



Analytical Methods

Sensitive determination of quinoline yellow using poly (diallyldimethylammonium chloride) functionalized reduced graphene oxide modified grassy carbon electrode



Li Fu^b, Yuhong Zheng^b, Aiwu Wang^c, Wen Cai^d, Haitao Lin^{a,*}

^a Yunnan University of TCM, Kunming City, Yunnan 650500, PR China

^b Institute of Botany, Jiangsu Province and Chinese Academy of Sciences, Nanjing Botanical Garden, Mem. Sun Yat-Sen, Nanjing 210014, PR China

^c Department of Physics and Materials Science and Centre for Functional Photonics (CFP), City University of Hong Kong, 88 Tat Chee Avenue, Kowloon, Hong Kong Special Administrative Region

^d Center of Super-Diamond and Advanced Films (COSDAF) and Department of Physics and Materials Science, City University of Hong Kong, Hong Kong Special Administrative Region

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ABSTRACT

A novel poly (diallyldimethylammonium chloride) functionalized reduced graphene oxide (PDDA-RGO) nanocomposite modified electrode was fabricated, and applied for the electrochemical determination of quinoline yellow (QY). The formation of PDDA-RGO nanocomposite was confirmed by SEM, FTIR and Raman spectroscopy. The electrocatalytic activity of PDDA-RGO nanocomposite to the oxidation of QY was evaluated using cyclic voltammetry and differential pulse voltammetry. Under the optimal conditions, the proposed sensor exhibited excellent electrochemical performance towards detection of QY. The linear range is from 0.01 to 10 μM , and the detection limit is down to 0.002 μM ($S/N = 3$). The proposed sensor also exhibited excellent anti-interference property, repeatability and stability. In addition, the proposed electrochemical sensor was successfully applied to determination of QY in soft drink.

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1. Introduction

Quinoline yellow (QY) is a water soluble acid dye which has been widely used in coloring fibers, leather, paper, agrochemicals, fertilizers, detergents, ink and food industry (Capitán-Vallvey, Valencia, & Nicolas, 2000; Khanavi et al., 2012). However, studies have shown that QY has detrimental effects on children's behavior (Macioszek & Kononowicz, 2004; Mekkawy, Ali, & El-Zawahry, 1998). Recently, the human lymphocytes in vitro study also confirmed that the QY is a genotoxic material (Macioszek & Kononowicz, 2004). Therefore, there is a great need to develop a sensitive, quick and reliable method for determining QY in different type of samples. Up to now, several analytical methods have been developed for QY detection, including spectrophotometric method (Capitan-Vallvey, Valencia, & Nicolas, 2000; Ozgur & Koyuncu, 2002), spectrofluorometry (Shahabadi, Maghsudi, & Rouhani, 2012), high-performance liquid chromatography (HPLC) (García-Falcón & Simal-Gándara, 2005; Khanavi et al., 2012; Oka

et al., 2003a, 2003b; Zou, He, Yasen, & Li, 2013) and UV–vis detector (Oka et al., 2003a, 2003b). Although these methods are capable for detecting QY, but they have their own drawbacks. For example, HPLC is time-consuming and required skilled operators; spectrophotometric method is not very sensitive and easily suffers from the interference species. In order to avoid these problems, electrochemical methods have attracted more attentions due to its sensitivity, accuracy, lower cost and simplicity (Choi, 2014; Gao, Wang, Yang, Sun, & Zhao, 2014; Ghorbani, Hosseinzadeh, Pashaei, & Hosseinzadeh, 2014; Ismail & Mohamed, 2014; Piech, Rumin, & Paczosa-Bator, 2014; Rouhani & Haji-ghasemi, 2009; Sabela, Kanchi, Ayyappa, & Bisetty, 2014; Sakac, Regusic, Sak-Bosnar, Jozanovic, & Breslauer, 2014; Zhang, Shi, & Wang, 2015). For example, hanging mercury drop electrode was employed for determining QY and the result showed a low detection limit of 0.05 μM (Nevado, Flores, & Llerena, 1997). However, the mercury electrode is toxic which limited its applications. Therefore, glassy carbon electrode (GCE) with surface modification has been chosen as an alternative electrode for determining QY. For example, Zhao and co-workers fabricated a multi-walled carbon nanotubes modified GCE for QY determination. The results display a linear

* Corresponding author.

E-mail address: haitaolinutcm@yahoo.com (H. Lin).

detection range from 1.29 to 34.5 μM with a limit of detection of 0.86 μM (Zhao, Zhang, Wu, Chen, & Zhou, 2011). Gao and co-workers demonstrated a polypyrrole/single-walled carbon nanotubes composites modified GCE for GY determination. The results indicate the proposed sensor has a linear range from 0.8 to 100 μM with a detection limit of 0.08 μM (Gao et al., 2014). Obviously, the fabrication of novel electrode modified materials with quick response, excellent conductivity, good sensitivity and high stability remains a challenge.

Graphene has attracted a great deal of interest since it has been discovered in 2004 due to its extraordinary properties, such as excellent electronic conductivity, large specific surface area and enhanced electrocatalytic activity (Novoselov et al., 2004). Therefore, graphene is considered as an excellent candidate for electrode surface modification for specific target molecule detection. However, most of the graphene used in the modification process is in the reduced form of graphene oxide (GO). The reduced graphene oxide (RGO) is prone to irreversible spontaneous agglomeration and lowers the performance of modified electrode. In order to overcome this problem, poly (diallyldimethylammonium chloride) (PDDA) has been studied for functionalizing GO to a solution-processable RGO (Xu et al., 2014).

In this work, PDDA functionalized graphene nanocomposite (PDDA-RGO) was synthesized via a simple wet chemical method and used for construction of PDDA-RGO modified GCE. The result PDDA-RGO/GCE was used for the detection of QY. Several factors affecting the electrocatalytic performances of the proposed QY sensor were also further optimized. We also studied the analytical applications of the sensor for determining QY in soft drinks.

2. Experimental

2.1. Reagents

Quinoline yellow, synthetic graphite (average particle diameter $<20\ \mu\text{m}$), poly (diallyldimethylammonium chloride) (20 wt% in water) (PDDA) and hydrazine hydrate (N_2H_4 50–60% in water) were purchased from Sigma–Aldrich. Other chemicals were of analytical reagent grade and used without further purification. The used water was doubly distilled.

2.2. Synthesis of PDDA-RGO nanocomposite

Graphene oxide (GO) was prepared with the modified Hummers method with little modification (Gan & Hu, 2011; Hummers & Offeman, 1958). PDDA-RGO composite was prepared by a simple wet chemical method according to the literatures with some modifications (Xu et al., 2014; Xue et al., 2011). Briefly, 5 mg GO was dispersed in 10 mL water and then 2 mL PDDA (0.5 wt%) was added. The aggradation of dispersion was resolved by 10 min sonication. Then 0.1 mL of N_2H_4 was added into dispersion, and the mixture was heated to 90 $^\circ\text{C}$ for 30 min. After cool down, the mixture was centrifuged three times followed by water washing process for removing excess PDDA and N_2H_4 . Finally, the PDDA-RGO was re-dispersed into 10 mL water to obtain 0.5 mg/mL PDDA-RGO. RGO without PDDA functionalization was also prepared using a similar method except adding PDDA.

2.3. Characterizations

The morphology of as-synthesized nanocomposite was observed using a ZEISS, SUPRA 55 field emission scanning electron microscopy (FESEM) measurements. Fourier transform infrared spectroscopy (FTIR, Nicolet iS5, Thermo Scientific, USA) was recorded on a Bruker Vertex 70 spectrometer ($2\ \text{cm}^{-1}$). Raman

spectroscopy was performed at room temperature using a Raman Microprobe (Renishaw RM1000) with 514 nm laser light.

2.4. Electrode preparation

A glassy carbon electrode (GCE, diameter of 3 mm) was successively polished with 0.3 and 0.05 μm Al_2O_3 slurry on an abrasive cloth and thoroughly cleaned with ethanol and distilled water under ultrasonication. For the electrode surface modification, a certain amount of catalyst dispersion (1 mg/mL) was dropped onto the GCE and dried at room temperature. Electrochemical measurements were performed on a CHI660D electrochemical workstation (USA), using a three electrode system. A platinum wire was used as the auxiliary electrode and an Ag/AgCl (3 M KCl) as the reference electrode. 0.1 M PBS at pH of 7 was used as supporting electrolyte for the determination of QY. After 1-min accumulation, the differential pulse voltammograms were recorded from 0.3 to 1.1 V, and the oxidation peak current at 0.72 V was measured for QY. The pulse amplitude was 50 mV, the pulse width was 30 ms and the scan rate was 50 mV/s.

3. Results and discussion

3.1. Characterization of PDDA-RGO nanocomposite

The surface morphology of the synthesized modifiers was observed using SEM. Fig. 1A and B showed the typical top-view SEM images of RGO and PDDA-RGO nanocomposite. As shown in the figure, the RGO displayed a stack layered morphology due to the Van der Waals interactions. In contrast, PDDA-RGO nanocomposite showed a well dispersed morphology with some wrinkles. Therefore, the surface functionalization of RGO with PDDA is an effective route for preventing agglomeration and could retain a large surface area of RGO.

The reduction and PDDA surface functionalization processes were confirmed using FTIR. Fig. 1C showed the FTIR spectra of GO, RGO and PDDA-RGO nanocomposite. As expected, the spectrum of GO showed peaks at 1720, 1592, 1412 and 1025 cm^{-1} , which are assigned to the C=O stretching of COOH groups, C=C vibrations, C–OH stretching vibrations and C–O vibrations from alkoxy groups, respectively (Ahmad et al., 2013; Li et al., 2013; Zhang et al., 2010a). After hydrazine reduction process, the intensity of these characteristic peaks significantly decreased or even vanished in the both RGO and PDDA-RGO nanocomposite, which indicates that GO has been successfully reduced. Meanwhile, two extra peaks are observed in the spectrum of PDDA-RGO nanocomposite at 844 and 1504 cm^{-1} , which are attributable to the N–C bond of PDDA, indicating the successful surface functionalization process (Fu, Zheng, Ren, Wang, & Deng, 2015b; Fu et al., 2015c; Wang, Yu, Dai, Chang, & Baek, 2011).

Fig. 1D displayed the Raman spectra of GO, RGO and PDDA-RGO. Three samples all displayed two characteristic bands at ~ 1570 and $\sim 1340\ \text{cm}^{-1}$, corresponding to the graphite (G band, first-order scattering of E_{2g} phonons by sp^2 carbon atoms) and diamondoid (D band, breathing mode of κ -point phonons of A_{1g} symmetry) bands, respectively (Fan et al., 2010; Fu, Cai, Wang, & Zheng, 2015; Fu & Fu, 2015; Fu, Wang, Zheng, Cai, & Fu, 2015a). The intensity ratio between D band and G band (I_D/I_G) increased clearly from GO to RGO, further confirmed the reduction process due to a decrease of sp^2 domain induced by the reduction (Zhang, Zhou, & Zhao, 2010b). Furthermore, the G band of PDDA-RGO nanocomposite showed a small shift from 1570 to 1588 cm^{-1} . This small shift can be ascribe to the electron transfer from RGO to the adsorbed PDDA (Hsiao et al., 2010; Qi et al., 2010; Rao, Eklund, Bandow, Thess, & Smalley, 1997; Wang et al., 2011).

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