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Polyelectrolyte functionalized gold nanoparticles-reduced graphene oxide nanohybrid for electrochemical determination of aminophenol isomers

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ABSTRACT

A green chemical method for preparation of gold nanoparticles-reduced graphene oxide nanocomposite is described. This can be readily accomplished through a two-step chemical reduction scheme by using poly(diallyldimethylammonium chloride), a cationic polyelectrolyte as a common reducer. Polyelectrolyte here also serves to stabilize gold nanoparticles and is beneficial to electrical communication, leading to the formation of well-characteristic nanohybrid. The prepared nanomaterial showed remarkable electrocatalytic ability as a result of the rational conjunction of graphene and gold nanoparticles, which was demonstrated by direct electrochemical determination of three aminophenol isomers on a modified glassy carbon electrode. Effective peak separation of three isomers was achieved due to the favorable electron-transfer network perfectly assembled on the electrode surface, thus enabling the simultaneous assay of multiple components featuring analogous chemical structure without chromatographic separation. The modified electrode was further used to detect *para*-aminophenol in paracetamol tablets. The present method is simple, eco-friendly and holds potential for electroanalytical and biosensing applications.

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1. Introduction

Graphene is a two dimensional carbon allotrope, with single-atom-layer thick through sp^2-hybridized carbon atoms perfectly arranged in a honeycomb lattice, and has been a major subject of material science and nanoelectrochemistry to date [1], because of its distinctive physicochemical properties that include high surface-to-volume ratio, rough mechanical property, and fast electron transfer rate [2]. Metal nanoparticles, such as gold nanoparticles (GNPs), have also attracted enormous attention in electrochemistry and biosensing areas, due to the large surface area and excellent electrocatalytic ability [3]. These nanoscale materials have opened a vast of application fields and would be continuously exploited to construction of versatile electrochemical sensors [4].

Nanoelectrochemistry commonly relies on the functionality of nanomaterials [5]. Often, graphene is prepared by oxidation and exfoliation of graphite; the resulting graphene oxide (GO) is subject to electrochemical [6,7] or chemical reduction [8] to produce graphene. Chemical methods for preparation of graphene from graphite usually involve the use of hydrazine as a reducing agent [9], which is a potent carcinogenic substance, and consequently harmful to environment and health [10]. On the other, electrochemical reduction for preparing GNPs has been widely used; this method however typically brings about nanoparticles with a wide size distribution [11], which may result in poor reproducibility and controllability. It is well known that chemical reduction to prepare gold nanoparticles is a prevailing strategy [12], for instance, sodium borohydride [13], citrate [14], and hydrazine [15] are frequently used. However, byproducts inevitably coexist with the final object as a result of the concomitant oxidation of such reducers, thus making subsequent treatments necessary [16], which is somewhat inconvenient. Besides, to avoid the aggregation of gold colloids, a variety of stabilizers including thiols, surfactants, and polymers are customarily used, which are adverse to the final product in view of electroanalytical purpose. Additionally, preparation of graphene-gold nanoparticles functionalized composite material has drawn widespread research interests in recent years, because the synergic electrocatalytic effect can be reaped, which is essentially in favor of electrochemical sensing applications [17–19]. Liu et al. proposed an electrochemical method to synthesize GNPs/graphene

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nanocomposite used for simultaneous determination of biomolecules [20]. Xu [21] reported on the preparation of three-dimensional GNP-embedded porous graphene thin film by electrostatic layer-by-layer assembly of bovine serum albumin-functionalized graphene nanosheets and GNPs that was combined with subsequent thermal annealing; the electrochemical property was tested by electrocatalytic oxidation of H_2O_2.

Poly(diallyldimethylammonium chloride) (PDDA) is a cationic polyelectrolyte, possesses good electrical conductivity and has proven to induce the reduction of GO [22], and to prepare of GNPs [23], and graphene-metal nanoparticles composite [24]. PDDA is also used to preparation of functionalized nanocomposite through electrostatic interaction with the nanosized materials such as carbon nanotubes and metal nanoparticles. More importantly, PDDA can render hydrophilicity to graphene, thus preventing the agglomeration of graphene sheets, which is of particular significance [25]. Similarly, the agglomeration of GNPs can also be avoided by interfusion of positively charged PDDA. Therefore, it may be reasonable that other than the reducers mentioned above, PDDA would be beneficial to producing functionalized gold nanoparticles-reduced graphene oxide (GNPs/rGO) nanohybrid, yet facilitates the fabrication of electrochemical sensors.

Aminophenol, comprising three isomers, is a class of important raw material for chemical processing and pharmaceutical industry; however, such compounds are toxic, and may cause severe damage to multiple organs of human body [26]. In general, determination of isomeric constituents faces a practical challenge, primarily due to the similar physiochemical property. This is also a case even though separation techniques such as chromatography [27] and electrophoresis [28] are widely used in analytical community. There have only been several examples thus far demonstrating the simultaneous determination of aminophenol isomers by electrochemical methods. Su [29] described a simple method for detection of these compounds in water samples by electrochemically pretreated screen-printed carbon electrode. Duan [26] reported the electroanalysis of aminophenol isomers using amino-functionalized ordered mesoporous silica modified carbon paste electrode (CPE). However, the electrode preparation was rather complicated, hence falling short of flexibility with respect to the practicability. Recently, nanocrystalline zirconosilicate modified CPE was presented by Kaur [30], with the analytical sensitivity at nanomolar concentrations level toward all of the isomers. Although elegant, this work was even more time-consuming and labor-extensive, since it took several days to prepare the electrode modifying material. Here, we show that direct determination of aminophenol isomers can be easily realized by electrochemistry on glassy carbon electrode decorated by PDDA functionalized GNPs/rGO nanocomposite. Furthermore, detection of *para*-aminophenol (*p*-AP) in pharmaceuticals has also been performed.

2. Experimental

2.1. Reagents and apparatus

Graphite oxide was purchased from XFNano (Nanjing, China). Chlorauric acid trihydrate ($HAuCl_4 \cdot 3H_2O$), PDDA (20 wt% aqueous solution), and aminophenol isomers were from Aladdin reagent (Shanghai, China). All other reagents obtained from Sinopharm reagent (Shanghai, China) were of analytical grade and used as received. Milli-Q water (18.2 MΩ cm) was used in the experiments.

Electrochemical measurements were carried out on a CHI-810C (Chenhua, Shanghai, China) electrochemical analyzer connected to a three electrode configuration, in which modified glassy carbon electrode (GCE, 2 mm diameter), platinum electrode and Ag/AgCl electrode were used as working electrode, counter electrode and reference electrode; respectively. Micrographs of the prepared nanomaterials were acquired on a JEM-2100F transmission electron microscopy (JEOL, Japan). UV absorption spectrum was performed on a Perkin Elmer Lambda 650 UV/VIS spectrometer.

2.2. Preparation of GNPs/rGO composite

A 0.5 mg mL^{-1} of graphene oxide (GO) aqueous dispersion was obtained by ultrasonic agitation (450 W) of graphite oxide for 2 h. The resulting solution was then centrifuged at 4000 rpm for 45 min

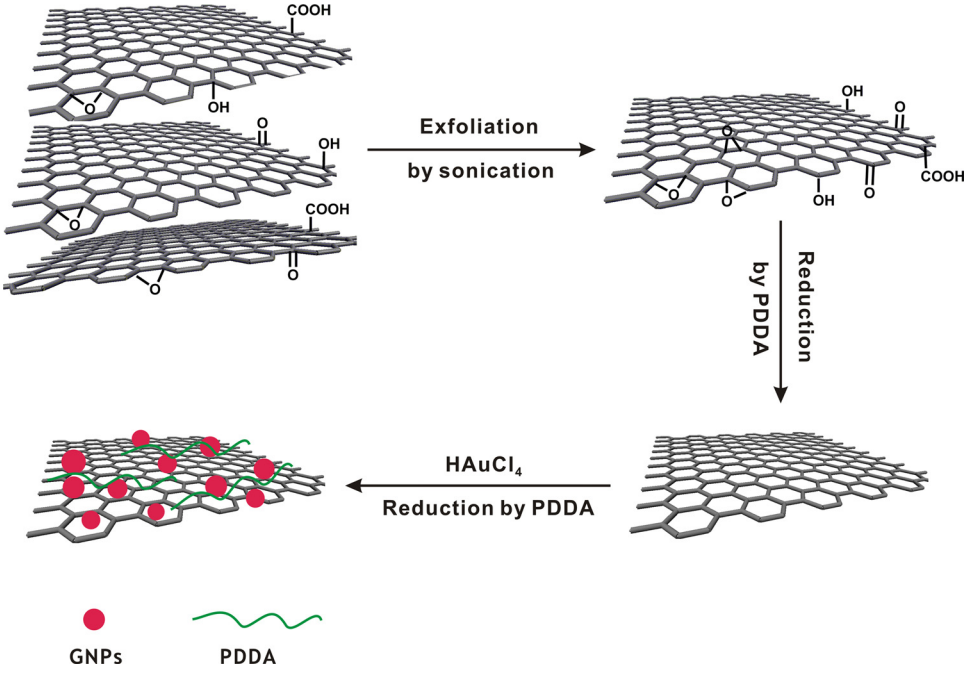

Fig. 1. Schematic illustration of the preparation of GNPs/rGO nanocomposite through PDDA-induced reduction. It is not to scale.

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