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High Sensitive Detection of Cd(II) and Pb(II) Based on Antimony-Film Covered Pencil Core Electrodes

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Abstract: In this paper, a pencil core was used as the substrate for an antimony-film electrode for simultaneous detection of Cd(II) and Pb(II) by square-wave anodic stripping voltammetry (SWASV). With the deposition time of 180 s, the low limit of detection (LOD) for the electrode was determined to be $0.075 \mu\text{g} \cdot \text{L}^{-1}$ for Cd(II), while $0.13 \mu\text{g} \cdot \text{L}^{-1}$ for Pb(II) with good reproducibility in low pH solutions (pH = 2). The results show a much lower LOD than that of bismuth-film pencil core electrode. Finally, this antimony-film electrode was successfully applied to determine Cd(II) and Pb(II) in tap water.

Key words: antimony film electrodes; anodic stripping voltammetry; heavy metal ions; pencil core electrode

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It is well known that heavy metal ions pose severe risks to the environment and human health. Cadmium can accumulate in various tissues and organs, especially for the kidney cortex^[1], leading to a vast assortment of illnesses, such as proteinuria, calciuria, aminoaciduria, glycosuria, and tubular necrosis^[2]. Lead has been shown to cause damage to various systems when absorbed into the body, including the hematopoietic, renal and skeletal systems, while the most significant effects occur with the central nervous system^[3-4]. Lead can interfere with brain development, slow down nerve conduction velocity, and trigger behavioral problems^[5-8], therefore, the detection of heavy metal ions is very important. Many methods for the detection of cadmium and lead have been reported previously, such as atomic emission spectrometry with inductively coupled plasma excitation (AES-ICP), X-ray fluorescence (XRF)^[10], electrothermal atomic absorption spectrometry (ETAAS)^[11], colorimetry^[12] and electrochemistry^[13]. Among them, elec-

trochemical method offers remarkable performance due to its simple instrument and easy operation.

Electrochemical methods have played an important role in detection of heavy metal ions, among them stripping voltammetry and chromopotentiometry are recognized as the most powerful tools in analyses of heavy metals^[14]. Stripping voltammetry commonly employs the mercury film electrode and hanging mercury drop electrode, which can form an amalgam with metal ions, and has been used for heavy metal ion detection for numerous years. However, due to the toxicity of mercury, some environmental-friendly materials have been reported to replace mercury. Recently, bismuth, a low toxicity material, has been introduced to substitute mercury as a promising electrode material for its ability to form alloys with different heavy metals similar to mercury amalgams^[15]. A bismuth-film electrode has been successfully used for the detection of different heavy metals, such as Cd(II), Pb(II), Zn(II), Cu(II), and Ni(III)^[16-18]. More re-

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cently, antimony, another new electrode material, has attracted attention to application with stripping analysis of heavy metals similar to bismuth film electrodes^[19]. Compared with the bismuth-film electrode, the antimony-film electrode offers a remarkable stripping performance in lower pH solutions ($\text{pH} \leq 2$) due to the favorable overpotential for hydrogen relative to the bismuth-film electrode. Therefore, the antimony-film electrode may be a more promising alternative in terms of performance for the determination of heavy metal ions.

Glassy carbon^[11, 20], gold^[21], silver^[22], iridium^[23], carbon paste^[24], boron doped diamond^[25] have been reported as a base for bismuth- or antimony-film electrodes. However, a pencil core rod has also been used as an electrode material for stripping analysis^[26-28]. The advantages of this material include its low cost, simple and fast preparation, renewable character, and high electrical conductivity^[29-30]. Here, by *in situ* depositing antimony on a pencil core electrode substrate, simultaneous detection of heavy metal ions Cd(II) and Pb(II) is reported. The pencil core antimony-film electrode displays a high sensitivity, $0.075 \mu\text{g} \cdot \text{L}^{-1}$ for Cd(II) and $0.13 \mu\text{g} \cdot \text{L}^{-1}$ for Pb(II), which are lower than $0.3 \mu\text{g} \cdot \text{L}^{-1}$ and $0.4 \mu\text{g} \cdot \text{L}^{-1}$, respectively, for Cd(II) and Pb(II) from pencil core bismuth-film electrode^[20], and $0.7 \mu\text{g} \cdot \text{L}^{-1}$ and $0.9 \mu\text{g} \cdot \text{L}^{-1}$, respectively, for Cd(II) and Pb(II)^[19] from glassy carbon antimony-film electrode. The pencil core antimony-film electrode also shows good reproducibility compared with the bismuth-film electrode, and is applied to the detection of the Cd(II) and Pb(II) in tap water.

1 Experimental

1.1 Apparatus

Electrochemical experiments were carried out using CHI 620B (ChenHua Instrument, Shanghai, China). A conventional three-electrode system was employed with a platinum wire as the counter electrode, and an Ag/AgCl ($3 \text{ mol} \cdot \text{L}^{-1}$ KCl) as the reference electrode. All experiments were performed at room temperature.

The pencil core rod (HB, 0.5 mm in diameter) was purchased from TOMBO (Japan). A copper wire was coiled around the pencil core rod and inserted in-

to a stock tube which was subsequently sealed with epoxy resin. The electrode was successively polished with 1000-mesh sand paper, then washed and sonicated in water before use.

1.2 Chemicals and Reagents

Standard stock solutions of antimony, Sb(III), lead, Pb(II) and cadmium, Cd(II), were provided by National Analysis Centre for Iron and Steel. Hydrochloric acid was obtained from the National Institute for Control of Pharmaceutical & Biological Products (Beijing, China). The supporting electrolyte consisted of a $0.01 \text{ mol} \cdot \text{L}^{-1}$ solution of hydrochloric acid ($\text{pH} = 2.0$). Deionized water (Millipore, Bedford, MA) was used throughout. The tap water was from our lab.

1.3 Procedures

The electrode was successively polished to a 1000-mesh sandpaper, washed and then sonicated in water before use. The three electrodes were immersed into a 10 mL electrochemical cell, containing $0.01 \text{ mol} \cdot \text{L}^{-1}$ hydrochloric acid and $0.5 \text{ mg} \cdot \text{L}^{-1}$ of Sb(III). The deposition potential of -1.2 V was applied while the solution was stirred. Following the preconcentration step, the stirring was stopped. After 5 s, the square-wave anodic stripping voltammetric measurement was performed by a potential scan from -1.2 V to 0.2 V with a frequency of 25 Hz, a pulse height of 25 mV, and a step increment of 4 mV. The standard solutions of Cd(II) and Pb(II) were introduced after the background signal was recorded. Prior to the next cycle, the electrode was cleaned for 60 s at 0.1 V under stirring.

2 Results and Discussion

2.1 Effect of Antimony Concentration

The concentration of Sb(III) could affect the thickness of antimony film, resulting in a different peak current of Cd(II) and Pb(II). Fig. 1 shows the square-wave anodic stripping responses for a pencil core graphite electrode in a solution containing $30 \mu\text{g} \cdot \text{L}^{-1}$ either Cd(II) or Pb(II) and $0.01 \text{ mol} \cdot \text{L}^{-1}$ hydrochloric acid ($\text{pH} = 2$) with different concentrations of Sb(III) in the range of 0.20 to $1.50 \text{ mg} \cdot \text{L}^{-1}$. With an increase in the concentration of Sb(III), the decrease in signal of Pb(II) was observed. However, the signal of Cd(II) increased first when the concentrations of Sb

(III) increased up to $0.5 \text{ mg} \cdot \text{L}^{-1}$, and then decreased with a further increase in concentration of Sb (III). This phenomenon was previously observed at antimony-film carbon paste electrodes, and could be attributed to the formation of a Pb-Sb intermetallic compound^[31]. Therefore, a concentration of $0.5 \text{ mg} \cdot \text{L}^{-1}$ of Sb(III) was selected for further experimentation.

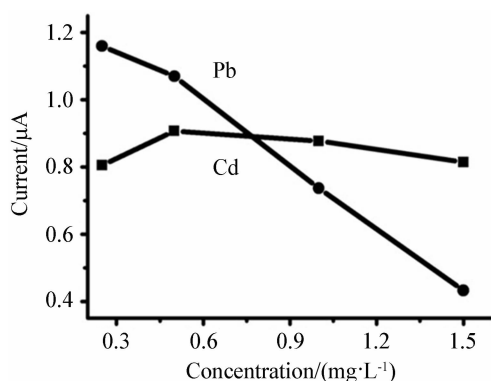


Fig. 1 Effect of Sb(III) concentration on the stripping voltammetric responses of Cd(II) and Pb(II) at an *in situ* antimony film deposited on a pencil core graphite electrode. The solutions contained $0.01 \text{ mol} \cdot \text{L}^{-1}$ hydrochloric acid and $30 \text{ } \mu\text{g} \cdot \text{L}^{-1}$ either Cd(II) or Pb(II). Deposition potential: -1.2 V ; deposition time: 240 s ; frequency: 25 Hz ; pulse height: 25 mV , step increment: 4 mV

2.2 Effect of the Deposition Potential

Fig. 2 shows the influence of the deposition potential on the peak currents of Cd(II) and Pb(II). With the more negative deposition potentials, the more heavy metal ions were deposited. But hydrogen generation occurred when the deposition potential was more negative than -1.4 V , which affected the signals of Cd(II) significantly. When the potential was -1.1 V the stripping signal of Cd(II) was highest, while the stripping signal of the Pb(II) increased with more negative deposition potential. To avoid the competitive generation of hydrogen and the codeposition of other metal ions from the real samples, the potential of -1.2 V was chosen as the optimized deposition potential for all subsequent experiments.

2.3 Effect of the Deposition Time

To enhance the electroanalytical performance,

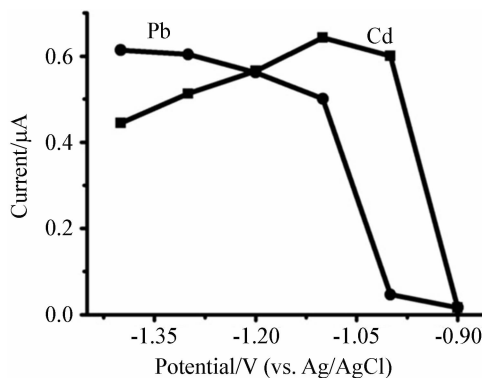


Fig. 2 Effect of deposition potential on the stripping voltammetric responses of Cd(II) and Pb(II) at an *in situ* antimony film pencil core electrode. The solutions contained $0.01 \text{ mol} \cdot \text{L}^{-1}$ hydrochloric, $30 \text{ } \mu\text{g} \cdot \text{L}^{-1}$ Cd (II) and Pb(II), and $500 \text{ } \mu\text{g} \cdot \text{L}^{-1}$ Sb(III). Deposition time: 240 s ; frequency 25 Hz ; pulse height: 25 mV , step increment: 4 mV .

the deposition time was also optimized. Fig. 3 displays the signals of Cd(II) and Pb(II) with different deposition time. When the deposition time is 180 s the signals of Cd(II) and Pb(II) are almost the same, and the signals for both heavy metal ions increased almost linearly when the deposition time is shorter than 300 s . This demonstrates that in this case the antimony coated pencil core electrode does not suffer from the saturation effect reported previously^[32] within 300 s .

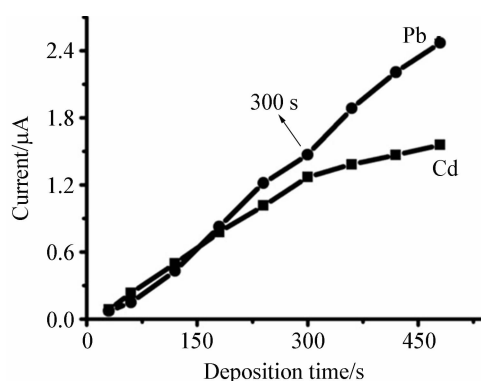


Fig. 3 Effect of deposition time on the stripping voltammetric response of Cd(II) and Pb(II) at an *in situ* antimony film deposited on a pencil core electrode. The solutions contained $0.01 \text{ mol} \cdot \text{L}^{-1}$ hydrochloric acid, $30 \text{ } \mu\text{g} \cdot \text{L}^{-1}$ Cd(II) and Pb(II), and $500 \text{ } \mu\text{g} \cdot \text{L}^{-1}$ Sb(III). Deposition potential: -1.2 V ; frequency 25 Hz ; pulse height: 25 mV , step increment: 4 mV

2.4 Calibration Data

Calibration was displayed on the antimony-film pencil core electrode for the simultaneous determinations of Cd(II) and Pb(II) with different deposition time (180 s, 300 s). As can be seen in Tab. 1, the results reveal good linear behavior for both metal ions when performed over different linear ranges, low and high concentrations. This may due to the different structures of Cd-Sb and Pb-Sb alloys^[33] under different ranges of concentration.

With a deposition time of 180 s, the LOD (3S/N) was determined to be $0.075 \mu\text{g} \cdot \text{L}^{-1}$ for Cd(II) and $0.13 \mu\text{g} \cdot \text{L}^{-1}$ for Pb(II). When the deposition time was 300 s, the LOD values of $0.048 \mu\text{g} \cdot \text{L}^{-1}$ for Cd(II) and $0.49 \mu\text{g} \cdot \text{L}^{-1}$ for Pb(II) were obtained. The results show a lower LOD than those of the $0.3 \mu\text{g} \cdot \text{L}^{-1}$ and $0.4 \mu\text{g} \cdot \text{L}^{-1}$ reported for Cd(II) and Pb(II), respectively, by bismuth-film electrode^[29]. With increasing deposition time the LOD of Cd(II) decreased, while the LOD of Pb(II) increased. The peak currents of both cations increased with longer deposition time, while the stability of the peak current of Pb(II) decreased, resulting in a higher LOD than that with the shorter deposition time. This may be attributed to the stronger competition from Cd(II) against Pb(II) to form alloys with antimony with longer deposition time. So for the rapidity and good detection limit, 180 s was chosen for detection with the real samples.

Fig. 4 reveals a good linear relationship of peak current with concentration when the deposition time was 180 s. The reproducibility for both Cd(II) and Pb(II) was also investigated when the deposition time was 180 s, and the relative standard deviation (RSD) was calculated to be 3.4% for Cd(II) and 1.3% for Pb(II) ($n = 9$) as shown in Fig. 5.

2.5 Determination of Cd(II) and Pb(II) in Tap Water

The antimony-film pencil core electrode was applied to detect Cd(II) and Pb(II) in tap water sampled from the lab. The water sample was diluted 20 times and the amounts of 10, 15, 20 $\mu\text{g} \cdot \text{L}^{-1}$ of Cd(II) and Pb(II) were added. The standard addition results are illustrated in Tab. 2. From the results, a reasonable

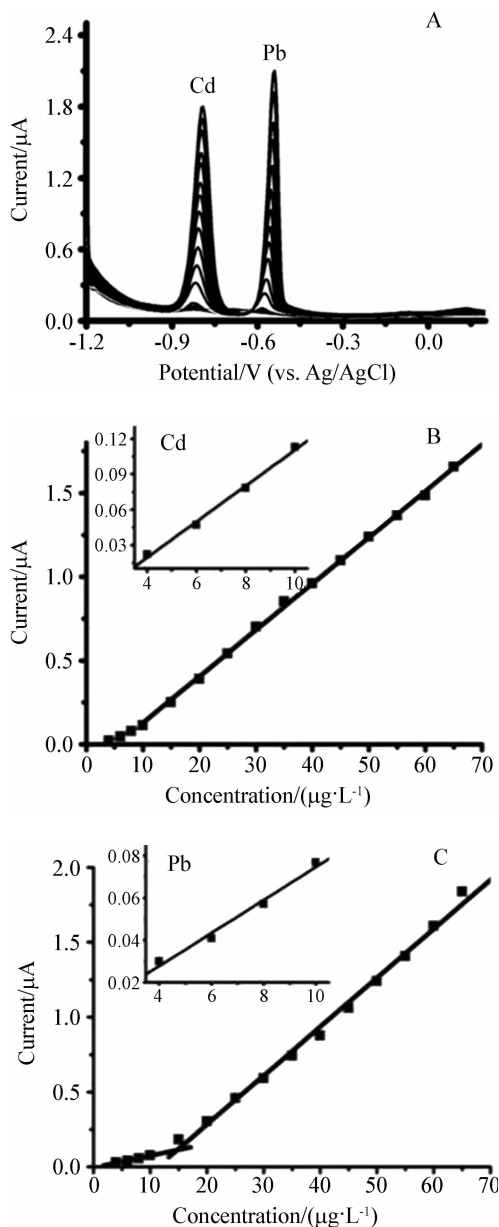


Fig. 4 SWASV data with increasing concentrations of Cd(II) and Pb(II) at an *in situ* antimony-film deposited on a pencil core electrode. The solutions contained $0.01 \text{ mol} \cdot \text{L}^{-1}$ hydrochloric acid, 4 to $65 \mu\text{g} \cdot \text{L}^{-1}$ Cd(II) and Pb(II), $500 \mu\text{g} \cdot \text{L}^{-1}$ Sb(III) (A). The linear relationship of peak currents and concentrations for Cd(II) (B) and Pb(II) (C). Deposition potential: -1.2 V ; deposition time: 240 s; frequency 25 Hz; pulse height: 25 mV, step increment: 4 mV

recovery was observed, indicating that the method employed is sufficient for practical application in real water sample detection.

3 Conclusions

Tab. 1 Results of Cd (II) and Pb (II) with different deposition time ($n = 3$)

| Deposition time/s | | Linear range/($\mu\text{g} \cdot \text{L}^{-1}$) | R | LOD/($\mu\text{g} \cdot \text{L}^{-1}$) |
|-------------------|----|--|--------------|---|
| 180 | Cd | 4~10, 15~65 | 0.997, 0.999 | 0.075 |
| | Pb | 4~10, 15~65 | 0.992, 0.997 | 0.13 |
| 300 | Cd | 4~10, 15~50 | 1.000, 0.998 | 0.048 |
| | Pb | 4~10, 15~50 | 0.992, 0.999 | 0.49 |

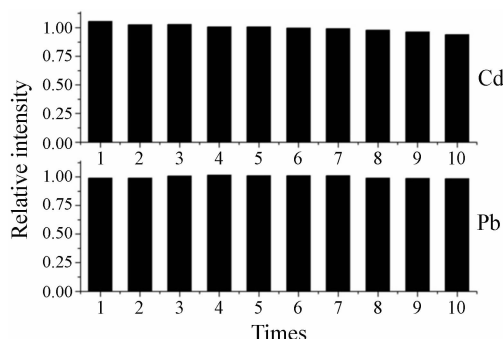


Fig. 5 The reproducibility in the stripping voltammetric responses of Cd(II) and Pb(II) at an *in situ* antimony-film pencil core electrode. The solutions contained $0.01 \text{ mol} \cdot \text{L}^{-1}$ hydrochloric, $30 \mu\text{g} \cdot \text{L}^{-1}$ Cd(II) and Pb(II). Deposition potential: -1.2 V ; deposition time: 240 s; frequency 25 Hz; pulse height: 25 mV, step increment: 4 mV

Tab. 2 The results of Cd (II) and Pb (II) by standard addition method at an antimony-film pencil core electrode in tap water sample

| Added/($\mu\text{g} \cdot \text{L}^{-1}$) | | Detected/($\mu\text{g} \cdot \text{L}^{-1}$) | | Recovery/% | |
|---|-------|--|-------|------------|-------|
| Cd | Pb | Cd | Pb | Cd | Pb |
| 10.00 | 10.00 | 10.41 | 10.39 | 104.1 | 103.9 |
| 15.00 | 15.00 | 14.66 | 15.98 | 97.73 | 106.5 |
| 20.00 | 20.00 | 19.41 | 20.78 | 98.55 | 103.9 |

In this work, the pencil core rod was used as a supporting substrate for an antimony-film electrode to detect heavy metal ions by SWASV. Its advantages, such as, low cost, easiness and rapid preparation, renewable character, low background current and high electrical conductivity, make it superior for the determination of Cd(II) and Pb(II) relative to previous reports. The antimony-film pencil core electrode was observed to perform with a low LOD and

good reproducibility. Owing to the low toxicity of antimony, inexpensive electrode substrate and simple operation, this method is promising for on-site monitoring of heavy metal ions in water.

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基于铋薄膜覆盖铅笔芯电极对Cd(II)和Pb(II)高灵敏度的检测

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摘要: 在铅笔芯上通过实时沉积铋薄膜, 使用方波阳极溶出伏安法(SWASV)对Cd(II)和Pb(II)进行同时检测. 沉积时间为180 s时, Cd(II)和Pb(II)的最低检测限分别为 $0.075 \mu\text{g} \cdot \text{L}^{-1}$ 和 $0.13 \mu\text{g} \cdot \text{L}^{-1}$, 较铋膜修饰的铅笔芯电极低. 在低pH值溶液(pH 2.0)中重现性好. 该电极可成功用于测定自来水中的Cd(II)和Pb(II)含量.

关键词: 铋膜电极; 阳极溶出伏安法; 重金属离子; 铅笔芯电极