steel has been of significant concern because of its importance in anti-corrosion and anti-oxidation performance. Chromium in stainless steels can rapidly form a very thin layer of chromium (III) oxide upon exposure to air, thus making stainless steels highly resistant to oxidation [31,32]. Besides its corrosion resistance, stainless steel has interesting characteristics, such as low-cost, environmentally friendly, high mechanical strength, good electrical conductivity and commercial availability [33–36].

In this study, we have developed stainless steels as new electrodes for electrochemical stripping analysis of trace heavy metals for the first time. Experimental parameters that influenced the electroanalytical signals were optimized, such as deposition potential and deposition time. The stainless steel electrode shows well-defined and sharp stripping peaks, high sensitivity, low background, good reproducibility, and wide electrochemical window in both cathodic and anodic regions.

2. Experimental section

2.1. Reagents

Type 304 stainless steel was obtained from Suzhou Qiangda Fastener Co., Ltd. All chemicals were purchased from Beijing Chemical Works (Beijing, China). 0.1 M acetate buffer solutions (pH 4.5) were prepared by mixing appropriate amount of 0.2 M CH_3COOH and CH_3COONa each and were used as supporting electrolyte throughout the experiments. Stock solutions (10 mM) of Cd^{2+} , Pb^{2+} , Cu^{2+} , and

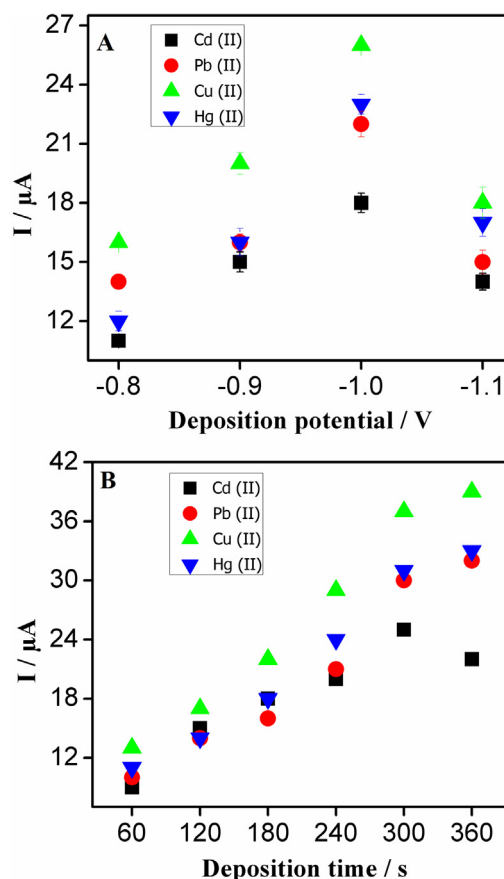


Fig. 2. Effect of (A) deposition potential, (B) deposition time containing $1 \mu\text{M Cd}^{2+}$, Pb^{2+} , Cu^{2+} , and Hg^{2+} ; SWASV settings: frequency 15 Hz, potential step 4 mV, and pulse amplitude 25 mV.

Hg^{2+} were prepared by doubly distilled water. Analytical grade reagents were used throughout the experiment.

2.2. Apparatus

Square wave anodic stripping voltammograms (SWASVs) were tested with a CHI 840B electrochemical workstation. Besides using type 304 stainless steel electrodes (3 mm in diameter) as working electrode, Ag/AgCl (saturated KCl) electrode and gold wire were used as reference and auxiliary electrode, respectively. The type 304 stainless steel electrode was fabricated by tightly packing stainless steel rod (3 mm in diameter) into the electrode cavity of Teflon tube and then the electrode was polished with abrasive papers and slurry of 0.5 and 0.03 μm alumina then sonicated and rinsed with double distilled water.

2.3. Square wave anodic stripping voltammetric measurement of Cd^{2+} , Pb^{2+} , Cu^{2+} , and Hg^{2+} at the stainless steel electrode

Type 304 stainless steel electrode was immersed in 0.1 M pH 4.5 acetate buffer solution containing Cd^{2+} , Pb^{2+} , Cu^{2+} , and Hg^{2+} . The deposition potential of -1.0 V was applied to the stainless steel electrode for 300 s while the solution was stirred. Following the preconcentration step, the stirring was stopped and after 10 s of equilibration time the responses were recorded by applying a square wave voltammetric scan with amplitude 0.025 V and frequency 15 Hz in the range from -1.0 to $+0.5 \text{ V}$. Before the next measurement, the electrode was cleaned by keeping the working electrode at the potential of 0.60 V for 30 s in a stirring solution and then the electrode was polished with abrasive papers and slurry of 0.03 μm alumina then sonicated and rinsed with double distilled water. To examine the effect of oxygen, CV was run in air saturated and oxygen free

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