Int. J. Electrochem. Sci., 11 **(2016)** 1907 - 1917

International Journal of
ELECTROCHEMICAL
SCIENCE
www.electrochemsci.org

Screen-printed Carbon Electrode Modified with Commercial Multilayer Graphene for Lead Detection in Soybean Sauces by Differential Pulse Stripping Voltammetry

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Received: 19 November 2015 / *Accepted:* 16 November 2015 / *Published:* 1 February 2016

A novel method to detect Pb^{2+} in soybean sauces was proposed by modifying commercially provided multilayer graphene on a disposable screen-printed carbon electrode to achieve increased electrochemical performance. Raman spectroscopy presented the low fluorescence background of graphene and revealed that the defects of sp^2 graphite were improved by graphene. The evident difference between the reduction and oxidation peaks of $K_3Fe(CN)_6$ was observed on the graphene modified screen-printed carbon electrode and could be compared with the electrochemical performance of a glass carbon electrode. To reuse the modified electrode, a new regeneration method was developed according to the largest stability constant of NaOH with Pb^{2+}. The key experimental parameters related to the fabrication of the electrode and the voltammetric measurements were optimized based on stripping signals where peak currents increased linearly with the Pb^{2+} concentration within the range of 5.0×10^{-7} and 5.0×10^{-6} mol/L by the standard addition method. The concentration of the soybean sauce sample was calculated to be 1.6×10^{-7} mol/L (0.032 mg/L) by extrapolation; by comparison, a concentration of 0.03576 mg/L was calculated through inductively coupled plasma mass spectrometry. Thus, the proposed electrode was demonstrated to be reliable and effective for detecting Pb^{2+} and could be further developed and combined with a palmtop electrochemical instrument to achieve in-situ detection.

Keywords: Graphene, Pb^{2+}, screen-printed carbon electrode, soybean sauce, stripping voltammetry

1. INTRODUCTION

Currently, graphite furnace atomic absorption, hydride atomic fluorescence spectrometry, flame atomic absorption spectrometry, the dithizone colorimetric method, and single-sweep polarography by Hg electrodes are the standard methods[1-3] used to detect lead in food. However, the main disadvantages of precise atomic spectrometry include the high cost of equipment and the high concentration of salinity in the soybean sauce matrices[4], which can cause significant background interference.

Electrochemical techniques employ significantly less expensive equipment. Hg is traditionally used as an electrode material for heavy metal detection because of its beneficial analytical properties[5]. However, a number of chemically modified electrodes have been developed to replace Hg electrodes because of its toxic components; these alternative techniques include layering ionic liquid[6-7], electrical polymer[8-10], nanomaterials[11-12], and graphene oxide[13-14] on different types of electrodes, including glass carbon electrodes[15-16], silver electrodes[17-18], gold electrodes[19-20], carbon paste electrodes[21-23] , and so on. Among these modified electrodes, bismuth film has been proved to form "fused" multicomponent alloys with heavy metal and present the advanced electrochemical performance to become an alternative electrode material in stripping analysis[24-26].

Screen-printed electrodes (SPEs), which are good for a single use, inexpensive, and mass-produced, are traditionally produced for blood glucose detection in combination with a portable meter[27]. SPEs are used in a variety of applications in environmental, industrial, and clinical analyses[28]. The working, counter, and reference electrodes could be integrated on one chip [29] through printing, which is beneficial for micro volume analysis. Thus far, screen-printed gold electrodes have been reported for on-site fuel electroanalysis of metal by stripping voltammetry[30] and screen-printed carbon electrodes(SPCEs) coated with bismuth film has also to be developed for stripping analysis of metal[24]. However, SPCEs do not usually exhibit ideal electrochemical performance because the carbon ink includes a number of organic solvents that could cause defects in graphite, which will block the establishment of the detection method with good repeatability and lower detection limit.

In this paper, SPCEs were modified with commercial multilayer graphene after simple pretreatment to improve their electrochemical performance. The cyclic volummetry experiments of $K_3[Fe(CN)_6]$ on the graphene modified SPCEs (graphene-SPCEs) has demonstrated that these modified SPCEs exhibited good electrochemical performance. The graphene-SPCEs combing with 5 μmol/L Bi^{3+} could detect lead concentrations as low as approximately 10^{-7} mol/L Pb^{2+} in soybean sauces[5]. Thus, modifying the carbon surface by graphene is a potentially attractive solution to the problem of SPCE which has not ideal electrochemical performance because this technique immobilizes a more organized sp^2 graphite on the SPCE[31].Several electrode-regeneration methods were also investigated. Reusing electrodes can increase their reparability because signal variations from different electrodes can be disregarded and the cost of testing may be further reduced. The main objective of this paper is to develop an effective method to detect Pb^{2+} in real samples using an graphene-SPCE that could be further developed for in-situ detection when combined with palmtop electrochemical equipment.

2. EXPERIMENTAL

2.1. Materials and Reagents

CH_3COOH, CH_3COONa, NaH_2PO_4, Na_2HPO_4, NaOH, KCl, NaCl, and HCl were purchased from Sinopharm Chemical Reagent Co.,Ltd. Lead and bismuth standard solutions (1 mg/L, in 1.0 mol/L HNO_3), Tris(hydroxymethyl)aminomethane, and sodium polyacrylate (Mw, ~5100) were obtained from Aladdin Industrial Corporation. Graphene paste was purchased from Suzhou Hengqiu Graphene Co.,Ltd. CH_3COOH–CH_3COONa buffer solution (0.2 mol/L; pH 4.2, 4.4, 4.6, 4.8, 5.0, 5.2, 5.4, 5.6), phosphorus buffer solution (0.2 mol/L; pH 5.8, 6.2, 6.6), and Tris–HCl(0.05 mol/L; pH 7.2, 7.6, 8.0, 8.4, 8.8) were prepared. All of other chemical reagents used were of analytical grade and applied without further purification. All solutions were prepared using ultrapure water (18.2 MΩ, Human, Korea)

2.2. Apparatus and Measurements

Electrochemical experiments were carried out at room temperature on a CHI 660 electrochemical station (CHI Instruments Inc., USA).All electrochemical experiments were performed with the SPCE, including carbon working, carbon counter, and Ag/AgCl reference electrodes (Ningbo Mxense Bio-Tech Co., Ltd.). The buffers were used as electrolytes in all experiments.Raman spectra were recorded using a LabRAM HR UV 800 Laser Micro Raman Spectrometer (Horiba, Japan). A laser excitation (He–Ne laser) of 632 nm was used to irradiate the samples and the spectra were then recorded by scanning from 300 cm^{-1} – 3800 cm^{-1} with integration time of 10 s and double integration. Data of real samples were determined and compared using an ELAN DRC-e inductively coupled plasma mass spectrometer (ICP-MS, Perkin Elmer).

2.3. Electrode Modification

The graphene–SPCE electrode was prepared according to the following procedures. Before modification, commercial multilayer graphene paste was first subjected to centrifugation at 13000 rpm for 10 min. Organic solvents were removed and rinsed using alcohol and water successively at 13000 rpm for 10 min. The rinsed graphene powder was added to 0.05% sodium polyacrylate to form a homogeneous dispersion under ultrasonic conditions. A 2 μL suspension was dropped onto SPCE and allowed to dry under ambient conditions overnight. The modified electrodes were rinsed with doubly distilled water two or three times to eliminate non-firmly adsorbed graphene.

Cyclic Voltammetric experiments of bare SPCE and graphene–SPCE were performed in 2 mmol/L $K_3Fe(CN)_6$ from -0.3 to 0.8 V at a scan rate of $0.1Vs^{-1}$.

2.4. Soybean Sauces Sample Preparation

The soybean sauce samples were purchased from a local market. A 5 mL sample was poured into a 100 mL Erlenmeyer flask bottle followed by 5 mL of concentrated HNO_3 and 2 mL of H_2O_2(30 % w/w). The solution was heated until most of the acid had evaporated, and the solution was cooled down to room temperature. Then, 10 mL of water was added to the flask, and the mixture was heated to boiling until nearly dry. Finally, the solution was transferred to a 25 mL volumetric flask bottle with buffer solution.

2.5. Lead Preconcentration and Differential Pulse Stripping Voltammetry

The graphene–SPCE was immersed in a home-made electrochemical cell, containing 5 μmol/L Bi^{3+} in 30 mL of buffer solution. Lead standard working solution or soybean sauce samples containing standard solutions were added to the electrochemical cell, and the electrode was preconcentrated at a potential of –1.2 V for 450 s under stirring. After allowing 15 s of standing, differential pulse stripping voltammetric measurements were performed from –1.2 V to 0 V and the electrical current of lead was recorded.

To reuse the graphene–SPCEs, the electrodes were rinsed by holding the electrode at + 0.3 V for 140 s to remove the residual lead and bismuth. The electrode was then transferred to 0.1 mol/L NaOH and stirred for 1 min. Finally, the regenerated electrode was rinsed by ultrapure water.

3. RESULTS AND DISSCUSSION

3.1. Characterization of Bare SPCE and Graphene-SPCE by Raman Spectroscopy

Figure 1 shows the Raman spectra of SPCE before (a) and after(b) modification with graphene. The characteristic peaks of sp^2 graphite material appear in both electrodes, including the D band at 1350 cm^{-1}, the G band at 1582 cm^{-1}, and the 2D band at 2700 cm^{-1}. However, the D band is more evident than the G and 2D bands in Figure 1(a),whereas the G band is more obvious than the D and 2D bands in Figure 1(b). The G mode (assigned to “in plane” displacement of the carbon strongly coupled to the hexagonal sheets) shows broadening, and the D mode at 1357 cm^{-1} appears when the graphite structure is disrupted[32]. Thus, the graphene–SPCE is more organized than the original SPCE. The $I_G/I_{2D}>1$ and broaden 2D band[Figure 1(b)] show that the graphene was multilayered. The modified graphene–SPCE [Figure 1(b)]reveals low background signals, whereas the carbon ink [Figure 1(a)] demonstrates a large fluorescence background, which originates from the adhesive organic solvents.

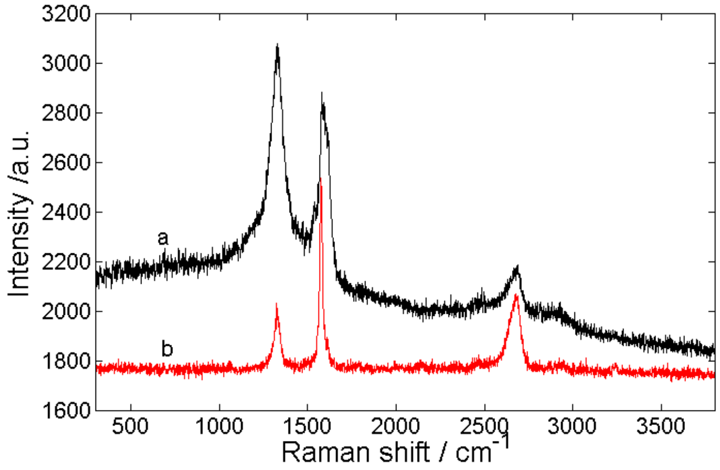

Figure 1. Raman spectra of bare SPCE (a) and graphene-SPCE(b)

3.2. Cyclic Voltammetric and Linear Sweep Voltammetric Behaviour of Graphene-SPCE

Figure 2 presents the cyclic voltammograms of glass carbon [Figure 2(a)], bare SPCE [Figure 2(b)], and graphene–SPCE electrode [Figure 2(c)] in 2 mmol/L $K_3Fe(CN)_6$ at a scan rate of $0.1Vs^{-1}$. The bare SPCE [Figure 2(b)] without electrochemical pretreatment exhibits a weak peak, which demonstrates electro inactivity in the potential window studied. The graphene–SPCE electrode[Figure 2(c)] reveals evident reduction and oxidation peaks of $K_3Fe(CN)_6$; the difference between the reduction and oxidation peaks(ΔE_p) is 89 mV. The ΔE_p of the glass carbon electrode is 170 mV[Figure 2(a)].Results thus far illustrate that the electrochemical behaviour of graphene–SPCE [Figure 2(c)] is better than that of the glass carbon electrode [Figure 2(a)]. The repeatability (RSD=7.6%[n=5]) of the graphene–SPCEs is further determined by comparing the reduction currents of five different electrodes in 2 mmol/L $K_3Fe(CN)_6$.

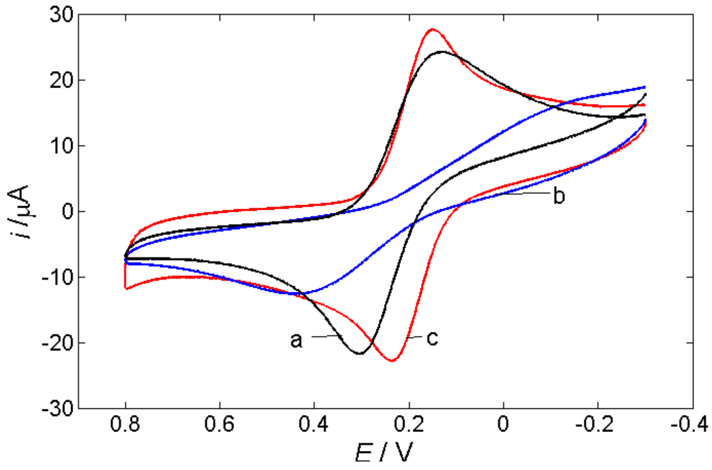

Figure 2. Cyclic voltammograms of glass carbon vs $Hg/HgCl_2$ (a), SPCE(b) and graphene-SPCE(c) vs Ag/AgCl in 2 mmol/L $K_3Fe(CN)_6$ from -0.3 to 0.8 V at a scan rate of $0.1Vs^{-1}$

Figure 3 shows the stripping performance of 10^{-5} mol/L Pb^{2+} on glass carbon(a) and graphene-SPCE(b) in pH 4.8 $CH_3COOH–CH_3COONa$ buffer solution. As shown in the figure, the oxidation of Pb^{2+} both yielded a sharp and well-defined peak on these two electrodes at high concentration (10^{-5} mol/L). Compared with glass carbon, the oxidation peak of Pb^{2+} was stronger on graphene-SPCE. This phenomenon can be attributed to the fact that the defect of graphite has been improved by graphene and make it present a good electrochemical performance. In addition, it could be inferred that the porous graphene will absorb more Pb ions to contribute the large stripping signal. Although the graphene-SPCE present a good electrochemical performance, the detection limit of Pb^{2+} can't be lower than 10^{-6} mol/L. Therefore, 5 μmol/L Bi^{3+} was introduced here[6] and it was added with Pb^{2+} to form "fused" alloys to realized the effective detection of Pb^{2+} in soybean sauces.

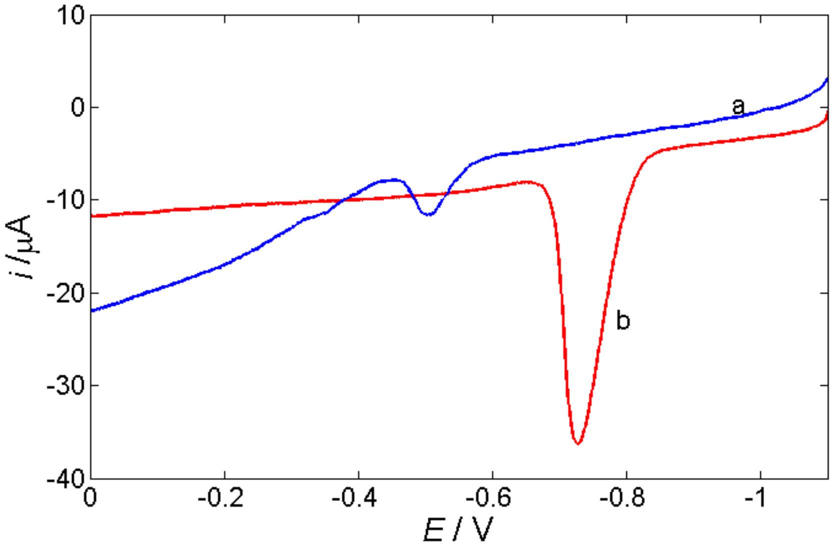

Figure 3. Linear sweep voltammograms of 10^{-5} mol/L Pb^{2+} solution on glass carbon vs $Hg/HgCl_2$ (a) and graphene-SPCE vs Ag/AgCl(b) in pH 4.8 $CH_3COOH–CH_3COONa$ buffer solution

3.3. Regenerated Method of Graphene-SPCEs

To reuse the graphene–SPCEs after stripping[Figure 3(b)], the electrodes were first regenerated at +0.3 V from 30 to 140 s. Figure 4(a) shows that most of the lead and bismuth are removed by this step. However, some residues remain on the electrode surface which was even regenerated about 140 s because weak oxidation peak appears at –0.4 V when the electrode is used to detect the buffer solution. Reports indicate that some reagents could bond with lead ions to form a stable complex, such as NaOH ($K_3=10^{13.3}$), tartaric acid ($K_3=10^{4.7}$), and thiosulfuric acid ($K_3=10^{6.4}$)[33]. NaOH, which presents a rather large stability constant, was thus selected as a cleaning reagent. Figure 4(b) indicates that the electrode exhibits no interference peaks after regeneration treatment with NaOH, thus demonstrating that the electrode could be used for preconcentration once more.

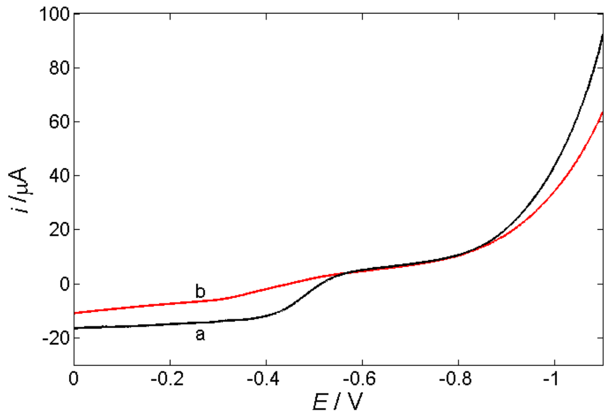

Figure 4. Linear sweep voltammograms of the preconcentrated graphene-SPCE before (a) and after (b) rinsed by NaOH in pH 4.8 $CH_3COOH–CH_3COONa$ buffer solution

3.4. Influence of Electrolytes and pHs

Figure 5 shows that the peak current values of Pb^{2+} are strongly related to the electrolytes and detection pHs. The peak currents are weak in $CH_3COOH–CH_3COONa$ buffer solution (0.2 mol/L, pH 4.2,4.4,4.6,4.8,5.0,5.2,5.4,5.6) compared with those in the phosphorus buffer solution (0.2 mol/L, pH 5.8, 6.2, 6.6) and Tris–HCl buffer solution (0.05 mol/L; pH 7.2, 7.6, 8.0, 8.4, 8.8). The strongest current appears at pH 8.4 in the Tris–HCl buffer solution. Cl ions in Tris–HCl buffer solution may be beneficial to the current signals of Pb^{2+}[15]. $PbCl_2$ and $Pb(OH)_2$ precipitate because the solubility products (K_{sp}) of $PbCl_2$ and $Pb(OH)_2$ are 1.6×10^{-5} and 1.2×10^{-15}, respectively. However, the concentration of Cl ions is approximately 2.92×10^{-3} mol/L, while that of lead ions is less than 5×10^{-6} mol/L. Thus, $PbCl_2$ does not form and trace $Pb(OH)_2$ is dissolved with the reduction of lead ions at –1.2 V. These findings support the supposition that Tris–HCl buffer and pH 8.4 are the best electrolyte and pH level, respectively.

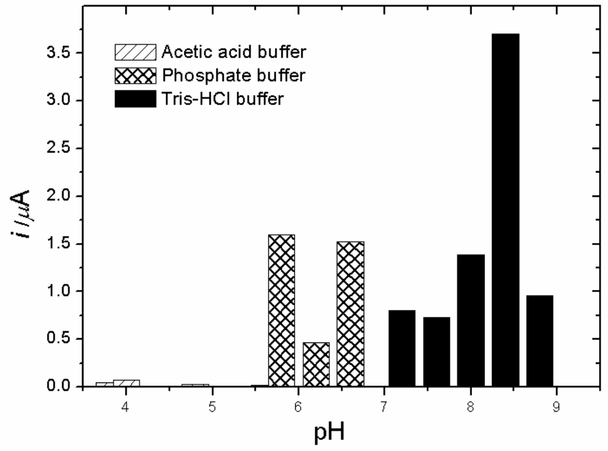

Figure 5. The peak current values of Pb^{2+} at different electrolytes and pHs: acetic acid buffer(0.2 mol/L, pH 4.2,4.4,4.6,4.8,5.0,5.2,5.4,5.6), phosphate buffer(0.2 mol/L, pH 5.8, 6.2, 6.6), tris-HCl buffer(0.05 mol/L; pH 7.2, 7.6, 8.0, 8.4, 8.8)

3.5. The Deposition Time and Potential

The deposition conditions were further investigated at 100, 200, 300, 400, 450, and 500 s with 1.0×10^{-6} mol/L lead ions. Figure 6(A) shows that the largest signals appear at 450 s, thus indicating that the current signal at 450 s is saturated. Deposition potentials were further investigated from –0.9 V to –1.3 V, and results reveal that –1.2 V is the best potential[Figure 6(B)].

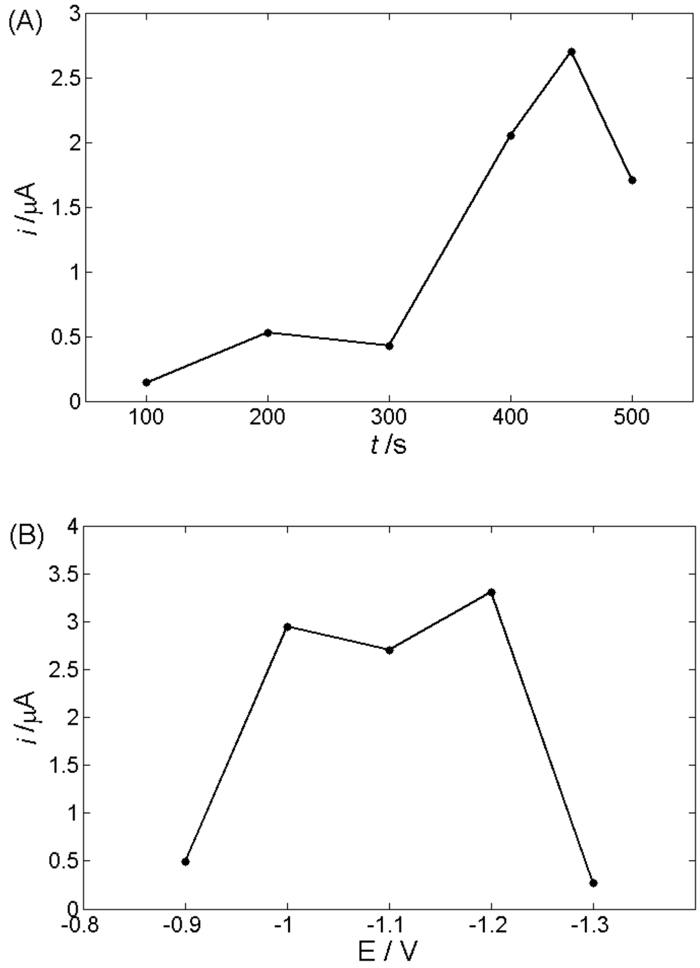

Figure 6. (A) The values of peak current at different times(100, 200, 300, 400, 450, and 500 s), and (B) different potentials(–0.9, -1.0, -1.1, -1.2, –1.3 V)

3.6. Evaluation of Mutual Inferences

The interference of foreign ions was investigated in a solution containing lead ions at a concentration of 1.0×10^{-6} mol/L (Figure 7). The influence of added interfering ions (Fe^{2+}, Zn^{2+}, Cu^{2+}, K^+, Na^+, Mn^{2+}, 100 times the Pb^{2+} concentration) on the lead ion peak currents is presented in Figure 6. The results indicate that Fe^{2+}, Zn^{2+}, Cu^{2+}, K^+, and Mn^{2+} exhibit weak inhibition of the Pb^{2+} signal. By contrast, Na^+ tends to increase the Pb^{2+} signal.

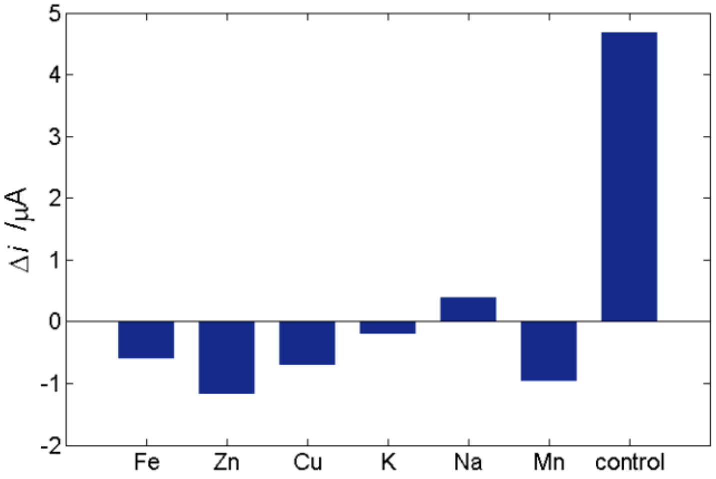

Figure 7. Relative signals for Pb^{2+} (1×10^{-6} mol/L) in the presence and absence of foreign ions (Fe^{2+}, Zn^{2+}, Cu^{2+}, K^{+}, Na^{+}, Mn^{2+})

3.7. Standard Addition Method and Real Sample Detection

Different concentrations of Pb^{2+} standard solutions (5.0×10^{-7}, 1.0×10^{-6}, 2.0×10^{-6}, 3.0×10^{-6}, 4.0×10^{-6}, 5.0×10^{-6} mol/L) were added to soybean sauce samples. Figure 8 shows that the concentrations of lead exhibits a linear relationship with the peak currents at concentrations between 5.0×10^{-7} and 5.0×10^{-6} mol/L ($y=3.74\times10^{6}x+0.61$, $R^2=0.9435$).

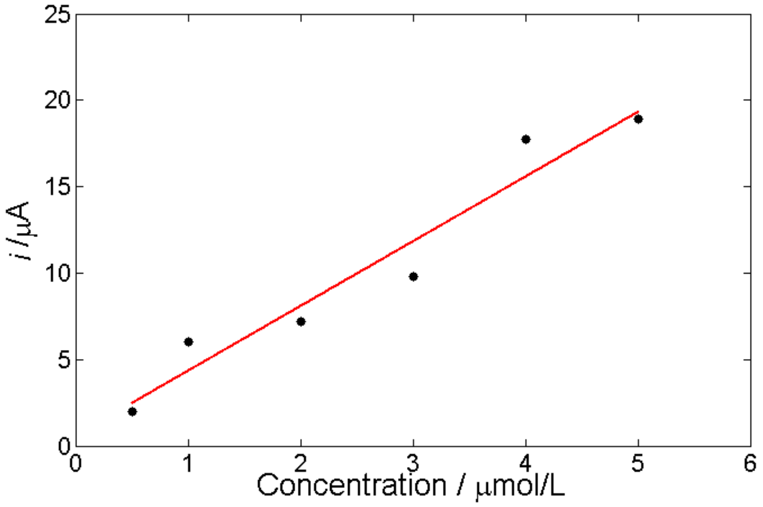

Figure 8. Calibration curve of soybean samples containing Pb^{2+} standard solutions (5.0×10^{-7}, 1.0×10^{-6}, 2.0×10^{-6}, 3.0×10^{-6}, 4.0×10^{-6}, 5.0×10^{-6} mol/L) on graphene-SPCE by standard addition method

The concentration of soybean sauce sample is calculated to be 1.6×10^{-7} mol/L (0.032 mg/L) by the extrapolation method that means the standard curve was extended to x-axis and the absolute value of x-axis is the concentration of soybean sauce sample. To test the accuracy of the new method, the concentrations of metal ions in the same soybean sauce sample was also determined by ICP–MS[34]. The ICP–MS result is 0.03576 mg/L. Comparison of different modified electrodes for determination of Pb^{2+}, most were developed for water samples[6,12,35-37] which have lower background inferences, although among these have low detection limit even to 0.1 μg/L[36]. And comparison of some methods developed for real samples (rice(>0.11 mg/L)[38], fuel(>2 mg/L)[30]) which have complex background can't satisfy the low detection concentration of 0.03 mg/L in soybean sauce. Therefore, the proposed graphene-SPCE is effective and satisfied for determination of Pb^{2+} in soybean sauce.

4.CONCLUSION

In this work, SPCEs were used for the first time as a kind of electrode material for Pb^{2+} detection in soybean sauces. The electrochemical performance of the SPCEs was improved by graphene, and the optimum detection conditions were investigated to achieve trace detection of Pb^{2+}. NaOH was considered to be a good regeneration reagent to rinse the lead and bismuth from the electrodes. The proposed electrode was successfully applied to the analysis of soybean sauce, and the calculated concentration was similar to the result obtained through ICP–MS.

ACKNOWLEDGEMENTS
The Analysis and Measurement Foundation of Zhejiang Province (2015C37068) and Foundation of Zhejiang Educational Committee (Y201432432) supported this work.

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