



Green preparation of reduced graphene oxide using a natural reducing agent

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Abstract

A simple and efficient method was introduced for the high-conversion preparation of graphene oxide (GO) from large graphite flakes (average flake size = 100 μm) using a simplified Hummer's method. Natural reducing agents such as lemon juice and vinegar were compared with hydrazine (N_2H_4) as potential reducing agents. Graphene was prepared by chemical reduction of GO because this method was low cost and could be used for large-scale graphene production. This one-pot graphene preparation was performed at room temperature. Different degrees of oxidation of graphite flakes were obtained by stirring graphite in a mixture of sulfuric acid and potassium permanganate at different oxidation times, and highly exfoliated GO sheets were produced. GO was subsequently reduced effectively by lemon juice, a new, green, and potential reducing agent with pH 2.3. This reduced GO exhibited a high electrical conductance of 24.6 μS attributed to its higher C/O ratio ($\approx 8:2$) compared with other samples. © 2015 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

Keywords: Natural reducing agent; Chemical reduction; One-pot preparation

1. Introduction

Malaysia weather is currently hotter than in previous years because of global warming attributed to several types of pollution induced by fossil fuel consumption. Consuming fossil fuels emits different compounds, such as sulfur dioxide, nitrogen oxides, ground-level ozone, particulate matter, carbon monoxide, carbon dioxide (CO_2), and volatile organic compounds that include benzene, certain heavy metals, and a number of other pollutants. These side products from fossil fuel combustion behave as an insulation layer that hinders heat from dissipating the surface of the Earth. According to the annual temperature anomaly simulated by the Providing Regional Climates for Impacts Studies, temperature is predicted to increase in future years; Table 1 summarizes the results of the annual temperature anomaly [1]. Therefore, green and renewable energy should be developed to address this potential problem.

Dye-sensitized solar cells (DSSCs) have emerged to global attention because of their low fabrication cost, high energy

conversion efficiency, and environmental friendliness. Nevertheless, the use of graphene in DSSCs could enhance their performance in energy conversion because of its excellent optical and electrical characteristics [2]. Certain researchers have reported a photoelectrical conversion efficiency of 7.02% by using a TiO_2 and graphene composite photo-electrode [3,4]. Thus, highly pure and highly conductive graphene should be synthesized in bulk for large-scale industrial production.

Graphene, which is a versatile two-dimensional (2D) material with sp^2 honeycomb lattice-structured C atoms, has attracted enormous global attention because of its unique characteristics. Several scientists and researchers have shown keen interest in the one C atom-thick graphene, and various solar-based studies have been performed because of the high conductivity and transparency of graphene. This material allows light to penetrate, and a universal 2.3% linear optical adsorption can be achieved by pristine graphene. Novel graphene sheets have significantly affected areas in modern chemistry, physics, material science, and engineering. Numerous efforts to obtain highly pure and highly conductive graphene have been embarked from various perspectives. Some of the typical methods used to synthesize graphene include chemical vapor deposition (CVD) [5–7], micromechanical graphite exfoliation [8,9], epitaxial growth on electrically insulated surface [10,11], and production of colloidal

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suspensions [12,13]. Decomposition of alcohol on a Cu surface is an example of CVD graphene synthesis [14]. Meanwhile, micromechanical exfoliation [15] is performed by peeling off using a Scotch tape, and epitaxial growth [16] involves growing graphene on electrically insulating surfaces, such as SiC. Colloidal suspensions [13] can be produced by dispersing GO in aqueous and various organic solvents. Table 2 shows the comparison of the aforementioned techniques.

However, industries demand the most economical and simple but most effective way of large-scale graphene synthesis. Thus, this work synthesized graphene by chemical exfoliation through a simplified Hummer's method [17]. Bulk graphite powder is oxidized using strong oxidizing agents, such as potassium permanganate (KMnO_4), to introduce oxygenated functional groups into the graphite structure. Oxygenated functional groups are introduced into the graphite structure to weaken the interlayer van der Waals forces. Reduction is then performed. The simplified Hummer's method does not use phosphoric acid and sodium nitrate; these compounds release harmful gas [18]. Lemon juice, vinegar, and N_2H_4 were used as potential reducing agents,

and their effects were compared to select the compound that produced high-quality graphene. Both GO and reduced GO (rGO) were characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffractometry (XRD), Raman spectroscopy, energy dispersive X-ray spectroscopy (EDX), tabletop scanning electron microscopy (SEM), and field emission-scanning electron microscopy (FE-SEM).

2. Methodology

2.1. GO synthesis

GO was synthesized via simplified Hummer's method. Approximately 3 g of graphite (graphite flakes, Sigma-Aldrich) was mixed with 70 mL of 0.5 M H_2SO_4 (Chemolab) in an ice bath. Exactly 9 g of KMnO_4 (Chemolab) was slowly added into the mixture, which was then stirred at a constant speed. The temperature of the suspension was maintained below 20 °C to avoid possible explosion. The temperature was then raised to 35 °C and stirred for 30 min after KMnO_4 was completely added in the mixture. Approximately 150 mL of deionized (DI) water was then added, and the temperature was raised to 95 °C. Approximately 500 mL of water and 15 mL of 30% hydrogen peroxide (Chemolab) were added to the suspension to terminate the reaction. The suspension was then washed with 10 mL of 1 M hydrochloric acid (Chemolab) and centrifuged at 7000 rpm for 15 min. The supernatant was decanted; the sediment was washed with DI water and centrifuged again. Washing was repeated twice; it was performed to remove the metal ions [18]. Reduction was then conducted. Lemon juice and vinegar were used as reducing agents to compare the effects of these environment-friendly reducing agents with N_2H_4 . Fig. 1 shows the experimental setup.

2.2. Characterization

The surface morphologies of GO and rGO were observed using an FEI Quanta 200F Environmental SEM at 5.0 kV with 10 mm working distance and a TM3030 tabletop SEM at

Table 1
Annual temperature anomaly simulated by the Providing Regional Climates for Impacts Studies [1].

Peninsular Malaysia	
Year	Temperature anomaly (°C)
2000	0.5
2007	0.6
2014	0.8
2021	0.9
2028	1.0
2035	1.2
2042	1.4
2049	1.9
2056	1.9
2063	2.3
2070	2.6
2077	3.0
2084	3.2
2091	3.4
2098	3.6

Table 2
Comparison of graphene synthesis techniques.

Synthesis technique	Processing steps	Advantages	Disadvantages	Year	Refs.
CVD	Requires precise parameter control	Can achieve single to few layers of graphene	Low productivity, difficult to achieve single-layer graphene, costly machine, lack of homogeneity on large areas	2010, 2013	[2–4]
Micromechanical exfoliation	Simple tool (scotch tape)	Able to obtain single-layer graphene	Hard to transfer and scale	2011, 2013	[5,6]
Epitaxial growth	Involves many machineries and tools, complicated procedures	Fairly good quality	Not transferable	2012	[7,8]
Colloidal suspension	Simple procedures	Wide variety of organic solvents can be used	High impurity	2009, 2010	[9,10]
Reduction of exfoliated graphene oxide	Simple procedures	Can be produced in bulk, simple procedure, transferable, scalable	Quality not as good as CVD-produced graphene	2013	[14,15]

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