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Concentration Enhancement of Liquid Phase Exfoliated Graphene with Addition of Organic Salts

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Abstract

A facile sonochemical route has been proposed in which liquid phase exfoliation of graphite into single and multi-layer graphene is obtained. An organic solvent ortho-dichlorobenzene (ODCB) is used for the graphene exfoliation and the effect of addition of organic salts in ODCB solvent is investigated. The characterization of the as prepared graphene is done using SEM, TEM and UV-Visible spectroscopy. The graphene film was prepared using vacuum filtration method and resistivity measurement was done using Four-Probe method. The application of graphene film in making flexible paper supercapacitor is demonstrated. It is observed that the concentration of graphene synthesized by liquid phase exfoliation method has been found to enhance appreciably with addition of EDTA disodium salt.

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Keywords: Concentration Enhancement, Graphene, ortho-dichlorobenzene, liquid phase exfoliation.

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1. Introduction

Graphene is the wonder material known for its exceptional properties not found in other materials[1]. High conductivity, high transparency and high flexibility are the some of its properties which makes it highly suitable for making transparent displays of the electronic devices like mobiles, smart phones, photovoltaic cells etc. Various methods have been used for the synthesis of graphene each having its own advantages and disadvantages. Some of

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the prominent routes for the synthesis of graphene are mechanical exfoliation, chemical vapor deposition (CVD), electrochemical method and liquid phase exfoliation method[2]. Graphene can be synthesized from graphite oxide by using Hummer's method in which graphite is converted to graphite oxide by oxidation and later reduced into reduced graphene oxide using various reducing agents like hydrazine hydrate, sodium borohydride. The reduced graphene oxide prepared by hummer's method posses many structural defects and impurities[3,4]. Therefore, this method is not suitable for the synthesis of high quality graphene. Hummer's method is also not safe because of the harmful chemicals used in the process. The recently graphene has been synthesized by liquid phase exfoliation of graphite in various solvents[5]. This method is also called sonochemical route because sonication is the main step through which graphite can be exfoliated into graphene nanosheets. Benzene, toluene and nitrobenzene have been used for liquid phase exfoliation of graphene[6]. Organic solvents like N-methyl-2-pyrrolidone (NMP) and *N*,*N*-Dimethylformamide (DMF) are the most commonly used for graphene exfoliation[7,8].The disadvantage of using these solvents is the production of low concentration of graphene, usually less than 0.01 mg/ml. ODCB is another organic solvent suitable for making graphene dispersion because it is already known to form stable single walled nanotube dispersions via π-π interactions. The sonopolymerization of ODCB is the main cause of the formation and stabilization of graphene dispersion. Sonopolymerization is the process of formation of sonopolymer[9-12], which helps in graphene exfoliation. For the exfoliation of graphene, the enthalpy of mixing of graphite dispersion in solvents should be close to zero and requires matching of surface energy of graphite with surface tension of the solvent. Based on this criterion, the solvents which have surface tensions in the range 40-50 mJ/m^2 are considered to be most suitable for synthesizing graphene. The graphene concentration enhancement has been reported by addition of organic salts in various solvents like NMP (N-methyl-2–pyrrolidone) and DMSO (Dimethyl sulphoxide) [13]. Therefore, in this paper, we report the effect of adding different salts like sodium citrate (SC), sodium tartrate (ST), potassium sodium tartrate (PST), etc. in ODCB solvent for concentration enhancement.

2. Experimental

2.1 Materials

Graphite extra fine powder, ortho-dichlorobenzene ($C_6H_4Cl_2$) (98%), potassium sodium tartrate ($KNaC_4H_4O_6.4H_2O$), sodium tartrate ($Na_2C_4H_4O_6.2H_2O$), sodium citrate ($Na_3C_6H_5O_7.2H_2O$), EDTA disodium ($Na_2C_{10}H_{14}N_2O_8.2H_2O$) and sodium hydroxide (NaOH), polyvinyl pyrrolidone (PVP) , were obtained from S. D. Fine chemicals limited, Mumbai, India. All the chemicals were used as received, without any modification or purification.

2.2 Method

As the surface tension of ODCB is 36.01 mJ/m^2 , it is suitable for making graphene dispersion. Accordingly, graphene dispersion was obtained by adding 2 gm of graphite powder as shown in Fig.1 (a) into 200 ml of ODCB solution. This mixture was sonicated for three hours 45 min in a water bath sonicator (500 W, 33 KHz). After sonication, dark black coloured solution was obtained which was centrifuged for 30 min at 4000 rpm. After centrifugation, the supernatants as shown in Fig.1 (b) were characterized using SEM and TEM analysis. For the study of effect of salt addition on graphene concentration, 0.5 gm of graphite powder, 1 gm each of organic salt was added to 50 ml of ODCB solvent and sonicated in different centrifugation tubes using water bath sonicator for 3 hours. After centrifugation at 3000 rpm for 30 minutes, the supernatants were used for characterization using UV-Visible absorbance spectroscopy. The Lambert-Beer's law was used to find the graphene concentration. TEM and SEM characterization was used for determination of the structure and morphology of the graphene nanosheets.