



Size separation of mechanically exfoliated graphene sheets by electrophoresis

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ARTICLE INFO

Article history:

Received 1 August 2017

Received in revised form

17 November 2017

Accepted 18 November 2017

Available online 21 November 2017

Keywords:

Size separation

Mechanically exfoliated

Graphene

Electrophoresis

ABSTRACT

A narrow size distribution of graphene sheets is critical to their application. Although several strategies have been demonstrated to classify the polydisperse graphene oxide (GO), a precursor of graphene sheets, into various specified monodisperse fractions, few reports have revealed the size separation of graphene sheets straightly. Herein, a straightforward and efficient approach for size separation of mechanically exfoliated graphene sheets is proposed. The original graphene sheets (S0) have been separated into two portions with smaller (S1) or larger (S2) lateral dimensions in a weak alkaline buffer via electrophoresis, based on the zeta potential of graphene colloid under different pH values. It was found that the electrophoresis velocities of graphene colloids increase when their diameters decrease. In this way, graphene sheets of different sizes and properties can be separated successfully. Further study on the as-separated components indicated that small-size graphene sheets (S1) display blue shift signals in both ultraviolet–visible and Raman spectroscopy, as well as a distinct fluorescent compared with large-size (S2) and original ones (S0). As electrode materials, S2 had a faster charge transfer and higher specific capacitance than that of S1 and S0. The feasible and effective method provided in this work to classify mechanically exfoliated graphene sheets are potentially beneficial to the broad applications of graphene.

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1. Introduction

Since its isolation in 2004, graphene has been the focus of numerous studies [1–5]. Typical synthesis routes of graphene include mechanical exfoliation [6,7], chemical vapor deposition (CVD) [8–11], redox reactions [12–15], and epitaxial growth [16,17], etc. However, regardless of the selected method, how to control the size of synthesized graphene sheets remains a challenge. In many cases, the obtained products are mixtures of sheets of varying sizes. Since graphene's performance is directly related to its size parameters, graphene sheets of varying size can be used in a range of fields. For instance, the nanometre-sized graphene can be used as quantum dot [18,19], giant graphene oxide sheets can be assembled into ultrastrong fibers [20], and other sizes graphene were used in supercapacitors [21], solar cells [22], sensors [23], photocatalysts [24], electrochemical catalysis [25]. Therefore, a highly

accurate separation and control for graphene with uniform sizes can significantly impact a variety of fields.

Currently, centrifugation is one of the effective size separation methods for graphene and its derivatives [26–28]. More specifically, sheets with large lateral size tend to move below the medium layer. However this method exhibits limitations such as a highly sophisticated medium preparation and a low yield. In fact, most of the reported work on the separation of graphene has been focusing on the graphene oxide (GO), a precursor of graphene, on which carries abundant functional groups, e.g., carboxyl and hydroxyl. For example, filtering separation [29] and pH-assisted selective sedimentation [30] are effective to separate GO. More importantly, the electrophoresis method [31,32] was reported to be a good technique for size separation of GO, based on the theory that the sufficient functional groups on their sheet surface tend to be ionized and offer driving forces for the electrophoresis movement.

However, for large-scale applications, separating the GO into mono-disperse fractions via electrophoresis is not competitive. On the one hand, the preparation of GO involves strong acid and oxidation of graphite to solution-processable GO. On the other hand, once the GO was classified into mono-disperse fractions, it is

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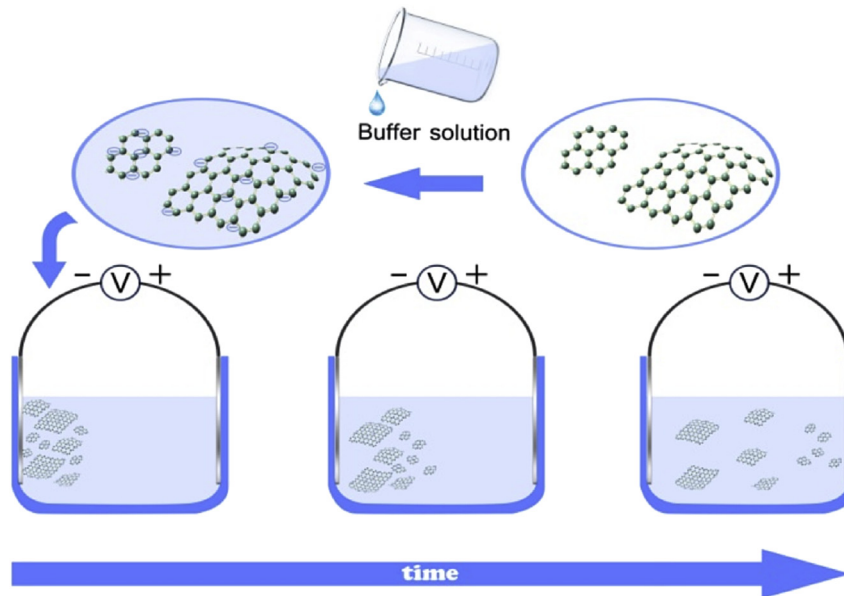


Fig. 1. Simulation illustration for electrophoresis.

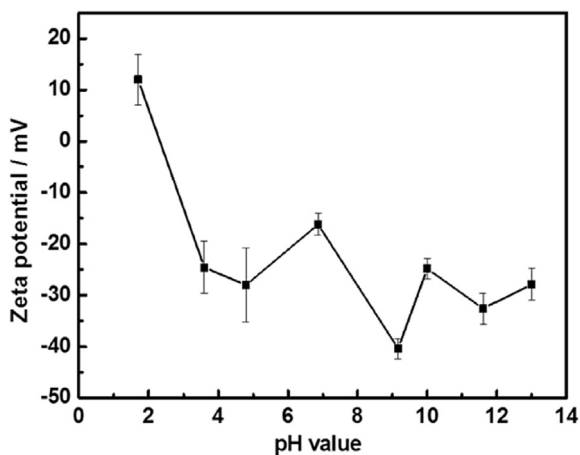


Fig. 2. Zeta potential of graphene in different pH buffer solutions.

required to further reduced to conjugated and neutralized graphene sheets (RGO), which also involve harsh reaction conditions. Interestingly, another preparation method of graphene, mechanically exfoliated graphene sheets, is more simple and safe, avoid acid alkali reaction process compared to GO and RGO. Despite of their feasible and safe preparation, no effective technique have been reported so far for the size separation of mechanically exfoliated graphene sheets, on which carries very few functional groups and ionization sites.

As reported in literature, many nanomaterials can be analysed or separated in microfluidics by electrophoresis [33,34]. And a dispersion system of conjugated graphene sheets was designed considering graphene shows very strong adsorption ability [35,36]. Graphene sheets adsorb charged ions to form micelles by adding different ions in dispersion medium. Under the effect of electric field graphene micelles move along the electromotive force. Inspired by these reports, in this study, we try to charge the mechanical exfoliated graphene sheets by adding ionization compounds to the system and separate the graphene sheets by electrophoresis in a U-tube. Studies have shown that graphene can

be successfully separated into different sizes by electrophoresis.

2. Experimental

2.1. Preparation of graphene sheets

Sample graphene was prepared via mechanical exfoliation of graphite dispersed in a liquid [37]. 0.1 g of graphite were dispersed in 80 mL of N, N-dimethylformamide by a mechanical stirring, introduced with zirconia balls (1.0, 3.0 and 5 mm in diameter, 100 g of weight respectively) into a poly (tetrafluoroethylene) vial, and milled in a planetary mill at a mild rotation speed 300 r.p.m. After ball-milling for 24 h, the obtained dark dispersion was subjected to centrifugation at 4000 r.p.m. for 30 min to remove any macroscopic aggregates using a centrifuge, giving a black suspension with single- and few-layer graphene sheets.

2.2. Electrophoresis

The freshly prepared graphene sheets (S0) were dispersed in solutions with varying pH values (See [Supporting Information](#)). Sucrose was added to the solutions to increase viscosity, and polyvinyl pyrrolidone was added as a surfactant (Fig. 1). Subsequently the graphene suspensions were placed in the middle position of U-tube electrophoresis apparatus. By applied potential difference of 200 V for 1 h, two fractions, the one close to the anode and the other one far from anode, were collected, filtrated, rinsed, and dried, obtaining sample S1 and S2, respectively.

2.3. Preparation of electrode

Graphene samples (about 4 mg) were wetted with a small amount of ethanol, applied to foam nickel films (1 cm × 1 cm), then pressed into thin slices in 5 MPa, dried at 60 °C, receiving graphene electrodes.

2.4. Characterization

The obtained samples were characterized using transmission electron microscopy (TEM), atomic force microscope (AFM), raman

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