



Optimization Study of Graphene Oxide Synthesis with Improvement of C/O Ratio†

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The present study was aimed to clarify detailed conditions of vigorous solid-state reaction as conventional method for graphene oxide. For optimization study, the effects of an initial concentration of KMnO_4 (0.75-7.5 % w/v), reaction temperature (0-120 °C) and an incubation time (1-48 h) were investigated. The obtained graphene oxide was characterized by Fourier transform infrared spectroscopy, X-ray diffraction spectroscopy, energy dispersive X-ray spectroscopy and scanning electron microscope (SEM) techniques. From the results, the optimum conditions for graphene oxide production were consisted of 4.5 % w/v KMnO_4 at 80 °C for 6 h. IR spectrum showed the characteristic peaks at wave number (cm^{-1}) of 3367 (O-H), 1719 (C=O), 1224 and 1049 (C-O). Energy dispersive X-ray spectroscopy of the graphene oxide revealed the improved C/O ratio of 1.10, indicating more polar functional groups bound on the surface of graphene oxide due to the increasing oxidation by MnO_4^- . The amorphous graphene oxide was obtained as confirmed by XRD pattern. Additionally, SEM image showed drastically differences in the surfaces between graphene oxide and its graphite powder, depicting a graphene oxide's smooth surface compared with that rough multilayer graphite powder. It could, thus, be gained a crucial factor affecting the graphene oxide synthesis from graphite powder under the optimized method.

Keywords: Graphite powder, Graphene oxide, Energy dispersive X-ray spectroscopy, Scanning electron microscope.

INTRODUCTION

Graphene oxide (GO) is a water-soluble nanomaterial prepared through extensive chemical attack of graphite to introduce oxygen-containing defects in the graphite stack. Graphene oxide sheets are composed of planar, graphene-like aromatic domains of random sizes interconnected by a network of cyclohexane like units in chair conformation which are decorated by hydroxyl, epoxy and carbonyl groups. These functional groups in graphene oxide impart water solubility to the individual sheets and removal of such groups resulted in flocculation and precipitation¹.

Functionalization of graphene oxide can fundamentally change their chemical and physical properties. The resulting chemically modified graphene oxide could then potentially become much more adaptable for almost limitless applications. There are many ways in which graphene oxide can be functionalized, depending on the desired application and amount of functional group on the surface of graphene oxide. For instance, the functionalizations of graphene oxide with polyhedral oligomeric silsesquioxane², 1-ethyl-3-(3-dimethyl

aminopropyl) carbodiimide and N-hydroxysuccinimide³, *p*-phenylene diamine⁴, N-(trimethoxysilylpropyl)ethylenediamine triacetic acid⁵, ethylenediamine⁶, 9-(4-aminophenyl) acridine⁷ and 2-amino-4,6-didodecylamine-no-1,3,5-triazine⁸ were reported.

Currently, graphene oxide preparation is relied on Hummers' method using KMnO_4 , NaNO_3 and H_2SO_4 under vigorous solid-state reaction. The C/O ratio obtaining from this method is substantially high. However, an optimum condition for higher C/O ratio of the graphene oxide is still limited. In this work, we reported an improvement of C/O ratio in graphene oxide by an optimization condition of the Hummers' method⁹.

EXPERIMENTAL

Sodium nitrate (NaNO_3), sulfuric acid (H_2SO_4 , 98 %) and hydrogen peroxide (H_2O_2 , 30 %) were purchased from Ajax FineChem Pty Ltd. Potassium permanganate (KMnO_4) and graphite powder (< 20 μm , synthetic) were purchased from Carlo Erba and Sigma-Aldrich, respectively. All chemicals were used as received without further purification.

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