

# SnO<sub>2</sub>/Reduced Graphene Oxide Nanocomposite for the Simultaneous Electrochemical Detection of Cadmium(II), Lead(II), Copper(II), and Mercury(II): An Interesting Favorable Mutual Interference

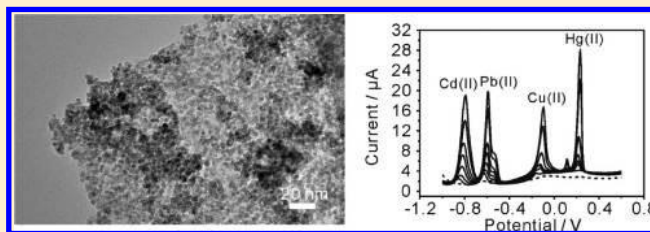
Yan Wei,<sup>†,‡,§</sup> Chao Gao,<sup>†</sup> Fan-Li Meng,<sup>†</sup> Hui-Hua Li,<sup>†</sup> Lun Wang,<sup>\*,§</sup> Jin-Huai Liu,<sup>\*,†</sup> and Xing-Jiu Huang<sup>\*,†</sup>

<sup>†</sup>Institute of Intelligent Machines, Chinese Academy of Sciences, Hefei 230031, People's Republic of China

<sup>‡</sup>Department of Chemistry, Wannan Medical College, Wuhu 241002, People's Republic of China

<sup>§</sup>College of Chemistry and Materials Science, Anhui Normal University, Wuhu 241000, People's Republic of China

A well-known gas sensing material SnO<sub>2</sub> in combination with reduced graphene oxide was used in heavy metal ions detection for the first time. This work reports the detailed study on the SnO<sub>2</sub>/reduced graphene oxide nanocomposite modified glass carbon electrode, which could be used for the simultaneous and selective electrochemical detection of ultratrace Cd(II), Pb(II), Cu(II), and Hg(II) in drinking water. The SnO<sub>2</sub>/reduced graphene oxide nanocomposite electrode was characterized voltammetrically using redox couples (Fe(CN)<sub>6</sub><sup>3-/4-</sup>), complemented with electrochemical impedance spectroscopy (EIS). Square wave anodic stripping voltammetry (SWASV) has been used for the detection of Cd(II), Pb(II), Cu(II), and Hg(II). The detection limit (3σ method) of the SnO<sub>2</sub>/reduced graphene oxide nanocomposite modified GCE toward Cd(II), Pb(II), Cu(II) and Hg(II) is 1.015 × 10<sup>-10</sup> M, 1.839 × 10<sup>-10</sup> M, 2.269 × 10<sup>-10</sup> M, and 2.789 × 10<sup>-10</sup> M, respectively, which is very well below the guideline value given by the World Health Organization. The chemical and electrochemical parameters that exert influence on deposition and stripping of metal ions, such as supporting electrolytes, pH value, deposition potential, and deposition time, were carefully studied. An interesting phenomenon of mutual interference was observed. Most importantly, we pose a potential for the use of gas sensing material in heavy metal ions detection.



## 1. INTRODUCTION

Heavy metals are extremely harmful pollutants in the biosphere due to their toxicity and even trace amounts of them pose a detrimental risk to human health. Thus, it is very important to search for a rapid, sensitive, and simple analytical method for the detection and monitoring of these environmental pollutants in water. There are already many mature methods to analyze heavy metal ions, such as atomic absorption spectroscopy, inductively coupled plasma atomic emission spectrometry, and inductively coupled plasma mass spectrometry. However, these spectrometric methods are expensive and not suitable for in situ analysis due to the ponderous and complicated instruments. On the contrary, the electrochemical method, as an alternative to these spectroscopic techniques, has been accepted as an efficient method to detect heavy metal ions due to their excellent sensitivity, short analysis time, portability, and low cost. Among all of the electrochemical methods, electrochemical stripping voltammetric analysis provides a powerful tool for the determination of metal ions, which possess high-sensitivity and can simultaneously analyze several heavy metal ions.<sup>1–5</sup> The use of the chemical modified electrodes tremendously improves the efficiency of accumulating target analytes and has been developed as a fascinating and effective way for the anodic stripping voltammetry determination of heavy metal ions. The nanostructured materials are extremely attractive to modify electrodes

for electrochemically detecting heavy metal ions due to their unique electronic, chemical, thermal, and mechanical properties in comparison with conventional materials.<sup>6</sup>

Graphene, as a “star” material, has been developed as an advanced nanoelectrocatalyst for constructing electrochemical sensors owing to its extraordinary electronic transport properties, large surface area, and high electrocatalytic activities.<sup>7</sup> In addition, most of the graphene used in electrochemistry is produced from the reduction of graphene oxide and usually has functional groups such as hydroxyl and carboxyl, which are advantageous for adsorbing heavy metal ions.<sup>8</sup> However, because of the van der Waals and π–π stacking interactions among individual graphene sheet interactions, the as-reduced graphene sheets tend to form irreversible agglomerates and even restack to form graphite when graphene dispersion solutions are dried.<sup>9–11</sup> By incorporation of nanoparticles into graphene sheets, the aggregation problem of graphene sheets could be minimized or prevented.<sup>12</sup> Recently, graphene-based nanosensors have been fabricated aiming to employ them in electrochemical detecting heavy metal ions, such as graphene decorated with metal nanoparticles<sup>13</sup> and conducting polymer.<sup>14,15</sup> However, very

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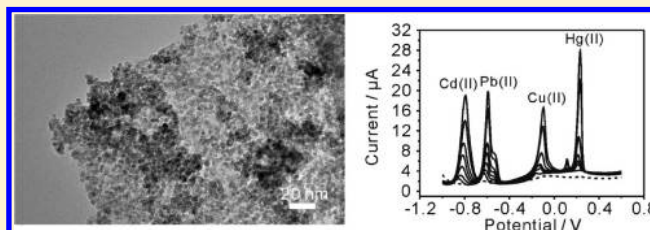
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few reports on the graphene decorated with metal oxides nanoparticles could be found in electrochemical detecting heavy metal ions.

Tin oxide ( $\text{SnO}_2$ ) is a well-known semiconductor and gas sensing material, and has been used as electrode material.<sup>16</sup> The  $\text{SnO}_2$  nanomaterials have been widely studied in the gas sensor<sup>17</sup> and anode material for lithium-ion batteries.<sup>17,18</sup> In addition, there have been a few reports about the preparation of  $\text{SnO}_2$ /graphene composites with application in gas sensor,<sup>19</sup> lithium batteries,<sup>20,21</sup> and supercapacitors.<sup>22</sup> However, to the best of our knowledge, both  $\text{SnO}_2$  and  $\text{SnO}_2$ /graphene nanocomposites have never been found in heavy metal ion detection. As we know,  $\text{SnO}_2$  is an oxide which may adsorb heavy metal ions,<sup>23</sup> and its nanoparticles possess unique properties such as high electrical conductivity and chemical sensitivity.<sup>19</sup> However, the  $\text{SnO}_2$  nanoparticles are easy to aggregate, and then it is hard to obtain the best performance of the  $\text{SnO}_2$  nanoparticles. Thus, we try to use reduced graphene oxide as template to prevent the  $\text{SnO}_2$  nanoparticles from aggregating and try to use  $\text{SnO}_2$  nanoparticles combined with reduced graphene oxide in electrochemical detecting heavy metal ions.

Although a lot of papers have been published on electrochemical sensing heavy metal ions, only a few works on the simultaneous and selective electrochemical sensing several target metal ions could be found in the literature. In this work,  $\text{SnO}_2$  was used in combination with graphene to fabricate a electrochemical platform for the simultaneous analysis of Cd(II), Pb(II), Cu(II), and Hg(II) in solution by square wave anodic stripping voltammograms (SWASV) for the first time. The  $\text{SnO}_2$ /reduced graphene oxide nanocomposite composed of 4–5 nm  $\text{SnO}_2$  nanoparticles was synthesized by a simple wet chemical method. Herein, the  $\text{SnO}_2$  nanoparticles not only prevented the graphene from gathering together but also acted as electrochemical catalyst in detecting heavy metal ions. Thus, the  $\text{SnO}_2$ /reduced graphene oxide nanocomposite modified glass carbon electrode showed enhanced sensing performance compared with single  $\text{SnO}_2$  and single graphene. Meanwhile, most of the papers reported that if a wide potential separation between the stripping peaks in simultaneous analysis of multiple heavy metal ions exists, there is no interference from each other. However, we observed an interesting phenomenon of mutual interference, even though a wide potential separation exists between the stripping peaks.

## 2. EXPERIMENTAL SECTION

**2.1. Chemical Reagents.** All reagents were commercially available from Sinopharm Chemical Reagent Co., Ltd. (China) with analytical grade and were used without further purification. Acetate buffer solutions of 0.1 M for different pH were prepared by mixing stock solutions of 0.1 M NaAc and HAc. Phosphate buffer solutions (PBS) of 0.1 M were prepared by mixing stock solutions of 0.1 M  $\text{H}_3\text{PO}_4$ ,  $\text{KH}_2\text{PO}_4$ ,  $\text{K}_2\text{HPO}_4$ , and NaOH. The water (18.2 M $\Omega$  cm) used to prepare all solution was purified with the NANOpureDiamond UV water system.

**2.2. Preparation of  $\text{SnO}_2$ /Reduced Graphene Oxide Nanocomposite.** Graphene oxide was first prepared by a modified Hummers' method.<sup>24</sup> Then the  $\text{SnO}_2$ /reduced graphene oxide nanocomposite was synthesized using the following one-step wet chemical method. Namely, 0.1 g of the dried graphene oxide was added into 500 mL of deionized water (DI water). The obtained mixture was sonicated for 90 min. At the same time, 2.4 g of

$\text{SnCl}_4 \cdot 3\text{H}_2\text{O}$  was dissolved into 20 mL of DI water and then 8 mL of the graphene oxide solution was added, in which the concentration of  $\text{SnCl}_4$  is about 0.24 M. The above mixture was stirred for 5 h and then centrifuged for 5 min at 8000 r min<sup>-1</sup>. In order to improve the crystallinity of the  $\text{SnO}_2$  and remove the residual water molecules and functional groups from the graphene oxide, the product was heated at 500 °C for 2 h under an argon atmosphere. For comparison, the single  $\text{SnO}_2$  and reduced graphene oxide were synthesized using the same process except the addition of the aqueous dispersion graphene oxide and  $\text{SnCl}_4 \cdot 3\text{H}_2\text{O}$ .

**2.3. Preparation of Modified Electrode.** Ultrasonic agitation (for 5 min) was used to disperse the  $\text{SnO}_2$ /reduced graphene oxide nanocomposite into alcohol to give a suspension. Prior to the surface modification, the bare glassy carbon electrode (GCE) was polished carefully with 1.0, 0.3 and 0.05  $\mu\text{m}$  alumina powder, respectively, and rinsed with DI water, followed by being sonicated in alcohol and DI water successively and dried under nitrogen. Then, an aliquot of 5  $\mu\text{L}$  of the mixture was coated on the electrode, and then the solvent was evaporated at room temperature to obtain the  $\text{SnO}_2$ /reduced graphene oxide nanocomposite film modified electrode. For comparison, single  $\text{SnO}_2$ , graphene oxide, and reduced graphene oxide modified glass carbon electrode were prepared using the same process.

**2.4. Cd(II), Pb(II), Cu(II), and Hg(II) Detection.** Square wave anodic stripping voltammetry (SWASV) was used for the detection under optimized conditions. Cd, Pb, Cu, and Hg were deposited at the potential of -1.0 V for 120 s by the reduction of Cd(II), Pb(II), Cu(II), and Hg(II) in 0.1 M NaAc-HAc (pH 5.0). The anodic stripping (reoxidation of metal to metal ions) of electrodeposited metal was performed in the potential range of -1.0 to 0.5 V at the following parameters: frequency, 15 Hz; amplitude, 25 mV; increment potential, 4 mV. The simultaneous and selective detection of Cd(II), Pb(II), Cu(II), and Hg(II) has been performed at the same experimental condition.

**2.5. Apparatus.** Electrochemical experiments were recorded using a CHI 660D computer-controlled potentiostat (ChenHua Instruments Co., Shanghai, China) with a standard three-electrode system. A bare glassy carbon electrode (GCE, diameter of 3 mm) or modified GCE served as a working electrode; a platinum wire was used as a counter-electrode with a saturated Ag/AgCl electrode (ChenHua Instruments Co., Shanghai, P. R. China) completing the cell assembly.

Scanning electron microscopy (SEM) images were obtained with a FEI Quanta 200 FEG field emission scanning electron microscope. Transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) and energy dispersive spectrometer (EDS) analyses were performed using a JEM-2010 microscope equipped with Oxford INCA EDS operated at 200 kV accelerating voltage (Quantitative method: Cliff Lorimer thin ratio section).

## 3. RESULTS AND DISCUSSION

**3.1. Morphologic Characterization of  $\text{SnO}_2$ /Reduced Graphene Oxide Nanocomposite.** Figure 1 shows the TEM images and phase maps of  $\text{SnO}_2$ /graphene nanocomposite. As seen, a graphene sheet was coated with lots of homogeneous and dense  $\text{SnO}_2$  nanoparticles having highly uniform size. Well-arranged crystalline lattice fringes (Figure 1b) suggest a well-crystallized polycrystalline structure of  $\text{SnO}_2$  nanoparticles. The adjacent lattice fringe spacing of about 0.345 and 0.267 nm correspond to



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