

Synthesis of Graphene Oxide (GO) and Reduced Graphene Oxide (RGO), its Characterization by XRD, UV-VIS Spectroscopy and measurement of DC conductivity

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ABSTRACT

Graphene Oxide (GO) was chemically synthesized using Modified Hummer's method. The GO was chemically reduced to Reduced Graphene Oxide (RGO) using hydrazine hydrate. The GO and RGO samples were characterized by the usual methods such as X-ray diffraction, UV-VIS and FT-IR. The XRD results showed 2θ is 24° and 26° with inter layer spacing equal to 0.358nm and 0.334 nm in GO and RGO resp. UV-VIS spectra of GO and RGO exhibit maximum absorption peak at 221nm and 247nm respectively. These peaks are attributable to π - π^* transition of the atomic C=C bonds. Thin films of GO and RGO were also deposited on glass substrate using spray pyrolysis technique (SPT). The DC conductivity of the thin film GO and RGO was studied by the Four Probe Method. The DC conductivity of RGO was found out to be twice as that of GO.

Keywords: Graphene Oxide (GO), Reduced Graphene Oxide (RGO), Thin film, DC conductivity.

1. Introduction

Graphene is an exciting material that is getting a lot of attention. Graphene has attracted much attention also from researchers due to its interesting mechanical, electrochemical and electronic properties. Graphene, a single atomic layer of sp^2 -bonded carbon atoms tightly packed in a two dimensional (2D) honeycomb lattice, has evoked great interest throughout the

scientific community since its discovery [1]. Graphene oxide (GO) have recently emerged as a new carbon - based nanoscale material that provides an alternative path to graphene [2]. It is a single-atomic-layered material made of oxidizing graphite crystals which available in large quantities at inexpensive prices. Structurally, the Graphene oxide is similar to a graphene sheet with its base having Oxygen-containing groups.

2. Experimental

2.1 Materials

Graphite Flakes (acid treated 99%), Potassium permanganate (99%), Hydrogen peroxide (40% wt), Sulphuric acid (98%), Hydrochloric acid (35%), distilled water, phosphoric acid, hydrazine hydrate, DMSO, Ethylene glycol.

2.2 Synthesis of Graphene Oxide (GO)

In this study, a modified Hummers' method was used to prepare GO. 3.0 g of graphite flakes was added to 120 ml solution of sulphuric acid and phosphoric acid in a 500 ml conical flask in ratio 9:1. The mixture was then stirred vigorously and maintained at 90°C for 2 hours in the interval of 30 sec in each 30min. The mixture was then allowed to cool to room temperature and kept below 10°C by placing the mixture in an ice bath. On adding 15 g of potassium permanganate (KMnO₄) gradually over a period of one and half hour the mixture showed yellowish green color. The mixture was again continuously stirred for another 6 hours in order to obtain homogeneous mixture of brownish (reddish) color.

It was then removed from ice-water bath and was placed in a warm-water bath maintained at 45°C and stirred for an hour. After this, the mixture was allowed to cool to room temperature. It was observed that the color of mixture changed to yellowish brown. Subsequently, 250 ml of distilled water was added to the mixture. The color of the mixture changed gradually from yellowish brown to reddish brown as the reaction proceeds. In order to stop the reaction, 20 ml of hydrogen peroxide was added to the mixture until the color changed from reddish brown to dark brown. This shows that oxidized graphite has been synthesized. The obtained mixture was left overnight. The mixture was washed by adding 100ml of HCl and 150 ml of water repeatedly three times. Finally, the mixture was filtered, oven-dried and pulverized. The powder obtained was sieved so as to obtain even particle size [4].

2.3 Synthesis of Reduced Graphene Oxide (RGO)

So as to reduce the grapheme oxide, 600 mg of GO was dispersed in 200 ml of distilled water (3 mg/ml). 0.2 ml of hydrazine monohydrate (N₂H₄) was added to the mixture of GO and distilled water in a beaker. The mixture was placed in microwave oven, stirred and maintained at 90°C. After this duration, the color of the solution changed from dark brown to black which indicated that RGO has been obtained. The obtained RGO was washed thrice and filtered in order to remove residual N₂H₄ from the solution. The black filtrate was then oven-dried.

2.4 Preparation of GO and RGO thin film deposition

The thin films of GO and RGO was deposited on glass substrate by using Spray Pyrolysis unit (Holmarc) . In order to deposit GO and RGO thin films, 0.5 g each of GO powder and RGO powder was dispersed separately in 20 ml of ethylene glycol (EG) and DMSO. EG and DMSO was chosen because GO and RGO have high dispersability in EG and DMSO.

The mixture was sonicated in the probe sonicator until the mixture becomes colloidal solution. After the GO and RGO was dispersed well in the solution then the solution is prepared for the thin deposition. The GO and RGO films were deposited using SPT. The deposition temperature was 200°C. The glass showed a dark coloration after deposition of GO and RGO films which indicated the decomposition of the precursors [4].

3. Characterization

The synthesized GO and RGO were characterized by X-Ray Diffraction Analysis (XRD), UV Spectroscopy Analysis and by DC conductivity measurements by four probe method.

3.1 XRD analysis of GO and RGO

The X-ray diffraction (XRD) study was performed by using X ray diffractometer (RIGAKU). The diffraction peak of pure graphite was found around 26° corresponding to the highly organized layer structure with an interlayer distance of 0.335 nm along the (002) orientation is shown as inset in Figure 1.

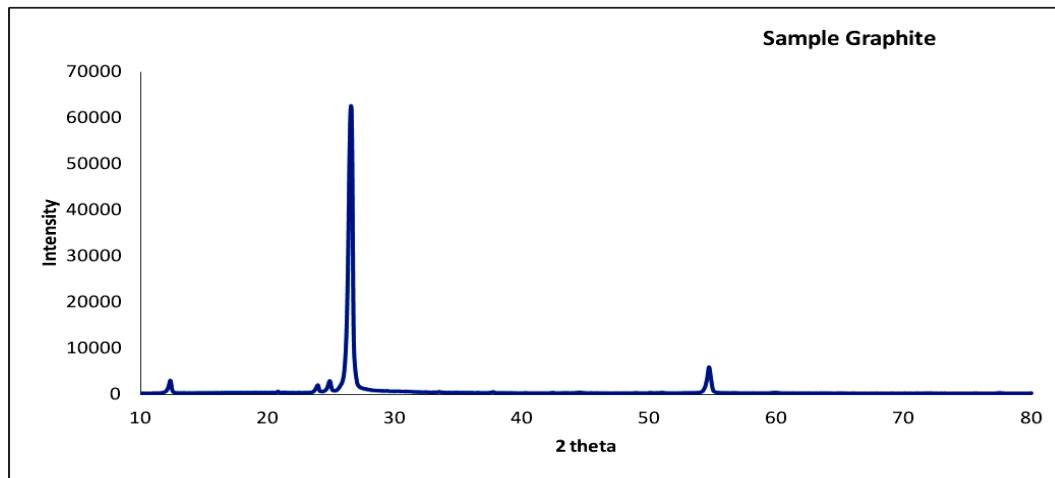


Figure 1: XRD of Graphite

The XRD pattern for synthesized GO by Modified Hummer's method is shown in Figure 2. The shift in peak at 26° to 24° in GO, showed that the product was completely oxidized after the chemical oxidation and exfoliation, indicating an increase in d-spacing from 0.335 nm to 0.358 nm.

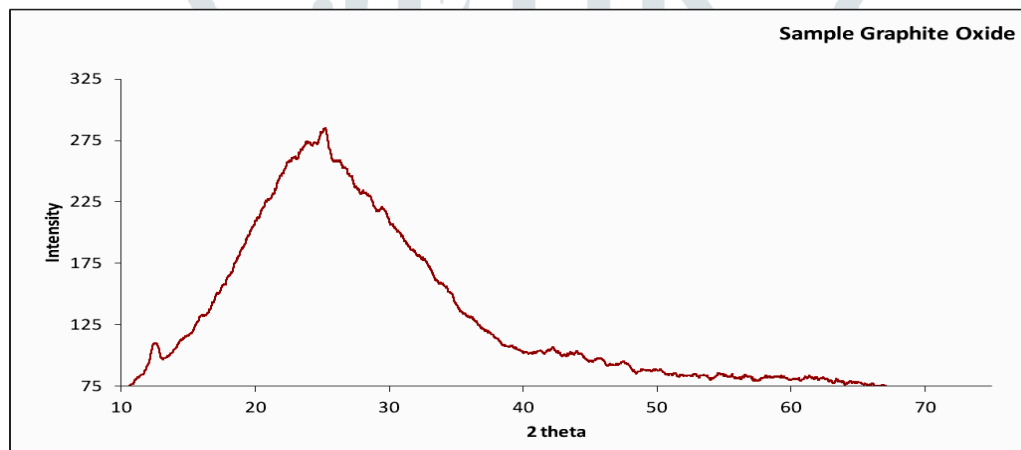


Figure 2: XRD of Graphene oxide

The XRD pattern for synthesized RGO by thermal method is shown in Figure 3. The peak at 24° for GO shifted to 26° for RGO. This showed that the product was completely oxidized after the chemical oxidation and exfoliation, indicating an increase in d-spacing from 0.358 nm to 0.334 nm.

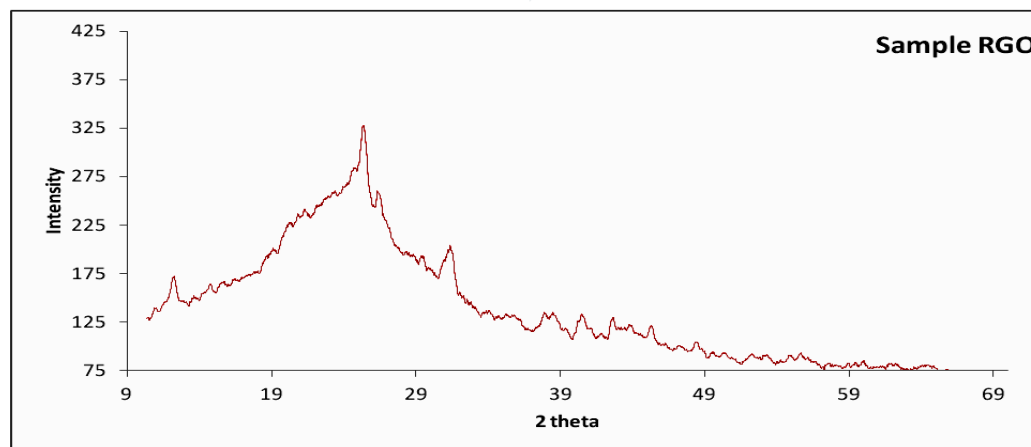


Figure 3: XRD of Reduced graphene oxide

3.2 UV Spectroscopic analysis of GO and RGO

UV-Vis study was performed by using UV 1800, Shimadzu. UV spectra of GO and RGO clearly indicates the transparency of GO and RGO films for visible lights. In figure 4 the peak at 221 nm corresponding to absorption intensity is stronger than that of pristine graphite. This peak is assigned to π - π^* transition. This is an indication that oxidative treatment involved in GO synthesis does not severely interfere with the conjugation in the system.

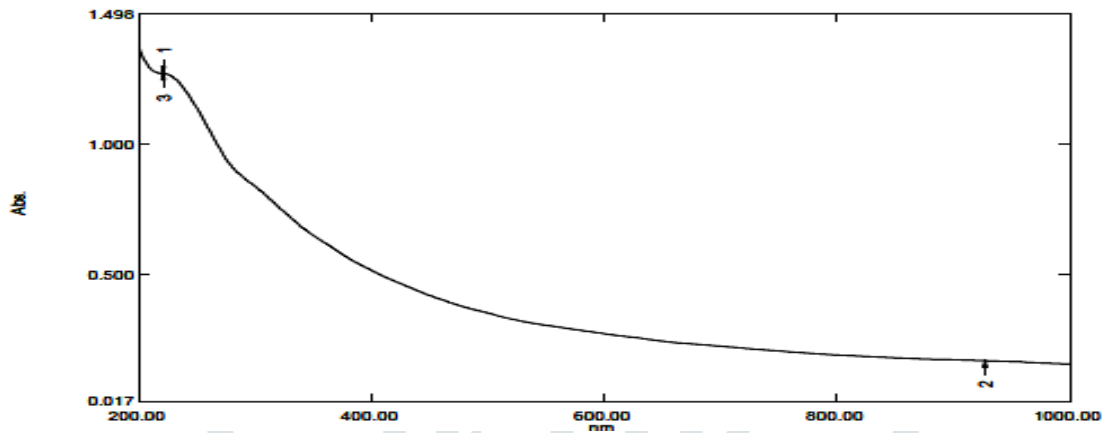


Figure 4: UV-Vis of Graphene Oxide

A similar shoulder was also seen for RGO in the figure 5. The peak at 247 nm in the case of RGO is due to π - π^* transition of aromatic C-C bond. The shifting of this peak from 221 nm for GO to 247 nm for RGO indicates the removal of oxygen.

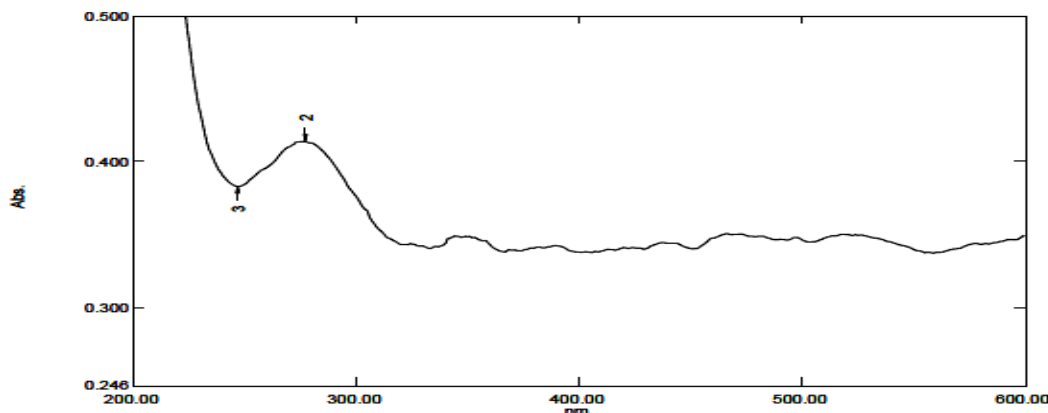


Figure 5: UV-Vis of Reduced graphene oxide

3.3 Electrical Conductivity study of GO and RGO

Figure 6 shows variation of conductivity with temperature. and the conductivity increase with the increasing temperature conductivity of RGO is greater than the GO, thus the conductivity of RGO is increase than that GO

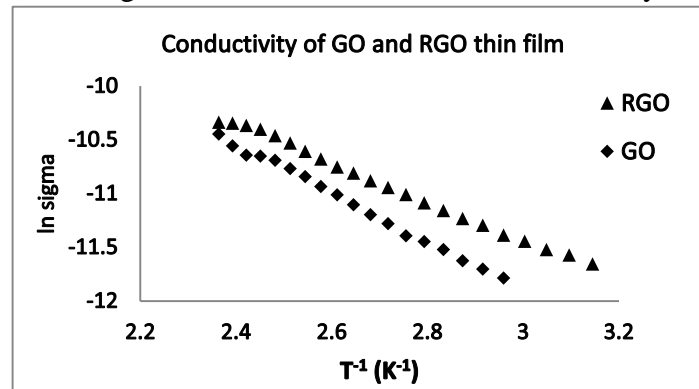


Figure 9: Conductivity of Graphene oxide and reduced graphene oxide

4. Conclusion

GO was prepared by modified Hummer's Method and RGO by chemical method that are confirm with the XRD and UV-VIS. Thin films of GO and RGO were also deposited on glass substrate using SPT. The DC conductivity of the thin films of GO and RGO was studied by the Four Probe Method. The DC conductivity of RGO was found out to be twice as that of GO.

5. REFERENCE

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