Structural, Optical and Morphological studies of Graphene Oxide produced by Improved Synthesis

Asutosh Swain, Solleti Goutham, Ch. Shilpa Chakra, K. Venkateswara Rao

Abstract*— Nanotechnology is a cutting-edge research in materials, energy, sensors, biotechnology and defence systems. One of the crucial bottlenecks of modern technology is cost and scalability. In this respect, Graphene Oxide (GO) has gained significant attention. GO is often described as an electrical insulator, due to the disruption of its sp2 bonding network. In order to recover the honeycomb hexagonal lattice and the electrical conductivity, the reduction of the GO has to be achieved. This remains an important property when mixing with polymers or ceramics matrixes to improve their electrical and mechanical properties. The GO used in this study was prepared by and modified Hummer's method. The structural properties were investigated by X-ray diffraction. The optical properties were studied by UV-Vis absorption spectra and FT-IR spectroscopy; furthermore, scanning and transmission microscopy analysis was done to study its morphology. In the study we successfully synthesized GO by modified Hummer's method, and the results indicated higher interlayer spacing.

Index Terms*— Graphene Oxide, Electrical insulator, X-ray diffraction, Modified Hummer's method.

I. INTRODUCTION

The research and synthesis of nanomaterials lies at the heart of nanotechnology, and an exciting nanomaterial that is undergoing high volume research exposure is Graphene oxide (GO). The precursor of GO is Graphite oxide, which was first produced in 1859 by Benjamin C. Brodie. Hummer and Offeman [1] developed a more efficient method in 1957. Currently a modified version of Hummer's method is in wide use, as it is effective and cheap. GO is essentially graphite that has been oxidized. Graphite, an allotrope of carbon, has layers of hexagonal carbon rings stacked on top of one another. The Oxidation of graphite changes the morphology and properties of graphite. The introduction of oxygen functionalities by oxidation intercalates the layers of graphite thereby increasing the interlayer distance making exfoliation easier. There are oxygen functional groups such as epoxides and hydroxyls on the carbon plane, which can be identified by FT-IR analysis [2-3]. Exfoliation of graphene sheets is easily confirmed by SEM image. Major developments on GO were made after the first time graphene was synthesized in 2004. GO has since become a valuable material in the field of nanoscience. This attention accredited to GO is due to its many interesting properties and potential applications. Important characteristics of GO include electrical conductivity, optical nonlinearity, permeability, hydrophilic nature, large surface area, high young's modulus, thermal conductivity and photo catalytic ability [4]. GO also has great potential for electron transport, and the applications of graphene-based photo catalysts on water splitting, oxidization of organic contaminants, photo reduction of CO2 into renewable fuels, toxic elimination of heavy metal ions, and antibacterial applications. The various aspects of this exciting material enable it to have a multitude of applications. Gas sensing applications for GO are being tested and research is being conducted on water purification using reverse osmosis with graphene oxide. GO materials are also great materials for constructing humidity sensors with ultrahigh sensitivity for widespread applications [5-6]. It can be used as battery electrodes and photo catalysts. Biomedical applications include drug delivery and biosensors. Water resistant coating using GO is being developed. It is also used to synthesis graphene and different types of chemically modified graphene (CMG) [7], which has its own multitude of applications. The structure and quality of graphene oxide is crucial for these applications. Hence, the synthesis method should produce standard quality of material. However, most methods are precise procedures and slight changes in temperature stirring time etc. can alter the morphology of the GO. Folding and cracking are present in many graphene oxide samples [8-9]. In this manuscript, we analyze the structural, optical and morphological properties of graphene oxide synthesized by modified Hummer's method [10].

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II. EXPERIMENTAL

A. Synthesis of Graphene Oxide

The GO was prepared by modified Hummer's method. In this method, 1.5 grams of graphite powder was taken in a beaker and kept in ice bath. Then 37 ml of H2SO4 and 3.8 ml of H3PO4were added and the solution was stirred for 20 minutes to remove clumps and homogenize the solution. The stirring allows H2SO4 to oxidize the graphite. Then 4.8 grams of KMNO4 powder was added very slowly while stirring the suspended solution. After 20 minutes of stirring, the ice bath is removed, and solution is stirred for 45 minutes while maintaining temperature at 35 °C. The suspended solution was then diluted with70 ml of doubly distilled water. After 30 minutes of stirring 15 ml of H2O2 (30%) was added to the solution. The final solution was then separated and washed using centrifugation with 10% HCl and doubly distilled water five times. The resulting product was dried in a vacuum oven at 80°C for 12 hours to obtain a powder form of GO.

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B. *Characterization*

For the characterization of GO it is necessary to have a complex analytical system capable to explain the mechanism of the reaction. To express the arrangement and properties of the substrates the composition and structure was studied using powder X-ray diffraction (Bruker D8 Advance) Cu Kα radiation, from which the layer distance was calculated. Scanning electron microscopy (SEM) measurement was performed by the help of Zeiss. Fourier transform infrared (FT-IR) spectra were confirmed on a Perkin Elmer FT-IR, the sample was placed on a universal pelletizer and pressed and the spectra was then recorded. The absorption spectroscopy in the UV-visible spectroscopy (Systronics 2202) with quartz cell was performed for optical characterization OriginPro 8 was used for graphical and data analysis.

III. RESULTS AND DISCUSSION

The GO prepared by modified Hummer's method, was analyzed by XRD, UV-vis absorbance spectroscopy, FT-IR spectroscopy, distribution of the particles on the surface were studied by Scanning and Transmission Electron Microscope. We chose to study GO because the structure and properties of GO depend on a particular synthesis method's degree of oxidation. Due to its vast applications GO has importance in electrical, technological, defense and biomedical industries.

A. *XRD*

The powder XRD pattern of GO is presented in Fig. 1. In this study, GO samples exhibited a peak (002) at 10.33° indicating higher interlayer spacing. The increase in interplanar distance of GO is due to structural defects and the existence of many oxygen functional groups such as hydroxyls. The XRD figure shows that the quality of GO material was obtained in the present system.

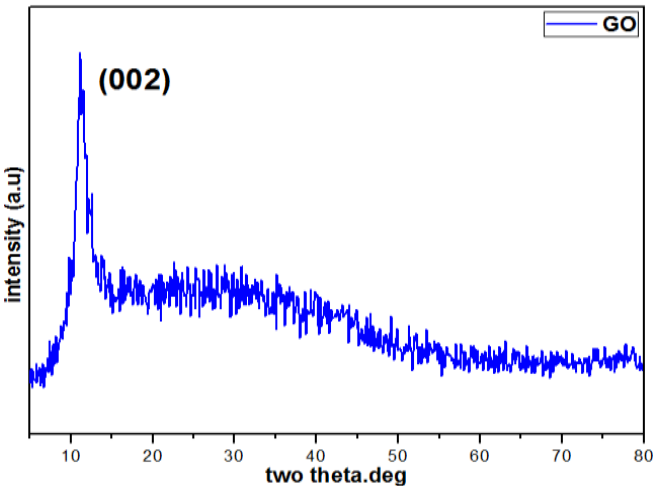

Figure 1. The XRD pattern of Graphene Oxide.

B. *UV*

UV-vis spectra of aqueous GO dispersion is presented in Fig. 2. Two kinds of characteristic peaks were observed in these spectra to identify GO. The first is a shoulder at 216 nm which is due to the C=C bond in an aromatic ring [11].The second characteristic feature is a broad shoulder peak at 255 nm and representing C=O [12]. At this absorption over 255 nm is expected to be caused by the conjugated ring [13].

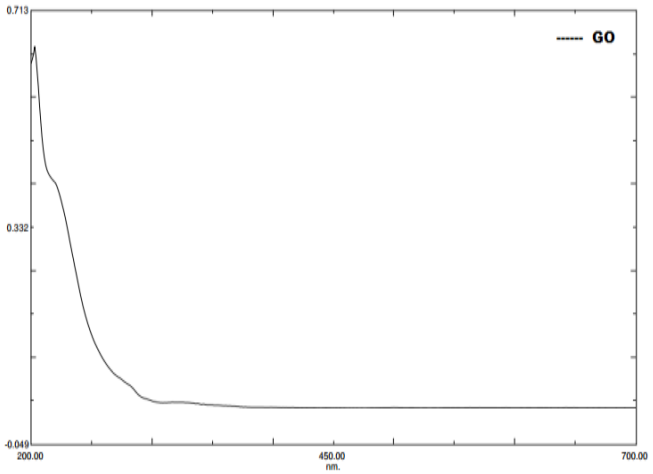

Figure 2. UV-visible spectra of aqueous dispersion of Graphene Oxide.

C. *FT-IR*

FT-IR measurements were performed to find the bonding interaction in graphene after oxidation process. Fig. 3 illustrates that the GO has a peak at 1154.9 cm-1 which is feature of the C-O bond and is a validation for the presence of oxide functional group. The peak in the range of 1602.9 cm-1shows the C=C bond. The absorbed water in GO is illustrated by a broad peak at 2926.7 cm-1 to 3467.4 cm-1, contributed by the O-H from H2O molecules[14].This supports the fact that GO is a highly absorptive material.

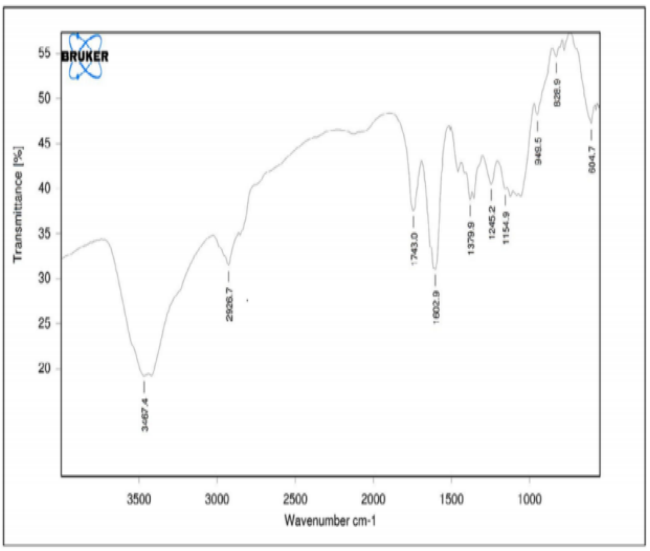

Figure 3. FT-IR spectra of Graphene Oxide

D. *SEM*

Fig.4 shows the SEM images of modified Hummer's method of synthesized GO, corresponding to well exfoliated, typical wrinkled surface morphology. The morphology of GO was studied through the SEM observation. Fig.4 shows the architectures of SEM images of freestanding GO Nano sheets, revealing a crumpled structure. This was the result of deformation upon the exfoliation and restacking processes [15].The results showed that the wrinkled structure has been observed in few layers GO Nano sheets.

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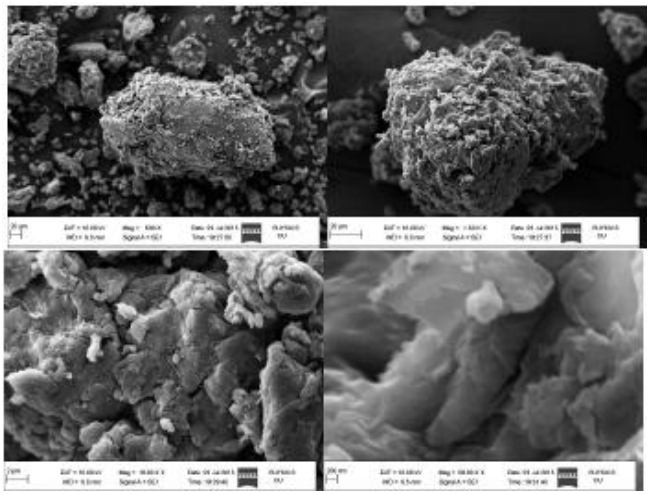

Figure 4. SEM images of Graphene Oxide*

E. TEM

Transmission electron microscopy (TEM) analysis was carried out using Hitachi H7500, which has a point resolution of 200-100 nm. TEM samples were prepared on Holey carbon-coated Cu 300 mesh grids.

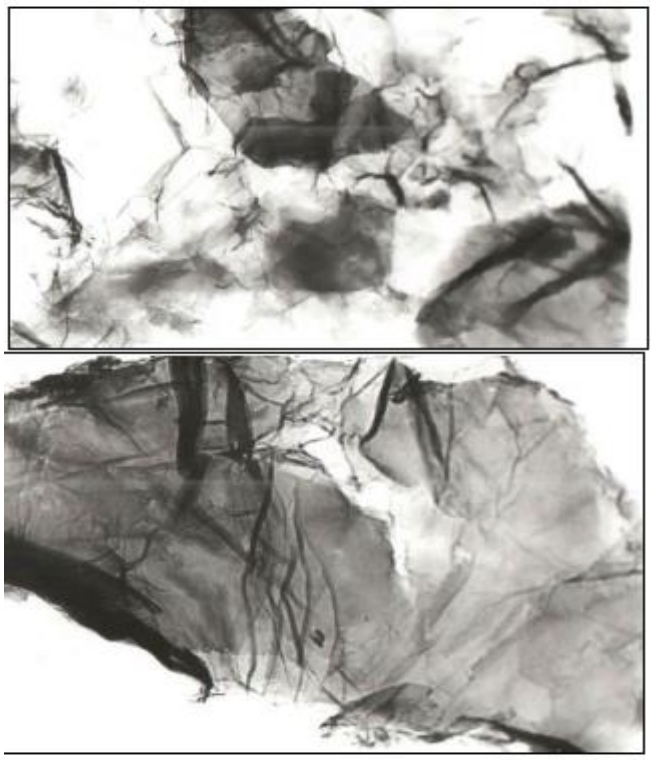

Figure 5. TEM images of graphene oxide (GO) sheets obtained by a modified Hummer's method.

The obtained TEM images (in Fig. 5) show morphology of the architecture of GO for the highly reduced graphene oxide (GO) sheets. These sheets were obtained by a simple modified Hummer's method at 80°C for 12 hours. The magnified image shows a well-exfoliated morphology.

IV. CONCLUSION

Present study and analyses of GO produced by modified Hummer's method has numerous advantages over the conventional methods. This method does not involve a large exothermal process and produces no toxic gases. Apart from having a unique structure GO also has a range of unusual properties. The formation of the GO was confirmed by the XRD and FT-IR analysis. Using TEM and SEM studies, structure and morphology were identified. For optical properties UV-visible analysis was performed and based on its results it is indicative that the modified Hummer's synthesis method produces GO with versatile advantageous.

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