Synthesis of Liquid Phase Exfoliation Graphene via Green Mixed Solvent Using Homogeniser

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ABSTRACT: The aim of this research is to study the potential of producing exfoliated graphene using green mixed solvents, where aqueous extracted soap nuts saponins as natural surfactant and water as liquid medium were used to replace the hazardous chemical used in typical method of production of graphene. In this work, the combine method of sonication and high-shear mixing in liquid phase exfoliation (LPE) using BANDELIN Ultrasonic Homogeniser HD2200.2 was chosen to exfoliate the graphite. The three dimensional hollow sphere graphene oxide and small quantity of monolayer graphene flakes was produced in this study. In conclusion, the great potential of producing exfoliated graphene via green mixed solvent in LPE method using homogenizer was proven in this research.

Keywords: *Graphene; Graphene Oxide; Liquid Phase Exfoliation; Surfactants*

1. INTRODUCTION

Graphene is a 2-dimensional carbon nanomaterial that exists as a single layer of carbon atoms tightly packed in a hexagonal or honeycomb lattice. In the last decade, graphene has attracted the advertency and interest in industry by its prodigious electrical and thermal properties combined with its physical strengths [1]. The production process of graphene often limited by its complex techniques, high production costs, low production and significant defects on structural network of produced graphene [2]. Liquid phase exfoliation (LPE) is widely used to obtain stable dispersion of defect-free monolayer and few-layer graphene sheets from graphite in liquid media [3]. Although LPE is considered as environmentally friendly technique, it also facing some challenges such as lower concentration of graphene obtained [4]. Therefore, in this study, the combined synthesis methods by sonication and high-shear mixing from LPE method was used for a better fabrication outcome.

Then, the toxicity of solvent used in LPE method is one of the important factors that need to be consider during exfoliation of graphene. Therefore, the exploration of less toxic solvent in exfoliation of graphene are necessary. Thus, in this study, natural surfactant and water as liquid medium were chosen due to neutral characteristic. Therefore, among the related natural surfactants, aqueous extracted soap nuts saponin is chosen as surfactants in green mixed solvent due to its eco-friendly characteristic and high content of saponin in shells.

2. EXPERIMENTAL

2.1 Materials

The graphite powder with particle size of <20μm was used in exfoliation process. Soap nuts saponins were extracted from bulk soap nuts using deionized water. Initially, 100g of bulk soap nuts were heated at 60°C for 4 hours in 1L of deionized water. After cooling to room temperature, the soap nuts shells were removed from the saponins using sieve. Green mixed solvent was prepared by combining saponins as natural surfactant and deionized water as neutral liquid medium. Then, 600mL of green mixed solvents were prepared in a 1000mL beaker by mixing different ratio of saponins with deionized water, as 0.05:0.95, 0.1:0.9, 0.2:0.8, and 0.3:0.7.

2.2 Synthesis of liquid phase exfoliation graphene

Graphene dispersion was prepared by exfoliating natural graphite powder in green mixed solvent. BANDELIN Ultrasonic Homogeniser HD2200.2 was used to provide both sonication force and high-shear mixing at the same time during exfoliation process. Initially, 6g of graphite powder was weighted and added to the green mixed solvent. The mixture will be sonicated and shear for 2 hours at 50% sonication power (100W) using homogenizer by setting on-time as 20 minute and off-time as 5 minute. Then, 10 mL of sample was pipetted from the exfoliated solution at every off-time, which means that total 6 samples were pipetted and placed in the test tubes. After sedimentation at room temperature for 1 hour, the samples were placed in a centrifuge tube for centrifugation for 30 minutes under 1000 rpm. The supernatants were pipetted and retained for characterization use.

2.3 Characterization

The characterization techniques involved in this

research study were UV- Visible Spectroscopy (UV-vis), Fourier Transform Infrared Spectroscopy (FTIR), X-ray Photoelectron Spectroscopy (XPS), X-ray Diffraction (XRD) and Transmission Electron Microscopy (TEM).

3. RESULTS & DISCUSSION

3.1 Time of exfoliation

The peak of absorbance of graphene suspension was detected between using UV-Vis Spectroscopy (UV-vis). By using Lambert Beer's law ($A = \alpha Cl$) where A represented the absorbance of exfoliated graphene dispersion at 660nm, C represented the concentration of exfoliated graphene, α = 1390 mL mg^{-1} m^{-1} represented the absorption coefficient of natural surfactant, and l = 0.01 m represented the cell length of cuvette [5], the concentration of exfoliated graphene under different ratio of green mixed solvent (soap nuts saponins : water) was calculated and plotted in Figure 1. The best ratio of soap nuts saponins with water in green mixed solvent was determined as 0.2:0.8, with maximum concentration at 0.03741 mg/ml after 2 hours of exfoliation using homogenizer. Other than that, the rising on concentration of exfoliated graphene can be observed with longer time of exfoliation. However, for ratio 0.3:0.7, the concentration of exfoliated graphene reached maximum value at 0.03237 mg/ml after 1 hour of exfoliation time, and started to drop dramatically

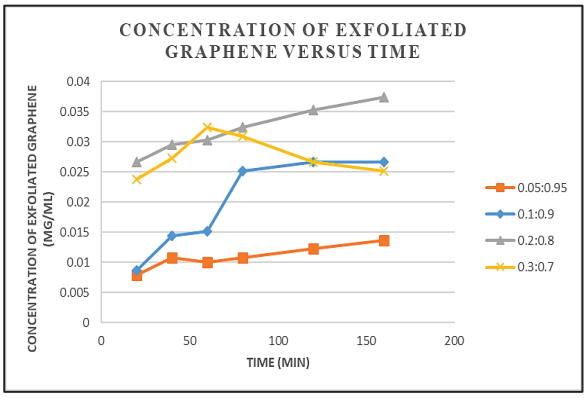

Figure 1: Concentration of exfoliated graphene versus time under different ratio of green mixed solvent

4. CONCLUSION

In conclusion, the objectives of this research study were achieved successfully. The existence of exfoliated graphene was supported by UV-Vis spectrum of exfoliated graphene at 660 nm which is the optical absorbance of graphene concentration. The LPE graphene was successfully produced in the form of graphene oxide by using green mixed solvent, where natural surfactant and water as liquid medium are used in this study instead of typical chemical surfactant method.

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