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MALONIC ACID ASSISTED REDUCTION OF GRAPHITE OXIDE: STRUCTURAL CHARACTERISATION

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Graphene is a thick sheet of sp^2 hybridized carbon atoms. It has received broad interest in many areas of science and technology because of its unique physical, optical, chemical, mechanical and thermal properties. There are many approaches to graphene preparation, including mechanical exfoliation, epitaxial growth, carbon nanotubes cutting, chemical vapor deposition [1]. Chemical reduction of graphite oxide (GO) is recognized as the most promising method for large scale and rapid production at low cost. However, the most popular chemical reducing agents, such as hydrazine, hydroquinone, sodium borohydride, lithium aluminium hydride, are hazardous, toxic and corrosive. The product obtained using these reagents has poor electrical conductivity, due to the defects remaining in crystal lattice and have negative impact on bio-related applications, too [2,3].

In this work, we present the thermal reduction of GO in the presence of malonic acid (MA), which is a green and inexpensive reducer decomposing thermally at 135 ℃. Graphite oxide was prepared from the natural graphite by the synthesis protocol reported by Yan et al. [4]. In a typical experiment, graphite powder was treated with conc. H_2SO_4, $K_2S_2O_8$ and P_2O_5. Later, this pre-oxidized graphite was subjected to oxidation by Hummers method using $NaNO_3$, H_2SO_4 and $KMnO_4$ [5]. The obtained GO was reduced by adding malonic acid with ratio of 1:3 or 1:5 and thermal annealing under Ar gas atmosphere for 30 min at different temperatures 200 ºC, 300 ºC, 600 ºC, 800 ºC. Reduced GO products were analyzed by Fourier Transform infrared (FTIR) and X-ray diffraction (XRD) analysis. Furthermore, the thermal decomposition of GO, malonic acid and GO:MA (1:3) mixture was investigated by TGA/DSC.

The results show that the level of GO reduction to graphene phase depends on the reduction conditions. Reduction of GO at 200 ºC and 300 ºC results formation of an amorphous product. XRD analysis of products obtained at 600 ºC and 800 ºC shows a similar ‘d’ spacing of 0.350 nm with a hexagonal structure. It indicates the formation of a more ordered graphitic structure and reestablishment of sp^2 network after annealing. Moreover, FTIR patterns of these samples exhibit a successful reduction of oxygen-containing groups (O–H, C=O, C–O–C, C–O).

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