Fast graphene preparation method by electrochemical exfoliation of natural graphite flakes

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Abstract

A simple and fast procedure to exfoliate natural graphite under galvanostatic condition is described.

Introduction

Graphene, a single layer of sp² carbon atoms, exhibits excellent physical and chemical properties such as high surface area, outstanding conductivity and high mechanical strength¹.

Graphene has been obtained by different methods including micromechanics cleavage, epitaxial growth of silicon carbide or metal, chemical vapor deposition (CVD), thermal exfoliation, chemical reduction of graphene oxide (GO) and electrochemical process². This work aims to propose a new electrochemical procedure to exfoliate natural graphite flakes under galvanostatic conditions to obtain graphene and graphene oxide (GO).

Results and Discussion

The electrochemical exfoliation was performed under galvanostatic condition in electrolyte solution containing 100 mmol L⁻¹ phosphate buffer, pH 9.2 and 100 mmol L⁻¹ Na₂SO₄. Graphite flakes 9 mm long (Nacional de Grafite Co.) were used as working electrode (anode) and a Pt sheet as the counter electrode. The current applied in the cell was ranged between 10 and 50 mA for 30 minutes. The exfoliated material was characterized by Raman spectroscopy (Fig.1)



Figure 1. Raman spectra of graphite (A), GO (B) and rGO (C) obtained by electrochemical exfoliation.

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The typical D (1338 cm⁻¹), G (1595 cm⁻¹) and 2D (2650 - 2690 cm⁻¹) graphitic bands are present in the spectra of the exfoliated samples. We observed that exfoliated material characteristics depend on the current applied intensity. GO is mainly produced at current values below 20 mA while graphene few layers (< 5) are obtained with higher current values. produced The GO by this method was electrochemically reduced at a glassy carbon (GC) electrode and its performance was evaluated for NO2⁻ detection. Figure 2 shows the voltammetric response of the bare GC electrode (A) and the modified GC electrode for NO2⁻ detection in 100 mmol L⁻¹ acetate buffer solution, pH 4.8.



Figure 2. Cyclic voltammograms of NO_2^- in acetate buffer solution, pH 4.8 recorded with bare GC electrode (A) and modified CG electrode (B). [NO_2^-]: (a) 0.0; (b) 0.5; (c) 1.0; (d) 2.0; (e) 4.0; and (f) 6.0 mmol.L⁻¹; Scan rate: 20 mV s⁻¹.

The voltammetric measurements indicate that the modified electrode shows better performance than bare GC electrode for NO₂⁻ detection, anticipating its oxidation potential and improving its analytical sensitivity.

Conclusions

The electrochemical exfoliation method proposed is fast and simple to produce GO and rGO and its nanomaterial shows good performance for electroanalytical applications.

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¹ Harima, Y.; Setodoi, S.; Imae, I.; Komaguchi, K.; Ooyama, Y.; Ohshita, J.; Mizotab, H.; Yano, J.; *Electrochimica Acta*, **2011**, 56, 5363. ² Xiong, Z.; DaCheng, Z.; Yao, C.; Xianzhong, S.; Yanwei, M.; *Chin Sci Bull*, **2012**, 57, 3045.