



Sensing of doxorubicin hydrochloride using graphene quantum dot modified glassy carbon electrode



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ABSTRACT

In this report, graphene quantum dot (GQD) was casted on the surface of the glassy carbon electrode and used for detection and determination of doxorubicin hydrochloride (DOX) in human plasma samples. It was found that GQD has been stably absorbed on glassy carbon electrode (GCE) by simple technique. The cyclic voltammetric results indicate that GQD modified GCE can remarkably enhance electroactivity toward the oxidation of DOX in phosphate buffer solutions. The electrochemical behavior was further exploited as a sensitive detection scheme for the DOX determination by differential-pulse voltammetry. Under optimized conditions, the concentration range and detection limit were 0.018–3.60 μM and 0.016 μM ($S/N = 3$), respectively. The method was successfully applied assay of the drug in human plasma samples.

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1. Introduction

Graphene quantum dots (GQDs) are graphene sheets that are smaller than 100 nm and have emerged as a significant research area in recent years [1–4]. Their various electronic and optoelectronic properties, due to quantum confinement and edge effects, make GQDs excellent candidates for the construction of nanoscale optical and electronic devices. GQDs are superior to common carbon materials because of the nature of nano-sized single layer graphene sheets, which endows them with an ultrahigh specific surface and makes them more sensitive to environmental changes. In the early research on GQDs, tremendous effort was devoted to developing methods for the preparation of GQDs and exploring their properties [5–8]. Their application in the analytical field has not been explored until very recently. Due to their novel properties, sensors based on GQDs can achieve a high level of performance [9–14]. Furthermore, GQDs increase contact area with the analyte,

which could increase the electrochemical active surface area to interact with some electroactive analytes. Since geometric surface area is a very important parameter in electrochemistry, modification of different substrates (such as glass, carbon, graphite, etc.) by GQDs can increase the rate of electrochemical reaction.

Due to these unique properties of GQDs, this study aimed at the easy bottom-up method for preparation of this material using pyrolyzing of citric acid and dispersing the carbonized products into alkaline solutions. Following, GQDs were applied for sensitive detection and determination of doxorubicin (DOX) as antineoplastic drug. The current study was an attempt to develop the application of low toxic materials for construction of electrochemical sensors and also aimed at low potential detection of analytes. To the best of our knowledge, this is the first report on the application of GQD as low toxic material toward low potential detection of analytes (DOX as a model analyte).

2. Experimental details

2.1. Chemicals and reagents

DOX was purchased from Exir Nano Sina Company (Tehran, Iran). The stock solution of DOX was prepared by dissolving an accurate

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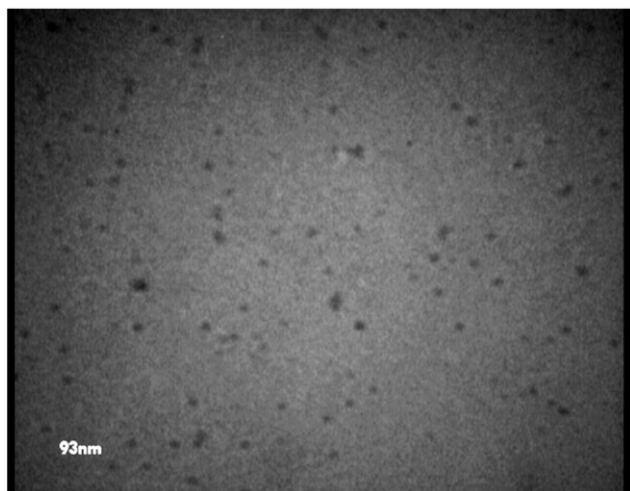


Fig. 1. TEM image of GQDs.

amount of DOX in an appropriate volume of 0.02 M phosphate buffer solution (PBS), pH = 4.0 (which was also used as supporting electrolyte).

Human plasma samples were obtained from the Iranian Blood Transfusion Research Center (Tabriz, Iran) and aliquots were transferred into microtubes and frozen at $-4\text{ }^{\circ}\text{C}$ until analysis. For extraction of plasma proteins, 2 mL of an aliquot volume of this sample was spiked with DOX, then acetonitrile with the volume ratio of 2:1 (acetonitrile:plasma) was added to precipitate plasma proteins. The mixture was centrifuged for 10 min at 6000 rpm to separate residues of plasma proteins residues.

2.2. Apparatuses

The transmission electron microscope (TEM) images were obtained on Leo 906, Zeiss (Germany). Electrochemical measurements were performed using a potentiostat/galvanostat AUTOLAB system with PGSTAT302N boards (Eco Chemie, Utrecht, Netherlands) at room temperature, using a standard electrochemical cell with three-electrodes. The working electrode was carbon paste electrode purchased from Azar electrode (Urmia, Iran). A platinum (Pt) wire was used as counter electrode and Ag/AgCl was the reference electrode. The system was controlled by a PC with Nova 1.7 operating software. The transmission electron microscope (TEM) images were obtained on Leo 906, Zeiss (Germany).

2.3. Synthesis and characterization of GQD

The GQDs were prepared by directly pyrolyzing CA [15]. In a typical procedure of GQDs preparation, 2 g CA was put into a 5 mL beaker and heated to $200\text{ }^{\circ}\text{C}$ using a heating mantle. About 5 min later, the CA was liquated. Subsequently, the color of the liquid was changed from colorless to pale yellow, and then orange in 30 min, implying the formation of GQDs. If the heating was kept on, the orange liquid would finally turn to black solid in about 2 h, suggesting the formation of GO. The obtained orange liquid for preparing GQDs was added drop by drop into 100 mL of 10 mg/mL NaOH solution, under vigorous stirring. After neutralized to pH = 7.0 with NaOH, the aqueous solution of GQDs was obtained. The black solid was dissolved with 50 mL of 10 mg mL^{-1} NaOH solution, and further neutralized with the same concentration of NaOH, resulting the aqueous solution of GO.

Fig. 1 presents the TEM image of GQDs and confirms that they have nearly spherical shape. Ninety percent of the particles represent dark

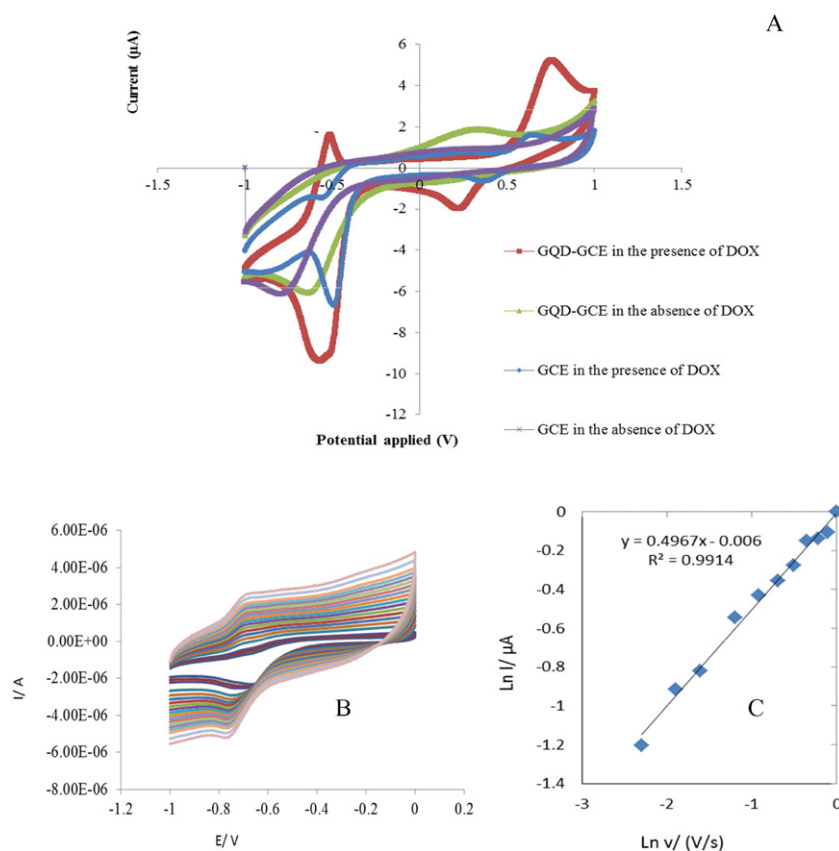


Fig. 2. (A) Cyclic voltammograms of bare GCE and GQD-GCE in the absence and presence of DOX (0.18 mM) in PBS (pH = 4.0). Potential sweep rate of 100 mV s^{-1} . (B) CVs of GQD-GCE in the presence of 0.1 M PBS (pH = 4.0) and $8\text{ }\mu\text{M}$ of DOX in different scan rates ($20\text{--}1000\text{ mV s}^{-1}$); (C) Dependence of anodic peak current on the potential scan rate in Neperian logarithm coordinates in the presence of 0.1 M PBS (pH = 4.0) at GQD-GCE.

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