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Sensitive and selective electrochemical detection of heavy metal ions using amino-functionalized carbon microspheres

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ABSTRACT

Amino-functionalized carbon microsphere (NH_2-CMS) was successfully synthesized via hydrothermal and amino-functionalized method for electrochemical detection of heavy metal ions for the first time. The as-prepared product was characterized by Scanning electron microscopy (SEM) and Fourier transform infrared (FTIR). The features of NH_2-CMS before and after heavy metal ion adsorption were studied using X-ray photoelectron spectroscopy (XPS). The morphology of synthesized NH_2-CMS was investigated by SEM images, and the presence of the amino group was confirmed using FTIR and XPS techniques. The NH_2-CMS modified glass carbon electrode (GCE) exhibited an excellent sensitive and selective detection of heavy metal ions simultaneously and individually, including Cd(II), Pb(II), Cu(II) and Hg(II) using square wave anodic stripping voltammetry (SWASV).

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1. Introduction

It is well-known that heavy metal ions, such as Cd(II), Pb(II), Cu(II) and Hg(II), are recognized as a great threat in the earth due to their toxicity and even trace amounts of them result in a serious condition to human health [1,2]. So far, all kinds of analytical methods have been applied to the detection of heavy metal ions [3–9]. Among the current developed strategies, electrochemical detection is highly favored by the characteristics of rapid detection, high sensitivity and selectivity [10–16]. However, related literature about simultaneous detection of heavy metal ions have been reported with precious metals [17,18] and complex synthesis [19], these modified materials for simultaneous detection of heavy metal ions are high cost or need more manpower and material resources. So it is significant to find the cheap and simple synthesis material for detection of heavy metal ions individually and simultaneously.

Carbon microspheres (CMSs) have been applied in many fields because of its low cost, good electrical conductivity and simple synthesis [20–24]. The amino group on the surface of functionalized CMS is bringing increased attractive force in adsorption of heavy metal ions [25]. In this study, we use amino-functionalized carbon microsphere (NH_2-CMS) modified glassy carbon electrode (GCE) for electrochemical individual and simultaneous detection of heavy metal ions, including Cd(II), Pb(II), Cu(II) and Hg(II) with square wave anodic stripping voltammetry (SWASV). CMS was prepared through the polycondensation reaction of glucose using hydrothermal synthesis, as reported previously [26]. NH_2-CMS was characterized using Scanning electron microscopy (SEM), Fourier transform infrared (FTIR) and X-ray photoelectron spectroscopy (XPS). The mutual interference was discussed in detail and the stability of the modified electrode was explored. Then, NH_2-CMS was successfully applied in the real water sample analysis. Finally, adsorption experiments were performed to give a reason for the stripping behaviors. The features of NH_2-CMS before and after heavy metal ion adsorption were studied using XPS. As a result, individual and simultaneous detection of heavy metal ions was achieved with NH_2-CMS.

2. Experiment Section

2.1. Chemical Reagents

All regents were bought from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). They were analytical grade and used without further purification. All the stock solution was diluted with the de-ionized (DI) water (18.25 MΩ cm) from the NANOpure®Diamond™ UV water system. Acetate buffer (HAc–NaAc) solutions (0.1 M) were prepared by mixing solutions of 0.1 M HAc and NaAc.

2.2. Apparatus

All electrochemical measurements were using a CHI660D computer-controlled potentiostat (ChenHua Instrument Co., Shanghai, China).

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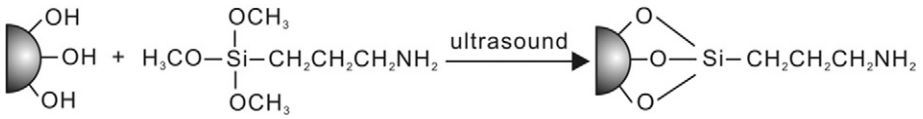

Fig. 1. Procedure of the amidogen functional carbon microspheres.

Experiments were operated in a conventional three-electrode cell using the modified bare glass carbon electrode (GCE, 3 mm diameter) as a working electrode, Ag/AgCl/KCl (3 M KCl saturated with AgCl) as a reference electrode and Pt wire as a counter electrode. The SEM images were obtained by a field-emission scanning electron microscope (FESEM, Quanta 200 FEG, FEI Company, USA). The FTIR was obtained using a Nicolet Nexus-670 FT-IR spectrometer. XPS analyses of the samples were conducted using a VG ESCALAB MKII spectrometer with an Mg Kα X-ray source (1253.6 eV, 120 W) as a constant analyzer.

2.3. Preparation of Amidogen Functional Carbon Microsphere (NH_2-CMS)

CMSs were prepared using a polycondensation reaction of glucose and hydrothermal synthesis, as previously reported. In simple terms, glucose (1 M) and cetyltrimethylammonium bromide (CTAB, 0.5 mmol) were dissolved in distilled water to form a homogeneous solution. The solution was transferred into a 50 ml Teflon-sealed autoclave and maintained at 180 °C for about 5 h. After reaction, CMSs were obtained through centrifugation, following being washed by distilled water and ethanol some time. Last, the products were dried in a vacuum oven at 60 °C for further use.

NH_2-CMSs were synthesized by CMS combining with reactive silane coupling agent. Firstly, 10 mg CMSs were dissolved in the 20 ml ethanol, 100 μl 3-aminopropyltrimethoxysilane (APTMS) and 100 μl ammonium hydroxide with ultra-sonication for 8 h. The process of synthesizing amino-functionalized carbon micro spheres is shown in Fig. 1 according to the previous work [27,28]. Then, NH_2-CMSs were obtained through centrifugation with distilled water washing several times. Finally, the products were dried in a vacuum oven at 60 °C. Using FTIR and XPS demonstrated the existence of the amino-functionalized in the following section in detail.

2.4. Fabrication of Modified Electrode

Bare GCE was polished with 1.0, 0.3 and 0.05 μm of alumina power before modification, respectively. Then, the polished GCE successively sonicated with 1:1 (V/V) HNO_3, alcohol and distilled water to remove any adsorbed substances on the surface of electrode. 5 mg NH_2-CMS was dissolved in 10 ml H_2O and sonicated for 1 min to form a homogeneous suspension. Following, 4 μl suspension was modified on the polished GCE while the bare GCE was drying. Lastly, the modified electrode was dried at room temperature in the air.

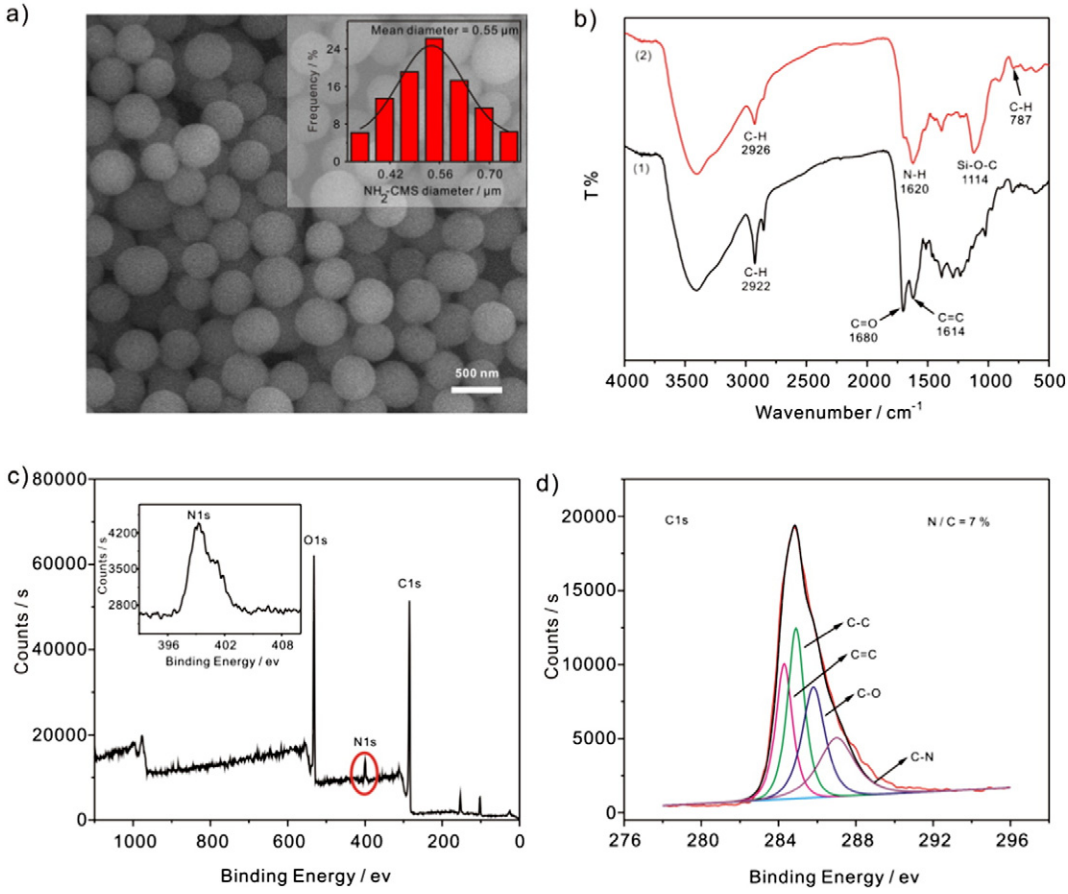

Fig. 2. a) SEM image of amino-functionalized carbon microspheres (NH_2-CMS). b) FTIR spectrum of (1) CMS and (2) NH_2-CMS. c) XPS spectra analysis of NH_2-CMS. d) Higher resolution data of the C1s.

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