Synthesis, characterization of graphene and graphene composites from vein graphite of Sri Lanka

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ABSTRACT

Graphite oxide (GO) and reduced graphite oxide (rGO) are value added products of graphite and have a great demand globally, especially in electronic applications and in contemporary research and development. Greater attention has been paid to search scalable chemical and physical methods for synthesizing GO and rGO. The present study is mainly focused on synthesis of high quality graphite oxide (GO), reduced graphite oxide (rGO/few-layers), and composites from natural vein graphite from Bogala mines for commercial purpose. The well known Hummers' method in preparation of graphite oxide was followed with modification. Particularly, the particle size of graphite as a raw material and the conversion temperature of GO to rGO have been optimized.

Particle size greatly affects on physical and chemical properties of the final products of interest, therefore, an attempt was made to optimize the particle size of commercial (natural) graphite and subsequently to determine the effect of particle size on properties of GO and rGO. Optimization of particle size reduction of graphite was achieved by ultra-sonication (with and without dispersive agents such as ethanol/surfactant) and ball milling technique (under optimized mill parameters and conditions). The properties of GO and rGO synthesized from commercial graphite and sized reduced graphite were investigated using an XRD, a UV-Vis, an FTIR and an SEM. The results clearly indicated that the ball milling for three hours of commercial graphite is rather accounted for efficient (~67% size reduction) particle size dispersion and homogenization. The instrumental characterization clearly showed that the GO and rGO synthesized from smaller sized graphite showed enhanced properties than the products synthesized from commercial (bigger sized) graphite. Therefore, ball-milled graphite was used to synthesize GO and then rGO for further study.

Thermal reduction under inert atmospheric conditions was employed for the synthesis of rGO from GO as a green technique, since it involves no hazardous contaminants as of the chemical reduction. The minimum thermal reduction temperature was systematically studied by high-temperature X-ray powder diffraction (HT-XRD) at specified temperatures. Results indicate that the optimum temperature required for effective reduction of GO to rGO under inert atmospheric condition was ranging from 475-500 °C. This minimum temperature of ~475 °C was found to be rather sufficient to produce high quality rGO with lesser amount of oxygenated functional groups.

The structure property and the electrochemical performance of GO and rGO synthesized from optimized conditions have been investigated in detail with the aid of modern characterization techniques of XPS, TG/DTA, BET, AFM and charge/discharge by Land Battery Tester

Facility to ascertain the suitability of GO and rGO to be used as an electrode material. The XPS and FTIR analysis of rGO showed that the functional groups in rGO layers are less and TGA/DTA revealed that the thermal stability of rGO is quite significant and capable of withstand against the temperatures below 214 °C that further ensures the diversified and endurable applications of rGO. The XRD, Raman spectroscopy, BET and the AFM have shown that the rGO has few layers (2-6) of graphene. The electrochemical performance of the rGO has shown an initial higher capacity in the range of 1400-1700 mAh g^{-1} and the stable capacity of around 450 mAh g^{-1} for 100 cycles.

Composites of GO/NiO and subsequent rGO/NiO at difference ratios of 1:4, 2:3, 1:1, 3:2, and 4:1 were prepared from a simple technique of direct mixing nano-sized (≤50 nm) NiO powder to be used as an advanced electrode material. Properties were characterized by X-ray diffraction, FTIR, X-ray photoelectron spectroscopy, Raman spectroscopy, and SEM. It was observed that the optimum interaction between rGO and NiO could be observed for the composite ratio of 1:1. The FTIR, XPS, and Raman analysis also revealed that the addition of nano-sized NiO has very less impact on the internal structure of rGO thus enabling rGO layers to be free stand with NiO. The SEM analysis of morphology of the composite of 1:1 shows that NiO particles are scattered on the surfaces of the graphene layers in rGO. The geometry of the composite would be beneficial in terms of electrochemical stability and high energy density in future applications as an electrode material for energy storage and other applications.