Introduction

The contamination of arsenic in the environment is a serious worldwide threat to human health. More than 20 arsenic compounds (such as As(III), As(VI) and As(0), etc.) are present in the natural environment and biological systems, and the trivalent state (As(III)) is much more toxic, more soluble and more mobile than the other valent state. Arsenic may cause acute toxicity, resulting in dysphasia, profuse diarrhea, dehydration, muscular cramps, and facial edema, or sub-acute toxicity as the loss of appetite, jaundice, and tingling of the hands and feet. Even low concentrations of arsenic species can lead to skin lesions, peripheral neuropathy, diabetes, renal system effects, cardiovascular disease, and cancer with prolonged exposure. WHO has fixed a limit of 10 ppb for As(III) in drinking water. It is thus necessary to find trace As(III) in polluted environments. The methods concerning the determination of As(III), such as atomic absorption spectrometry, atomic fluorescence spectrometry, atomic emission spectrometry, and inductively coupled plasma-mass spectrometry (ICP-MS) have been employed to detect As(III). However, the electrochemical method could provide more convenient means, such as a rapid analysis time as well as simple and low-cost for the arsenic detection. Especially, a stripping voltammetry technique has been widely used for detection of As(III). It is very important to choose proper electrode materials in electrochemical detection, since most materials of modified electrodes for the determination of As(III) are precious metals. It has been widely reported that Au and Au nanomaterials modified electrodes provided a more sensitive stripping voltammetry response than others. Fe3O4, as a magnetic particle oxide with low cost and ignorable toxicity, has been widely employed to perform the adsorption and determination of As(III). However, the poor conductivity of Fe3O4 has limited the sensitivity of electrochemical detection. Graphene, a basic building block of all graphitic materials, is a flat monolayer of carbon atoms tightly packed into two-dimensional honeycomb lattices. Due to its unique nanostructure and fascinating properties, such as its fast electron transportation, high surface area, good conductivity and excellent mechanical flexibility, graphene have become a sensational material in current research. The two-dimensional basal plane structure and high specific surface area make graphene an ideal support material for the deposition of nanoparticles. Graphene has become an ideal support material for the deposition of nanoparticles.
The use of SPCE is also a great simplification in the determination of As(III) and As(V), indicating that there is a strong interaction between Fe₃O₄ and arsenic. Screen-printed carbon electrodes (SPCE), as a disposable electrode, modified with nanoparticles have been shown to be highly effective in the analysis of arsenic. The use of SPCE is also a great simplification in the design and operation of heavy metal ions determination.

Therefore, based on the above mentioned, it is still desired to develop a facile, eco-friendly and efficient method to synthesize rGO/Fe₃O₄ composites and to employ them to modify SPCE for the electrochemical determination of As(III). Also, we have reported the determination of As(III) using a Fe₃O₄ modified electrode. Also, a Fe₃O₄-ion liquid composite modified electrode was employed to further perform the electrochemical detection of As(III). Excellent performance was obtained with a lower detection limit and good sensitivity. However, the room-temperature ion liquid with a high price was difficult to synthesize. Based on these points, the incorporation of reduced graphene oxide with good conductivity, a large surface area and good electronic properties could result in a new platform for the sensitive detection of As(III).

Preparation of rGO/Fe₃O₄ composites

The GO sheets were prepared using a previous method, and rGO/Fe₃O₄ composites were synthesized according to our reported research by the addition of GO. In brief, both polyethylene glycol (1.0 g) and GO (30 mg) were dissolved in 18 mL of ethylene glycol; the mixture was successively sonicated for 1 h to form a transparent solution. Then, 1.25 mmol of FeCl₃·6H₂O (0.675 g) and a mixture of NaAc (1.8 g), followed by the addition of 2.0 g anhydrous sodium acetate, were added into the above mixture solution under magnetic stirring for 1 h. After the autoclave was allowed to cool to room temperature, the precipitate was separated by a magnetic field, washed three times with distilled water and absolute ethanol, respectively, and then dried under a vacuum for 10 h at 60°C.

Results and Discussion

The morphology and structure characteristics of obtained rGO/Fe₃O₄ composites

The morphology and structure characteristics of obtained rGO/Fe₃O₄ composites were analyzed using a Fourier-transform infrared (FTIR) spectrometer (Nicolet Co., USA) in the specular transmission mode. The Raman spectrum was recorded with a Jobin Yvon LABRAMHR spectrometer with argon ion laser-beam focusing on the sample surface at room temperature.

Experimental

Chemicals and apparatus

All of the reagents and solvents, which were used in the synthesis of Fe₃O₄, were of analytical grade and used as received without further purification. Graphite was purchased from Alfa Aesar (Tianjing, China). Impurity-free nitrogen was purchased from Nanjing Special Gases Factory Co., Ltd. All experiments were carried out at a room temperature. All electrochemical measurements were performed with a CHI 660D computer-controlled potentiostat (ChenHua Instruments Co., Shanghai, China). The electrochemical cell consists of a three-electrode arrangement with bare or modified SPCEs serving as the working electrode, with carbon used as the counter electrode, a silver pseudo reference electrode completed the circuit.

The morphologies of the nanomaterials were verified by field-emission scanning electron microscopy (FESEM, Quanta 200 FEG, FEI Company, USA). X-ray diffraction (XRD) patterns of the samples were recorded on a Philips X’pert PRO SUPER diffractometer (Netherland) with Cu Kα radiation. A pH meter (Mettler Toledo FE20, Switzerland) was used for measuring the pH of the solution. X-Ray photoelectron spectroscopy (XPS) analyses of the samples were conducted using a Thermo ESCALAB 250 spectrometer with an Mg Kα X-ray source (1486.6 eV, 150 W). The structure characteristics of the samples were analyzed using a Fourier-transform infrared (FTIR) spectrometer (Nicolet Co., USA) in the specular transmission mode. The Raman spectrum was recorded with a Jobin Yvon LABRAMHR spectrometer with argon ion laser-beam focusing on the sample surface at room temperature.
composites were investigated by SEM and XRD as shown in Fig. 1. Figure 1(a) shows a representative SEM image of as-synthesized rGO/Fe₃O₄ composites; it can be observed that the size of Fe₃O₄ microspheres is ca. 400 nm, and the Fe₃O₄ is well distributed on the surface of rGO sheets, forming the composites.

The XRD pattern of as-prepared rGO/Fe₃O₄ composites is shown in Fig. 1(b). It can be observed that no typical diffraction peak of GO appears in the pattern of rGO/Fe₃O₄, and that the diffraction peak at 2θ = 26.2° is resulted from rGO, thus confirming the effective reduction of GO by ethylene glycol. The Bragg reflections of (220), (311), (440), (422), (511) and (440) appear in rGO/Fe₃O₄ in Fig. 1(b), matched well with those of Fe₃O₄, which confirms the formation of rGO/Fe₃O₄ composites. Figure 1(c) shows the high-resolution Fe 2p spectrum; the two peaks at 724.9 and 710.8 eV correspond to Fe 2p₁/₂ and Fe 2p₃/₂ of Fe₃O₄, respectively, confirming the chemical state as Fe³⁺.

The electrochemical behaviors of the rGO/Fe₃O₄ modified SPCE

According to the Randles-Sevcik equation, \( I = (2.69 \times 10⁵)n^{3/2}A^{1/2}D^{1/2}v^{1/2}, \) where \( I \) is the reduction peak current, \( n \) is the electron-transfer number, \( A \) is the apparent electrode area (cm²), \( D \) is the diffusion coefficient of [Fe(CN)₆]³⁻ (cm² s⁻¹), \( C \) is the concentration of [Fe(CN)₆]³⁻ (mol L⁻¹) and \( v \) is the scan rate (V s⁻¹). The electrochemical active surface of bare, Fe₃O₄, and rGO/Fe₃O₄ modified SPCE are calculated to be 0.0781, 0.0896, and 0.148 cm². It is apparent that the effective area of rGO/Fe₃O₄ is the biggest.

The electrochemical behaviors of rGO/Fe₃O₄ composites modified SPCE are studied using cyclic voltammetry (CV) and electrochemistry impedance spectra (EIS). Cyclic voltammogram of Fe₃O₄/SPCE showed the maximum stable and well-defined redox peaks in Fig. 3(a). This is the reason that the reduced graphene oxide with good conductivity improved the electron transfer of a redox probe, while the redox peak of Fe₃O₄/SPCE reduced. That of bare SPCE is the smallest due to poor conductivity of Fe₃O₄. The corresponding EIS of different electrodes is also shown in Fig. 3(b). It can be observed that the impedance value of SPCE is the largest, while that of Fe₃O₄/SPCE decreased. The value of rGO/Fe₃O₄/SPCE is

FT-IR, Raman spectra of rGO/Fe₃O₄ composites

The absorption band at 1630 and 1050 cm⁻¹ of rGO/Fe₃O₄ composites is assigned to the stretching vibration of the C≡C and C–O of reduced graphene oxide in Fig. 2(a); the peak at around 580 cm⁻¹ is attributed to Fe–O, which demonstrates successful loading of Fe₃O₄ in the rGO sheets. Raman spectra of both GO and rGO/Fe₃O₄ have two strong peaks at 1334 and 1589 cm⁻¹, which correspond to the well-documented D and G bands, respectively. The intensity ratios (G/D) of rGO/Fe₃O₄ (1.157) are much higher than that of GO (0.92), indicating the presence of localized sp³ defects within the sp² carbon network after reduction of the exfoliated GO.

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the smallest among three electrodes, which matches with the cyclic voltammogram in Fig. 3(a).

Optimum experimental conditions
In order to obtain the maximum response of rGO/Fe₃O₄/SPCE towards the determination of As(III), the important voltammetric parameters influencing the stripping response of As, such as the amount of rGO/Fe₃O₄ modified SPCE, pH of solution, deposition time, and deposition potential, were optimized in a 10-ppb As(III) solution, respectively.

The amount of rGO/Fe₃O₄ modified SPCE
Since the amount of materials plays important roles in the performance of the modified electrode, the quantities of rGO/Fe₃O₄ dispersed in an ethanol solution was investigated in a 0.1 M NaAc–HAc buffer solution (pH 5.0) at 120 s (deposition time) and −0.6 V (deposition potential). The corresponding results are shown in Fig. 4(a). It can be observed that the SWASV response of the electrode towards As(III) reaches the maximum value when the amount of the composites modified electrode is 9 μg, and then decreases with increasing rGO/Fe₃O₄, due to the excess of rGO/Fe₃O₄, resulting in slow electron transfer. Therefore, 9 μg of the composite is chosen for electrode modification.

pH of supporting solution
The stripping behavior of metal ions is influenced by the pH of solution, due to the hydrolysis of metal ions. The effect of the pH was investigated in the range of 3.0 to 6.0 as shown in Fig. 4(b) as follows: supporting electrolyte, 0.1 M NaAc–HAc; deposition time, 120 s; deposition potential, −0.6 V; the amounts of composites modified electrode, 9 μg. As can be seen in Fig. 4(b), the stripping current of As increased with increasing pH values until the pH reached 5.0, and then decreased with increasing pH. Thus, the NaAc–HAc solution with pH 5.0 is selected for subsequent electrochemical analysis.

Deposition time
The deposition time is an important factor that affects the detection limit and the sensitivity. The deposition time was studied in 10 ppb As(III) in the range of 30 to 210 s, as shown in Fig. 4(c) (others as follows: the pH of solution, 5.0; supporting electrolyte, 0.1 M NaAc–HAc; the amount of composites modified electrode, 9 μg). The results are shown in Fig. 4(c). It can be seen that the peak current for As(III) gradually increases from the beginning of −0.3 V, and reaches the maximum at potential of −0.6 V. However, when the accumulation potential is more negative than −0.6 V, the stripping current increases slowly. Thus, −0.6 V is chosen as the optimal potential.

Deposition potential
The potential in the range of −0.3 to −0.8 V at rGO/Fe₃O₄/SPCE was studied in a 10-ppb As(III) solution (others as follows: deposition time, 120 s; the pH of solution, 5.0; supporting electrolyte, 0.1 M NaAc–HAc; the amount of composites modified electrode, 9 μg). The results are shown in Fig. 4(d). It can be seen that the peak current for As(III) gradually increases from the beginning of −0.3 V, and reaches the maximum at potential of −0.6 V. However, when the accumulation potential is more negative than −0.6 V, the stripping current increases slowly. Thus, −0.6 V is chosen as the optimal potential.

The detection of ultratrace As(III) through rGO/Fe₃O₄ modified SPCE
The detection of As(III) is performed by SWASV in an acetate buffer solution containing different concentrations of As(III)
under the optimized conditions. Figure 5(a) shows the stripping responses of As(II) to As(III) in the range from 2 to 20 ppb. It is found that the peak current increases with increasing concentrations of As(III). In addition, since the stripping potentials of As shifts to a more positive with increase of the peak current, this could be the reason that the As of high concentration affects the stripping of As on the surface of the electrode. Figure 5(b) refers to the linear relationship between the voltammetric currents and the concentrations of As(III). The lowest detectable concentration for the rGO/Fe3O4 modified SPCE is 2 ppb, four-times lower compared with Fe3O4.15 The detection limit of 0.3 ppb (3δ method) is obtained, and is lower than the standard of WHO (10 ppb).

The same modification electrodes were employed three times, and the obtained relative standard deviations were all less than 3.5%, which showed good repeatability of the modified electrode.

**Interference analysis**

Cu(II) shows the major interference in the detection of As(III) among various metal ions. Thus, Cu(II) was firstly chosen for interference studies. The SWASV responses of the rGO/Fe3O4 modified SPCE are shown for different concentrations of As(III) in the presence of Cu(II) (200 ppb) in Fig. 6. The stripping peak current for Cu(II) at about -0.16 V is almost the same, while the stripping peaks of As with a little position shift increase with increasing concentrations of As(III). The differences of the stripping potential of Cu(II) compared with the paper16 might be due to the different electrodes used and modifications of different materials shown in Fig. 6(a). A glassy carbon electrode modified with a Fe3O4/Gd2O3 composite was employed in the detection of Cu(II) in reported paper;10 however, the screen-printed carbon electrode modified with rGO/Fe3O4 in this report was used for an interference analysis of Cu(II). The sensitivity is 0.01 μA ppb⁻¹, and obviously less than that (1.922) of the rGO/Fe3O4 modified SPCE in the absence of Cu(II). The decrease of sensitivity might be due to competition for active sites at the Fe3O4/rGO surface by the Cu(II), or the formation of intermetallic compounds.

In the experiment, metal ions including Pb(II), Cr(III), Ni(II), K(I), Ca(II), Fe(III), Hg(II), and As(III) ions (5 ppb) (the molar ratio of metal ions and As(III) is 80:1) were chosen as interfering ions for investigating the selectivity of the Fe3O4/rGO modified electrode, respectively. The results indicated that little stripping peak current signal could be observed for each metal ion mentioned above in Fig. S1. As can be seen, the metal ions showed no interference toward the stripping peak current of As(III).

### Table 1 Analysis of soil samples

| Sample | Found/ nM | Added/ nM | After added/ nM | Mean recovery, % |
|--------|-----------|-----------|-----------------|-----------------|
| Sample 1 | 53.4 | 40 | 92.6 | 99.1 |
| Sample 2 | 64.5 | 80 | 145.2 | 100.5 |

**The analysis of real sample**

In order to evaluate the real application performance of the proposed sensor, the Fe3O4/rGO modified SPCE was used to detect As(III) in the soil sample solution. The soil samples (1 g) were digested with concentrated HCl, HNO3, HF and HClO4 solutions. The procedure was as follows: the soil samples of conical flask were treated with 4 ml of nitric acid for half an hour, 9 ml of hydrochloric acid for 20 min, 9 ml of hydrofluoric acid for half an hour, and 5 ml of perchloric acid for 1 h at about 100 - 140°C, respectively. Then, the white precipitate was separated, centrifuged and dried in vacuum, and diluted with 0.1 M NaAc-HAc buffer (pH 5.0). Also, the concentration of As(III) was allowed to fall within the linear range of the Fe3O4/rGO modified SPCE calibration curve. As(III) standard solutions with different concentrations were added into the sample solutions, and the recoveries were subsequently evaluated as shown in Table 1. It is suggested that this Fe3O4/rGO modified SPCE could have a potential application for detecting As(III) in real samples.

### Conclusions

Fe3O4/rGO composites were prepared by a one-pot method, and characterized by SEM, XRD, FTIR, etc. The electrochemical behaviors of Fe3O4/rGO modified electrodes were investigated by CV, EIS and SWASV, respectively, and the proposed electrode exhibited lower detection limit and a higher sensitivity for As(III). These excellent electrochemical properties of the composite electrode might be ascribed to its large surface area and the good conductivity ability of reduced graphene oxide. It is expected that this Fe3O4/rGO modified electrode could offer a potential for fabricating electrochemical sensors.
Acknowledgements

This research was financially supported by the National Science Foundation of Anhui Province (1608085MB43) and the Natural Science Foundation of the Education Department of Anhui Province (2016).

Supporting Information

This material is available free of charge on the Web at http://www.jsac.or.jp/analsci/.

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