Simple synthesis of large graphene oxide nano-sheets via electrochemical method coupled with oxidation process

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Abstract: Graphene is an emerging 2-D carbon material containing sp^2 hybridised carbon atoms arranged in hexagonal array.[1-4] Graphene is a future material due to its excellent physico-chemical properties such as high surface area (2630 m^2/g), thermal conductivity (5000 $Wm^{-1}K^{-1}$), Young's modulus (1 TPa), electron mobility ($2.5*10^5$ $cm^2V^{-1}s^{-1}$), relatively highest electrical conductivity at room temperature (of the order of 10^6) and chemical durability.

In this paper, we report a simple two step approach for the synthesis of graphene oxide nanosheets. The first step is a pre-treatment step involves electrochemical exfoliation of graphene from graphite electrode in a mixture of H_2O_4+H_3PO_4. The second step is the oxidation step where, oxidation of exfoliated graphene sheets was performed using $KMnO_4$ as the oxidising agent. The oxidation was carried out for different time periods between 1-12 h at ~ 60 °C. Different graphene oxide batches prepared were characterized using an array of spectroscopic and microscopic techniques such as XRD, FESEM, TEM, FT-IR and UV-vis spectroscopy. Raman, CV and TGA techniques were used to study degree of oxidation in the as-synthesized graphene oxide batches. As-prepared GO nano-sheets were characterized using an array of spectroscopic and microscopic techniques. The UV-visible absorption spectrum showed an intense peak at 230 nm and an adjacent band at 300 nm corresponding to π-π* and n-π* transitions in all samples. Boehm titration was used to quantify the functional groups present on the GO surface. Overall GO nano-sheets obtained after 6 h of oxidation (GO 6h) was found to be the best. XRD pattern of GO 6h revealed characteristic peak at 2θ=8.88º with the corresponding interplanar spacing between the layers being 0.995 nm which is among the best with respect to the previous methods reported in the literature. Raman spectroscopy showed that the degree of defect (I_D/I_G) ratio for GO 6h was found to be 0.942 which is higher than those obtained for GO (Hummer's) (0.863) synthesized using improved Hummer's method. The electrochemical property of GO nano-sheets was analysed using CV, which also provide information about the oxidation level

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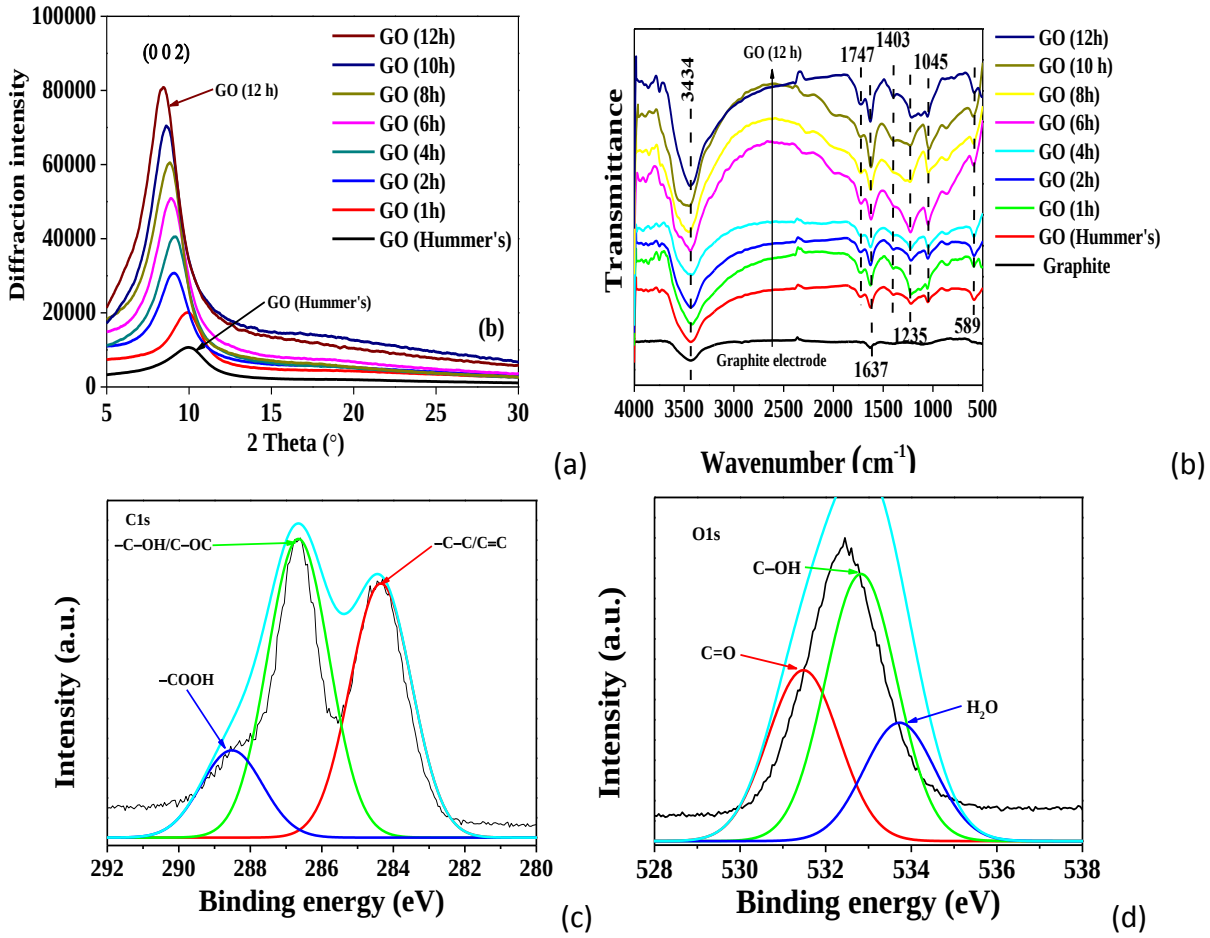

Fig. (a) XRD patterns ofdifferent GO batches, (b) FT-IR Spectra of different GO batches, (c) XPS C1s spectra of GO 6h, (d) XPS O1s spectra of GO 6h.

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