



An electrochemical sensor for hydrazine and nitrite based on graphene–cobalt hexacyanoferrate nanocomposite: Toward environment and food detection



Xiaoli Luo^a, Jianbin Pan^b, Kemei Pan^a, Yanyan Yu^c, Anni Zhong^a, Shanshan Wei^a, Jin Li^a, Jiayan Shi^a, Xinchun Li^{a,*}

^a Division of Pharmaceutical Analysis, School of Pharmacy, Guangxi Medical University, 22 Shuangyong Road, Nanning 530021, PR China

^b State Key Laboratory of Analytical Chemistry for Life Science, School of Chemistry and Chemical Engineering, 22 Hankou Road, Nanjing University, Nanjing 210093, PR China

^c School of Pharmaceutical Sciences, Sun Yat-sen University, 132 Waihuan East Road of Higher Education Mega Centre, Guangzhou 510006, PR China

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ABSTRACT

Hydrazine and nitrite are well known as carcinogenic substances, which however, are commonly used in chemical industry and food processing. Monitoring of such two species in water and food is necessary. Here, we report a convenient electroanalytical method for detection of hydrazine and nitrite by facile preparation of graphene–cobalt hexacyanoferrate nanocomposite through a two-step electrode surface functionalization. The morphology and constituent of the prepared nanocomposite were characterized by scanning electron microscopy, transmission electron microscopy, and X-ray photoelectron spectroscopy. Cyclic voltammetry was used to profile the electrochemical behaviors of hydrazine and nitrite, revealing the synergetic electrocatalytic function of reduced graphene oxide and cobalt hexacyanoferrate nanoparticles. This was confirmed by the decreased oxidation overpotentials and increased current response of the target analytes, and thus offered a premise for electroanalysis. The quantitative measurements were carried out by amperometry and differential pulse voltammetry, respectively. Moreover, the analytical utility was demonstrated by determination of water samples and pickled food; the average recovery was found to be from 92.2% to 108%, with the relative standard deviation (RSD) less than 7.8%. The present method opens an avenue for detection of potential carcinogens in relation to environment and food territory.

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1. Introduction

Environmental pollution and food safety are currently a focus of considerations worldwide. Hydrazine is a highly carcinogenic substance and may cause water source contamination, owing to its wide-range applications involved with industrial process [1]. For example, hydrazine can function as corrosion inhibitor, antioxidant, catalyst, reducing agent, photographic developer, rocket propellant, and is frequently used as the starting material for the synthesis of insecticides, explosives, dyestuffs and pharmaceuticals [2,3]. Nitrite is widely used as food preservative, which is however detrimental to human body, because it can bind with hemoglobin to produce methemoglobin, leading to the reduced oxygen transport capability of blood [4]. Furthermore, nitrite can be readily converted to potent carcinogenic *N*-nitrosamines in the

stomach [5]. Therefore, humans exposed to hydrazine and nitrite would bear severe health risk. In this regard, detection of these poisonous substances is of practical importance.

A large number of analytical methods have been developed for the determination of hydrazine and nitrite, including spectrophotometry [6], fluorimetry [7], chemiluminescence [8], chromatography [9], capillary electrophoresis [10], flow injection analysis [11], and electrochemistry [12–15]. Among these techniques, electrochemical methods have drawn widespread attention, due to the particular merits such as low cost, easy operation, high sensitivity and considerable selectivity [14,16]. However, electrooxidation of hydrazine on conventional electrodes commonly faces substantial limit because of sluggish electrode kinetics and high overpotential [17]. Likewise, electroanalytical determination of nitrite generally suffers from poor sensitivity and accuracy as a result of electrode contamination by electrochemical intermediates [18]. Chemically modified electrodes bring about much flexibility and create many opportunities to address these

* Corresponding author.

E-mail address: xchleegxmu@163.com (X. Li).

problems [12]. Particularly, nanomaterials have been intensively studied and explored to fabricate electrochemical sensors for hydrazine and nitrite assay. Among them, carbon nanotubes (CNTs) [19,20], graphene [21,22], metal nanoparticles [23,24], and metal oxide [25] are the typical representatives. It should be noted that no efforts thus far, have been devoted to simultaneous determination of the two kinds of carcinogens by using an electrochemical sensor.

Graphene has attracted tremendous research interests in recent years [26,27]. It has been widely used for electrochemical sensing, due to its unique physicochemical features including large surface area, wide potential window, and remarkable electrical conductivity [28], thus enabling the direct analysis of a host of analytes pertaining to environment, food, pharmaceuticals, and biomolecules [29]. On the other hand, transition metal hexacyanoferrate (MHCF) is an important class of mixed-valence compound, and has distinctive electrochemical attributes. For instance, MHCF possesses notable electron mediating ability. A variety of MHCFs have been prepared and used for electrochemical sensors [30–35]. We previously described a miniaturized amperometric detector coupled to a microfluidic device using CNTs–cobalt hexacyanoferrate (CoHCF) modified graphite electrode; further a microchip electrophoresis–electrochemistry method was proposed [36].

The combination of graphene and nanoscale MHCFs may be promising for electrochemical sensing, because the synergetic electrocatalytic effect can be expected through rationally assembling of different nanomaterials. Thus, the analytical sensitivity and selectivity can be largely improved. A graphene–CoHCF modified electrode was presented for detection of prostate specific antigen [37]. In this work, thermal exfoliation of graphite oxide and chemical preparation of CoHCF were used, and then the graphene–CoHCF composite dispersion was dropped onto glassy carbon electrode (GCE). This method maybe led to poor stability and

reliability. Recently, Sattarahmady [38] reported graphene–CoHCF modified carbon paste electrode for determination of captopril, an antihypertensive drug. Specifically, CoHCF was also chemically prepared and graphene was produced by hydrazine reduction method. As aforementioned, the use of hydrazine is harmful to health, yet causes environmental nuisance.

We herein describe a green analytical method to determine hydrazine and nitrite using CoHCF nanoparticles–graphene modified glassy carbon electrode. This protocol was fulfilled by means of a two-step electrochemical preparation of reduced graphene oxide (rGO) and CoHCF nanoparticles. The prepared sensor exhibited favorable electrooxidation ability for the two analytes. Furthermore, the practicability was tested by determination of hydrazine in water samples and nitrite in pickled meat. To the best of our knowledge, this work reported, for the first time, an electrochemical method for the determination of hydrazine and nitrite.

2. Experimental

2.1. Chemicals and reagents

All reagents were of analytical grade and used as received otherwise special statement. Potassium dihydrogen phosphate, potassium nitrate, potassium hydroxide, sodium acetate, and glacial acetic acid were obtained from Sinopharm reagent (Shanghai, China). Potassium ferricyanide, potassium chloride, cobalt chloride hexahydrate (99.99% purity), hydrazine sulfate, and sodium nitrite were from Aladdin reagent (Shanghai, China). Graphite oxide was purchased from XFNano (Mesh size: 0.5–5 μm , Nanjing, China). Both of 50 mM stock solutions of hydrazine and nitrite were prepared in water, and were diluted with supporting electrolyte to desired concentrations for analysis. Ultrapure water

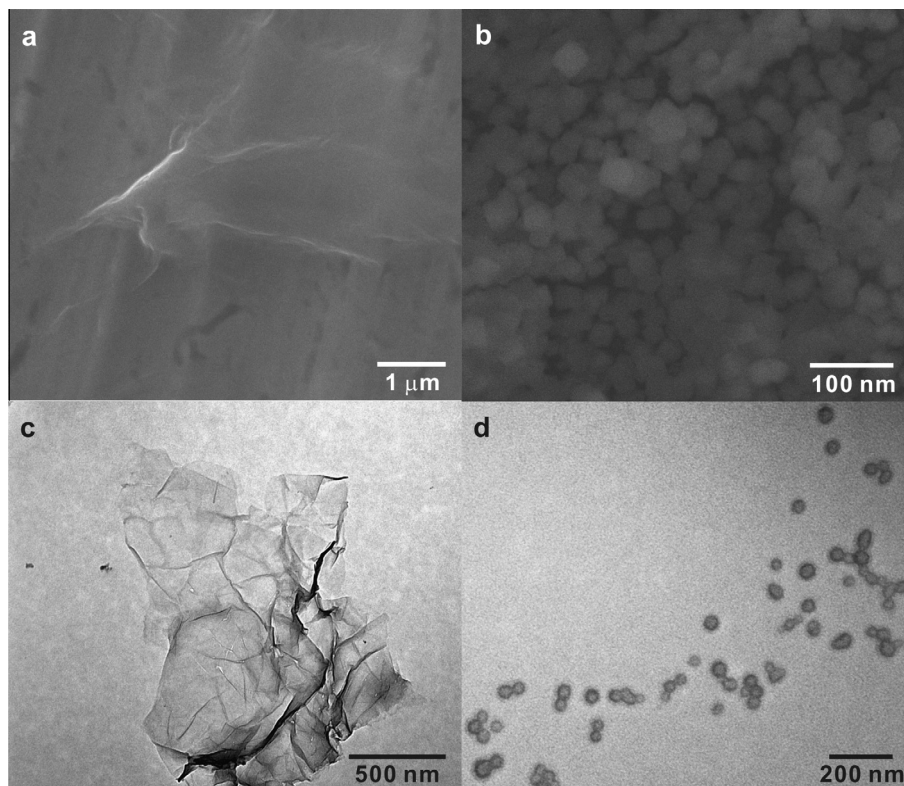


Fig. 1. SEM (a and b) and TEM (c and d) images of the electrochemically reduced graphene oxide (a and c) and cobalt hexacyanoferrate nanoparticles (b and d).

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