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ORIGINAL PAPER

A novel tin-bismuth alloy electrode for anodic stripping voltammetric determination of zinc

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Abstract We report on a novel tin-bismuth alloy electrode (SnBiE) for the determination of trace concentrations of zinc ions by square-wave anodic stripping voltammetry without deoxygenation. The SnBiE has the advantages of easy fabrication and low cost, and does not require a pre-treatment (in terms of modification) prior to measurements. A study on the potential window of the electrode revealed a high hydrogen overvoltage though a limited anodic range due to the oxidation of tin. The effects of pH value, accumulation potential, and accumulation time were optimized with respect to the determination of trace zinc(II) at pH 5.0. The response of the SnBiE to zinc(II) ion is linear in the 0.5–25 μM concentration range. The detection limit is 50 nM (after 60 s of accumulation). The SnBiE was applied to the determination of zinc(II) in wines and honeys, and the results were consistent with those of AAS.

Keywords Tin-bismuth alloy electrode · Anodic stripping voltammetry · Zinc · Wine · Honey

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Introduction

The determination of heavy metal is usually performed by atomic absorption spectrometry (AAS), inductively coupled plasma-mass spectrometry (ICP-MS), inductively coupled plasma-optical emission spectrometry (ICP-OES) and atomic fluorescence spectrometry (AFS). However, these methods require expensive and bulky devices, high cost of equipment maintenance and long analysis time [1, 2]. Comparatively, due to the relatively low cost, more portable instrument and the combination of an effective accumulation step with advanced measurement procedures, stripping analysis is recognized as an extremely sensitive electrochemical technique for measuring trace metals [3]. Anodic stripping voltammetry (ASV) is the most widely used form of stripping analysis. For many years, mercury electrode was the best choice of electrode material in heavy metal detection for not only does it have a high hydrogen overvoltage and a highly reproducible and readily renewable surface, but also can form diluted amalgams which prevent the formation of intermetallic compounds [4, 5]. However, its use has been completely banned in many countries due to its hypertoxicity [6]. Also, in the last five decades, various solid electrodes such as the glassy carbon electrode (GCE) [7], graphite electrode [8], gold electrode [9], silver electrode [10] and iridium electrode [11] have been investigated for their possible ability to substitute Hg electrode. But none of these electrodes was found to be acceptable for their relatively low hydrogen overvoltage (usually below −0.8 V [5]), large background current, or poor precision and resolution [12].

Recently, bismuth-based electrodes were introduced as a favorable alternative mercury-free electrode used for stripping voltammetry analysis of trace metals [13, 14]. Bismuth offers the closest behavior to mercury but with very low

toxicity [15]. Both in situ or ex situ bismuth film electrode (BiFE) [16–19] and bulk bismuth electrode (BiBE) [20–22] display a very attractive stripping behavior. Some concerns were also focused on tin film electrode because the position of tin and bismuth is catercorner in periodic table of the elements and tin has analogical character of bismuth [23–25]. Based on the bismuth and tin electrodes, the tin/bismuth film electrode, another type of electrode, has been suggested by in situ depositing tin and bismuth on a poly (p-aminobenzene sulfonic acid) coated glassy carbon electrode [26]. Compared with the traditional bismuth and tin film electrode, the electrode shows a higher stripping current response. However, the preparation of this kind of modified electrode is relatively complex.

Zinc is an essential micronutrient involved in human metabolism. More than 100 specific enzymes require it for their catalytic function. However, the insufficient or excess of essential elements in human body may cause many disorders and diseases [27]. The lack of zinc will cause a number of neuropathologies such as Alzheimer's disease (AD), amyotrophic lateral sclerosis (ALS), Parkinson's disease, hypoxia-ischemia and epilepsy while an excessive intake will result in a series of symptoms such as nausea, anaemia and lethargy [28–30]. The upper limit of zinc per day for adults is 40 mg set by Food and Nutrition Board of the International Zinc Nutrition Consultative Group (IZiNCG) and even lower for young children [31]. Hence, considerable interests have been focused on the determination of Zn^{2+} in many fields, especially in food control.

As a lead-free solder, commercial Sn-Bi alloy is a silvery white solid in normal atmospheric temperature and low hardness metal. With a low melting point, it is usually used in welding industry and manufacturing industry to manufacture all kinds of plastic and rubber mould. Until now, to our best knowledge, no studies have been reported the Sn-Bi alloy as electrode material to detect heavy metal. In this paper, commercial Sn-Bi alloy wire was used to fabricate Sn-Bi alloy electrode (SnBiE) for anodic stripping voltammetric determination of Zn^{2+} without any deoxygenating and electro-plating pre-treatment. Experimental parameters, including the pH values of the buffer solution, the accumulation potential and the accumulation time have been investigated in detail. Additionally, the practical application of this electrode has been carried out for determination of Zn^{2+} in wines and honeys.

Experimental

Reagents

Sn-Bi alloy wires (Sn:Bi, 42:58 wt%, 1 mm in diameter) were purchased from KAIT Electronic Material Co., Ltd, Guangzhou, China (http://www.kaitelectronic.com/Default.aspx). A 0.01 M stock standard of Zn^{2+} solution was prepared by dissolving zinc nitrate hexahydrate (Sinopharm Chemical Reagent Beijing Co., Ltd, China) (http://www.crc-bj.com/) into de-ionized water, acidified to pH 2.0 with nitric acid and then diluted to various concentrations of working solutions. 0.1 M acetate buffer solution (pH 5.0) prepared with sodium acetate and acetic acid was used as the supporting buffer solution for Zn^{2+} determination. All other chemicals were analytical reagents and used without further purification. Deionized water (18.2 MΩ cm specific resistance) obtained with a Pall Cascada laboratory water system (http://www.pall.com/default.asp) was used throughout.

Apparatus

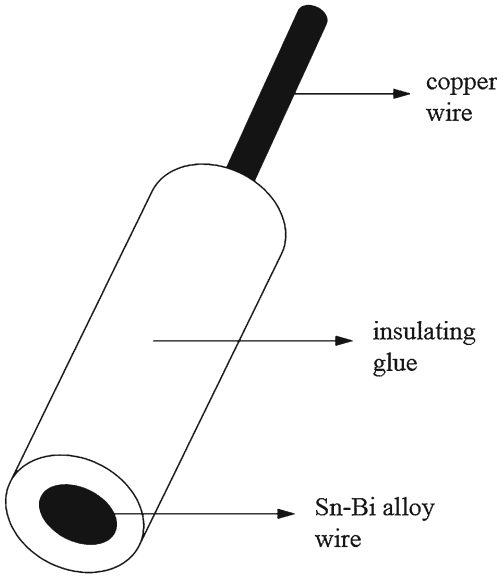
All electrochemical experiments were carried out in a conventional three-electrode cell controlled by a CHI 660D Electrochemical Work Station (CH Instruments, Inc) (http://chi.instrument.com.cn/). A Sn-Bi alloy wire electrode was used as working electrode. A platinum foil was applied as the counter electrode, and a saturated calomel electrode (SCE) served as the reference electrode. The pH measurements were performed at an E-201-C Model pH meter (Shanghai Leici Instrument Factory) (http://www.lei-ci.com/). Atomic absorption spectrometric (AAS) measurements were conducted with a GGX-900 atomic absorption spectrophotometer (Kechuang Haiguang Instrument Co., Ltd, China) (http://www.kchaiguang.com/). All electrochemical experiments were carried out at room temperature and non-deoxygenating condition.

Preparation of SnBiE

Sn-Bi alloy wire was first polished with metallographic abrasive paper and washed with deionized water. Then it was sonicated in alcohol and deionized water for 2 min, respectively. After that, it was contacted using a copper wire and sealed with an insulating glue to make a disc electrode (Scheme 1). Finally, the fabricated electrode was put into the oven to be dried at 60 °C for 1 h for the following experiments.

Analytical procedure

The analysis of Zn^{2+} was performed in 25 mL beaker containing 20 mL 0.1 M acetate buffer solution (pH 5.0) without removal of oxygen. It had two main steps including accumulation and stripping out. First, Zn^{2+} ions were reduced to zinc under −1.4 V for 60 s onto the SnBiE under stirring conditions. Then, the reduced zinc was oxidized at around −1.2 V during an anodic square wave potential sweep from −1.4 to −0.8 V. After each measurement, the



Scheme 1 Schematic diagram for the construction of the SnBiE

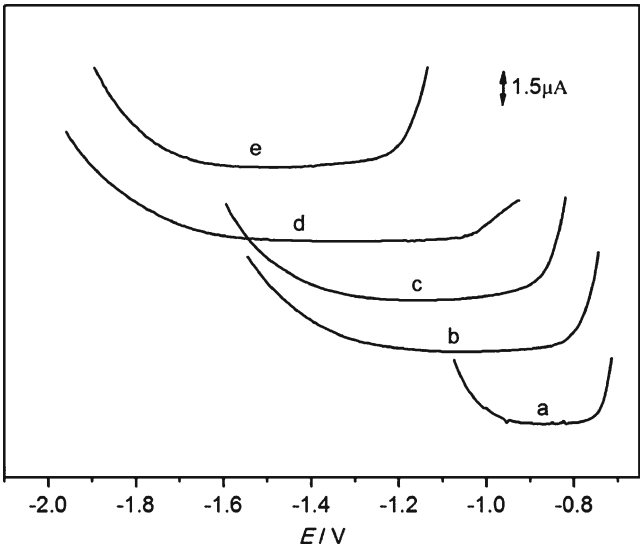


Fig. 1 Stripping voltammetric potential windows of the SnBiE in different electrolytes with different pH values: **a** 0.1 M hydrochloric acid, pH 0.96, **b** 0.1 M sodium acetate + 0.1 M hydrochloric acid, pH 3.00, **c** 0.1 M acetate buffer, pH 5.00, **d** 0.1 M sodium acetate, pH 7.97, **e** 0.1 M sodium hydroxide, pH 12.86. Increment potential of each step, 5 mV; pulse amplitude, 25 mV

electrode was cut to obtain a renewable surface and then directly used for the following experiment.

The parameters applied for the square wave anodic stripping voltammetry (SWASV) were as follows: accumulation potential, −1.4 V; accumulation time, 60 s; equilibration time, 4 s; increment potential of each step, 5 mV; pulse amplitude, 25 mV.

Sample preparation

Prior to analysis, wines and honeys purchased from local supermarket were accurately weighed and put into porcelain crucibles. They were heated in a muffle furnace at 200 °C with the door slightly ajar to allow the smoke to escape. Then, the temperature of furnace was set to 500 °C with the door closed for 8 h (more than 12 h for honeys) to ash. Subsequently, 6 M hydrochloric acid solution was added to dissolve the ash by gently boiling the solution. After cooling, the obtained sample solutions were adjusted to pH 5.0 with sodium hydroxide solution and then quantitatively transferred to the volumetric flasks.

Results and discussion

The potential window of the SnBiE

The applicable potential window of solid electrodes strongly depends on the inherent properties of the electrode materials and the pH of the electrolyte. The effect of pH values of different electrolyte on the accessible potential window of the SnBiE is presented in Fig. 1. At pH 0.96, 3.00, 5.00, 7.97 and 12.86, the potential windows extended from −0.75 to −1.01 V, −0.82 to −1.30 V, −0.90 to −1.35 V, −1.02 to −1.72 V and −1.22 to −1.75 V, respectively. The widest useful potential window was recorded in alkaline solution (pH 7.97, curve d). The cathodic limit of the potential window of the SnBiE was extended from −1.01 to −1.75 V with the pH form 0.96 to 12.86, which is mainly depended on the hydrogen evolution. Compared with the BiFE [32] and the BiBE [14], the overvoltage of the hydrogen evolution at the SnBiE is more negative. This is probably due to the existent of Sn. Sn and Bi are in catercorner position at the periodic table of chemical elements and form molten alloy easily, which probably provides an especial crystal plane to prohibit the generation of hydrogen. Considering that Sn is oxidized at more negative potential than Bi, the anodic limit of the potential window of the SnBiE is mainly limited by the oxidation potential of Sn. The produced Sn and Bi ions are easily hydrolyzed at higher pH values, which may facilitate and accelerate the oxidation process and result in the shift of the anodic potential limit. These situations are similar to that of BiBE [14].

Voltammetric behavior of the SnBiE

The voltammetric behaviors of SnBiE in 0.1 M acetate buffer solution (pH 5.0) were shown in Fig. 2. In the potential range from −1.4 to −0.7 V, SnBiE has a low background current and no other redox peak can be seen in the blank buffer solution. However, with the addition of Zn^{2+} solution, the oxidation peak of zinc at −1.119 V (i_{Pa}=2.8 μA) was observed at SnBiE during the positive potential scan, while the reduction peak occurred at

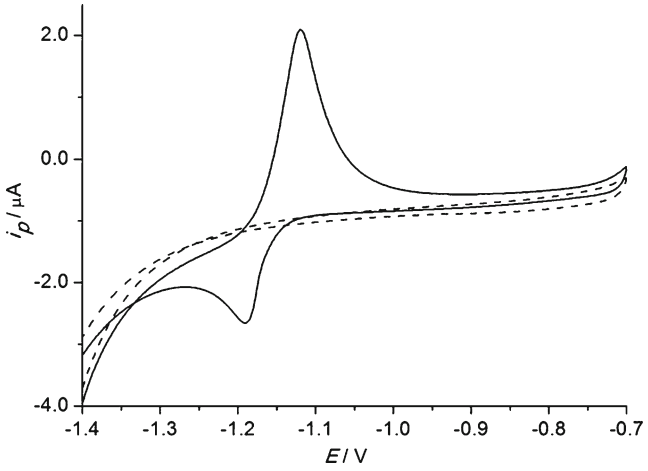


Fig. 2 Cyclic voltammograms of the SnBiE in 0.1 M acetate buffer solution (pH 5.0) without (*dash line*) and with (*solid line*) 0.4 mM Zn^{2+}. Scan rate, 50 mV·s^{-1}

−1.190 V (i_{Pc}=1.7μA) during the negative potential scan. The presented voltammetric behavior of the SnBiE demonstrates that the alloy electrode possesses relatively better interfacial electrochemical characteristics, which implies the possibility of direct voltammetric detection of Zn^{2+} in the appropriate potential region of the SnBiE.

ASV of Zn^{2+} at the SnBiE

According to the literature [33], at bare glassy carbon electrode (GCE), the stripping peak of zinc was not be observed. However, in 0.1 M acetate buffer solution (pH 5.0) containing the same concentration of Zn^{2+} (0.5 μM), there is an obvious stripping peak at −1.175 V at SnBiE (Fig. 3). In the

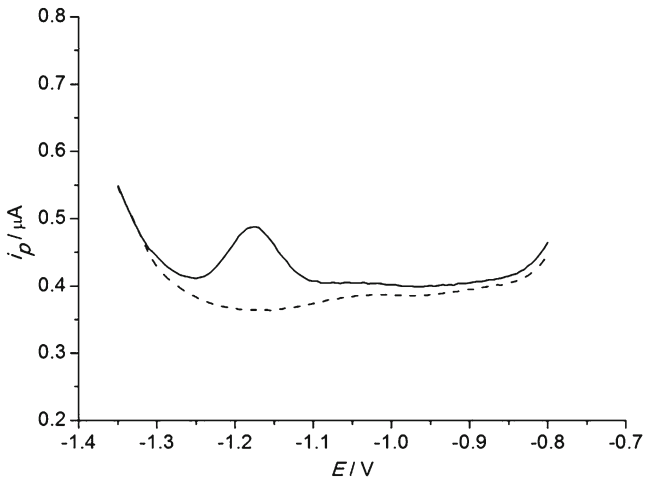


Fig. 3 SWASV obtained in 0.1 M acetate buffer solution (pH 5.0) without (*dash line*) and with (*solid line*) 0.5 μM Zn^{2+} at the SnBiE in the potential range of −1.35 to −0.8 V. Accumulation potential, −1.4 V; accumulation time, 60 s; equilibration time, 4 s; increment potential of each step, 5 mV; pulse amplitude, 25 mV

accumulation step of the ASV, the accumulated Zn^{2+} was reduced at −1.4 V and electrochemically deposited at the electrode surface. Then the deposited zinc was oxidized, i.e., electrochemically stripped off. Surface area of the electrode and its properties would affect the stripping efficiency. It is well known that Bi forms binary or multi-component "fusing" alloy with zinc, allowing its accumulation at the electrode surface [15]. Therefore, it can be inferred that the Sn-Bi alloy forms alloy with zinc, which results in better response to Zn^{2+} at SnBiE than GCE.

Optimization for determination of Zn^{2+} at the SnBiE

Effect of pH values of the buffer solution

The effect of several supporting electrolytes, such as phosphate buffer, citrate buffer, acetate buffer (NaAc-HAc, NaAc-HNO_3, NaAc-HCl) in a concentration of 0.1 M was evaluated for their suitability for Zn^{2+} determination at the SnBiE electrode. The electrochemical behaviors of Zn^{2+} varied little with different electrolytes. The relatively better current and peak shape of Zn^{2+} was found in acetate buffer solution. Furthermore, different concentrations of acetate buffer solution, in the range of 0.05–0.3 M, were tested at constant concentration of Zn^{2+} (5 μM). The best performance can be obtained in 0.1 M acetate buffer solution due to its relatively better current response. Therefore, 0.1 M acetate buffer solution was selected for further experiments.

Considering that the analyte of Zn^{2+} is hydrolyzed in the alkaline solution and Sn-Bi alloy can be dissolved in strong acidic solution, the pH values of the buffer solution are controlled in the moderately acidic environment. Figure 4a shows pH dependency on the stripping current of Zn^{2+} in 0.1 M acetate buffer solution. The maximum current was obtained at pH 5.0 and the stripping current decreased gradually with a lower pH value. This probably ascribed to the effect of hydrogen dissolving which caused high background current from the initial scan that partly overlapped the stripping peak current. Moreover, the response current decreased with the pH values above 5.0, which was due to a low background current but a lower response current simultaneously. Therefore, in this work, 0.1 M acetate buffer solution of pH 5.0 was selected as the optimum experimental condition.

Effect of accumulation potential

The effect of accumulation potential on the stripping peak current of 5 μM Zn^{2+} was examined over the potential range of −1.2 to −1.6 V. As shown in Fig. 4b, the peak current increased greatly with changing potential from −1.2 to −1.4 V, probably due to the fact that Zn^{2+} can be reduced

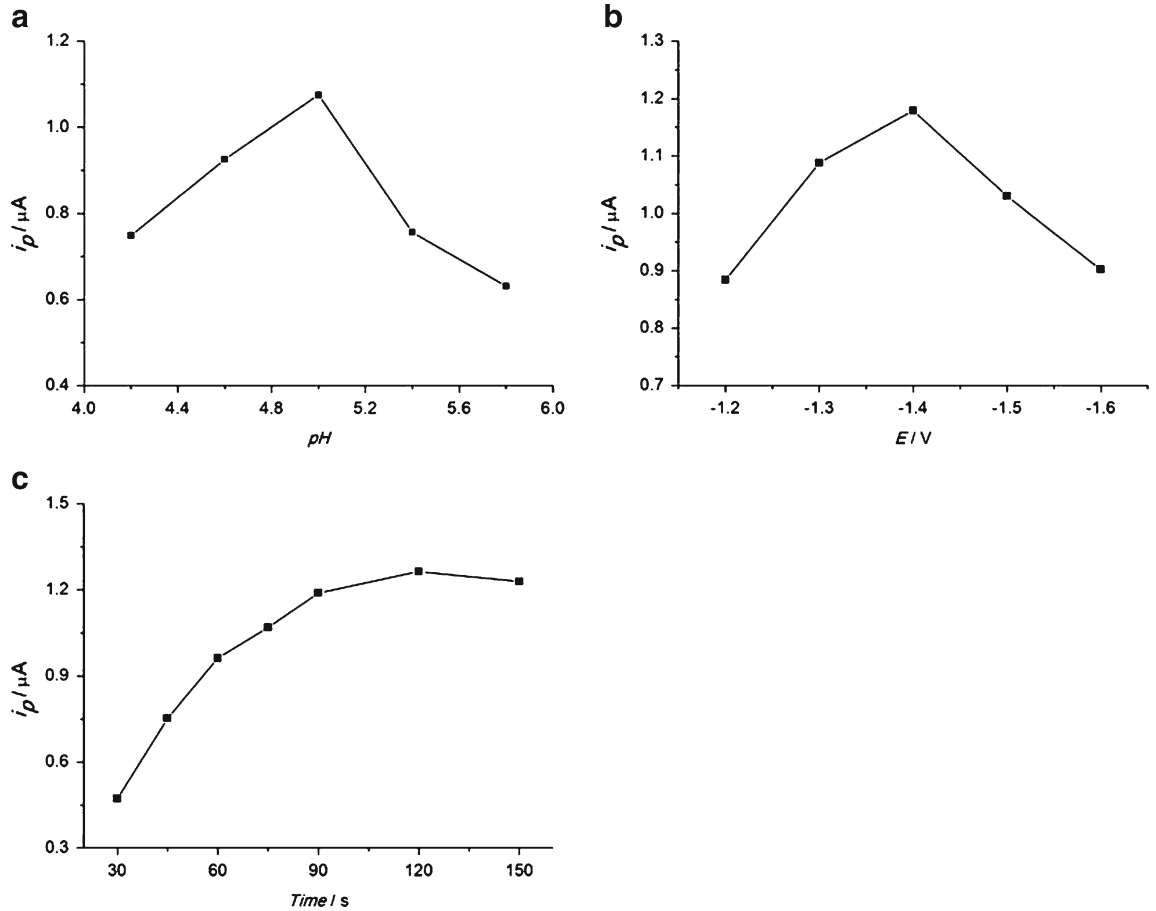


Fig. 4 Effect of (**a**) pH values of buffer solution, (**b**) accumulation potential and (**c**) accumulation time on the stripping peak current of 5 μM Zn^{2+} at the SnBiE; Other conditions are the same as in Fig. 3

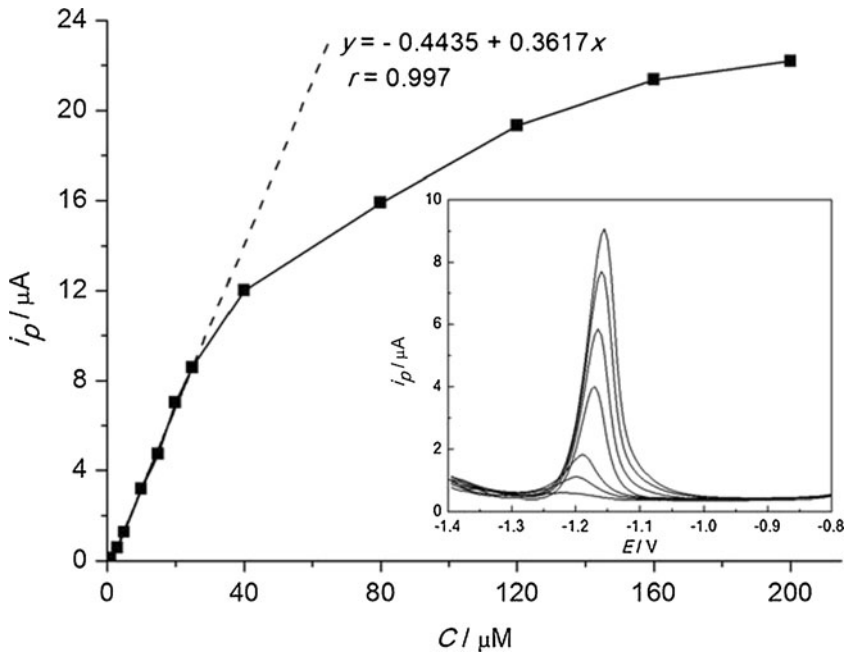


Fig. 5 Calibration curve of Zn^{2+} at the SnBiE. The concentrations of Zn^{2+} for the inset curves are 0.5, 3, 5, 10, 15, 20, and 25 μM from bottom to top, which are in the linear range. Other conditions are the same as in Fig. 3

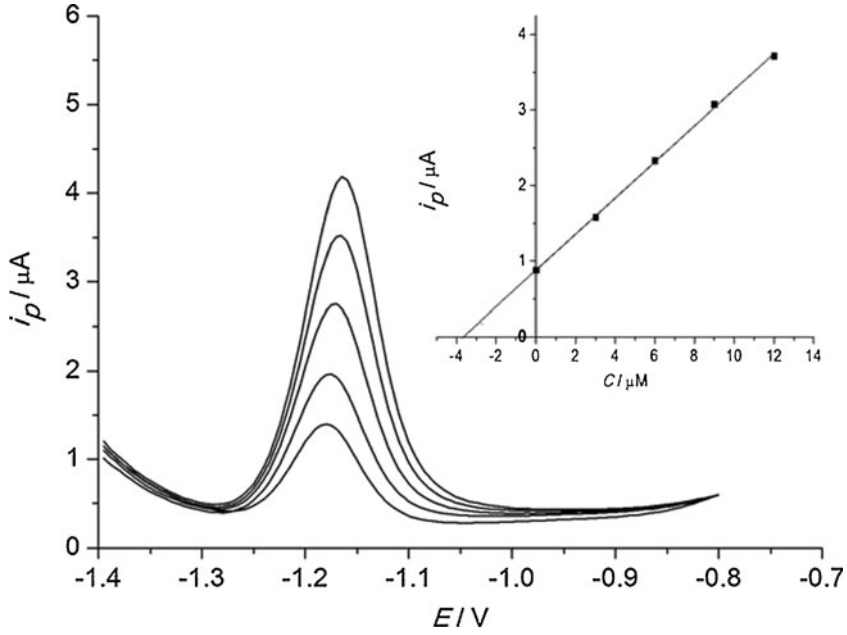


Fig. 6 SWASV of Zn^{2+} in Chinese yellow wine (sample 3) at the SnBiE by using standard addition method of standard additions of 0, 3, 6, 9 and 12 μM Zn^{2+} (from bottom to top). The inset is the fitted curve of Zn^{2+} measurement obtained from standard addition. Other conditions are the same as in Fig. 3

more efficiently at more negative deposition potentials. Experiments also showed that the peak current decreased at a potential more negative than −1.4 V and caused a relatively higher background current (not shown), which might be attributed to the co-hydrogen evolution at such negative potentials. Therefore, −1.4 V was adopted in the following experiments.

Effect of accumulation time

The effect of the accumulation time upon the peak current of Zn^{2+} was investigated. The corresponding results are shown in Fig. 4c. The variation of the peak current is depicted with the accumulation time for 5 μM Zn^{2+} in the range from 30 s to 150 s. The response current increased rapidly with the accumulation time up to 90 s, and then tended to increase slowly. Though a longer accumulation time can increase the amount of Zn^{2+} on the electrode surface and improve the sensitivity of practical determination, a short analysis time of 60 s is sufficient for determination of Zn^{2+} in wines and honeys. Additionally, for determination of lower concentration of Zn^{2+} in other matrixes, a prolonged accumulation time is recommended.

Calibration curve

The calibration curve for determination of Zn^{2+} at the SnBiE was established in Fig. 5 by using SWASV. In Fig. 5, for 60 s of accumulation, a proportional relationship between the oxidation peak current and the concentration of Zn^{2+} was obtained in the range of 0.5 μM to 25 μM by fitting the following regression equation:

$$y = -0.4435 + 0.3617x, r = 0.997$$

where y is the stripping peak current in μA, x is the concentration of Zn^{2+} in μM. The sensitivity of the SnBiE to Zn^{2+} is 0.36 μA/μM. The detection limit was given by the equation $C_L = 3s_{bl}/S$, where s_{bl} is the standard deviation of the blank measurements and S is the sensitivity of the calibration graph. The detection limit of Zn^{2+} for 60 s accumulation was calculated to be 0.05 μM, which is lower than that at Bi-modified carbon nanotube electrode [34], Hg-coated glassy carbon electrode [35], the polymer-coated Bi film electrode [36], while higher than that at the tin-film electrode (with 120 s of accumulation) [25] and the BiBE (with 180 s of accumulation) [20]. Although a lower detection limit can be obtained at the SnBiE with a prolonged accumulation time, a short analysis time is sufficient for determination of Zn^{2+} in real samples.

Moreover, due to the soft property of Sn-Bi alloy, the SnBiE is very easy to be cut to get a renewable surface after each measurement, which can effectively avoid the memory-effect of the solid electrodes. Therefore, it is potentially applied to the disposable measurement in food control, medical treatment and environmental monitoring.

Table 1 Comparison of the SnBiE and AAS for determination of Zn^{2+} in real samples

Sample	Detected by SnBiE	Detected by AAS
Red wine 1	0.79±0.03 (mg L^{-1})[a]	0.83 (mg L^{-1})
Red wine 2	1.40±0.02 (mg L^{-1})	1.42 (mg L^{-1})
Chinese yellow wine 1	4.86±0.11 (mg L^{-1})	4.80 (mg L^{-1})
Chinese yellow wine 2	3.15±0.15 (mg L^{-1})	3.10 (mg L^{-1})
Honey 1	0.80±0.02 (μg g^{-1})	0.83 (μg g^{-1})
Honey 2	0.75±0.03 (μg g^{-1})	0.78 (μg g^{-1})

[a] average value of three determinations ± standard deviation

The reproducibility of eight SnBiEs and one SnBiE with nine measurements for 5 μM of Zn^{2+} by using this refresh method were estimated. The results reveal that the SnBiE has satisfactory reproducibility and repeatability with relative standard deviation of 6.9% and 3.0%, respectively.

Study of interferences

The effect of possible interfering species was studied by analyzing a standard solution (5 μM Zn^{2+}) to which amounts of interfering ions were added under the optimized conditions. Interfering ions were added at different concentrations (max. 2,000 fold) higher than the concentration of Zn^{2+} until they produced a change in height of peak current of less than 5% of the initial height. The results show that more than a 2000-fold excess of Na^{+}, K^{+}, NO_3^{-} and Cl^{-}, less than a 1500-fold excess of Ca^{2+}, Mg^{2+} and SO_4^{2-}, 100-fold As^{3+}, 80-fold Cd^{2+}, 40-fold S^{2-}, 30-fold Pb^{2+} and 20-fold Sn^{2+} have no influence on the peak current of 5 μM Zn^{2+}. The Sb^{3+} can not stay stable in the supporting buffer solution because it was hydrolyzed immediately when added to the cell. The monitored Zn^{2+} voltammetric peak current was changed with a quantity of As^{3+}, Cd^{2+}, Pb^{2+} or Sn^{2+} added to the solution, which probably due to the strong competition effect with Zn^{2+} at the limited active sites of the electrode surface in the deposition procedure. For S^{2-}, it may form a deposit of SnS on the electrode surface during the stripping procedure and result in a deterioration of the electrode. Additionally, 5-fold of Bi^{3+} was found to cause 13% increase of the peak current of Zn^{2+} for it is probably formed a bismuth film on the SnBiE surface. 1-fold of Ag^{+} was found to cause 70% decrease of the peak current of Zn^{2+} which was due to the formation of Sn-Ag binary alloy [37]. 1-fold of Cu^{2+} was found to cause 75% decrease of the peak current of Zn^{2+} which was probably attributed to the formation of the intermetallic compound between Cu and zinc deposited in the electrode [15, 33]. However, the effect of interferences for real samples analysis was eliminated effectively by using the standard addition method (see below) or using masking reagents.

Real sample analysis

To demonstrate the performance of the novel SnBiE in real sample analysis, the contents of Zn^{2+} in wines and honeys were analyzed by using the standard addition method because the content of zinc in local wines or honeys is larger than that of Cu. Figure 6 shows the SWASV responses to Chinese yellow wine (sample 3) at SnBiE. The voltammetric peak current of Zn^{2+} in Chinese yellow wine can be detected at SnBiE, indicating the possibility of successful determination of Zn^{2+} in real samples by using standard addition method. The concentration of Zn^{2+} in real sample can be calculated from fitted curve (inset of Fig. 6). These results show that the SnBiE exhibits high sensitivity and good selectivity for the determination of Zn^{2+} under the optimum experimental conditions. Additionally, in order to illustrate its accuracy in practical analysis, the comparison was carried out between this novel SnBiE and atomic absorption spectroscopy (AAS) for detection of Zn^{2+} in real food samples. As can be seen from Table 1, the results obtained from the SnBiE are in accordance well with those detected by AAS, which indicated the capability of the SnBiE for determination of Zn^{2+} in real samples.

Conclusions

Sn-Bi alloy as a novel solid electrode material had been used to fabricate solid electrode for sensitive determination of Zn^{2+} in wines and honeys. The electrode is easy fabrication, low cost and no requirement of any pre-treatment modification prior to the analysis or cleaning step after each measurement. Moreover, it exhibits a high hydrogen overvoltage. Experimental parameters were optimized, such as pH values of the buffer solution, accumulation potential and accumulation time. Under optimized experimental conditions, excellent linear dynamic range (0.5–25 μM) with a correlation coefficient of 0.997 and a detection limit of 0.05 μM with 60 s of accumulation was obtained. Wines and honeys as real samples were determined and the results obtained from the suggested SnBiE were in good agreement with by AAS. Further work will be carried out by our laboratory to establish a disposable sensor based on the promising electrode to determine zinc and other heavy metals in such as food control, medical treatment or on site environmental monitoring applications.

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