

Green synthesis of dimension-controlled silver nanoparticle–graphene oxide with in situ ultrasonication

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Abstract

A green chemical approach to control the dimensions of Ag nanoparticle-decorated graphene oxide (AgNP–GO) composites was proposed by in situ ultrasonication of a mixture of AgNO₃ and GO solution with the assistance of vitamin C acting as an environmentally friendly reducing agent at room temperature. The AgNP–GO composites were characterized by X-ray diffraction, transmission electron microscopy, energy-dispersive spectroscopy, Fourier transform infrared spectroscopy, Raman spectra and ultraviolet–visible absorption spectra. The results demonstrated that Ag nanoparticles with an average diameter of ~15 nm were uniformly dispersed on the surface of GO nanosheets by in situ ultrasonication of 1 min with vitamin C. Increasing the ultrasonication times resulted in Ag nanoparticles with tunable dimensions ranging from 15 to 55 nm being formed on the surface of GO nanosheets. The amount of silver nitrate and the ultrasonication time play a key role in the control of the dimension of Ag nanoparticles on GO, and a formation mechanism of the as-prepared AgNP–GO composites is proposed. This study provides a guide to controlling the dimensions of AgNP–GO composites, which may hold promise as advanced materials for various analytical applications such as catalysis, sensors and microchips.

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1. Introduction

For a decade, the development of graphene has received considerable attention because it possesses unique properties, such as a two-dimensional extended honeycomb network of *sp*² hybridized carbon atoms [1–3], high electron mobility (200,000 cm² V⁻¹ s⁻¹) [4], a large theoretical

specific surface area (2630 m² g⁻¹) [5], excellent mechanical, chemical and thermal properties [6] and high electrical conductivity (10³–10⁴ S m⁻¹) [7]. Based on these unique properties, graphene has become a promising advanced building material for potential applications in many technological fields, including nanoelectronics [8,9], nanocomposites [10,11], energy storage devices [12,13], transparent electrodes for displays [14,15] and solar cells [16,17]. Yet the superb properties of graphene have been significantly improved by exploiting the hybridization of graphene, which is an important step in the development of

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multifunctional materials with synergistic properties [18–27]. In particular, noble metal nanoparticles (Au, Pt, Pd, etc.) on graphene composites have attracted tremendous interest because of the novel optical, electronic, mechanical and catalytic properties of the resulting materials [28–31]. Therefore, their fabrication has become a topic of scientific and technological importance. In addition, the dispersion of metal nanoparticles on graphene sheets potentially provides a new way to develop catalytic [32,33], magnetic [34,35] and optoelectronic materials [36,37]. Among them, Ag nanoparticle (AgNP)-decorated graphene has been demonstrated to be the most promising material for optoelectronics [38,39], catalysis [40,41] and electrochemistry [42]. However, challenges in the fabrication of metal nanoparticle/graphene composites remain. For example, the use of surfactant or stabilizing agent molecules, which are introduced and strongly adsorbed on the surface of metal nanoparticles, result in a discrepancy in the performance of metal nanoparticle/graphene composites [43–45]. In addition, AgNP–graphene composites are usually obtained from the in situ reduction of silver precursors with hazardous reducing agents such as sodium borohydride, formaldehyde and hydrazine on the preformed graphene or decoration graphene with pre-fabricated AgNPs, which involves multi-steps and requires complex manipulation [18,46,47]. Accordingly, the syntheses of metal nanoparticle–graphene oxide (GO) nanocomposites employing GO as a starting precursor has been attracting a great deal of attention. This is because GO consists of oxidized graphene sheets that have their basal planes mostly decorated with hydroxyl, epoxide, carbonyl and carboxyl groups [48], which allows AgNPs to interact with the GO sheets through physisorption, electrostatic binding or through charge-transfer interactions [49]. In particular, carboxylic moieties on a GO surface can stabilize the silver cations that can directly attach to the carboxyl groups. Thus, the reduction takes place on the surface of the exfoliated sheets, giving one-step formation of Ag nanoparticles attached to the exfoliated GO sheets without the need for functionalization [18]. Fernandez-Merino et al. [50] have investigated the deoxygenation efficiency of GO suspensions by vitamin C at 95 °C and concluded that vitamin C can compete in terms of reducing ability with the widely employed, but highly toxic, reducing agent hydrazine used for graphene synthesis. Thus far, the efforts have been focused on the effective performance of the metal nanoparticle–GO or –graphene composites [38–42]. However, few studies have comprehensively investigated the effects of synthesis parameters on controlling the dimension and morphology of metal nanoparticle–GO composites. To resolve this gap, we propose a fast, green, low-cost, large-scale and environmentally friendly preparation of dimension-controlled Ag nanoparticle-decorated graphene oxide (AgNP–GO) composites at room temperature. Herein, the AgNP–GO was prepared by in situ ultrasonication at room temperature with the assistance of vitamin C (ascorbic acid), a natural antioxidant essential for many

metabolic functions in living organisms [51], which acts as a nontoxic reducing agent. The AgNP–GO composites were characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), energy-dispersive spectroscopy, Fourier transform infrared (FT-IR) spectroscopy, Raman spectra and ultraviolet–visible (UV–vis) absorption spectra. The possible formation mechanism of the dimension-controlled AgNP–GO is proposed. Composites were prepared at various concentrations of silver precursors (AgNO_3) under different ultrasonication times in order to explore the influence of ultrasonic waves on the dimension and morphology of the AgNP–GO composites.

2. Experimental

2.1. Materials

Graphite powders were obtained from Unichem Chemical Reagent Co. Ltd. (South Korea). AgNO_3 , NaNO_3 and vitamin C were provided by Guangdong Guanghua Sci-Tech Co. Ltd. (China). All the other chemicals and reagents were of analytical grade and purchased from Sigma–Aldrich unless otherwise stated. All solutions were prepared using ultrapure water (Millipore, 18.2 M Ω cm). A Buehler sonic cleaner (UltraMet 2005) was used for the ultrasound source. The ultrasonic frequency used was 20 kHz \pm 50 Hz.

2.2. Synthesis of AgNP–GO composites

Graphite oxides were prepared by a modified Hummers' procedure as previously reported [52,53] and exfoliated into GO nanosheets by ultrasonication for 1 h. AgNP–GO composites were prepared by mixing AgNO_3 aqueous solution at various concentrations of 0.1, 0, and 0.6 M, and 50 ml of GO suspensions (0.4 mg ml $^{-1}$) under a sonication bath for 30 min. Afterwards, vitamin C (5 mM) was added slowly to the reaction mixture under continuous ultrasonication for another 30 min. The suspension was then washed with deionized water three times and dried at 60 °C for 12 h to obtain the as-prepared AgNP–GO composites. In addition, in order to investigate the effect of an ultrasonic wave on the morphology of the AgNP–GO composites, composite samples with 0.1 M AgNO_3 were prepared with an analogous experimental process, but the ultrasonication time was adjusted for 1, 5, 10, 20 and 30 min, respectively. Table 1 summarizes the sample information at various experimental conditions, corresponding to the names of the samples.

2.3. Characterization

The morphology and nanostructure of the prepared samples were observed using Philips Tecnai 12 BioTWIN TEM operated at 120.0 kV. The TEM samples were prepared under ambient conditions by depositing droplets of the aqueous suspensions onto carbon films supported by

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